

FRACTOGRAPHY AND MATERIALS SCIENCE

Gilbertson/Zipp, *editors*

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FRACTOGRAPHY AND MATERIALS SCIENCE

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Foreword

The symposium on Fractography and Materials Science was held on 27–28 Nov. 1979 in Williamsburg, Va. The American Society for Testing and Materials, through its Committee E-24 on Fracture Testing and Subcommittee E24.02 on Fractography and Associated Microstructures, sponsored the event. The symposium chairmen were L. N. Gilbertson, Zimmer, U.S.A., and R. D. Zipp, International Harvester, both of whom also served as editors of this publication.

Related ASTM Publications

Crack Arrest Methodology and Applications, STP 711 (1980), \$44.75,
04-711000-30

Fracture Mechanics: Twelfth Conference, STP 700 (1980), \$53.25,
04-700000-30

Fracture Mechanics Applied to Brittle Measurements, STP 678 (1979), \$25.00,
04-678000-30

Fracture Mechanics: Eleventh Conference, STP 677 (1979), \$60.00,
04-677000-30

Elastic-Plastic Fracture, STP 668 (1979), \$58.75, 04-668000-30

Fractography in Failure Analysis, STP 645 (1978), \$36.50, 04-645000-30

Developments in Fracture Mechanics Test Methods Standardization, STP
632 (1977), \$24.75, 04-632000-30

Fractography—Microscopic Cracking Process, STP 600 (1976), \$27.50,
04-600000-30

Toughness and Fracture Behavior of Titanium, STP 651 (1978), \$28.50,
04-651000-30

Evaluations of the Elevated Temperature Tensile and Creep Rupture Prop-
erties of 12 to 27 Percent Chromium Steels, DS 59 (1980), \$24.00,
05-059000-40

A Note of Appreciation to Reviewers

This publication is made possible by the authors and, also, the unheralded efforts of the reviewers. This body of technical experts whose dedication, sacrifice of time and effort, and collective wisdom in reviewing the papers must be acknowledged. The quality level of ASTM publications is a direct function of their respected opinions. On behalf of ASTM we acknowledge with appreciation their contribution.

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SUMMARY

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Introduction

This symposium was organized to demonstrate the importance of utilizing state-of-the-art and new fractographic principles in materials science. These principles are applied in the upcoming text to a variety of metals, including iron, aluminum, titanium, copper, nickel, and tungsten-base alloys, and various nonmetals, including polymers, ceramics, and glasses.

The papers contained in this volume demonstrate that fracture analysis is more than just examination of the fracture surface. Variables such as the microstructure, stress conditions, and the environment control the fracture surface topography in materials. All of the papers presented here discuss at least one of these variables and its influence on the resulting fracture morphology. By correlating these variables with fractography, a more complete and detailed understanding of fracture characteristics in materials is made possible. This is necessary to comprehend more fully the complexities involved in fracture processes.

This volume should serve as a background reference and a guide for investigators interested in evaluating fracture surface topographies for a variety of materials. The high degree of sophistication needed to interpret complex fractographs should become evident as the reader becomes familiar with this document. We believe that the information contained within provides a firm foundation for continued advancement in fractography and demonstrates the level of refinement that has taken place recently in this field. We also think that the work presented here can be still further refined to provide for better understanding of fracture behavior in materials.

L. N. Gilbertson

Zimmer, U.S.A., Warsaw, Ind. 46580; symposium chairman and editor.

R. D. Zipp

International Harvester, Hinsdale, Ill. 60521; symposium chairman and editor.

Environment

Microstructural Origin of Flutes and Their Use in Distinguishing Striationless Fatigue Cleavage from Stress-Corrosion Cracking in Titanium Alloys

REFERENCE: Meyn, D. A. and Brooks, E. J., "Microstructural Origin of Flutes and Their Use in Distinguishing Striationless Fatigue Cleavage from Stress-Corrosion Cracking in Titanium Alloys," *Fractography and Materials Science*, ASTM STP 733, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 5-31.

ABSTRACT: Postfracture analysis does not always distinguish striationless low-stress fatigue from stress-corrosion cracking (SCC), since both are characterized by cleavage, together with other less distinct fracture modes. Studies of identical specimens of Ti-8Al-1Mo-1V broken under both conditions suggest that the presence of certain microplastic fracture features called flutes may be uniquely characteristic of SCC, and absent from low-stress striationless fatigue fractures. Some new observations concerning the microstructural origins of flutes verify that they arise from a tendency toward planar slip in α and α - β alloys and from the presence of multiple cleavage during crack propagation under certain circumstances, including SCC.

KEY WORDS: titanium alloys, stress-corrosion cracking, fatigue, fractography, fracture mechanisms, cleavage, flutes, hydrogen embrittlement, sustained load cracking, materials science, materials

Both aqueous stress-corrosion cracking (SCC) and low-stress fatigue cracking (LSFC) in alloys that are susceptible to SCC, such as Ti-8Al-1Mo-1V, create substantially similar fracture surfaces that consist mostly of cleavage facets. Under sufficiently low cyclic crack-tip stresses, LSFC leaves no striations to serve as unmistakable signs of fatigue. Hence, failure analysis in these alloys can be uncertain where the fracture surface consists mainly of cleavage. Differentiation between the two cracking mechanisms can often be made by experienced fractographic analysts by noting a smoothed, tear-ridge-free appearance in LSFC, in contrast with a greater abundance of tear ridges and somewhat more microplastic deformation at cleavage facet boundaries in

¹ Metallurgists Naval Research Laboratory, Washington, D.C. 20375.

SCC. However, more cut-and-dried qualitative differences are preferable in making such distinctions.

A feature of SCC fracture surfaces of α and α - β titanium alloys, once mistaken for cleavage [1],² but since identified as flutings [2], river patterns [3, 4], and striations [5], seems to provide such a qualitative differentiation. The term "flutes" is preferred for these features as it avoids confusion with other applications for the latter two terms.

Not all those who use fractography for materials research or failure analysis understand exactly what flutes are and what causes them to form. Some new observations of fluting under conditions of mechanical overload fracture, SCC, sustained load cracking (SLC) in inert environments, fatigue, and corrosion fatigue conditions will be presented to familiarize readers with a variety of flute characteristics and to provide new information concerning fluting mechanisms and the conditions that give rise to fluting. The following discussion includes a review of significant prior work, a summation of conditions and parameters important to flute formation, some comments on mechanisms of flute initiation and formation, and, finally, an assessment of the significance of flutes as a diagnostic fractographic feature in α and α - β titanium alloys.

Materials and Methods

Materials

A review of numbers of fractographs in the literature made it clear that flutes produced by SCC look similar in most near- α and α - β alloys such as Ti-5Al-2.5Sn, Ti-8Al-1Mo-1V, and Ti-6Al-4V. Alloy Ti-8Al-1Mo-1V was therefore selected for flute fractography studies in two metallurgical conditions: the β -annealed and furnace-cooled and the as-received "mill-annealed" condition. One other alloy of unusually high interstitial content, Ti-0.35O was chosen for examination of flutes formed under mechanical overload conditions.

The microstructures of both the beta-annealed (β A) and the mill-annealed (MA) Ti-8Al-1Mo-1V material are shown in Fig. 1. The β A material consists mainly of colonies or packets of similarly aligned α plates, with interplate β -phase. This is usually called coarse Widmanstätten alpha. The colonies or packets behave like single grains in many respects; for example, large cleavage facets consist of cleavage on a common plane through all the plates in a single facet, since they are all of essentially the same crystallographic orientation within a colony. However, the β -phase between the alpha plates does not cleave, and this constitutes a site for diversion of cracks. The MA material contains some fine Widmanstätten-like microstructures, but it consists mostly of a mixture of primary alpha (irregular grains) and so-called transformed

² The italic numbers in brackets refer to the list of references appended to this paper.

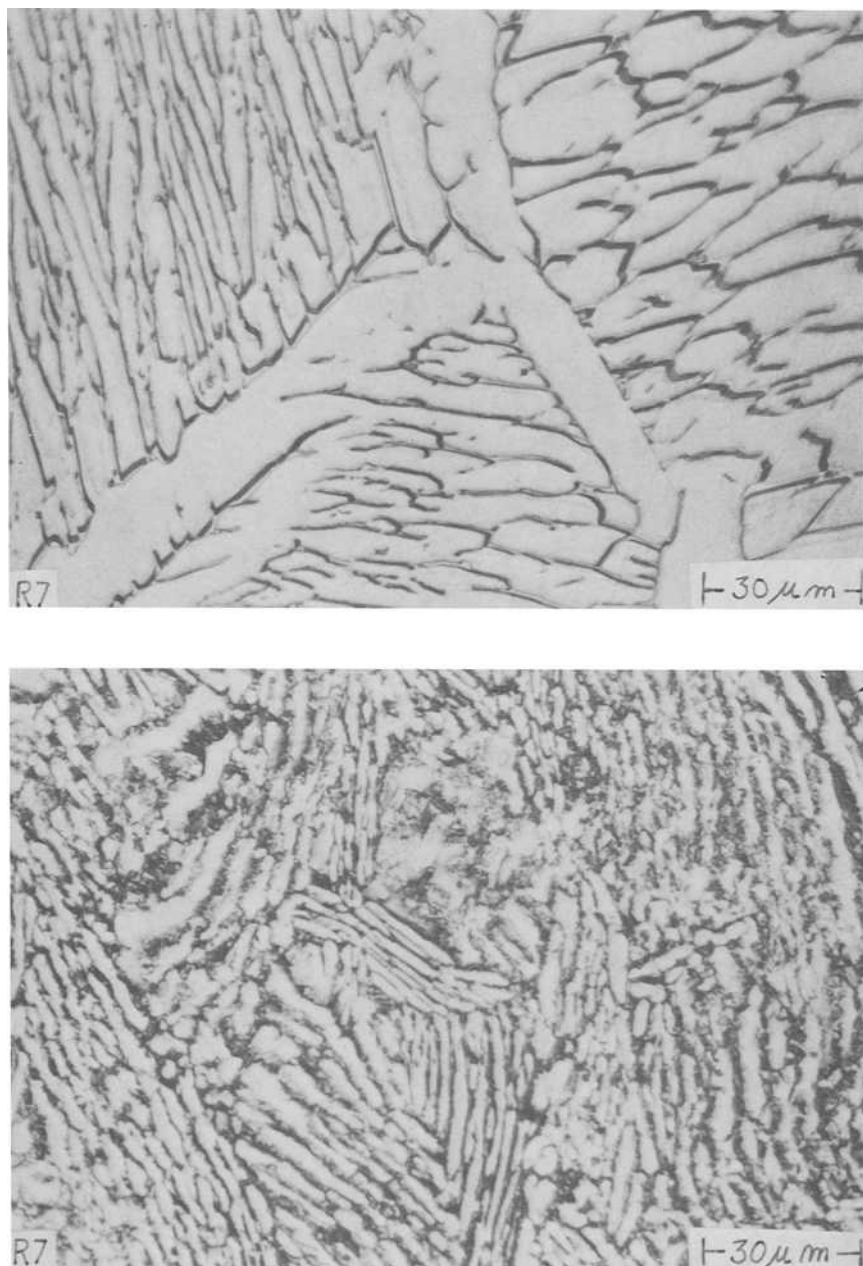


FIG. 1—Microstructures of Ti-8Al-1Mo-1V alloy, etched with Kroll's reagent, $\times 700$: (top) β annealed (1065°C , 4h) and furnace cooled; (bottom) α - β hot worked, mill annealed.

beta, a fine dispersion of alpha-phase in a skimpy beta matrix. The Ti-0.35O alloy (not shown) consists of very large angular and platelike grains with no β -phase.

The following presentation will refer both to α plates, which are platelike α grains, and to cleavage plates, which result from multiple cleavage through one or more α grains to produce partly isolated cleavage elements. This is illustrated in Fig. 2 for a single plate-shaped α grain, with some other features whose significance will be apparent later. In the hexagonal close-packed (HCP) crystal of the α -phase, the (0001) planes are often called basal planes, and planes and generalized surfaces perpendicular to (0001) are termed prismatic.

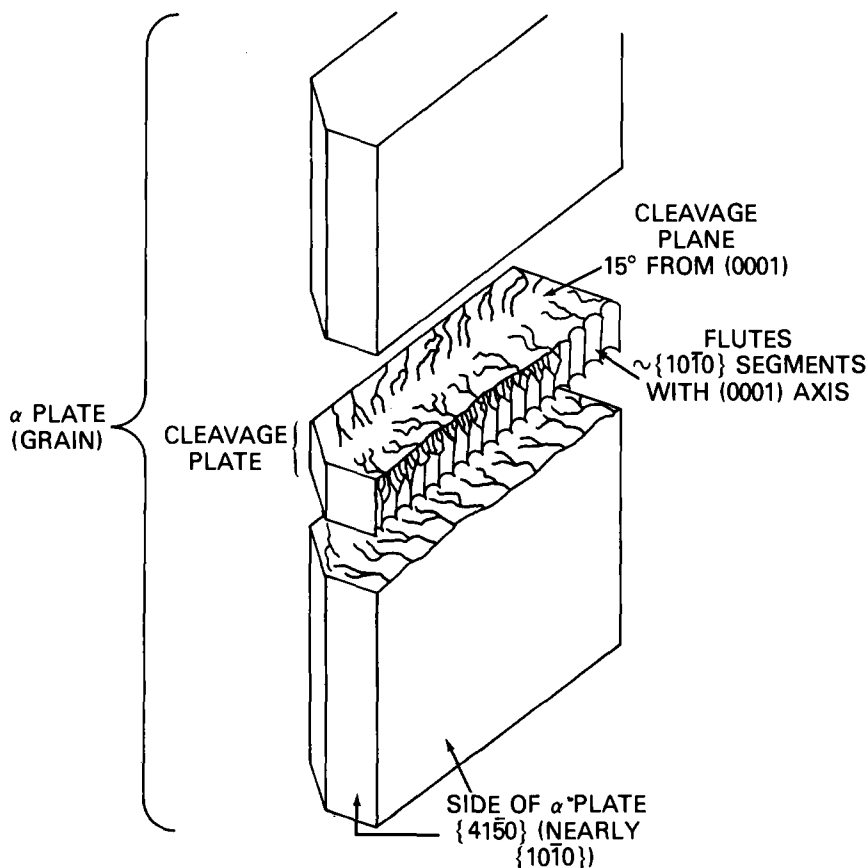


FIG. 2—Schematic illustration of a platelike α grain, cleaved into three parts on a plane 15 deg from the basal plane showing what is intended by the terms α plate and cleavage plate. Flutes are shown on one side to illustrate the geometrical relationship with the cleavage plane.

Experimental Methods

The Ti-0.35O alloy was notched, clamped in a vise, and broken off by a hammer blow. The specimens of Alloy Ti-8Al-1Mo-1V were either of the notched and fatigue precracked cantilever bend type or of the fatigue-precracked single edge-notched type. Stress-corrosion cracking tests were conducted by immersion in either 3.5 percent sodium chloride (NaCl) aqueous solution or in reagent-quality methanol. Sustained load cracking tests were conducted in vacuum [133 to 13.3 μPa (10^{-6} to 10^{-7} torr)]. The specimens were held at a constant load so that initial values of the stress-intensity factor were approximately equal to the threshold value, K_{Isc} or K_{Isc} , and were allowed to crack until fracture occurred. Fatigue experiments were conducted at approximately 30 Hz with sinusoidal waveform. The ratio of maximum to minimum stress, R , was in the range of nearly zero (zero-tension cycling) to 0.9 (tension-tension cycling). Most of the fatigue experiments were conducted in air, some in 3.5 percent NaCl solution.

The specimens were examined in a scanning electron microscope (SEM) at magnifications up to $\times 20\,000$. This technique visualizes flutes much better than does transmission electron fractography with replicas. Considerable use was made of stereoscopic imaging to evaluate the topographical characteristics of fluting.

Results

Description and Occurrence of Flutes

Figure 3 shows the most perfect examples of flutes that can be obtained, epitomizing the major topographical characteristics of flutes. This example was not produced by SCC but was caused by a mechanical overload fracture of Ti-0.35O. This high oxygen content produces deformation characteristics that lead to cleavage and planar slip, which results in fractures analogous to those caused by SCC in other alloys, as will be discussed later. The flutes clearly resemble striations in their regularity and parallelism but are of a variety of widths, from fractions of a micrometre to many micrometres in a single field of view. The tear ridges that delineate the flutes are extremely straight, merging into one another very abruptly to form so-called river patterns. Not all flutes have river-patterned tear ridges, for example, the coarse group at the upper center. The concavity of flutes is apparent even without the aid of stereoscopic examination in this case, and some impression can be gained that they are angular in cross section, as if this shape were determined by crystallographic characteristics. The fluted surfaces are approximately perpendicular to cleavage facets and in general are found more often at the torn-off edges of cleavage elements than elsewhere.

Figure 4 shows well-developed flutes at the edges of a group of cleavage

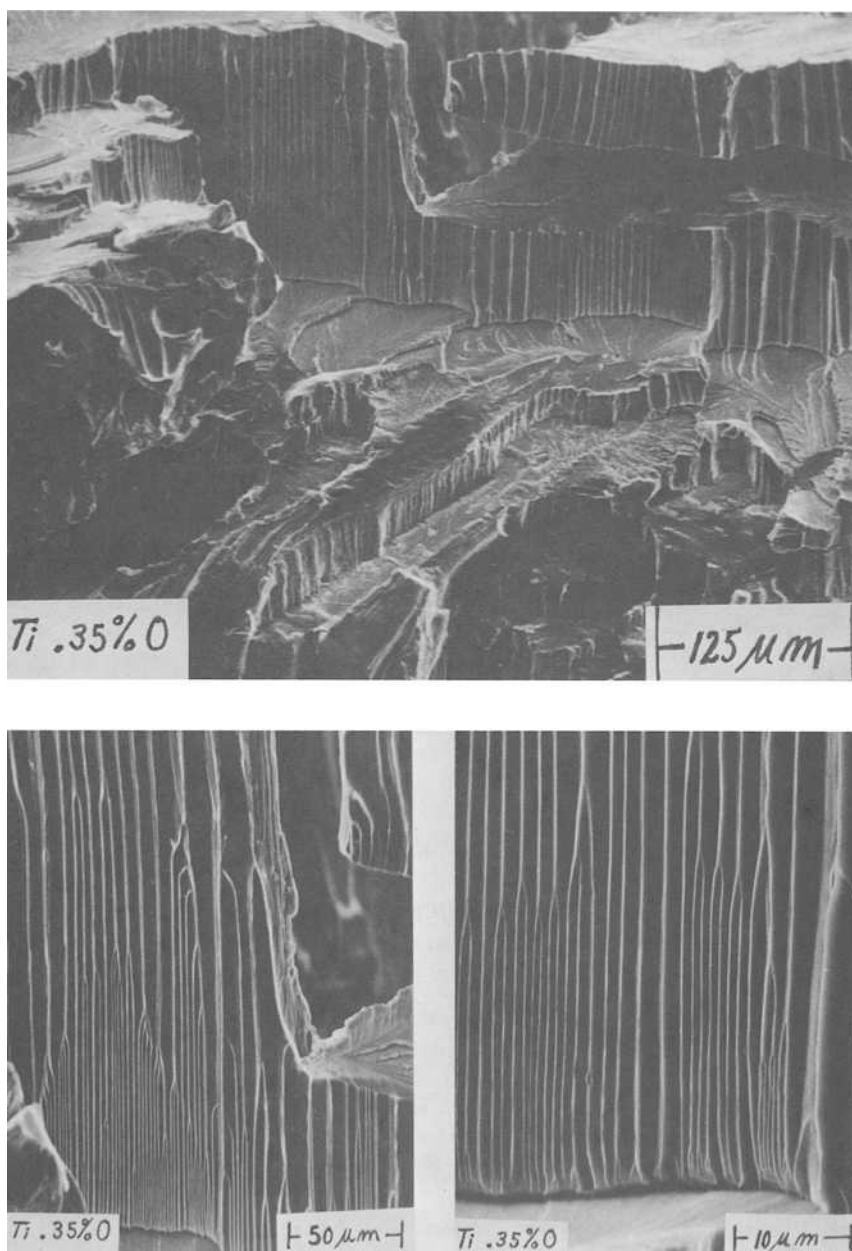


FIG. 3—Flutes and cleavage resulting from a mechanical overload fracture of Ti-0.35O alloy: (top) $\times 200$, (bottom, left) $\times 300$, (bottom, right) $\times 1500$. All the fractographs were taken by SEM.

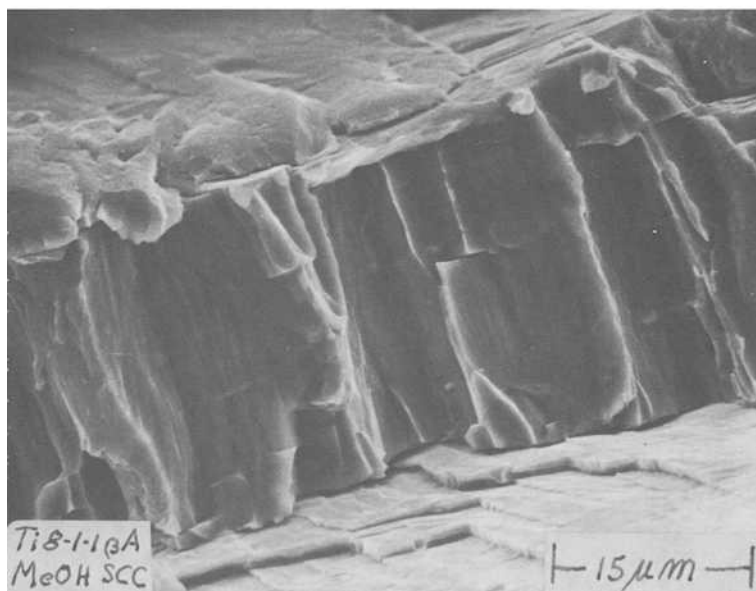
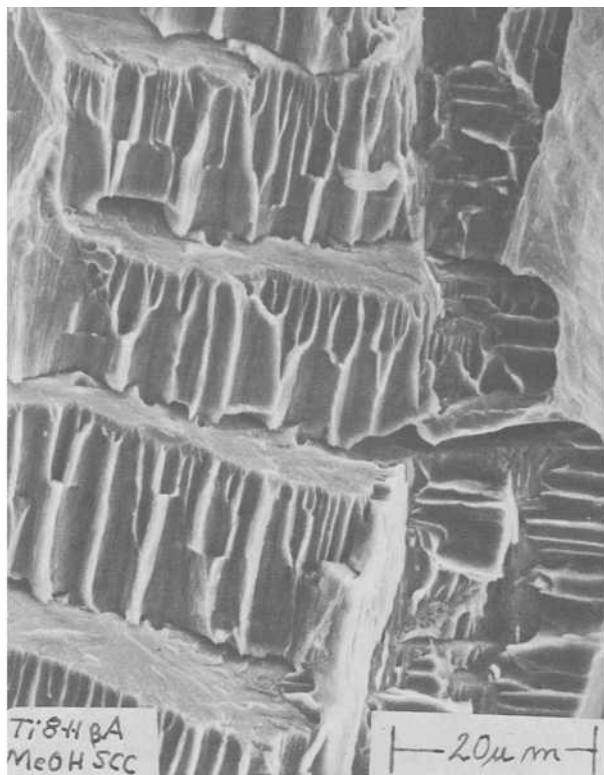


FIG. 4—Flutes and cleavage resulting from SCC of β -annealed Ti-8Al-1Mo-1V alloy in methanol: (top) $\times 1350$, (bottom) $\times 1730$, both from different areas.

plates formed by multiple cleavage in a single large α grain in β -annealed Ti-8Al-1Mo-1V cracked by SCC in methanol. This alloy has a relatively low interstitial content, and the resulting flutes are not as regular as those in Fig. 3. Alloy Ti-8Al-1Mo-1V does not develop flutes under mechanical overload fracture conditions; the presence of an aggressive environment and relatively slow crack growth rates seem necessary for fluting. The concave cross section and straight surface slip markings are particularly apparent in Fig. 4 (*bottom*), which again shows that not all flutes have tear ridges arranged in river patterns. The thin cleavage steps at the bottom of Fig. 4 (*bottom*) have very small, poorly developed flutes.

Figure 5 is a very complex photomicrograph illustrating some characteristics of flutes that bear on their microstructural formation mechanisms. The material is β -annealed Ti-8Al-1Mo-1V cracked by SCC in salt water. The figure is displayed as a stereo pair, and its examination in stereoscopic perspective is very worthwhile because certain characteristics will not be readily apparent otherwise. This can be done by holding two ordinary 50-mm (2.5-in.) diameter magnifying glasses up to the eyes like a pair of spectacles, viewing the figure straight down from about 125 mm (5 in.), eye height, and moving the glasses until the two pictures converge. Figure 5 (*bottom*) is a map of the principal divisions in Fig. 5 (*top*), and the letters C, F, and G indicate areas of cleavage, flutes, and grain-boundary fracture, respectively. The two regions outlined in heavy black are individual alpha grains whose grain-boundary surfaces are exposed.

The flutes in Fig. 5 vary from extremely fine to rather coarse and have all the characteristics already mentioned. However, the large group near the lower left does not obviously occur at the torn edge of a cleavage plate, and, in fact, the two small groups in the outer grain appear to start from the grain surface and penetrate the grain as intact tubular microvoids or tunnels of possibly rhombic or hexagonal cross section. One member of the group of large flutes at the lower left is askew and slightly bent, as if a local deviation into a grain of different orientation occurred. It is also somewhat out of the plane of the others.

Figure 6 shows flutes continuing as tubular voids, penetrating down the interface between two alpha plates of identical crystallographic orientation which have been cross sectioned at different levels by cleavage. It is probable that, had the flute-making process been completed by rupture along the boundary, an appearance much like that of Fig. 4 would have resulted. The degree of plasticity achieved suggests that the interface might be occupied by a thin layer of β -phase, but that is not certain. Figure 7 shows a cleavage plate that consists of double cleavage through a number of plates, and the cross section thus exposed shows the grain boundaries and grain boundary β -phase. At some of the grain boundaries small voids are forming, and the close-up in Fig. 7 (*bottom*) shows that the voids are forming at the α - β interface and appear to be propagating down the interface approximately perpendicular to the cleav-

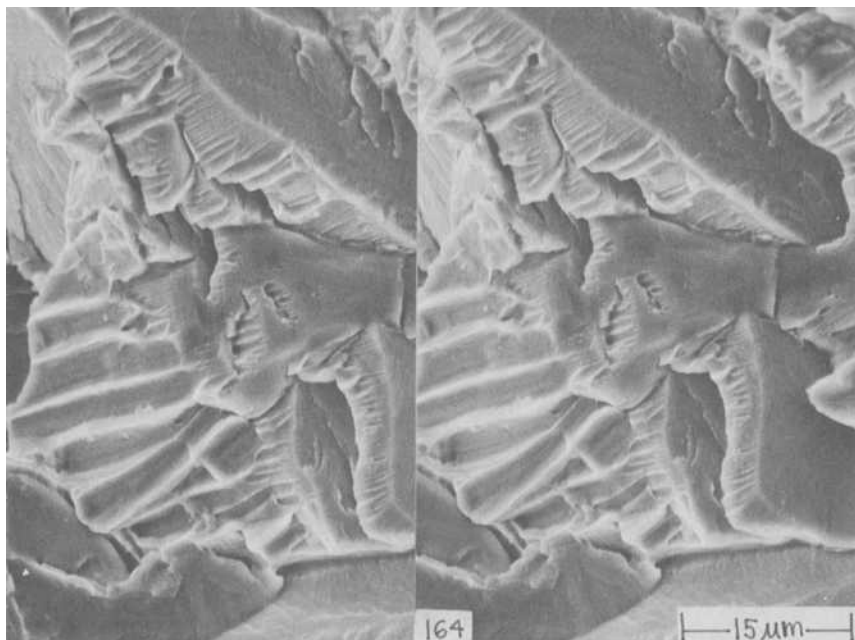


FIG. 5—Stereofractograph showing cleavage, flutes, and grain boundary fracture from SCC of β -annealed Ti-8Al-1Mo-1V in salt water: (top) $\times 1500$, (bottom) sketch indexing features in top.

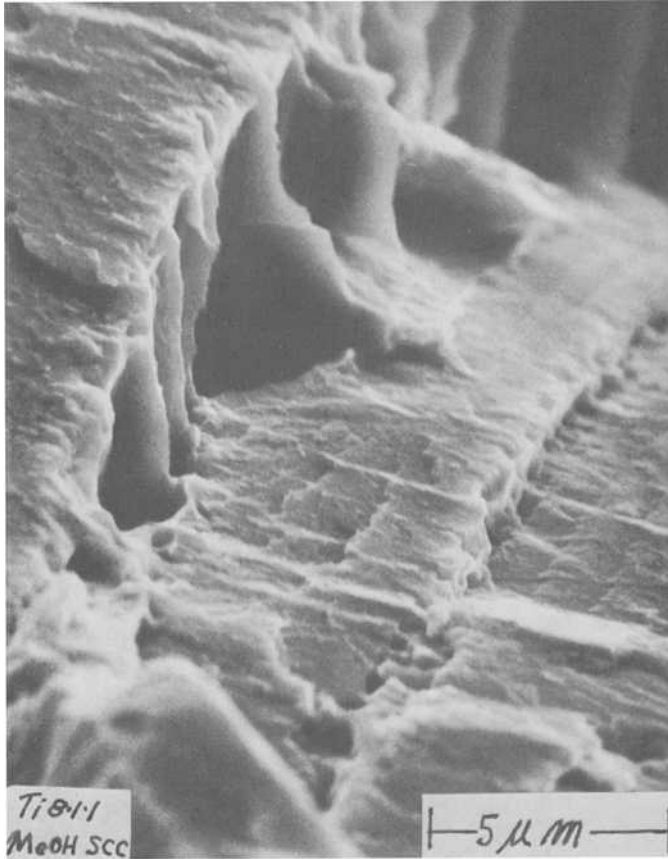


FIG. 6—Flutes and tubular voids penetrating between two cleaved α grains: SCC of β -annealed Ti-8Al-1Mo-1V in salt water, $\times 6400$.

age planes of the adjoining grains. Very little imagination is required to visualize the flutes that would result if this process continued to rupture, and at the top edge of the cleavage plate in Fig. 7 (*top*) are flutes which apparently have so formed at another α - β interface. Similar embryonic flutes appear at other locations in Fig. 7 (*top*). In all these locations the tubular microvoids are forming at the interface by joint deformation of α - and β -phases, not within the ductile β -phase itself. It even appears that most of the deformation is occurring in the α -phase, but this may possibly be an impression produced by the angle of view.

The previous illustrations have been derived from titanium alloys of relatively coarse microstructure. The much finer microstructure in alloys and heat treatments more likely to be found in structural applications are simulated by the mill-annealed Ti-8Al-1Mo-1V. Figure 8 shows examples of flutes in this

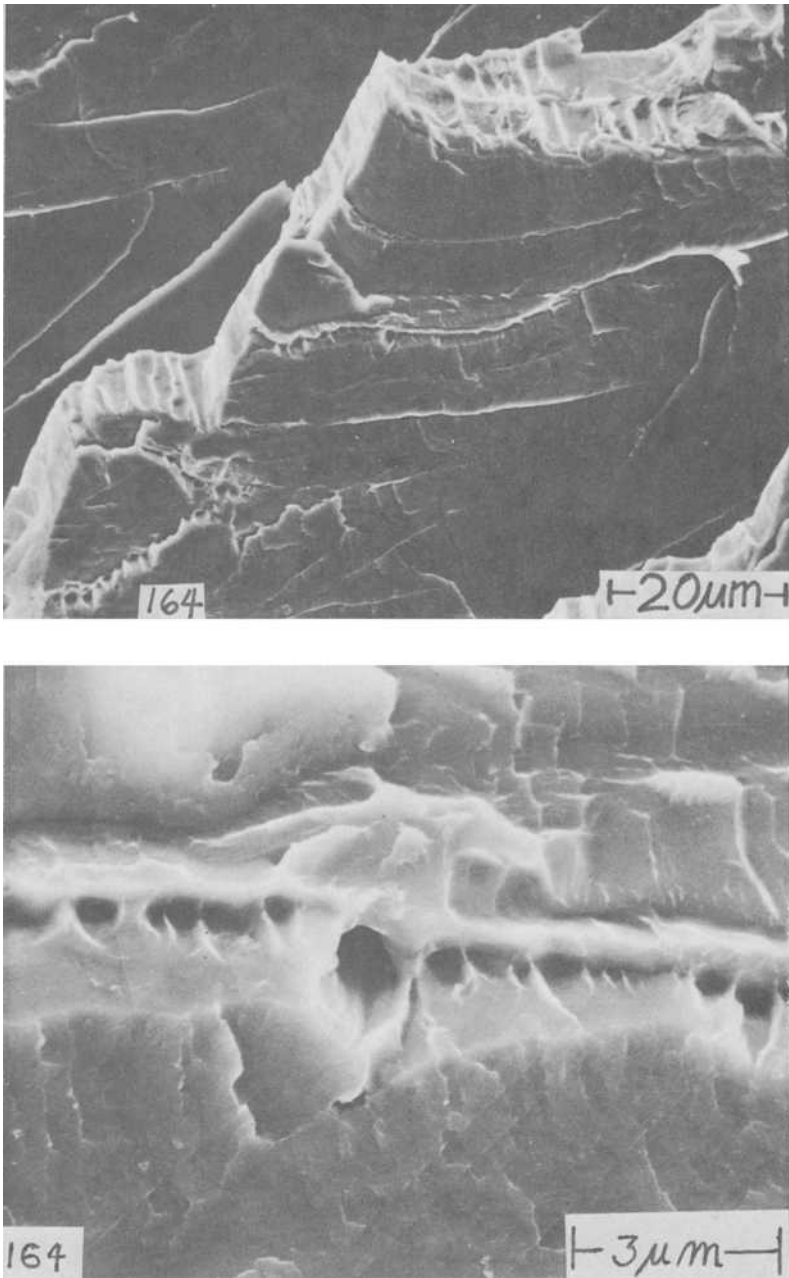


FIG. 7—Flutes at torn edges of cleavage plates and tubular voids penetrating at α - β interfaces: SCC in β -annealed Ti-8Al-1Mo-1V in salt water; (top) $\times 1150$, (bottom) $\times 9000$.

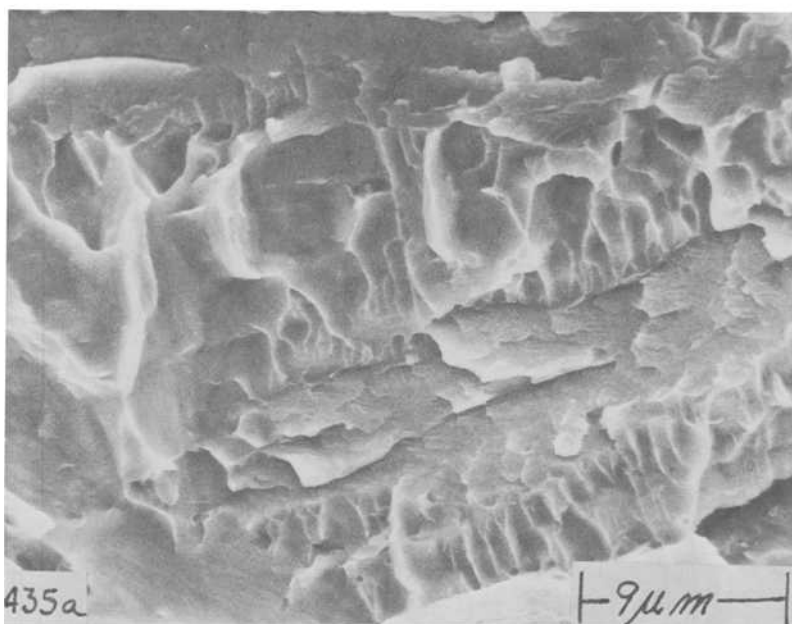
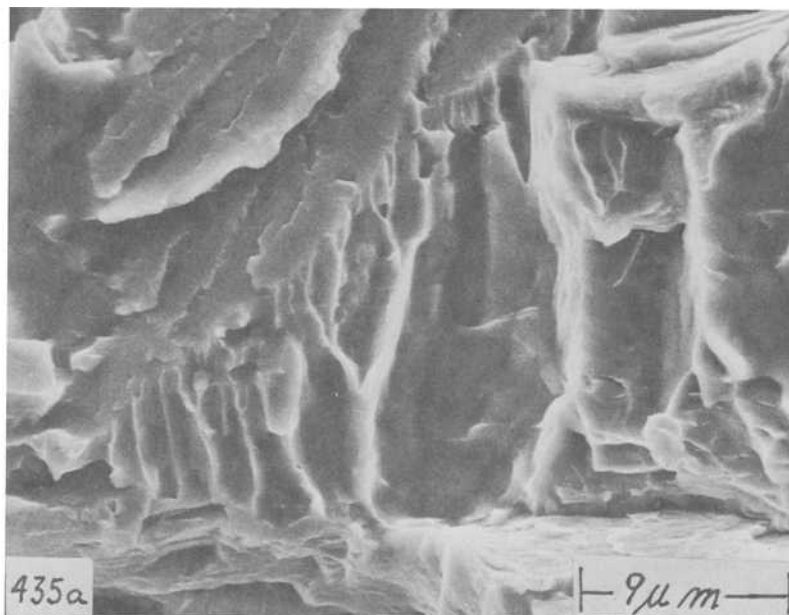


FIG. 8—Flutes and cleavage in fine-grained mill-annealed Ti-8Al-1Mo-1V: SCC in salt water, $\times 3000$.

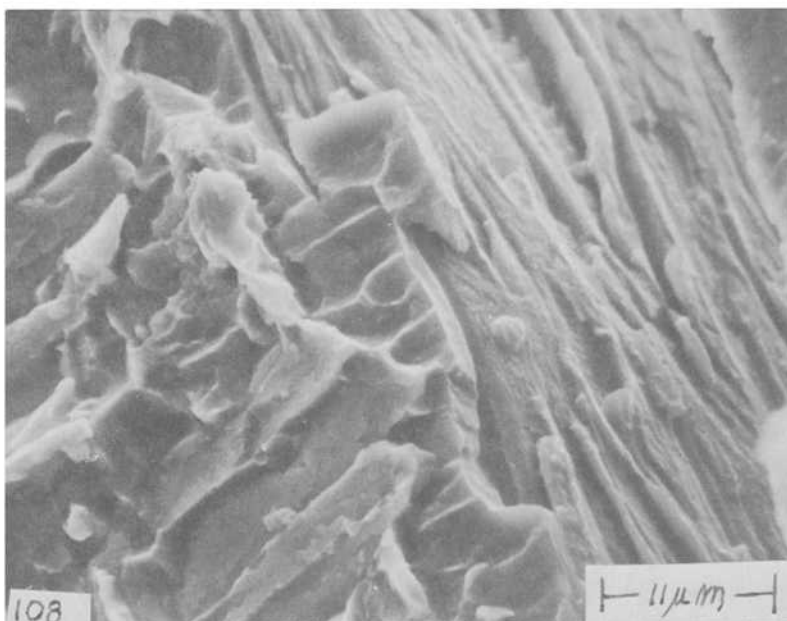
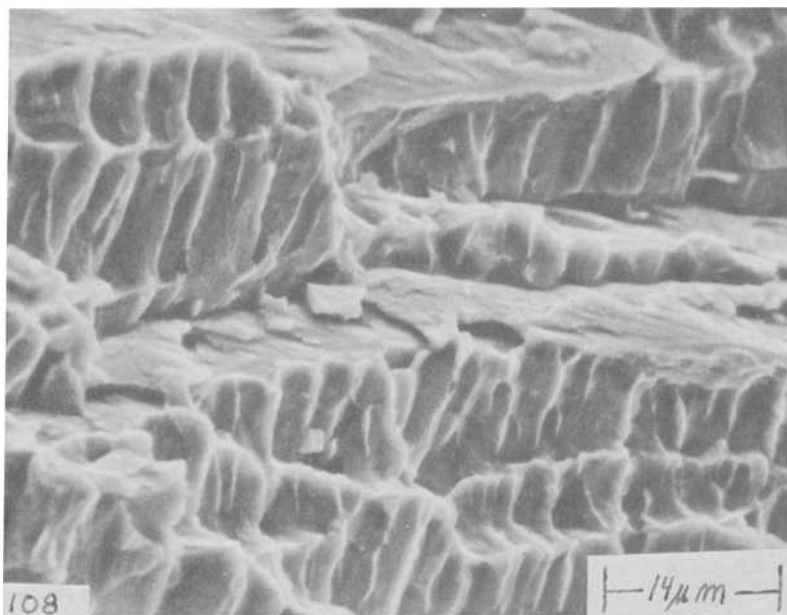


FIG. 9—Flutes and cleavage in β -annealed Ti-8Al-1Mo-1V; SLC in vacuum; flutes are not common in this case, (top) $\times 1650$, (bottom) $\times 2100$.

alloy cracked by SCC in salt water. The flutes are smaller, more poorly developed, and not as easy to find, primarily because higher magnifications must be used to search for them. However, they are generally readily found at the torn-off edges of cleavage plates, in cleavage steps, and in areas of jumbled or chaotic topography.

Some specimens of Ti-8Al-1Mo-1V and of Ti-6Al-4V broken by sustained load cracking in inert environments (air and vacuum) were examined for evidence of fluting. Figure 9 shows that flutes can be found, but very few such instances were discovered. These photomicrographs were taken on the SLC fracture surface of β -annealed Ti-8Al-1Mo-1V; the other materials showed no well-developed flutes; the broken-off edges of cleavage elements usually consisted of ordinary dimples or, in the alloys of high hydrogen content, of α - β interface cracking.

Fatigue Aspects

Fatigue produced flutes or flutelike features only under certain conditions. No flutes of the river-patterned type were found on specimens of Ti-8Al-1Mo-1V fatigued in air, even in β -annealed specimens. Specimens fatigued in salt water did show some flutes. Low amplitudes with low stress ratios (approximately zero-to-tension loading) caused some flute formation near the initiation surface in notched specimens, but further along the fracture surface none was seen. Figure 10 illustrates some of the better examples found on the fracture surface near the notch. As in the case of SCC, they were generally associated with cleavage. Flutes also appeared in specimens fatigued in salt water at sufficiently high stresses and low cyclic frequency that SCC was a major component of the cracking process. Specimens fatigued in salt water at very low cyclic stress amplitude with high stress ratios, and thus high average crack-tip stresses, might also be expected to show typical SCC fractures, but such experiments have not been done.

Low-amplitude fatigue fracture surfaces in either air or salt water had a characteristic, shown in Fig. 11, that was absent from SCC and SLC fractures. The broken-off edges of cleavage plates looked very angular and jagged, which suggests separation by a brittle cracking process. Figure 11 (*top*) shows this appearance for β -annealed Ti-8Al-1Mo-1V fatigued in air, Fig. 11 (*bottom*) for the mill-annealed alloy. When the brittle cracking process results in linear features such as those in Fig. 12, these might at first be mistaken for flutes, but closer examination should show that they are not the same but are composed of brittle fracture steps.

Figure 13 shows another aspect of typical fatigue fracture features that very superficially resemble flutes, especially at lower magnification, but the magnification of Fig. 13 clearly shows typical brittle fracture patterns and a square, blocky cross section totally unlike flutes. Figure 14 shows an example that is even more deceiving at low magnification [Fig. 14 (*top*)], but, at higher magnification [Fig. 14 (*bottom*)], the blocky, brittle fracture character is evident.

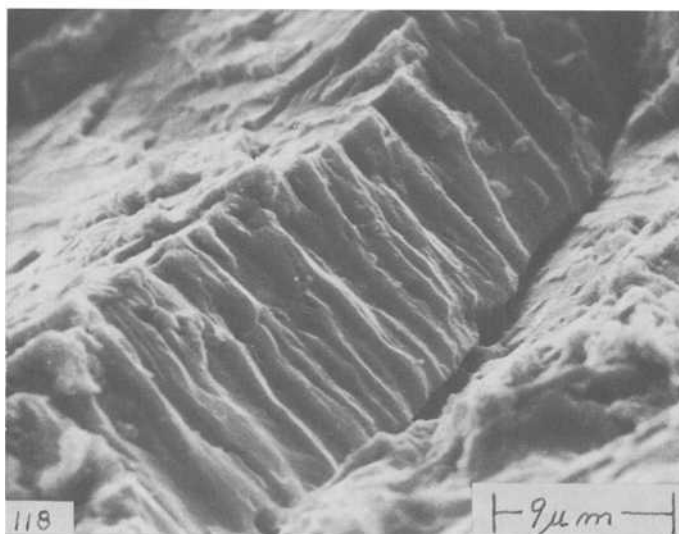


FIG. 10—Flutes occurring near the notch in the fracture surface of fine-grained mill-annealed Ti-8Al-1Mo-1V: corrosion fatigue in salt water; $K_{max} \sim \Delta K = 10 \text{ MPa } \sqrt{\text{m}}$; flutes are not common in this case; (top) $\times 2900$; (bottom) $\times 2600$.

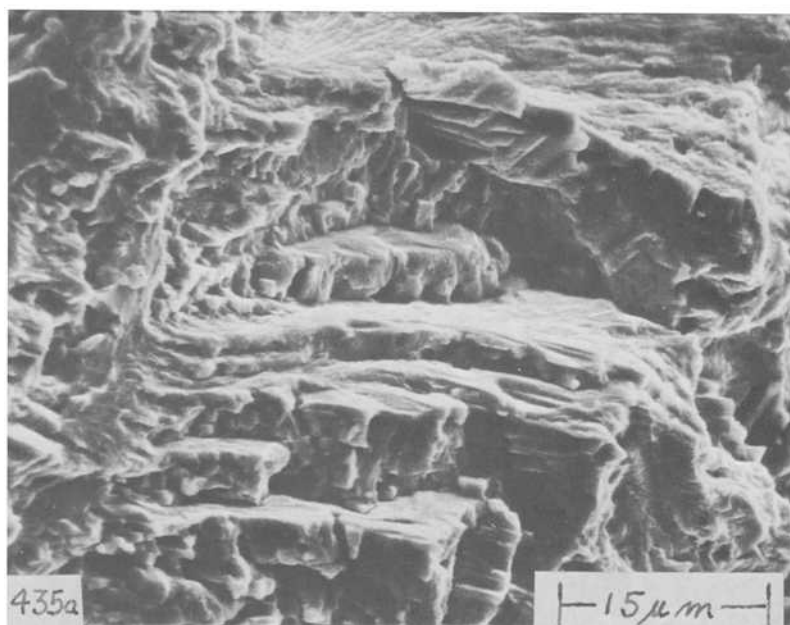
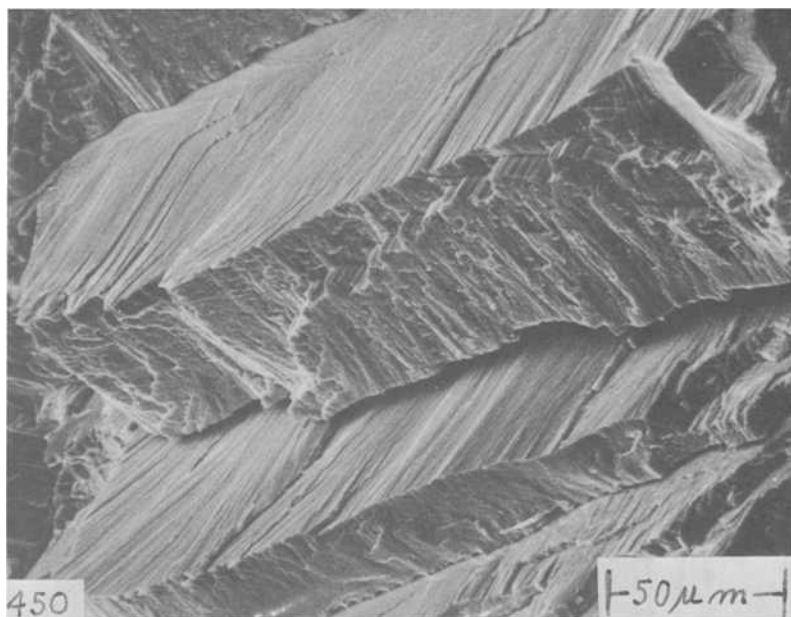


FIG. 11—The typical shattered, brittle cracking appearance of the broken-off edges of cleavage steps in low-stress striationless fatigue: (top) β -annealed Ti-8Al-1Mo-1V, $\times 460$; (bottom) mill-annealed Ti-8Al-1Mo-1V, $\times 1850$.

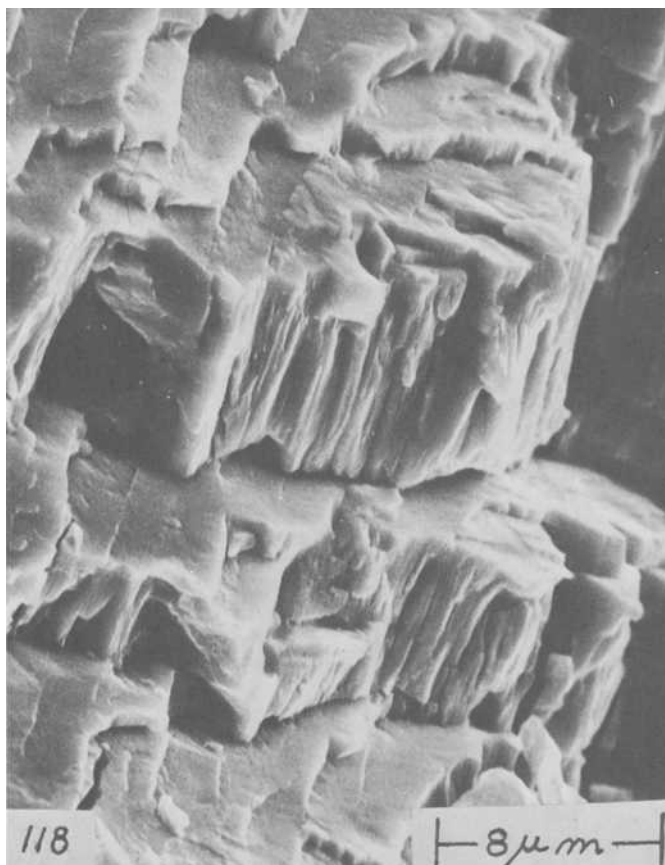


FIG. 12—Pseudofluting at the edges of cleavage steps in mill-annealed Ti-8Al-1Mo-1V: corrosion fatigue in salt water, $\times 3700$.

Discussion

Review of Previous Work

Scully and various co-workers brought attention to the characteristic features of SCC fractures in α and α - β titanium alloys that are here called flutes; these features were previously mistaken for cleavage river patterns [1] but this view was later corrected [5]. Hickman et al [6] attributed similar features to ductile tearing.

Aitchison and Cox [2] demonstrated by precise matching of identical features on mating fracture surfaces that flutes (also called striations in their paper) are the ruptured halves of tubular voids, resulting like ordinary irregular microvoids from internal necking by slip, which in the case of flutes is substantially restricted to the $\{10\bar{1}0\} \langle 11\bar{2}0 \rangle$ system. They also suggested the

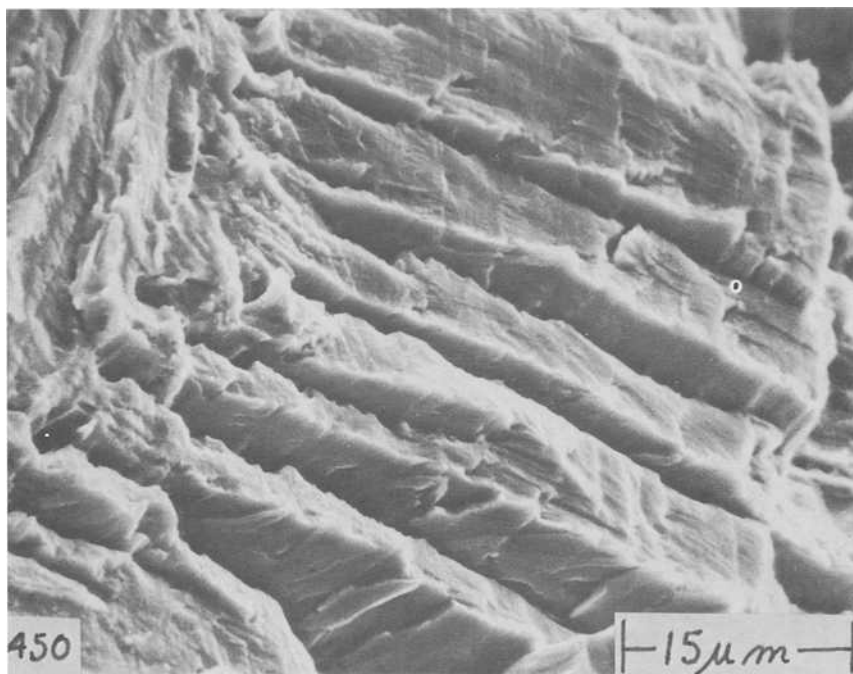


FIG. 13—An arrangement of separate cleavage elements, producing a river-pattern impression: air fatigue in β -annealed Ti-8Al-1Mo-1V, $\times 2000$.

term “flutings” for such features. Spurrier and Scully [5] arrived at substantially similar conclusions, attributing such “striations” to some type of planar slip mode, usually facilitated by the presence of cleavage facets that provided free surfaces at the ends of the tubular voids, reducing constraints on the slip processes which would otherwise be forced to operate three-dimensionally, thus producing more irregular voids. The effect of the environment in producing flutes was considered to be twofold: first, it encouraged cleavage; second, it caused generation and absorption of an interstitial, probably hydrogen, which altered slip characteristics in such a way as to encourage planar slip. Considerable experimentation on commercially pure titanium showed that increased interstitial content, oxygen or hydrogen, correlated with fluted fractures even when caused by purely mechanical overload fracture in the absence of active environments.

Wanhill [3, 4] observed that in addition to SCC in aqueous and methanolic environments, which can cause hydrogen absorption, liquid metals (mercury) also caused fluted fractures. He thus concluded that no specific causative agent gave rise to fluting. From precision matching and stereographic measurements of flutes, he concluded that flutes result from geometrical and mechanical factors that encourage preferential operation of intersecting $\{1010\}$

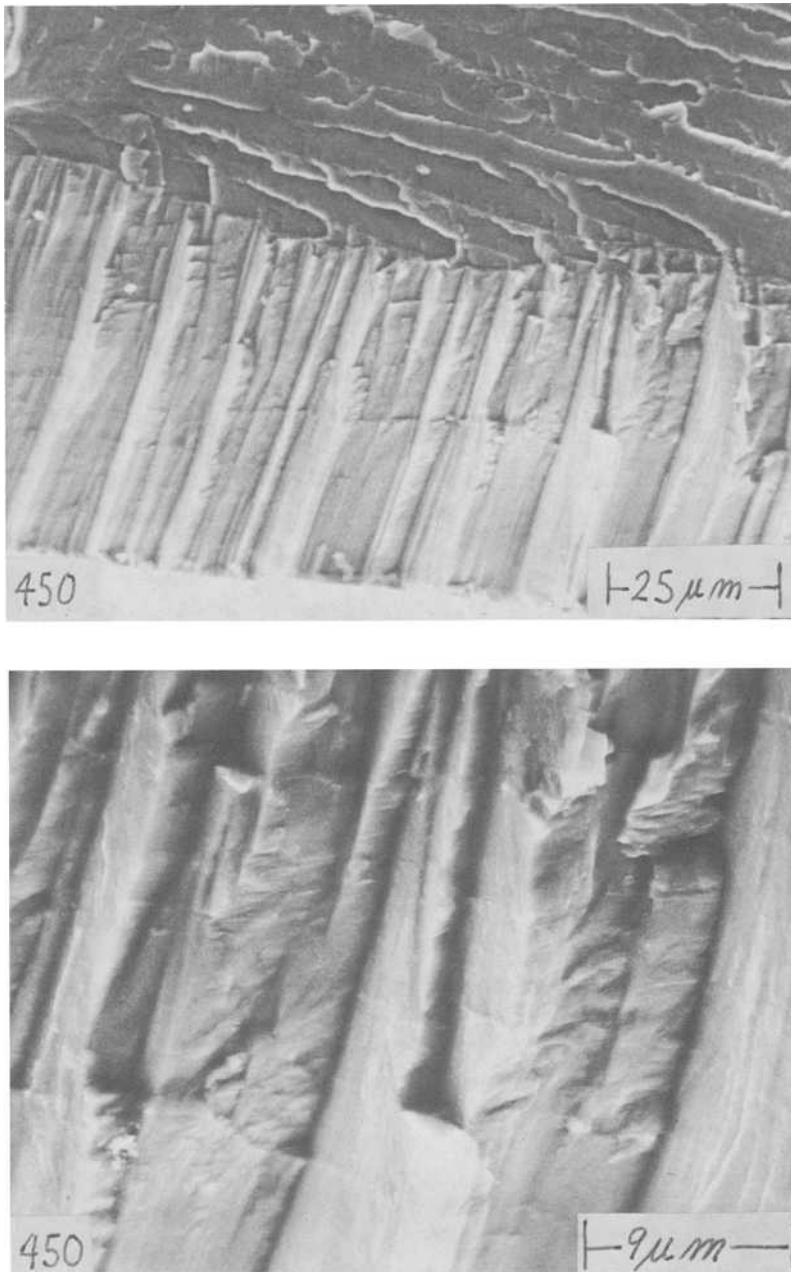


FIG. 14—The same effect as in Fig. 12, but more easily confused with flutes: air fatigue in β -annealed Ti-8Al-1Mo-1V; (top) $\times 900$, (bottom) $\times 3000$.

$\langle 11\bar{2}0 \rangle$ slip systems during tearing between cleavage elements to form tubular voids on prismatic surfaces. The effect of the environment was to create numerous cleavage surfaces that relaxed hydrostatic strains. He pointed out that the crystallographic texture in relation to the stress axis and crack plane will influence the occurrence of flutes and the ease with which they are observed. If (0001) is predominantly parallel to the crack plane, the flutes will be in surfaces perpendicular to the crack plane, not easily seen without tilting the specimens. If (0001) is perpendicular to the crack plane, the flutes will abound and be easily seen, but the associated cleavage elements will not be obvious.

Wanhill [7] has also pointed out that flutes occur under certain conditions of fatigue in aqueous environments, but these were not usually so well developed into river-line patterns or so numerous and regular as in SCC of the same alloys. Wanhill termed flutes in the fatigue case columnar slip surfaces rather than river patterns, which was his nomenclature in the SCC case. He noted that lower cyclic frequencies encouraged flutes, which became coarser and less river-patterned with increasing cyclic stress. The conclusion was reached that SCC-type, noncyclic cracking mechanisms were the cause of fluting amidst otherwise typical fatigue fracture modes.

The view that these flutes arise from tunneling by chemical dissolution at crack tips [8] has been advanced, but the results of the present investigation and that in Ref 5 just discussed, that flutes form even under SLC and mechanical overload conditions, particularly in alloys of high oxygen content, renders this supposition unnecessary. It also does not easily explain the straight slip traces and ample evidence of deformation around partly formed flutes. The authors did corroborate the general finding that flutes are associated with cleavage.

Van Stone et al [9] have studied the mechanisms of purely mechanical fluting in Ti-5Al-2.5Sn, caused by mechanical overload fracture at cryogenic temperatures. This alloy showed little tendency for formation of river-patterned flutes at room temperature; only equiaxed dimples occurred. At cryogenic temperatures the commercial alloy with about 0.16 percent oxygen showed extensive river-patterned fluting, whereas the extra-low interstitial (ELI) alloy containing 0.05 percent oxygen seemed to have flutes of more columnar, non-river-pattern type. Flutes were found to initiate at various slip band and twin intersections with twin and grain boundaries and to propagate along such boundaries. Low temperatures appeared to suppress all but $\{10\bar{1}0\}$ slip and to make even that system difficult to activate, which would encourage twinning. In this case cleavage, which was evidently not observed, did not play a role in fluting.

Chesnutt and Williams [10] have observed copious fluting from mechanical overload fracture of unalloyed titanium of unspecified oxygen content, verifying the origin of this fluting as tubular plastic deformation voids by precise matching studies in the SEM. Very similar river-line patterns, containing very clear fatigue striations, were also found under fatigue conditions. However,

these were different in detail, with linear cleavage elements running parallel to and in the plane of the pseudoflutes, and precise matching studies proved that they originated entirely from flat crack propagation, as they were not halves of tubular voids. The authors emphasized that the safest way to distinguish flutes was by precise stereo-fractographic matching to demonstrate their semitubular topography and exact correspondence of tear ridges on mating fracture surfaces.

Parameters Favoring Fluting

A summary of the parameters and conditions that appear to give rise to fluting in titanium alloys will be helpful in determining their causative mechanisms and in diagnosing fractures characterized (or not) by flutes. In the absence of aggressive environments flutes seem to occur under mechanical overload fracture only in alloys of high alpha stabilizer content (especially interstitials such as oxygen) or in other alloys at cryogenic temperatures [9]. No direct fractographic evidence is available concerning effects of aluminum in low-oxygen alloys, but because aluminum also causes planar slip, which renders cross slip difficult, and causes cleavage to occur [11], it should have similar effects. The effect of low temperatures seems similar to that of high oxygen content in restricting cross slip, but instead of producing cleavage, cryogenic temperature seems to encourage flutes by causing copious twinning [9].

The flute-making effect of aggressive environments seems to be to produce cleavage on planes approximately perpendicular to the prismatic fluting surfaces, thus providing favorable geometrical and mechanical conditions for fluting [3]. In the case of a corrosive environment, such as salt water, there may be the additional effect of absorption of the interstitials [5].

If the conditions for fluting are only marginally present, such as under cyclic stressing or monotonic loading in inert environments in alloys of low interstitial or moderate aluminum content, then reduced crack-tip stresses seem to increase the probability of fluting [1]. Alloy Ti-8Al-1Mo-1V, for example, does not flute when rapidly loaded to fracture in inert environments, but it does to a slight degree if held at a relatively low crack-tip stress and allowed to crack by SLC. Also, fluting is observed to some degree at low fatigue crack stresses in salt water but not at higher stresses [1].

A high degree of cold work can cause cleavage and fluting under SCC conditions in pure titanium of low oxygen content, which, in the annealed condition, would fail by intergranular cracking [12]. The effect of cold work may be to raise yield strengths by locking up dislocation sources, which would make cleavage more probable and hence provide conditions favorable for fluting.

Flutes appear to be more copious in alloys of strongly anisotropic crystallographic texture when the basal plane is parallel to the stress axis and perpendicular to the crack plane [3]. Such an orientation favors formation of tubular

voids by intersecting slip on $\{10\bar{1}0\} \langle 11\bar{2}0 \rangle$ systems and hides the cleavage facets from view, as they tend to be seen end on.

Mechanisms of Fluting

A discussion of fluting mechanisms must take account of the factors just discussed. The mechanisms for fluting at cryogenic temperatures in the absence of extensive cleavage are apparently specialized and related to extensive twinning and planar slip; the reader is referred to Ref 9 for this discussion. Fluting at the tips of intergranular SCC cracks in methanol-hydrochloric acid is also a peculiar circumstance and not very well understood [5]. This discussion will concentrate on the most common cases, where fluting is strongly associated with cleavage.

The most probable mechanisms, proposed by Aitchison and Cox [2] and further developed by Wanhill [4], are based on geometrical and mechanical

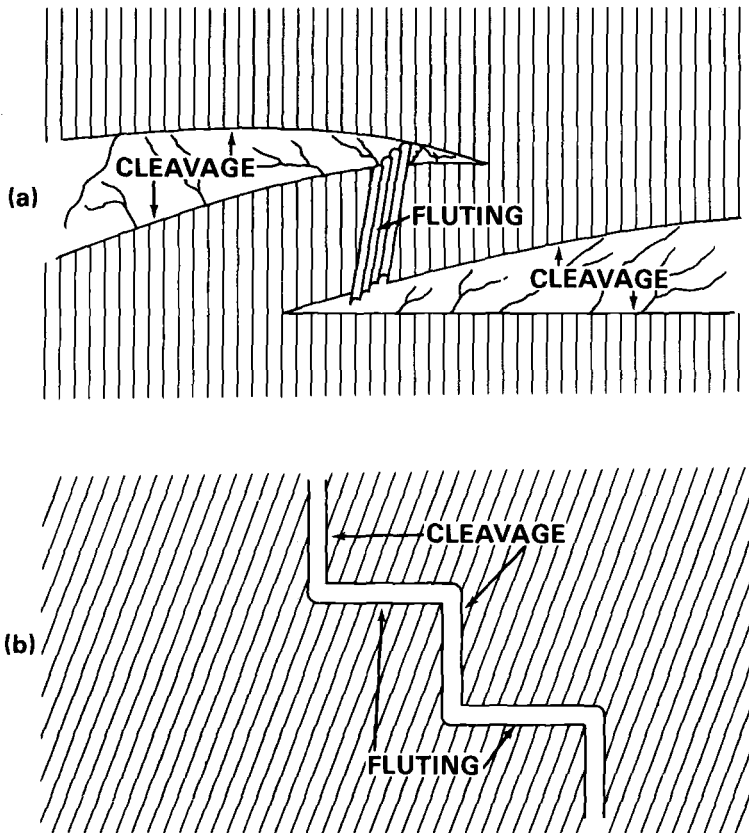


FIG. 15—Sketches illustrating flute mechanisms: (a) tearing between overlapping cleavage; (b) tearing between stepped cleavage elements.

circumstances created by cleavage in an HCP crystal structure. The simplest case, possibly that envisaged by Aitchison and Cox, is illustrated in Fig. 15a. Here, two cleavage cracks have overrun each other, and the three-dimensional fracture process can only be completed by tearing between them, there being no orthogonal cleavage plane available in the HCP α -phase. In alloys with significant oxygen or aluminum content, slip is planar [11], cross slip is difficult, and the formation of tubular microvoids by intersecting $\{10\bar{1}0\} \langle 11\bar{2}0 \rangle$ slip should be easier than the formation of ordinary microvoids, which would require very complex multiple and cross-slip processes. A slightly modified version of Wanhill's explanation of the process is that small plastic tunnels initiate preferentially at the most highly stressed cleavage surface, since there is always some bending component, probably at jogs and steps in the cleavage surface. These would tend to coalesce in the direction of tube propagation as strain increased, resulting in the observed broadening and proportional deepening of the tubes and in a consequent river pattern of the resulting tear ridges. Wanhill has suggested that $(0001) \langle 11\bar{2}0 \rangle$ slip is required at the propagating ends of these voids, and he points out that the addition of α stabilizers, such as oxygen and aluminum, tends to promote such slip. Figure 15b illustrates the staircase effect achieved by alternation of the cleavage and fluting elements. Examples such as those in Fig. 15a and b can be seen in the fractographs presented earlier. It should be emphasized that fluting apparently is dependent upon the prior existence of free surfaces approximately transverse to the prismatic slip surfaces, to permit easy development of columnar prismatic slip. This is thought to be the primary reason why flutes occur under conditions that cause multiple cleavage, such as SCC, liquid metal embrittlement (LME), certain kinds of corrosion fatigue, and mechanical fracture in unusually brittle alloys such as Ti-0.35O.

Figure 16a illustrates the particular case envisaged by Wanhill [3]. The cleavage in Grain B, propagating from right to left, hits the boundary with Grain A. If the boundary is resistant to cracking, because of β -phase or because the adjacent grain is misoriented for cleavage, the crack can continue by fluting down to another cleavage element. No river patterns are shown in order to simplify the drawing, but they would be fine at the top and coalesce and coarsen in the downward direction. Figure 16b shows a possible variant of this process, which may have caused the incomplete fluting in the central grain whose surface (grain boundary) is exposed in Fig. 5. The cleavage in Grain A, propagating from left to right, hits Grain B, and fluting is initiated into Grain B, instead of into the same grain in which the cleavage occurred. Subsequently, a grain boundary crack between Grains A and B relieves stresses that might otherwise complete the fluting fracture.

Figure 17 shows still another mechanism for flute formation, differing from the preceding mainly in the special characteristics conducive to fluting provided by a peculiarity of α - α and α - β interfaces in titanium alloys. Straight, flat segments of α grain boundaries, and particularly the flat sides of platelike

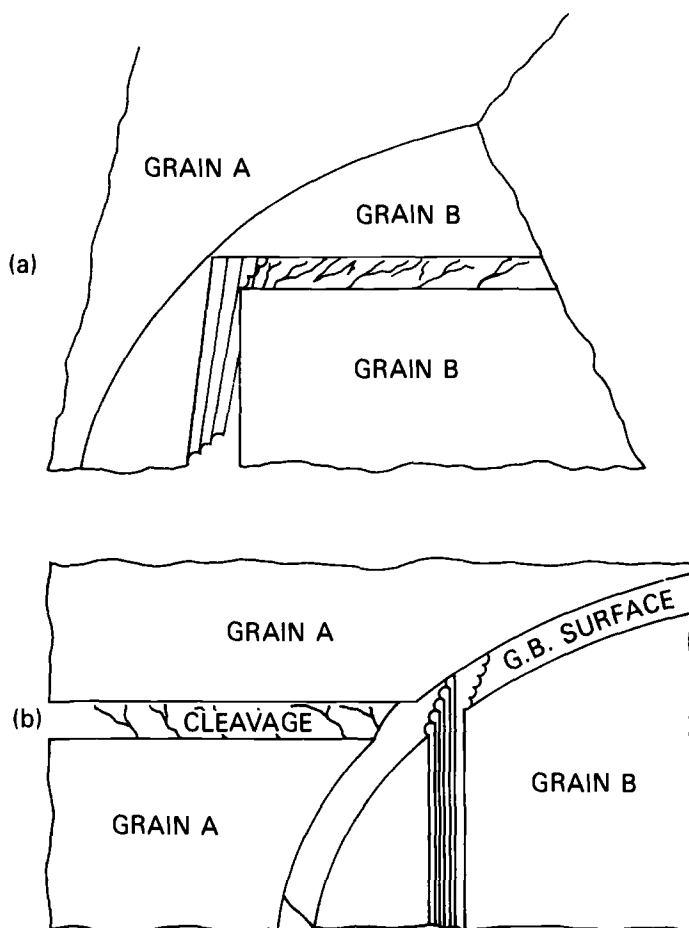


FIG. 16—Sketches illustrating fluting mechanisms: (a) fluted tearing initiating at the intersection of cleavage with the grain boundary; (b) Same as (a) except the tearing is in a different grain and there is a complication with a grain boundary fracture.

α grains in the Widmanstätten microstructure, have a very strong tendency to lie on planes of the type $\{41\bar{5}0\}$ [13], which are prismatic planes rotated 11 deg from $\{10\bar{1}0\}$ about $[0001]$. This is illustrated in Fig. 2. When cleavage on (0001) or 15 deg from (0001) intersects such a boundary, its favorable orientation for cracking by fluting along prismatic surfaces should result in an easy fracture path. Such fluting along α - α or α - β interfaces appears to have happened in the upper part of Fig. 5 (*top*) and in Figs. 6, 7, and 8 at several locations. Some of these observations are based on the suggestive shapes of the torn-off edges and on the appearance, in some cases, of possible β -phase at the fluted boundaries.

There is some evidence that chemical activity may play some part in fluting

under SCC conditions. The experiments of Spurrier and Scully [5] in particular seem to point to the possibility that local absorption by the alloy ahead of the crack tip of a chemical reaction product may change its slip and cleavage characteristics locally to favor fluting. The agent suggested was hydrogen, which in high concentrations is known to cause the formation of hydride precipitates on prismatic planes under plastic straining [14]. However, numerous complex experiments by Spurrier and Scully [5] and by Wanhill [3, 4] have not determined conclusively the role of hydrogen, and the fractures of titanium alloys thermally charged with hydrogen [15] or exposed to hydrogen gas during cracking [16, 17] usually exhibit flat, brittle α - β interface cracking with or without cleavage, and, except for small tear ridge formations at the edges of α - β interface facets, flutelike features do not seem characteristic of such fractures.

Cox [18] and Wanhill [19] have discussed directional, crystallographic etch pit formation under SCC conditions in zirconium and titanium alloys, respectively, and Cox appears to have suggested that such pits might serve as nuclei for flute formation. No evidence for this type of initiation has been found in the present study, but if such fluting nuclei form (directional etch pits) it is at least possible that they might be distorted beyond recognition by the deformations attending flute propagation.

Distinguishing SCC from Striationless Fatigue

The foregoing results and discussion make it clear that fracture surface flutes, with or without characteristic river-line patterns, can occur under a variety of conditions, in various types of α and α - β titanium alloy systems. Nonetheless, fractures *characterized* by cleavage and river-patterned flutes occur

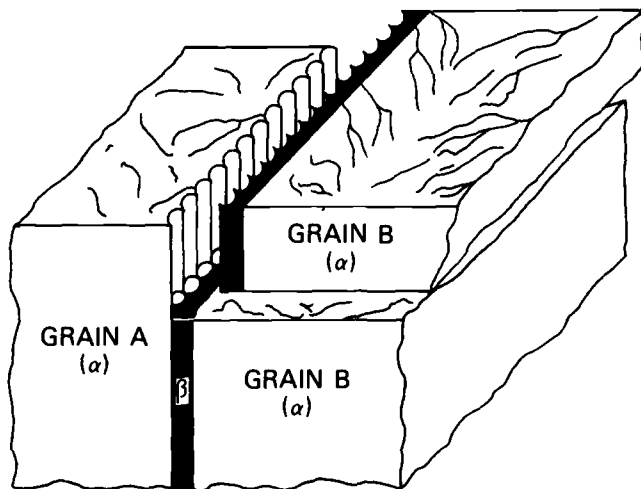


FIG. 17—Sketch illustrating fluting along an α - β interface.

under more limited circumstances. In no case shown in the literature nor in the present investigation was it found that striationless fatigue or corrosion fatigue fracture surfaces were characterized by cleavage with copious river-patterned flutes, as distinct from surfaces having scattered flutes or only flutes of the straight columnar slip type [1]. Further distinction between fatigue and other fracture surfaces that might consist mostly of cleavage with some flutes is provided by the kind of fragmented, brittle, broken-off cleavage plate and step edges discussed earlier in reference to Fig. 11. Such characteristics were only found under fatigue conditions.

Certain corrosion fatigue conditions are so little different from SCC that difficulty may be experienced in making fractographic distinctions. When the stress intensity factor, K_I , during any part of a fatigue load cycle exceeds the SCC threshold, $K_{I_{SCC}}$, and the cyclic frequency is low enough that most of the crack propagation is due to SCC, then few, if any, striations may be seen, and the fracture will, for practical purposes, be typical of SCC. Likewise, very small fatigue amplitudes with a high stress ratio with the average value of $K_I \sim K_{I_{SCC}}$ may produce SCC-type fractures. Under such conditions, however, attempts to distinguish the presence of a fatigue aspect in the fracture process may be entirely academic, since SCC is, in truth, the major cause of fracture.

An intermediate case is represented by the conditions of Fig. 10. In this case, fatigue with $R = 0$ (zero-tension fatigue) in salt water has produced clearly river-patterned flutes in scattered locations near the notch only, even though $K_{I_{max}}$ (about $10 \text{ MPa} \sqrt{\text{m}}$) was considerably below $K_{I_{SCC}}$ (about $20 \text{ MPa} \sqrt{\text{m}}$). It is believed that this was caused by multiple initiation and propagation of fingerlike cracks, which later linked up by tearing between cleavage elements as the local stresses rose. Once a relatively continuous crack front is achieved, such extensive, isolated, and highly stressed unbroken ligaments are seldom found. Despite the presence of river-line flutes near the notch, however, the additional presence of many sharp, brittle-looking broken-off cleavage plate and step edges gives ample warning that fatigue was the main operative mechanism.

Summary

1. Flutes, also termed striations or (slip) river patterns, are ruptured halves of tubular voids which form on prismatic surfaces by planar, intersecting slip on $\{10\bar{1}0\}$ planes in the α -phase or at α - α and α - β interfaces. They are primarily associated with near-basal cleavage, the role of which is to relax three-dimensional stresses and facilitate columnar slip on prismatic planes.

2. High α stabilizer content, especially interstitial oxygen, but also aluminum, and cryogenic temperatures promote fluting by restricting cross slip and promoting columnar slip. Conditions such as SCC and LME, as well as high α stabilizer content, promote fluting indirectly by causing multiple cleavage. Stress-corrosion cracking may also have a specific chemical effect of promoting absorption of interstitial reaction products at the crack tip.

3. The river-pattern aspect of flutes arises from the coalescence of the associated tear ridges in the direction of tubular void propagation and has no connection with cleavage river patterns. Flutes that do not have river-patterned tear ridges might also be termed columnar voids.

4. Corrosion fatigue fracture surfaces consisting mainly of cleavage and devoid of striations may be distinguished from SCC by the paucity of *river-patterned* flutes and by the presence at broken-off edges of cleavage plates and steps of a characteristic shattered, brittle cracking appearance.

Acknowledgments

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Influence of Microstructure and Environment on the Fatigue Crack Growth Fracture Topography of Ti-6Al-2Sn-4Zr-2Mo-0.1Si

REFERENCE: Ruppen, J. A. and McEvily, A. J., "Influence of Microstructure and Environment on the Fatigue Crack Growth Fracture Topography of Ti-6Al-2Sn-4Zr-2Mo-0.1Si," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 32-50.

ABSTRACT: An examination of fatigue fracture surfaces as influenced by microstructure (β processed versus $\alpha + \beta$ processed) and environment (air versus vacuum at 298 and 811 K) for a high-temperature creep-resistant near- α titanium alloy, Ti-6Al-2Sn-4Zr-2Mo-0.1Si, has been undertaken utilizing scanning electron microscopy. Results show that fracture topography is very sensitive to microstructural and environmental causes. Beta processing leads to a more irregular fracture surface than $\alpha + \beta$ processing. At low growth rates (10^{-6} mm/cycle) in air, shear mode growth predominates, resulting in large transgranular facets, whereas at growth rates of 10^{-5} in./cycle, striated and dimpled fracture features were present. At elevated temperatures, where oxidation effects were quite pronounced, particularly at low growth rates, the fracture surface was smoother than at room temperature, but faceting persisted to higher growth rates. In vacuum, however, the degree of faceting and striation formation was markedly reduced. Finally, the observed fractographic features influenced by microstructure and environment are discussed in terms of the mechanism of crack growth.

KEY WORDS: titanium, fatigue crack growth, environment, temperature, fractography, materials, materials science.

Microstructure and environment are known to influence the fracture behavior of titanium alloys [1-15].² The influence of microstructure and environment is reflected in the fracture topography and can often be related to structure-sensitive fracture mechanisms that affect crack growth characteristics. It is important to establish these correlations between fractographic features and the microstructure and to relate them to the separation process that occurs during fatigue crack growth. The present work describes the effect of the processing history, temperature, and environment on the fatigue crack growth

¹ Graduate research assistant and professor, respectively, School of Engineering, Department of Metallurgy, University of Connecticut, Storrs, Conn. 06268.

² The italic numbers in brackets refer to the list of references appended to this paper.

fracture characteristics of a creep-resistant, near- α titanium alloy, Ti-6Al-2Sn-4Zr-2Mo-0.1Si.

Material and Experimental Procedure

Two pancake forgings were used in this investigation. One of these was $\alpha + \beta$ forged prior to β heat treatment and aging, and the other was β forged prior to $\alpha + \beta$ heat treatment and aging. The microstructures developed as a result of the processing and heat treatment are shown in Fig. 1. Both heat treatments resulted in colonies of similarly aligned α platelets and regions of coarse α -phase. (The α -phase was separated by the body-centered cubic β -phase.) In addition, the β heat-treated forging is characterized by large prior β grains (700 μm) outlined by grain boundary α -phase and regions of α platelets arranged in a basket-weave morphology. Table 1 lists the pertinent microstructural and mechanical properties.

Fatigue crack growth testing was carried out with compact tension specimens of 6.3-mm thickness and a half-height-to-width ratio, h/W , of 0.6. Crack length was measured using a $\times 15$ optical telescope, and crack growth rates were determined using a 7-point incremental polynomial method [16]. The stress-intensity factor, ΔK , was calculated using the following formula [16]

$$\Delta K = \frac{\Delta P(2 + \rho)}{B \sqrt{w}(1 - \rho)^{3/2}} \quad (0.886 + 4.64\rho - 13.32\rho^2 + 14.72\rho^3 - 5.60\rho^4)$$

$$\rho = a/W$$

valid for $a/W > 0.2$

where B is the specimen thickness, a is the crack length, and P is the applied load. The crack growth data were obtained in air and in vacuum 2.7×10^{-3} Pa (2×10^{-5} torr) at 298 and 811 K under constant amplitude loading with a sine waveform and a load ratio, R , of 0.05, at 30 Hz. Elevated temperature was obtained in air by induction heating and in vacuum by radiation heating utilizing a tantalum heating element. The fracture surface topography was studied using scanning electron microscopy (SEM).

Results

The fatigue crack growth results are shown in Fig. 2 and cover the range of growth rates from 10^{-6} to 10^{-3} mm/cycle. Although the processing history had little effect on the fatigue crack growth rates, it did influence the fracture topography and crack extension characteristics. Under all test conditions, crack bifurcation, induced by the microstructure, occurred. This type of cracking contributes to the tortuous crack growth behavior and is common in titanium alloys having colonies of similarly aligned α platelets [2, 4-6, 14, 15]. An ex-

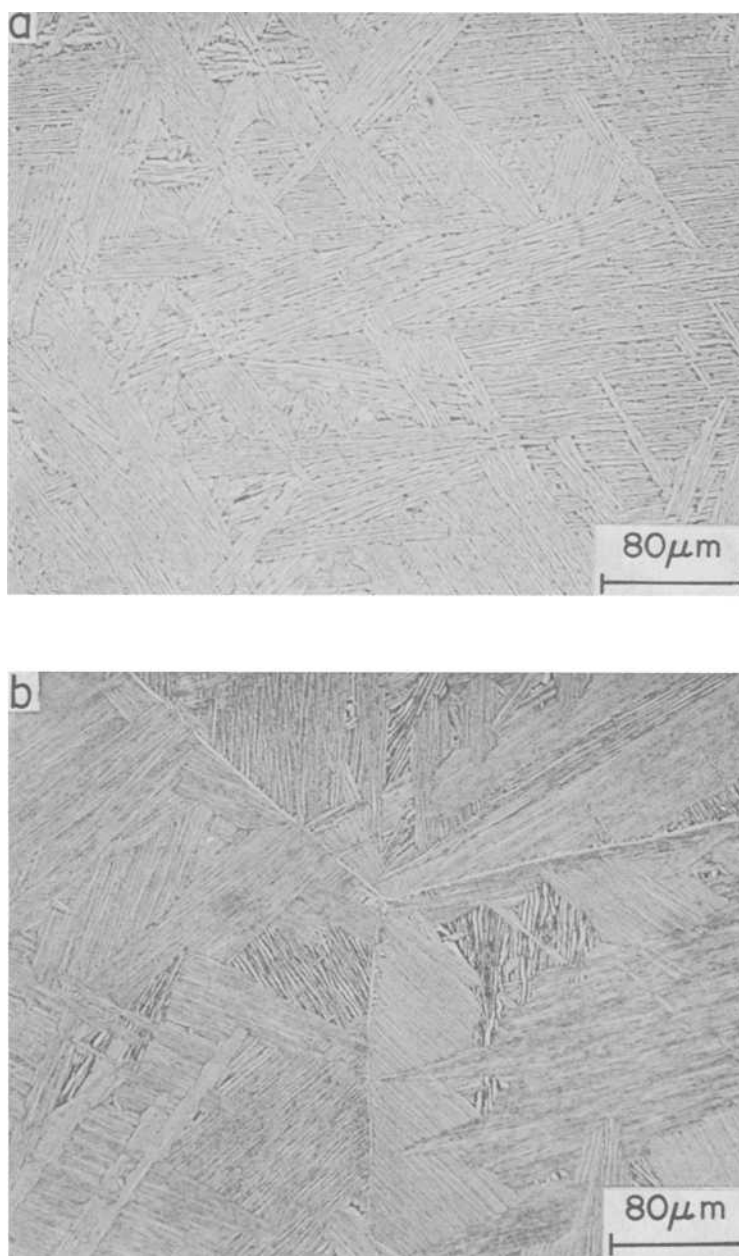


FIG. 1—Alloy Ti-6Al-2Sn-4Zr-2Mo-0.1Si microstructures (a) β forged and $\alpha + \beta$ heat treated; (b) $\alpha + \beta$ forged and β heat treated.

TABLE 1—Microstructural and mechanical properties.

Properties	$\alpha + \beta$ Forged	β Forged
α Platelet size, μm	1	5
Colony size, μm	...	50
Prior β grain size, μm	700	...
Tensile strength, MPa	1021	980
Yield strength, MPa	918	897

ample of the influence of microstructure on crack path is shown in Fig. 3. In this case the mode of cracking can be either crystallographic, as at A where the crack propagated in a direction perpendicular to the length of the α platelets, or interfacial, as along a colony boundary at B and an α - β interface at C. The fracture characteristics for the four test conditions and two processing histories are shown in Figs. 4 through 11.

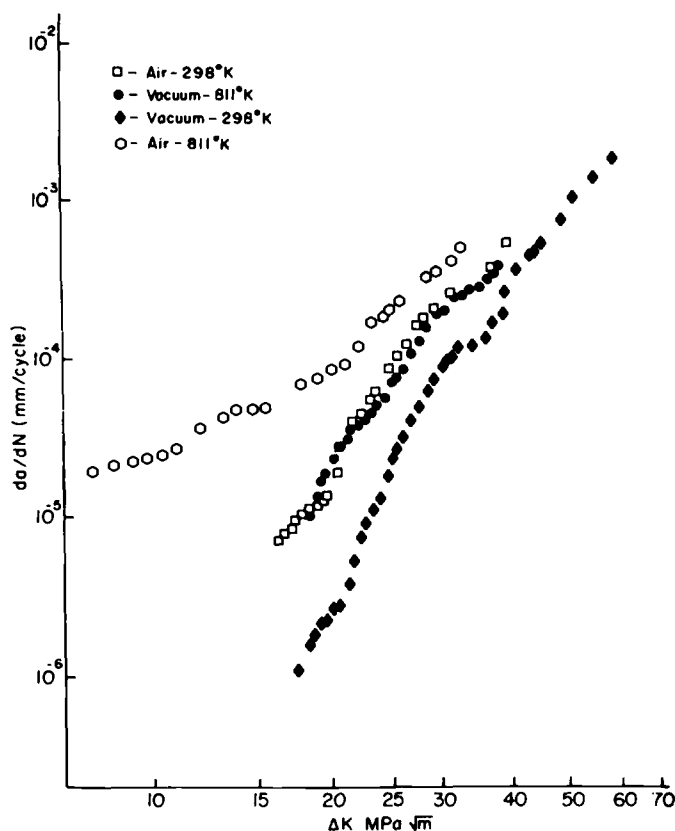


FIG. 2—Fatigue crack growth rate curves as a function of stress intensity and environment.

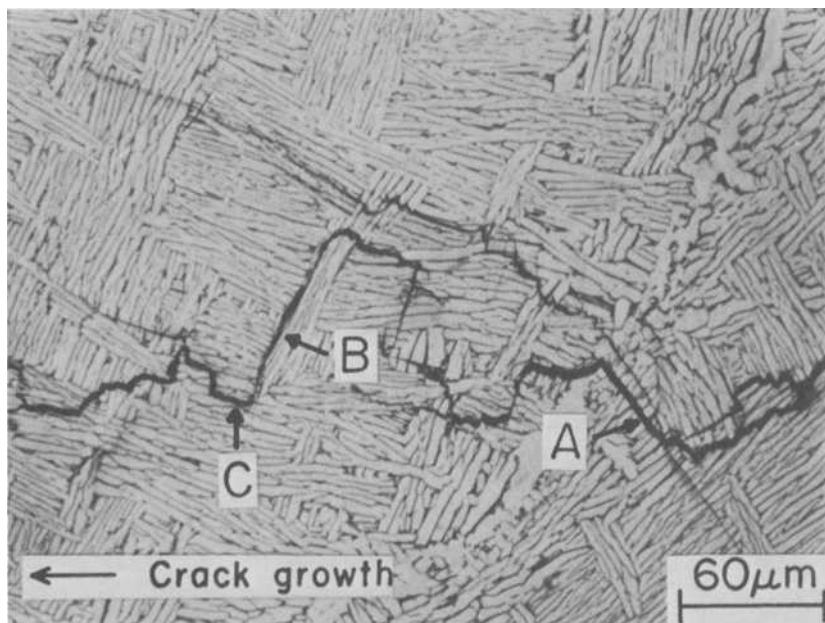


FIG. 3—Photomicrograph of β forged material illustrating (a) cracking perpendicular to α platelets; (b) cracking along a colony boundary, and (c) cracking along an α - β interface.

$\alpha + \beta$ Forged

Air Environment—The fracture surface appearance was dependent on the magnitude of the stress-intensity factor and concomitant crack growth rate. The general transition in fracture topography that occurred with increasing ΔK level at room temperature is shown in Fig. 4a, c, and e.

Below ΔK levels of $25 \text{ MPa } \sqrt{\text{m}}$, the fracture surface was characterized by large transgranular facets of varying orientation to the macroscopic crack plane, which were of the order of the prior β grain size. These planes were usually near the close-packed plane (0001) [17] and, as will be discussed later, resulted from a microstructural dependence on the fatigue fracture mechanism. Intermixed with these facets were regions exhibiting pronounced secondary cracking (Fig. 4b).

Under intermediate ΔK conditions (25 to $38 \text{ MPa } \sqrt{\text{m}}$), the amount of facetting steadily decreased, and more ductile fracture modes, which gave rise to striations and dimples, became apparent. Beyond ΔK levels of $38 \text{ MPa } \sqrt{\text{m}}$ the fracture mode involved striation, microvoids, and dimple formation (Fig. 4d). The final separation occurred primarily along prior β grain boundaries, with the intergranular facet surface exhibiting dimples (Fig. 4f).

At elevated temperature the fracture characteristics and fatigue crack growth rates, especially at low ΔK levels, were extremely sensitive to the inten-

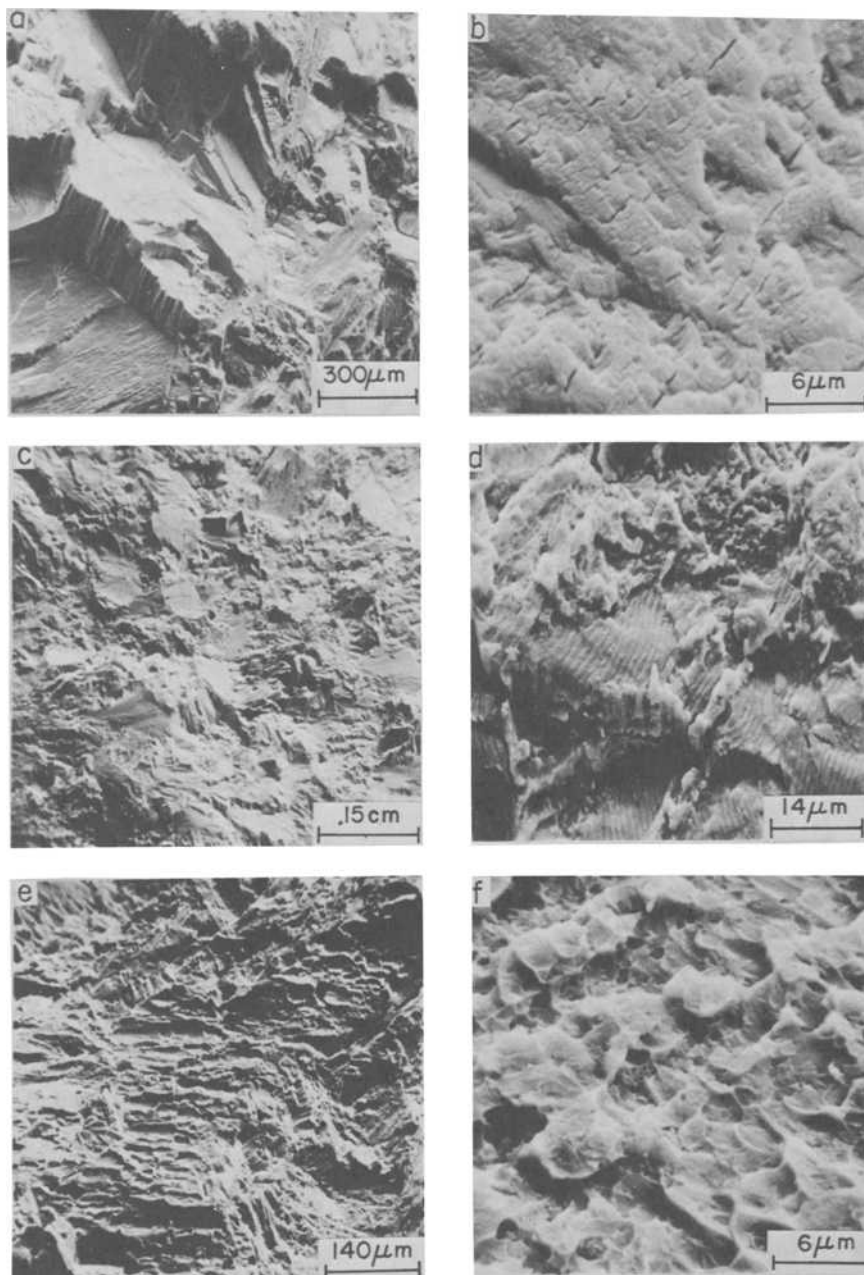


FIG. 4—Fatigue crack propagation fracture topography of $\alpha + \beta$ forging in an air environment at room temperature. The crack growth direction is from left to right; (a) and (b) $da/dN = 5 \times 10^{-6}$ mm/cycle; (c) $da/dN = 1.0 \times 10^{-4}$ mm/cycle; (d) $da/dN = 5.0 \times 10^{-4}$ mm/cycle; (e) $da/dN = 2.0 \times 10^{-3}$ mm/cycle; (f) fast-fracture region.

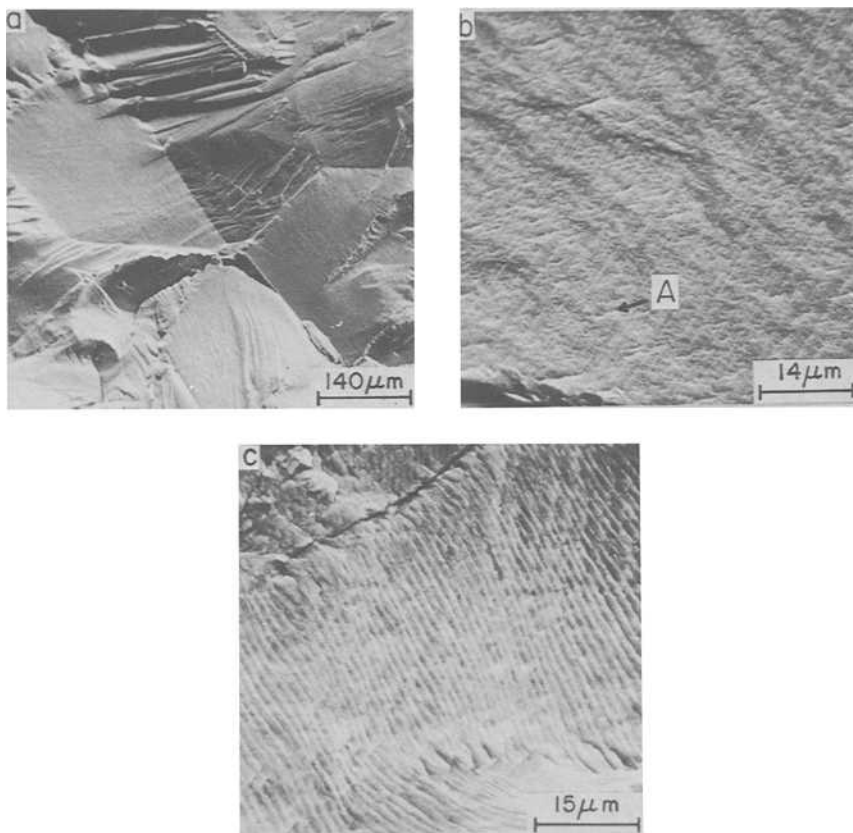


FIG. 5—Fatigue crack propagation fracture topography of $\alpha + \beta$ forging in an air environment at 811 K. The crack growth direction is from right to left; (a) and (b) $da/dN = 1.8 \times 10^{-5}$ mm/cycle; (c) $da/dN = 7.6 \times 10^{-4}$ mm/cycle.

sity of environmental action. Below stress-intensity levels of $25 \text{ MPa } \sqrt{\text{m}}$ ($da/dN = 2 \times 10^{-4}$ mm/cycle), the fracture surface was virtually 100 percent faceted (Fig. 5a), and the influence of oxidation was clearly evident from the deep purplish color and macroscopically smoother fracture surface (compare Figs. 4a and 5a). At higher growth rates the crack path was more tortuous, with an increased tendency for cracking along prior β grain boundaries and striation (Fig. 5c), and the dimple formation was similar to that observed at room temperature, which was indicative of the small influence of temperature on fatigue-crack propagation rates.

Vacuum Environment—Typical low-growth-rate room-temperature fracture characteristics are shown in Fig. 6a through d. In contrast to the air results, at low ΔK levels, a ductile-appearing fracture mode was present (Fig. 6b), and the degree of faceting and irregularity of the fracture surface was

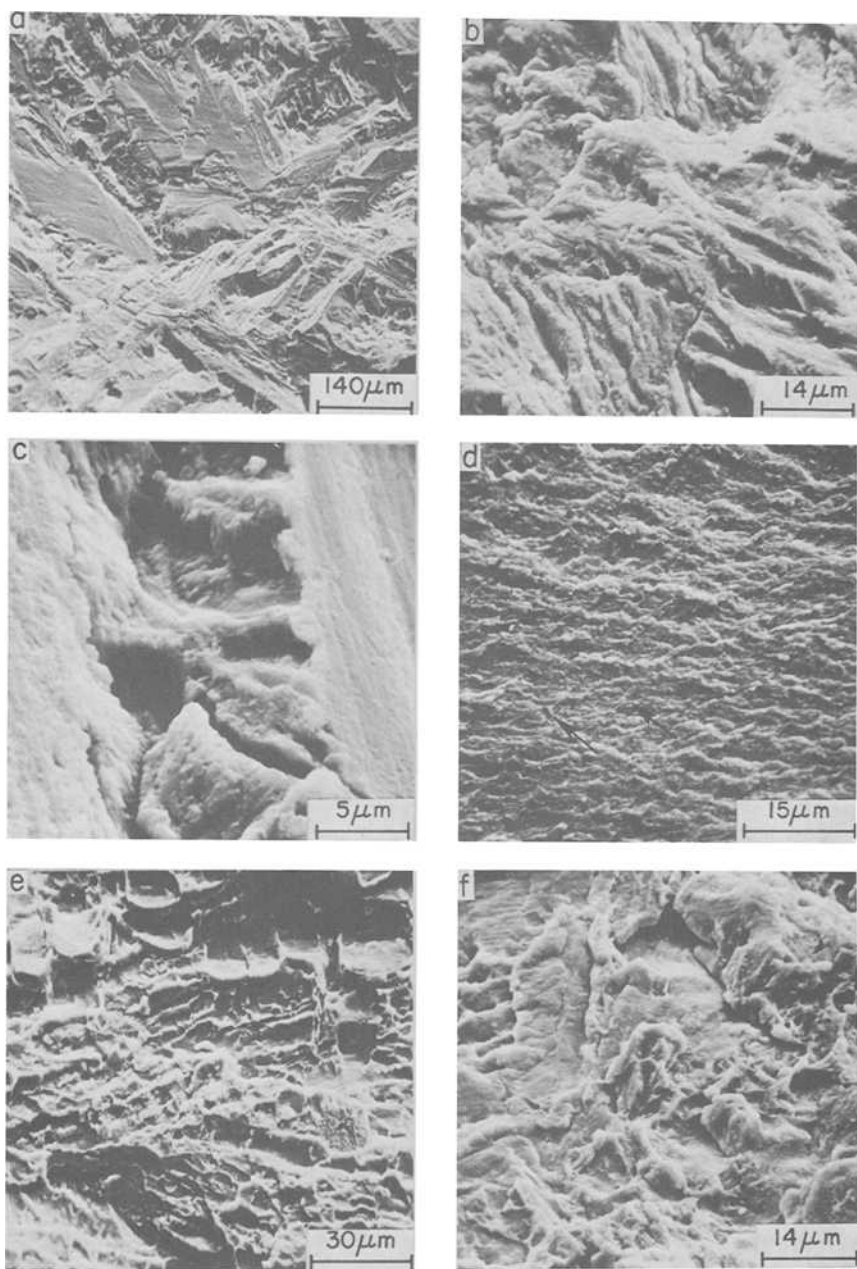


FIG. 6—Fatigue crack propagation fracture topography of $\alpha + \beta$ forging in vacuum at room temperature. The crack growth direction is from left to right; (a) $da/dN = 1.7 \times 10^{-6}$ mm/cycle; (b) and (c) $da/dN = 5.1 \times 10^{-6}$ mm/cycle; (d) facet morphology, $da/dN = 2 \times 10^{-6}$ mm/cycle; and (e) $da/dN = 7.6 \times 10^{-4}$ mm/cycle.

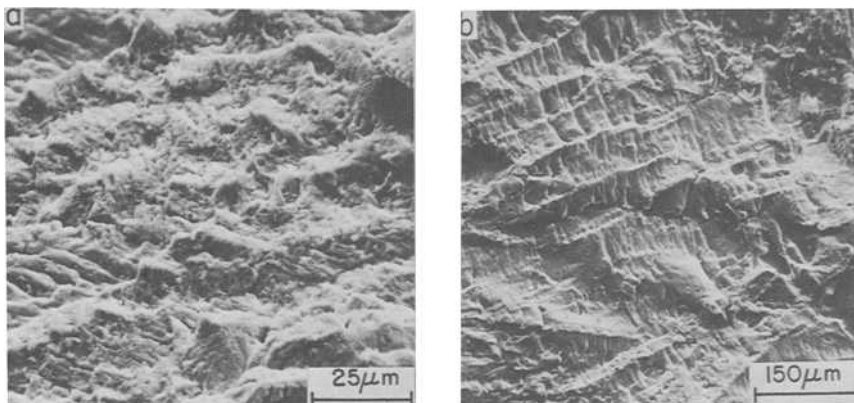


FIG. 7—Fatigue crack propagation fracture topography of $\alpha + \beta$ forging in vacuum at 811 K. The crack growth direction is from right to left; (a) $da/dN = 5.1 \times 10^{-6}$ mm/cycle, and (b) $da/dN = 2.3 \times 10^{-3}$ mm/cycle.

lower. However, secondary cracking was still observed. At higher crack growth rates (2×10^{-4} mm/cycle) the fracture surface consisted of voids and dimpled rupture (Fig. 6e and 6f).

Increasing the test temperature promoted crystallographic faceting, but not to the extent observed in air. In the 10^{-6} mm/cycle crack growth rate regimen, facets intermixed with a ductile-appearing fracture (Fig. 7a), similar to that observed at room temperature (Fig. 6b), were present. As the growth rate increased, the separation process resulted in a predominately flat and blocky fracture, characteristic of the underlying microstructure (Fig. 7b), whereas at the highest growth rates (2×10^{-3} mm/cycle) voids, dimples, and blocky areas comprised the fracture surface. Finally, a major effect of a vacuum environment (at room or elevated temperature and for both forging conditions) was the suppression of striation formation. In only a few regions of the fracture surface were striation-like markings observed (see Fig. 10e).

β Forged

Many of the observations just described for the $\alpha + \beta$ forged material apply to the β forging as well. Rather than repeat similar observations, however, only the differences between the two forging conditions in fracture appearance will be presented.

Air Environment—The most striking difference in fracture appearance occurred in the low-crack-growth-rate microstructure-sensitive regimen. In this region, the $\alpha + \beta$ forging exhibited a more irregular fracture surface and consisted of larger facets (compare Fig. 4a with Fig. 8a, and Fig. 5a with Fig. 9a), and this difference was clearly related to the respective prior β grain and colony sizes of the forgings.

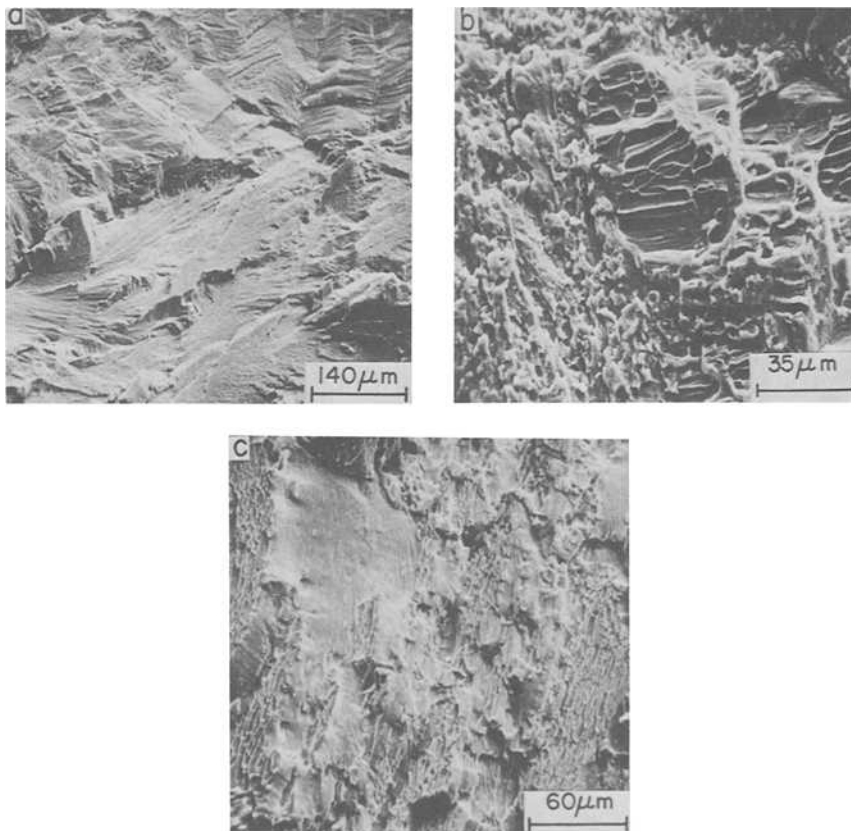


FIG. 8—Fatigue crack propagation fracture topography of β forging in an air environment at room temperature. The crack growth direction is from left to right; (a) $da/dN = 2.5 \times 10^{-5}$ mm/cycle; (b) $da/dN = 1.5 \times 10^{-3}$ mm/cycle; and (c) $da/dN = 2.5 \times 10^{-3}$ mm/cycle.

At higher growth rates, 10^{-4} to 10^{-3} mm/cycle, the fracture mechanisms were the same (striation, microvoid, and dimple formation), but the β forged condition demonstrated elongated dimples corresponding to the underlying α - β platelet microstructure (Fig. 8b and c) and more secondary cracking at elevated temperature (Fig. 9c).

Vacuum Environment—A comparison of β (Fig. 10a and b) and $\alpha + \beta$ forging (Fig. 6a and b) illustrates the difference in the room-temperature fracture topography characteristics: the β forging fracture surface is clearly not as rough. In addition, at a stress-intensity level of $25 \text{ MPa } \sqrt{\text{m}}$, a sharp transition from structure-sensitive to structure-insensitive fracture mode occurred. Below this ΔK level, traces of the α - β colony microstructure were evident (Fig. 10c), but upon reaching a ΔK level of $25 \text{ MPa } \sqrt{\text{m}}$ a ductile fracture composed of dimples and voids developed (Fig. 10d and e).

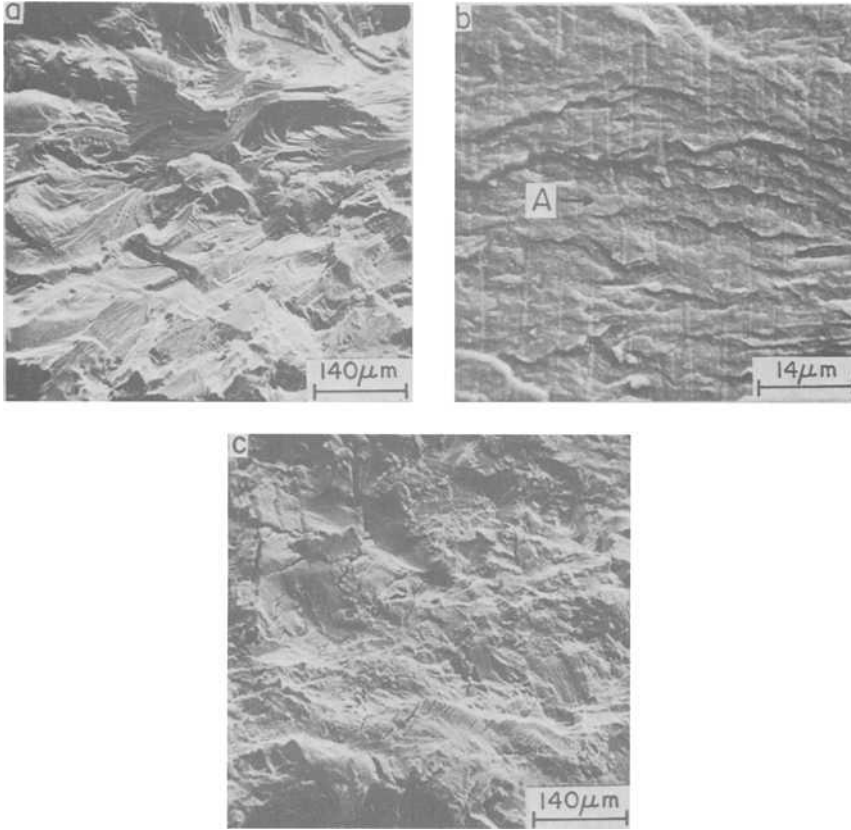


FIG. 9—Fatigue crack propagation fracture topography of β forging in an air environment at 811 K. The crack growth direction is from right to left; (a) and (b) $da/dN = 2.5 \times 10^{-5}$ mm/cycle, and (c) $da/dN = 2.5 \times 10^{-3}$ mm/cycle.

Finally, Fig. 11 reveals the fracture topography characteristics at 811 K. At low growth rates, cleavage-like regions intermixed with a feathery ductile fracture were present (Fig. 11b). As the growth rate increased, the predominant effect of temperature was to promote a serrated fracture (Fig. 11c), whereas at higher ΔK levels the familiar ductile fracture developed (Fig. 11d).

Discussion

The influence of microstructure and environment on fracture topography was evident throughout the crack-growth-rate spectrum but was most apparent at lower crack propagation rates in the form of crystallographic facets.

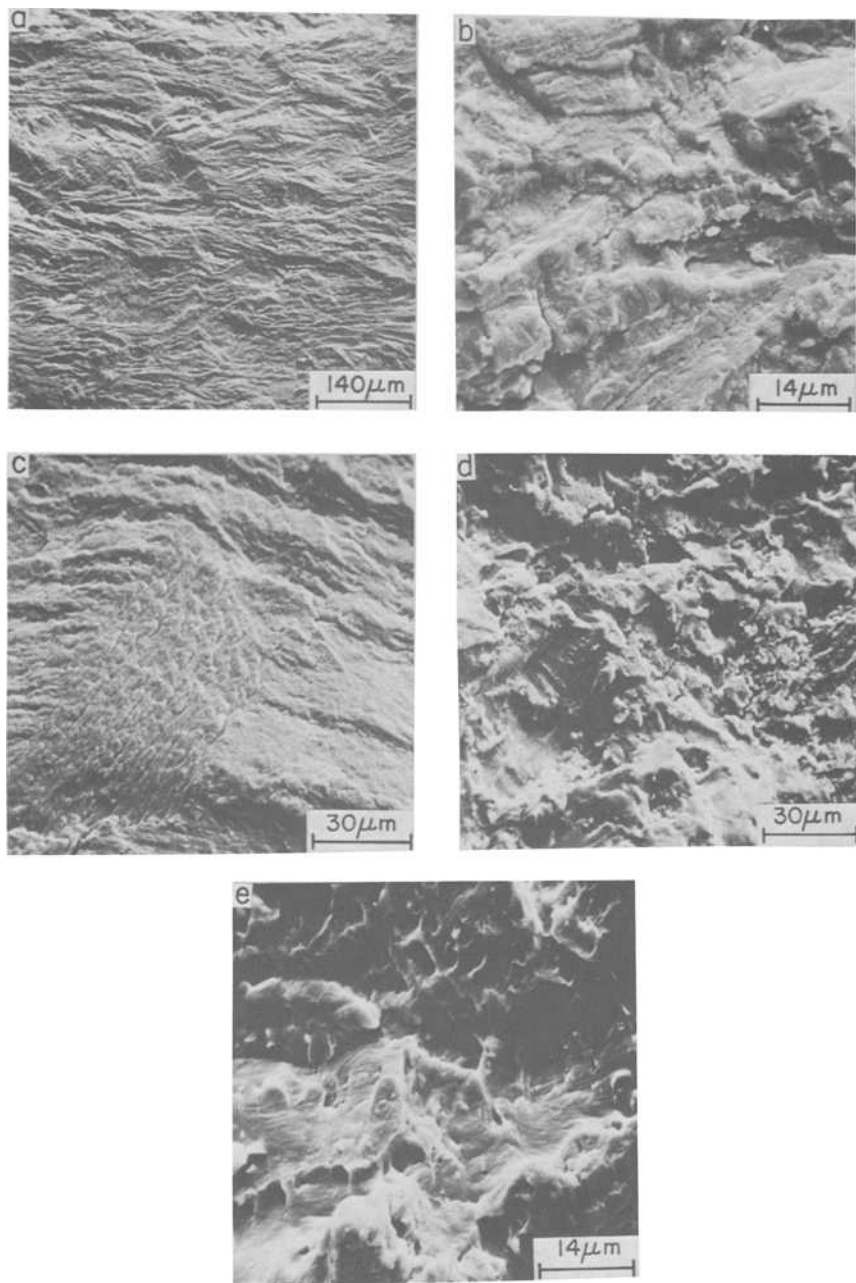


FIG. 10—Fatigue crack propagation fracture topography of β forging in vacuum at room temperature. The crack growth direction is from right to left; (a), (b), and (c) $da/dN = 7.6 \times 10^{-6}$ mm/cycle, (d) $da/dN = 1.3 \times 10^{-4}$ mm/cycle, and (e) $da/dN = 5.1 \times 10^{-4}$ mm/cycle.

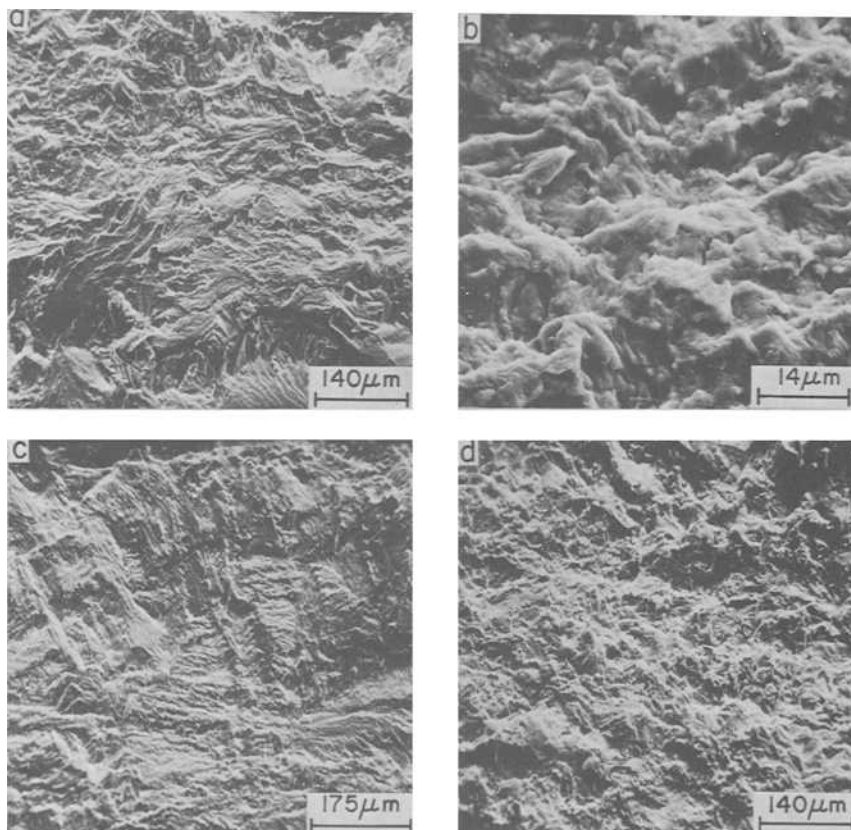


FIG. 11—Fatigue crack propagation fracture topography of β forging in vacuum at 811 K. The crack growth direction is from left to right; (a) and (b) $da/dN = 8.9 \times 10^{-6}$ mm/cycle; (c) $da/dN = 6.4 \times 10^{-5}$ mm/cycle; and (d) $da/dN = 1.0 \times 10^{-3}$ mm/cycle.

The fact that a parallel set of α platelets (colony) transforms from the β -phase, according to the Burgers relation [18, 19]

$$\begin{matrix} \{110\}_{\beta} \parallel (0001)_{\alpha} \\ \langle 111 \rangle_{\beta} \parallel \langle 1\bar{1}20 \rangle_{\alpha} \end{matrix}$$

means that they are of a single orientation, and it is possible for adjacent colonies of the same prior β grain to have a common plane [20]. Thus, large facets of the order of the prior β grain size can form in the $\alpha + \beta$ forged material, and, indeed, observations showed that the facets were related to the scale of the microstructure, colony, and prior β grain size for the β and $\alpha + \beta$ forging. Furthermore, the irregularity of the fracture surface was a direct consequence of this crystallographic faceting and extremely tortuous crack path. In addition, through this mechanism of crystallographic crack branching and tortu-

ous crack path extension, the net fatigue crack growth rate can be reduced (even though crack propagation on a common plane through one or more colonies can occur two to three times more rapidly than the prevailing net crack growth rate).

Thus, it is of interest to consider the nature of these facets and attempt to correlate crack propagation rates with these fractographic features. In air, cleavage-like facets exhibited lineal markings (similar to those observed by Yoder et al [6] in the Ti-6Al-4V alloy) with their spacing of the order of the α platelet width, which suggests that these markings correspond to traces of the α - β interfaces (Fig. 5b and Fig. 9b). Many small parallel cracks (marked A) and river-line patterns also characterized the facet surface. In vacuum, however, in addition to many straight parallel cracks (marked with arrows), the facet surfaces exhibited a ductile rumpled appearance (Fig. 6d). In the case of the $\alpha + \beta$ forging, adjoining facets were connected by small fracture segments (Fig. 6c). Since the facets correlate with the prior β grain size, and grain boundary α -phase is present along the prior β grain boundaries, it is probable that these segments correspond to fracture of grain boundary α -phase.

As shown in Fig. 12, intense shear bands can extend completely across a prior β grain and are coincident with many secondary cracks (marked A). Thus, in agreement with previous studies [4, 6, 8, 21, 22], this crystallographic

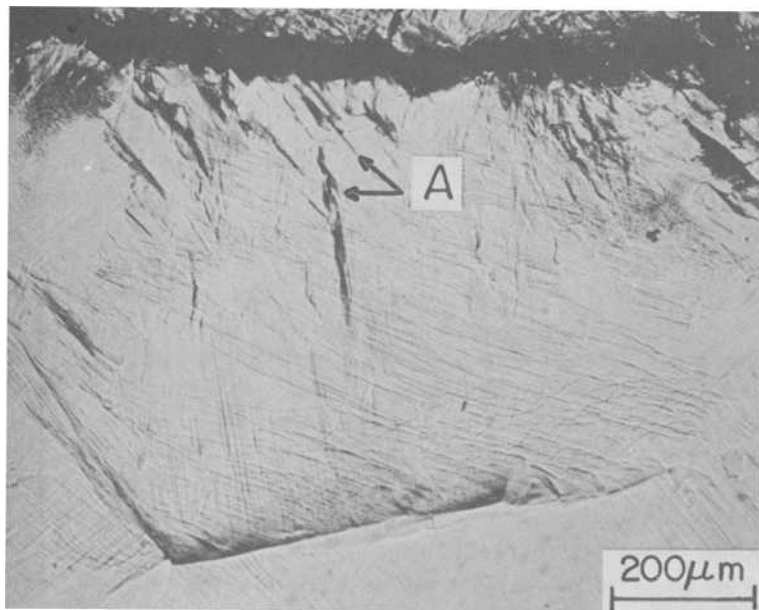


FIG. 12—A photomicrograph illustrating slip bands and slip-band cracking (marked A) produced during fatigue crack growth; also note the blockage of slip at the prior β grain boundary ($\alpha + \beta$ forging).

faceting appears related to a Stage-I cracking mechanism, wherein the straight cracks or branches follow shear bands. Furthermore, the influence of environment on facet morphology, described earlier, and the increased tendency for facet formation and higher crack growth rates in an air environment, particularly at elevated temperature, suggests that Stage-I cracking and facet formation is an environmentally sensitive process and can have a pronounced influence on fatigue crack growth rates.

The work of several investigators also supports this view. Neumann [23], using copper single crystals, found that in air a Stage-I crack could be induced at the tip of a growing Stage-II crack by sufficiently reducing the load amplitude, but in vacuum no Stage-I crack propagation occurred. For nickel-base superalloys, Duquette and Gell [24] showed that vacuum Stage-I cracks propagated by a local ductile rupture mechanism, whereas in air gaseous oxygen adsorbed on the fatigue fracture surfaces lowered the surface energy during crack growth and accounted for both the faster growth rates and the cleavage appearance. Similarly, the increase in Stage-I propagation rates of an aluminum-zinc-magnesium alloy in the presence of moist laboratory air in comparison with dry nitrogen was interpreted as embrittlement of the material by water vapor [25].

Thus, it appears that Stage-I crack extension in vacuum was a difficult process and occurred in a ductile manner (Fig. 6*d*). The significant increase in crack growth rates at low ΔK levels in air is attributed to easier Stage-I crack propagation, an increase in low-energy facet-failure mode, and the embrittling effects of environment along the crack front. The latter explanation is supported by an observed increase in surface hardness from an initial value of 345 VHN to a value of 420 VHN after elevated temperature exposure. In fact, the smoother and less tortuous fracture appearance that occurred at low ΔK levels at elevated temperature may have resulted from rupture of an epitaxial oxide layer.

As noted previously, one other significant influence of environment on fracture topography was the suppression of striation formation in vacuum. This effect has been attributed to enhanced slip reversal in vacuum [26]. The slip steps produced by fatigue-cycling do not immediately oxidize, consequently reverse slip can occur and striation formation is suppressed.

An alternative explanation, worthy of further investigation, is the influence of crack closure on striation formation. Although the effect of environment on closure levels is not precisely clear [27–31], the closure stress has been reported to be larger in vacuum than in an air environment [29–31]. In this situation, through contact of the fracture surfaces, crack closure may be an additional impediment to striation formation. In addition, if this mechanism is indeed operative, we would expect striation formation to be dependent on the stress ratio, R .

Finally, the transition from structure-sensitive to structure-insensitive growth has been explained on the basis of the size of either the reverse plastic

zone [1, 3, 6-9, 32, 33] or the maximum plastic zone [34] in relation to the scale of the microstructure. Theoretical predictions of plane stress monotonic plastic zone size are usually calculated using the expression

$$r_y = \alpha' \left(\frac{K}{\sigma_y} \right)^2 \quad (1)$$

where α' can take a range of values from 0.159 to 0.382 [35-38]. In addition, the reverse plastic zone size is often taken as one fourth of the monotonic plastic zone. However, Lankford et al [35] have shown that for many materials a good estimate of the maximum monotonic plastic zone size can be obtained using $\alpha' = 0.06$ to 0.10, and the ratio of the maximum monotonic to cyclic plastic zone size is of the order 10:1, rather than 4:1.

With this in mind, Table 2 lists the predicted plastic zone sizes as a function of stress-intensity level for the various values of α' . The present results have shown that crystallographic faceting predominated at low stress-intensity levels, but beyond $\Delta K = 25 \text{ MPa } \sqrt{\text{m}}$ faceting rapidly diminished, and in the case of vacuum testing (β forging at 298 K) a sharp transition from structure-sensitive to structure-insensitive growth occurred ($\Delta K = 25 \text{ MPa } \sqrt{\text{m}}$). From Table 2, the best correlation with this transition and reverse plastic zone size ($0.25 r_y$) results in $\alpha' = 0.318$. In this case, the reverse plastic zone size just exceeds the colony size ($50 \mu\text{m}$). It is also clear that, when crack-tip plasticity was well contained within individual colonies or prior β grains, facet formation was promoted, and as crack-tip plasticity increased the degree of faceting decreased.

Conclusions

1. Beta processing led to a more irregular-faceted fracture surface than $\alpha + \beta$ processing.
2. The microstructure-sensitive region was the one most affected by environment.
3. Facet formation was microstructurally dependent and enhanced by increased temperature and an air environment.
4. Testing in vacuum markedly reduced the irregularity and degree of faceting and suppressed striation formation. The influence of crack closure on striation formation warrants further study.
5. Although differences in fracture topography occurred between the two forging conditions, these differences were not sufficient to alter the fatigue crack growth rates.

Acknowledgment

The financial support provided by the U.S. Air Force Office of Scientific Research (Contract No. 74-2703) is gratefully acknowledged.

TABLE 2—Theoretical plastic zone size predictions.

α'	Plastic Zone, μm	$\Delta K = 15 \text{ MPa} \sqrt{\text{m}}$	$\Delta K = 20 \text{ MPa} \sqrt{\text{m}}$	$\Delta K = 25 \text{ MPa} \sqrt{\text{m}}$	$\Delta K = 35 \text{ MPa} \sqrt{\text{m}}$
0.06	r_y	16	28	44	87
	$0.25 r_y$	4	7	11	21
0.10	$0.1 r_y$	1.6	2.8	4.4	8.7
	r_y	26	47	74	145
0.154	$0.25 r_y$	6	11	18	36
	$0.1 r_y$	2.6	4.7	7.4	14.5
0.318	r_y	42	75	117	231
	$0.25 r_y$	10	18	29	57
0.382	$0.1 r_y$	4.2	7.5	11.7	23.1
	r_y	84	150	235	462
	$0.25 r_y$	21	37	58	115
	$0.1 r_y$	8.4	15	23.5	46.2
	r_y	101	181	253	535
	$0.25 r_y$	25	45	70	138
	$0.1 r_y$	10.1	18	25.3	53.5

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A Fractographic Investigation of Stress-Corrosion Cracking in High-Strength Steel Alloys

REFERENCE: Fraser, F. W. and Metzbower, E. A., "A Fractographic Investigation of Stress-Corrosion Cracking in High-Strength Steel Alloys," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 51-69.

ABSTRACT: The fracture separation processes of stress-corrosion cracking (SCC) specimens of steels with yield strengths in the 700 to 1400 MPa (100 to 200-ksi) range were investigated by scanning electron microscopy. Plates from a variety of processes were tested and examined. These included the electric furnace, electroslag remelt, vacuum induction melt-vacuum arc remelt, and electric furnace-vacuum arc remelt processes. The materials were studied both in the as-received condition and after heat treatment.

Stress-corrosion cracking tests were carried out on single-edge-notched, fatigue-pre-cracked specimens by the cantilever beam method. The specimens were fractured under stress in an aqueous 3.5 percent sodium chloride solution while galvanically coupled to zinc. The modes of fracture at the fatigue-SCC interface, in the SCC zone, and in the overload area were investigated, and a correlation was drawn between the fracture mode, the microstructure, and the resistance of the steel alloy to stress-corrosion cracking.

KEY WORDS: high-strength steels, HY-130, HY-180, stress-corrosion cracking, fractography, microstructure, steel processing, heat treating, materials, materials science.

The degree of susceptibility of high-strength steels to stress-corrosion cracking (SCC) in an aggressive environment, such as seawater, depends on paths through the microstructure that favor the propagation of a stress-corrosion crack. Since the levels of residual sulfur and phosphorus and the presence of nonmetallic inclusions in the microstructure are thought to influence SCC resistance, the quality of the steel, which is controlled by the processing method, becomes an important factor.

The objective of this investigation was twofold: (1) to investigate the effect of different steel processing methods and heat treatments on the dominant SCC fracture process and the microstructure of HY-130 and HY-180 steel

¹ Chemist and head of Welding Metallurgy Section, respectively, Naval Research Laboratory, Washington, D.C. 20375.

plates; (2) to correlate the fractographic and microstructural data with the susceptibility of the steel to SCC.

Materials and Procedure

The steels used in this investigation were produced either by the electric-arc method [electric furnace steels (EF)] or by induction melting in a vacuum [vacuum induction-melted steels (VIM)]. Melting in a vacuum, rather than in air, reduces the inclusion count and the amount of trapped gases in the solidified ingot. To obtain premium quality steel, the as-cast ingots are refined either by vacuum-arc remelting (VAR) or by electroslag remelting (ESR). In the VAR process the ingot (the electrode) is remelted by heat generated by the arc, and the molten steel collects in a water-cooled crucible. Benefits accruing from this refining process include a reduction in hydrogen, oxygen, and nitrogen content and improved cleanliness of the steel (fewer nonmetallic inclusions). Also, center porosity and segregation in the resulting ingot are practically eliminated. The ESR method is a variation of the consumable electrode process in which metal droplets from the remelted electrode filter through a slag blanket. The slag floats on top of the molten metal pool, protecting it from oxidation and rapid heat loss by radiation. Also, by controlling the chemistry of the slag layer, various elements can be removed selectively from the molten steel droplets. Premium melting stock and premium melting practices that minimize sulfur, phosphorus, and oxides have been singled out by Brown [1]² as being significantly beneficial to SCC resistance.

Table 1 lists the steels investigated, the manufacturing processes and heat treatments, the yield strengths, and the resistance of each specimen to stress-corrosion cracking (K_{Isc} values). In the HY-130 series two specimens were tested as received, and two were heat treated before testing. One was given a prolonged stress-relief treatment in a deliberate attempt to embrittle the microstructure, thereby producing a worst-case condition. The second was heat treated to produce a coarse-grain microstructure, similar to that developed in base metal, immediately adjacent to the fusion zone of a weldment. The HY-180 steels were tested both in the as-received condition and after being tempered to lower yield strengths. Table 2 gives the chemical composition of each of the as-received specimens [2]. The K_{Isc} values reported in Table 1 were determined in a previous investigation [2] by the cantilever beam method [3]. Notched and fatigue precracked specimens were tested in 3.5 percent nonflowing sodium chloride (NaCl) solution while galvanically coupled to zinc. The SCC crack plane was perpendicular to the width or long transverse (T) direction and parallel to the rolling or longitudinal (L) direction (T-L orientation). The SCC testing was performed at room temperature.

One fracture surface of each specimen was cleaned ultrasonically and fractographic analysis was carried out by scanning electron microscopy (SEM).

² The italic numbers in brackets refer to the list of references appended to this paper.

TABLE 1—Test conditions and properties of HY-130 and HY-180 steel specimens.^a

Material	Process	Heat Treatment	Yield Strength		K_{Isc} , 100 h ^b	
			MPa	ksi	MPa $\sqrt{m}$	ksi $\sqrt{in}$
HY-130 5Ni-Cr-Mo-V	EF-ESR	as received	986	143	112	102
	EF	as received	938	136	101	92
	EF	stress relieved	910	132	97	88
		510°C (950°F) 100 h air cooled				
HY-180 10Ni-Cr-Mo-Co	EF	coarse grain	1027	149	90	82
		980°C (1800°F) 1 h; WQ				
		570°C (1050°F) 1 h; WQ				
9Ni-4Co-0.2C	VIM-VAR	as received	1282	186	132	120
	VIM-VAR	tempered	952	138	138	126
		620°C (1150°F) 1 h; WQ				
	EF-VAR	as received	1282	186	85	77
	EF-VAR	tempered	786	114	79	72
		680°C (1250°F) 1 h; WQ				

^a Key to abbreviations used:

ESR—electroslag remelt

EF—electric furnace

VIM—vacuum-induction melt

VAR—vacuum-arc remelt

WQ—water quench

^b Hold time between load increments.

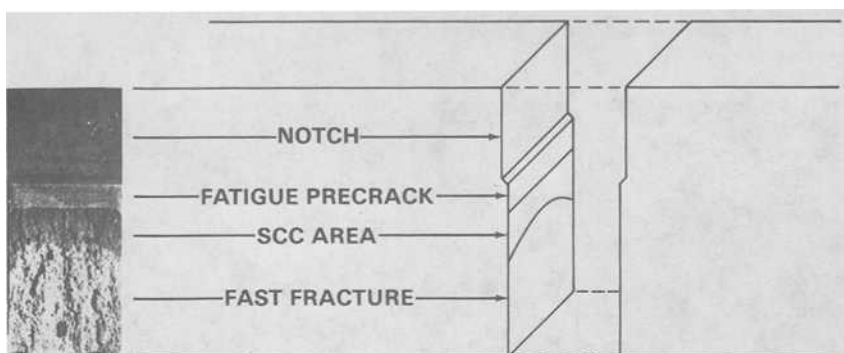


FIG. 1—A cantilever-beam test specimen depicting a typical fracture surface and the areas analyzed (fatigue–SCC interface, SCC area, and fast-fracture area).

The dominant modes of fracture were identified at the fatigue–SCC interface and in the SCC and fast-fracture areas. Figure 1 is a schematic drawing of a test specimen depicting a typical fracture surface and the areas analyzed. Upon completion of the fractographic analysis, the specimens were sectioned longitudinally for metallographic analysis. The metallographic sections were mounted, polished, and etched with 2 percent nital, and the microstructures near the fracture edge were identified by optical microscopy. Microhardness measurements were made along the fracture edge of the metallographic specimens using a Vickers microhardness tester under a 300-g load. All hardness measurements were converted to the Rockwell hardness C scale (HRC).

Results

HY-130 Steel

The dual-processed EF–ESR specimen fractured predominantly by microvoid coalescence (MVC) in all of the areas surveyed. There was no evidence of intergranular (IG) fracture or cleavage at the fatigue–SCC interface (Fig. 2, *top*), however, in the SCC zone, fracture showing cleavage-like features occurred in isolated areas in the plane of the main SCC crack and was generally accompanied by secondary cracking (Fig. 2, *bottom*). On the other hand, cleavage-like fracture predominated in the SCC area of the unrefined EF specimen tested as received, and secondary cracks were found both at the fatigue–SCC interface and throughout the SCC area (Fig. 3). In both specimens fast-fracture occurred by MVC. Globular sulfide inclusions on the surface of the EF–ESR specimen (Fig. 4) had no effect on the fracture mode. The refining of the EF–ESR specimen produced a homogeneous microstructure composed of tempered martensite of a uniform grain size (Fig. 5, *top*). The unrefined EF specimen, on the other hand, consisted of tempered martensite, acicular ferrite, and bainite (Fig. 5, *bottom*). Average hardnesses of the EF–ESR and EF specimens were 33 and 31 HRC, respectively.

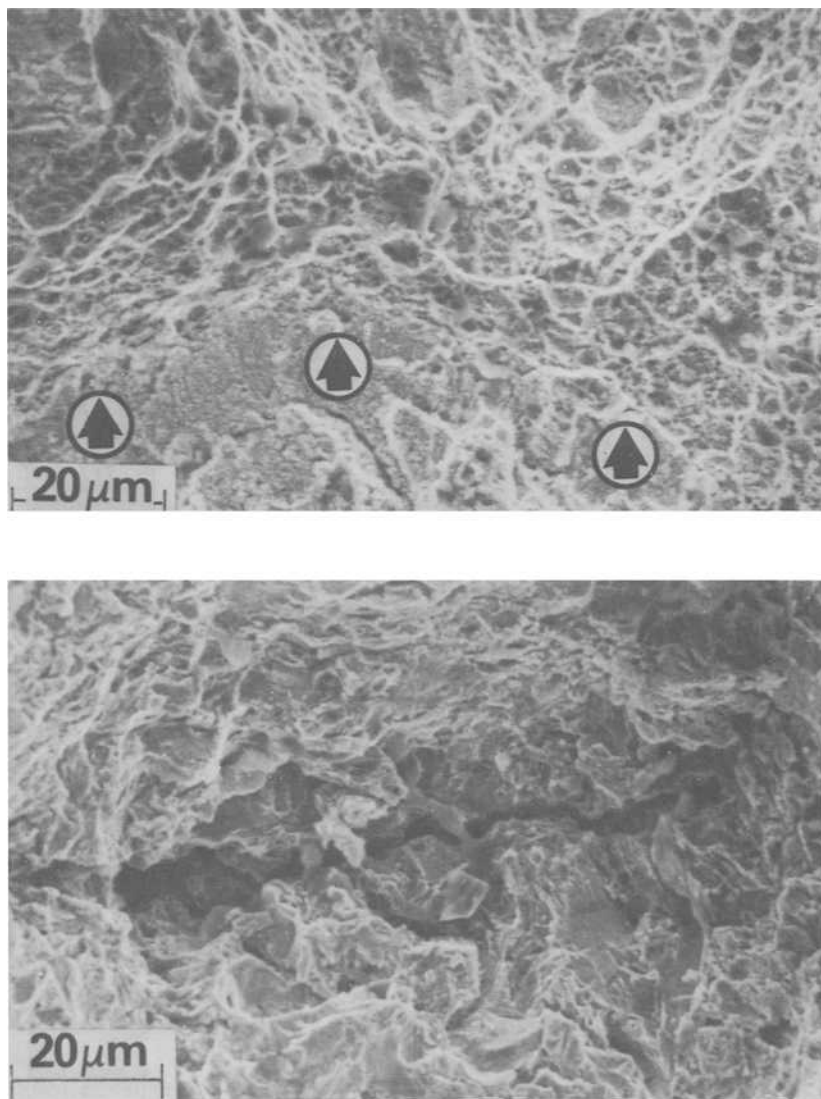


FIG. 2—(Top) HY-130 EF-ESR specimen, as received; fractured by MVC. The arrows indicate the fatigue-SCC interface. (Bottom) the SCC area showing cleavage-like fracture and secondary cracks.

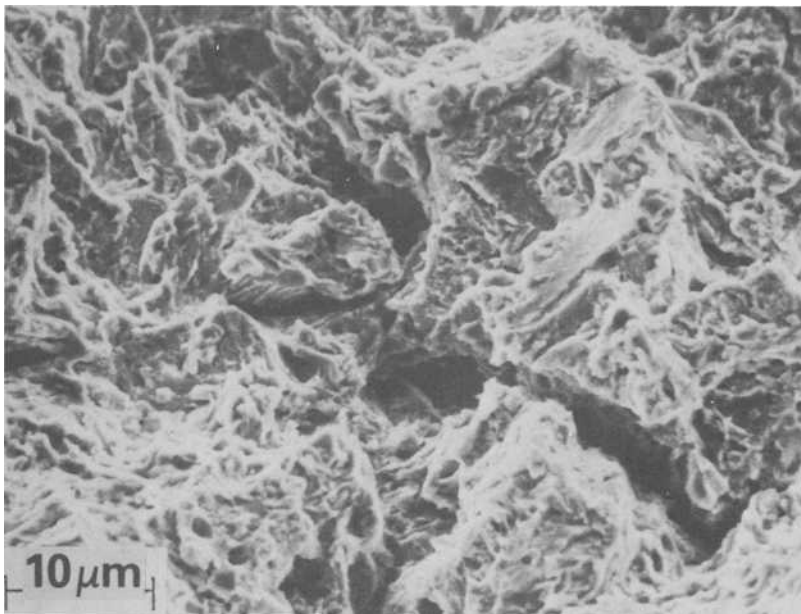


FIG. 3—HY-130 EF specimen, as received; the SCC area shows cleavage-like fracture and secondary cracks.

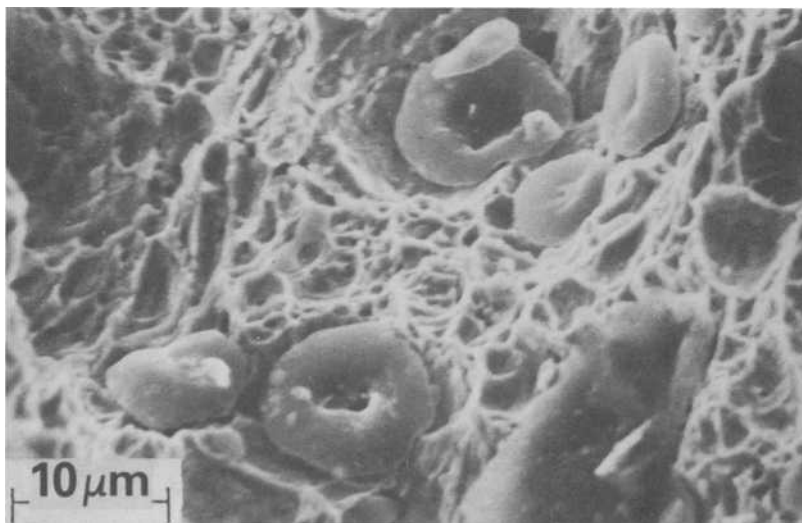


FIG. 4—HY-130 EF-ESR specimen, as received; the fast-fracture area shows MVC and globular sulfide inclusions.

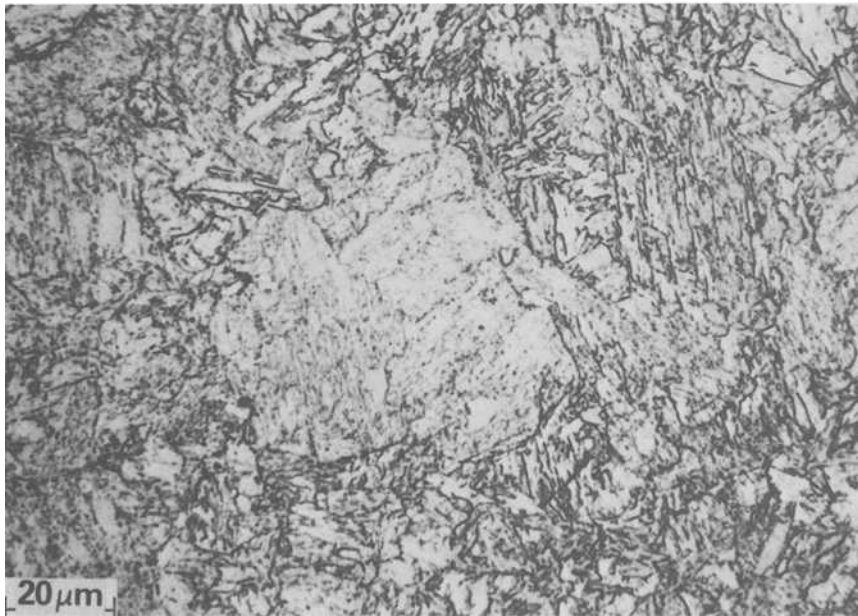


FIG. 5—(Top) *HY-130 EF-ESR specimen, as received; the homogeneous, tempered martensitic microstructure is of a uniform grain size. The specimen is etched with 2 percent nital. (Bottom) HY-130 EF specimen, as received; tempered martensite, acicular ferrite, and bainite. The specimen is etched with 2 percent nital.*

Cleavage-like fracture predominated throughout the SCC area of the EF-stress-relieved specimen (Fig. 6, *top*) including the fatigue-SCC interface. Fast fracture occurred by a combination of MVC and cleavage similar to that seen in the SCC area, and short secondary cracks running parallel to the direction of the primary SCC crack were found on the fracture surface (Fig. 6, *bottom*). A polished and etched cross section of this specimen disclosed heavy, irregular banding (Fig. 7). Manganese sulfide (MnS) stringers and heavy agglomerates of precipitates were found within the dark-etched bands. The hardness of the bands was 37 HRC whereas the hardness of the adjacent matrix was 31 HRC. The prolonged heat treatment also led to the spheroidization of carbides. The aligned longitudinal microcracks on the fracture surface may be due, at least in part, to the morphology of the sulfide inclusions. Where the sulfides were encountered as globules on the fracture surface, as in the EF-ESR specimen (Fig. 4), there was no evidence of microcracking.

The EF-coarse-grain specimen fractured predominantly by a combination of cleavage and IG separation in the SCC area (Fig. 8) and by MVC in the fast-fracture area. Extensive secondary cracking was found at the fatigue-SCC interface. The microstructure consisted of martensite, ferrite, and aggregates of bainitic ferrite and carbides (Fig. 9). The hardness of the coarse-grain specimen was 36 HRC.

HY-180 (10 Nickel) Steel

In the as-received condition, the VIM-VAR plate fractured by IG separation and cleavage in the SCC area (Fig. 10) and by MVC in the fast-fracture area. Although deep secondary cracks were found at scattered locations along the fatigue-SCC interface, fine, shallow microcracks were generally found throughout the SCC area. The martensitic microstructure contained branched microcracks within the SCC area (Fig. 11). The hardness was 45 HRC.

Tempering the VIM-VAR plate to a lower yield strength resulted in the coalescing of precipitated carbides within the martensitic microstructure (Fig. 12). Correspondingly, the fracture mode changed from fracture by cleavage and IG separation to fracture by MVC throughout both the SCC and the fast-fracture areas. A minor amount of secondary cracking occurred at the fatigue-SCC interface (Fig. 13). The hardness of the tempered specimen was 36 HRC.

HY-180 (9 Nickel) Steel

As in the 10 nickel VIM-VAR specimen, fracture in the SCC area of the 9 nickel EF-VAR specimen, tested as received, occurred by a combination of IG separation and cleavage. The highest incidence of IG fracture was found in the vicinity of the fatigue-SCC interface (Fig. 14). Secondary cracking, which

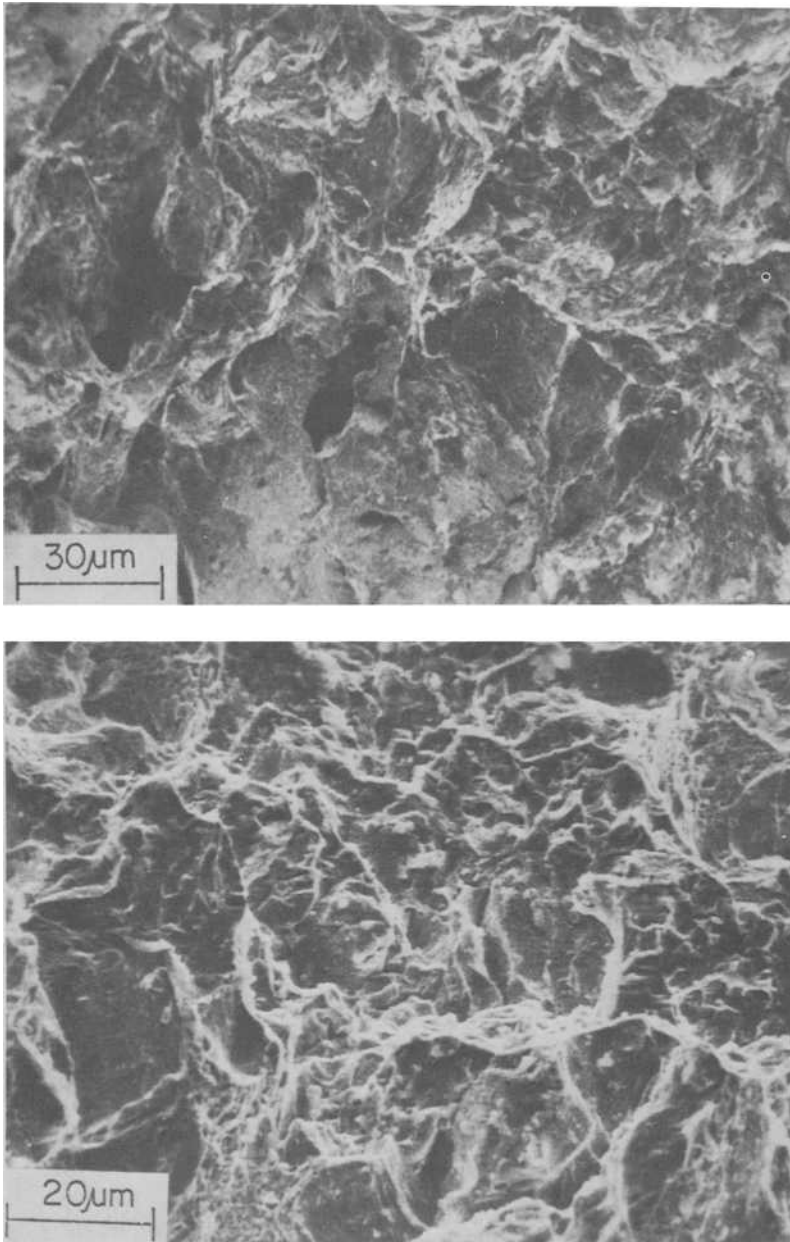


FIG. 6—(Top) HY-130 EF-stress-relieved specimen; the SCC area shows cleavage-like fracture. (Bottom) the fast-fracture area shows cleavage-like fracture and MVC with secondary cracks running parallel to the primary SCC crack.

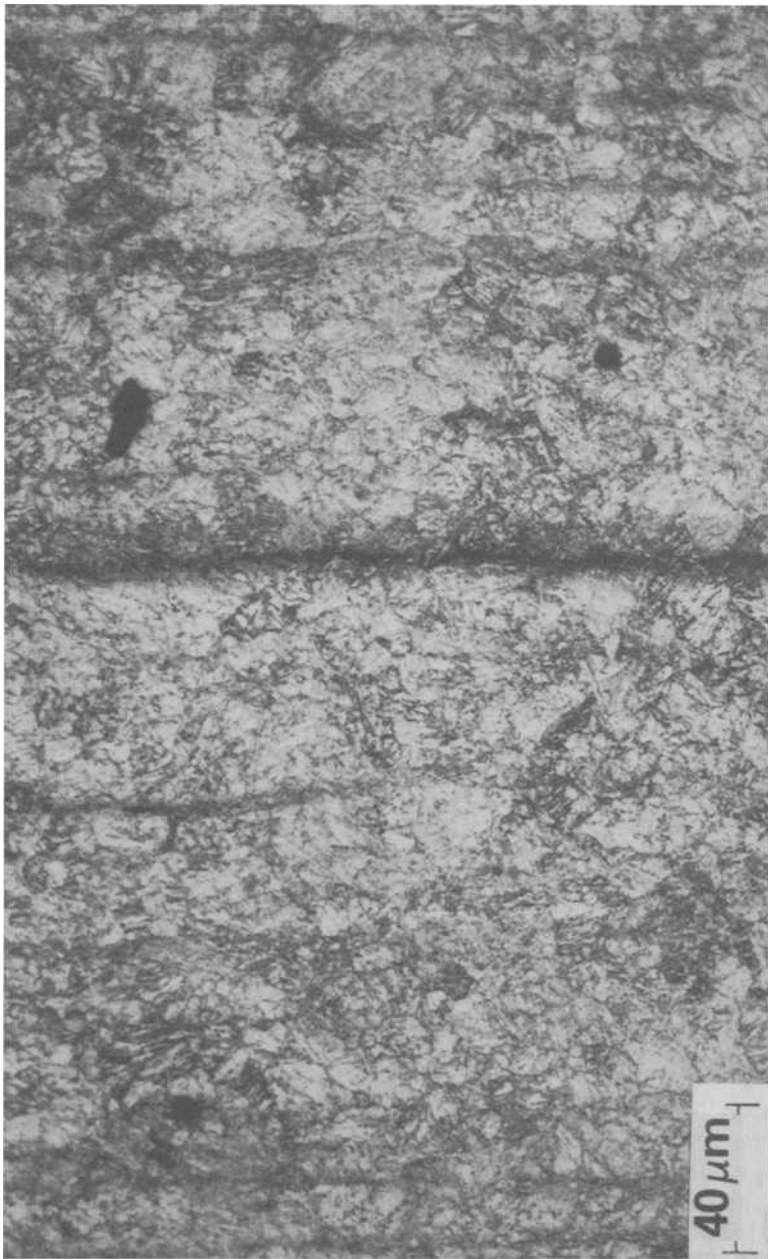


FIG. 7—HY-130 EF-stress-relieved specimen. A cross section shows heavy irregular banding. The specimen is etched with 2 percent nital.

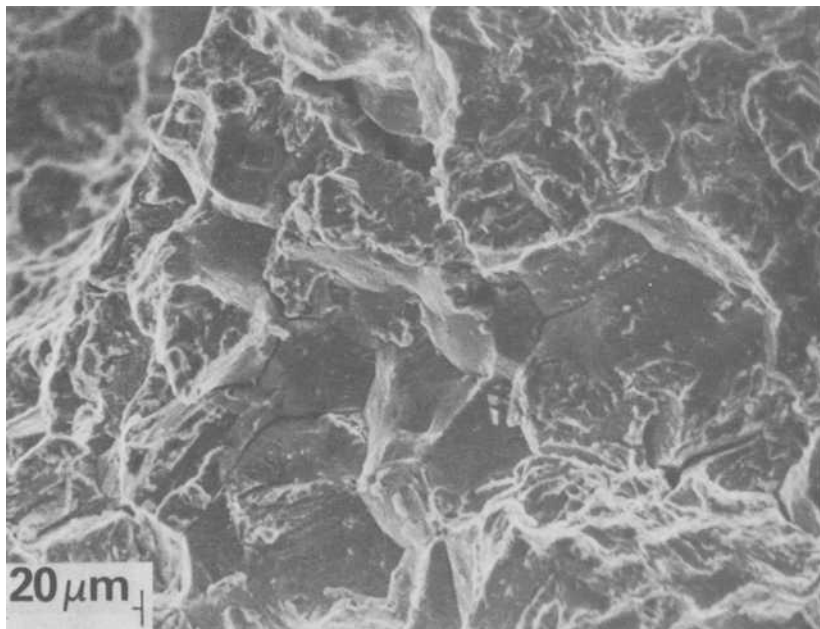


FIG. 8—HY-130 EF-coarse-grained specimen. The SCC area shows cleavage and IG separation.

was most prevalent at the fatigue–SCC interface, was found to some extent in all areas on the fracture surface. Fast-fracture occurred by MVC. The microstructure was tempered martensite with a heavy precipitate of carbides and contained discontinuous, intergranular, and transgranular microcracks (Fig. 15). The intergranular cracks coincided with chain-like precipitates within the grain boundaries. The hardness of this specimen was 44 HRC.

The heat-treated EF-VAR specimen fractured by MCV in both the SCC and fast-fracture areas. A minor amount of secondary cracking occurred in all areas on the fracture surface. The microstructure was composed of heavily tempered martensite and bainite (Fig. 16) with a hardness of 34 HRC.

Discussion and Conclusions

In the HY-130 series, the dual-processed EF-ESR specimen exhibited a greater resistance to SCC than did the EF unrefined specimen, as indicated by the K_{Isc} values in Table 1. Electroslag remelting processing of the EF ingot resulted in a homogeneous microstructure of tempered martensite with a uniform grain size. Transgranular fracture by MVC, with little secondary cracking in the SCC area and none at the fatigue–SCC interface, indicates relatively little embrittlement of this specimen during SC testing. On the other hand, the coarse, nonuniform structure of the unrefined EF specimen, composed of

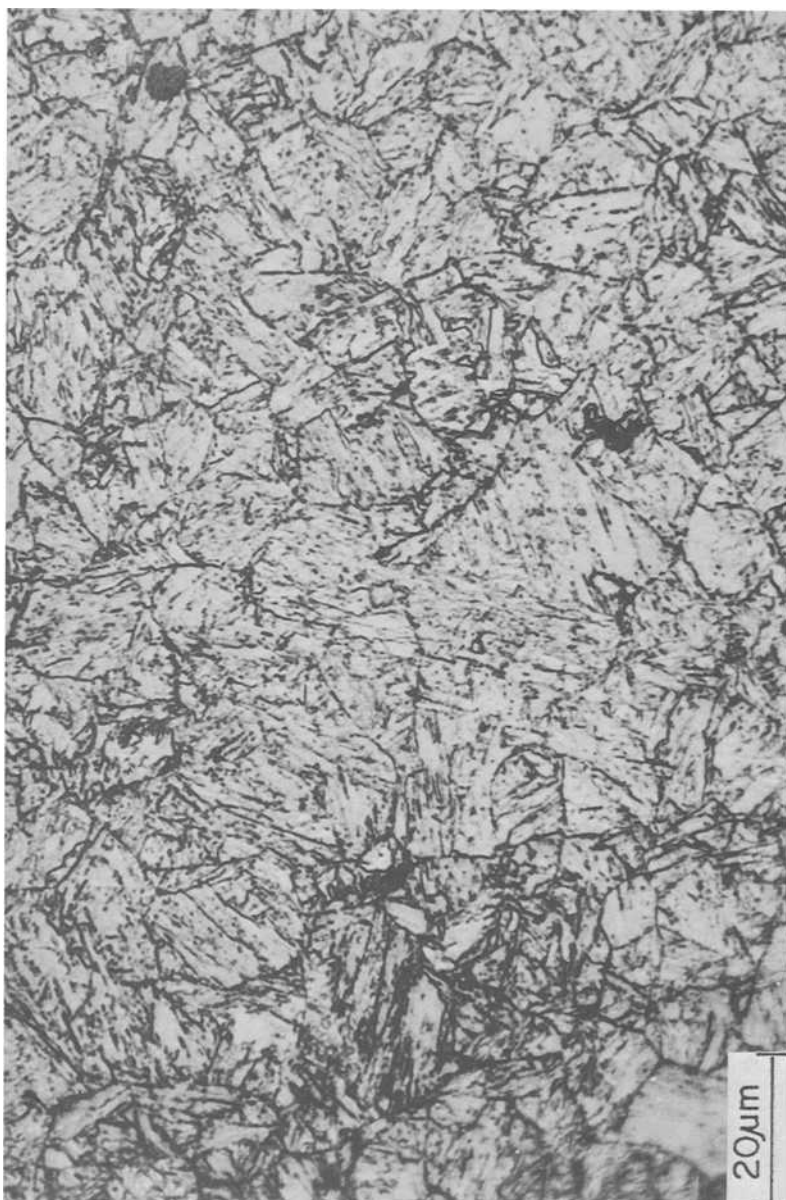


FIG. 9.—HY-130 EF—coarse-grain specimen, containing martensite, ferrite, and aggregates of bainitic ferrite and carbides. The specimen is etched with 2 percent nital.

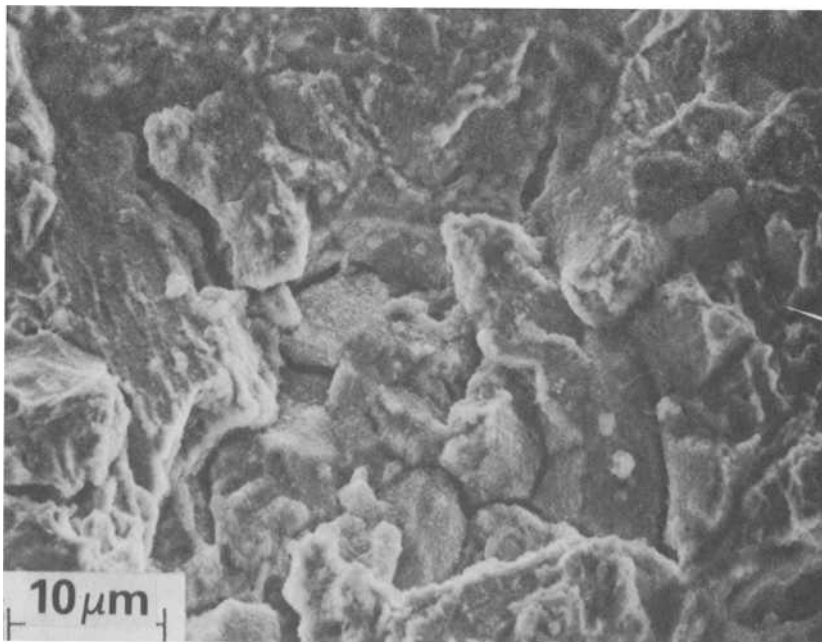


FIG. 10—HY-180 (10 nickel) VIM-VAR specimen, as received. The SCC area shows cleavage and IG separation.

martensite and acicular ferrite, exhibited a substantial amount of cleavage-like fracture and a lower K_{Isc} value.

Subjecting the EF specimen to a prolonged stress-relief heat treatment led to spheroidization of carbides along with intensified banding, which in turn resulted in the formation of hard areas within the microstructure. Cleavage-like fracture predominated throughout the SCC area and was found in substantial quantities in the fast-fracture area. In all of the other HY-130 specimens investigated, fast-fracture occurred exclusively by MVC. The increased incidence of cleavage-like fracture was again accompanied by a lowering of the K_{Isc} value.

Coarsening the grain size of the EF specimen to simulate the heat-affected zone (HAZ) of a weldment raised the hardness of the steel and produced substantial amounts of ferrite and coalesced carbides. In this instance the stress-corrosion crack was propagated by a combination of IG and cleavage-like fracture. The resulting K_{Isc} values was the lowest obtained in the HY-130 series. In view of the results obtained with the coarse-grained specimen, we might expect the coarse-grained, hard structures found within the HAZ of HY-130 weldments to be more susceptible to SCC than the unaffected base plate.

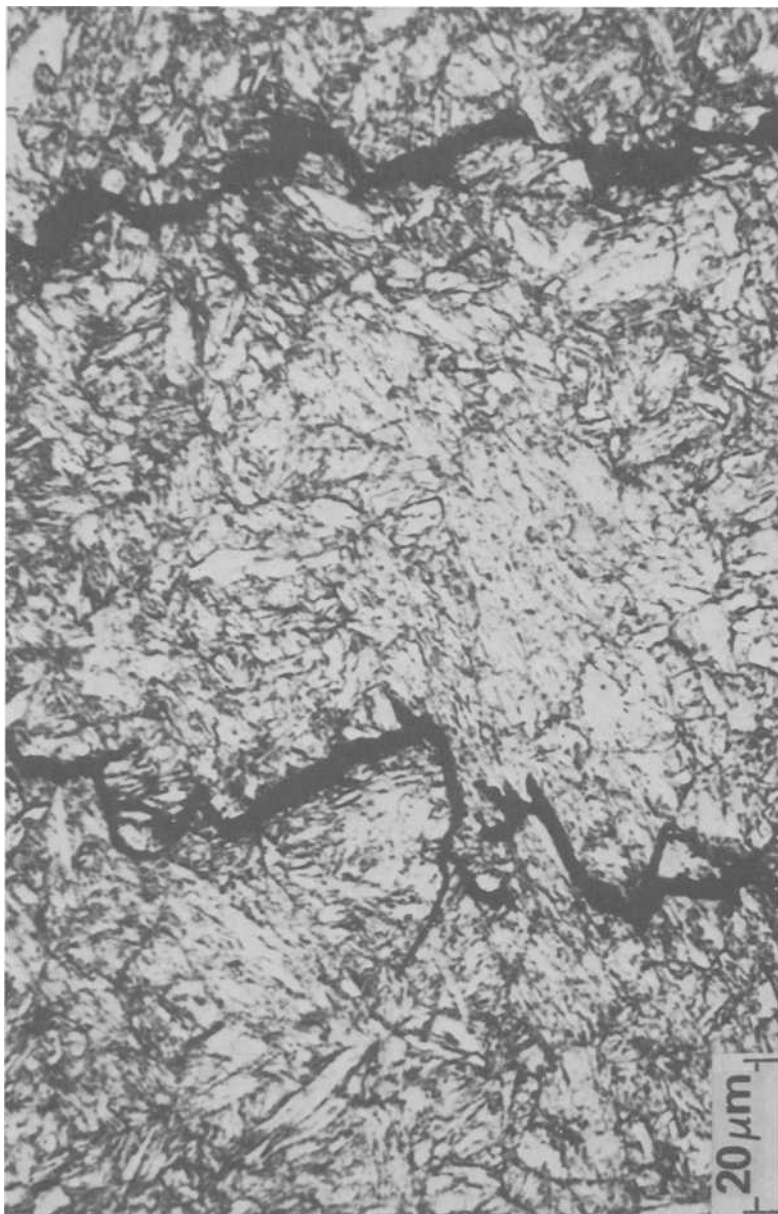


FIG. 11.—HY-180 (10 nickel) VIM-VAR specimen, as received, with a martensitic structure and branched microcracks within the SCC area. The specimen is etched with 2 percent nital.

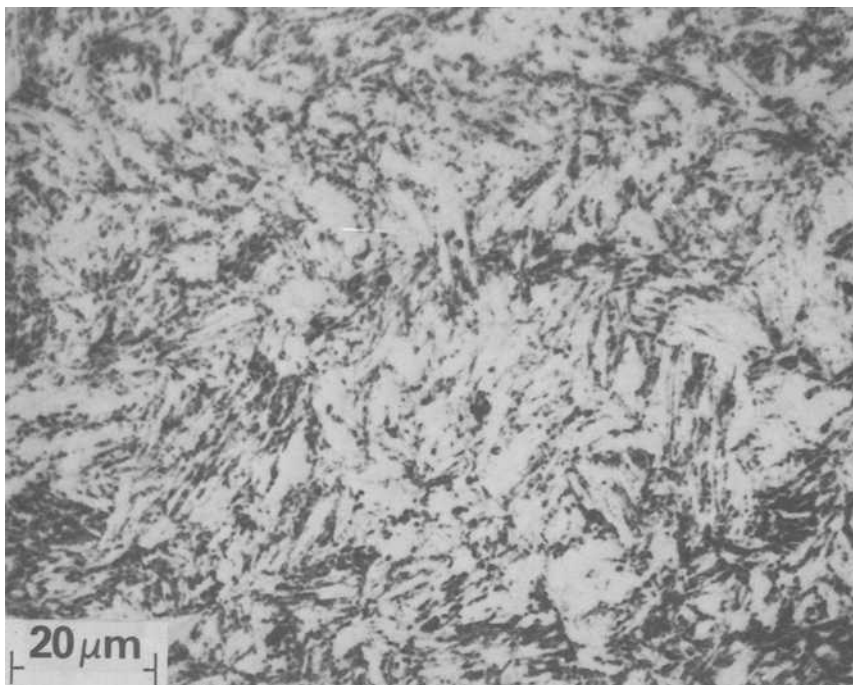


FIG. 12—HY-180 (10 nickel) VIM-VAR specimen, tempered, with martensitic structure with coalesced carbides. The specimen is etched with 2 percent nital.

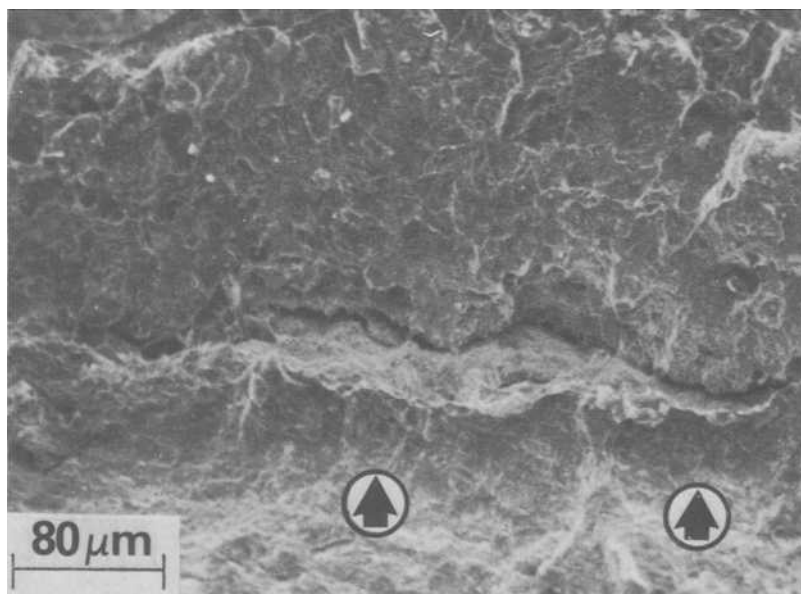


FIG. 13—HY-180 (10 nickel) VIM-VAR specimen, tempered, with secondary cracks at the fatigue-SCC interface (arrows).

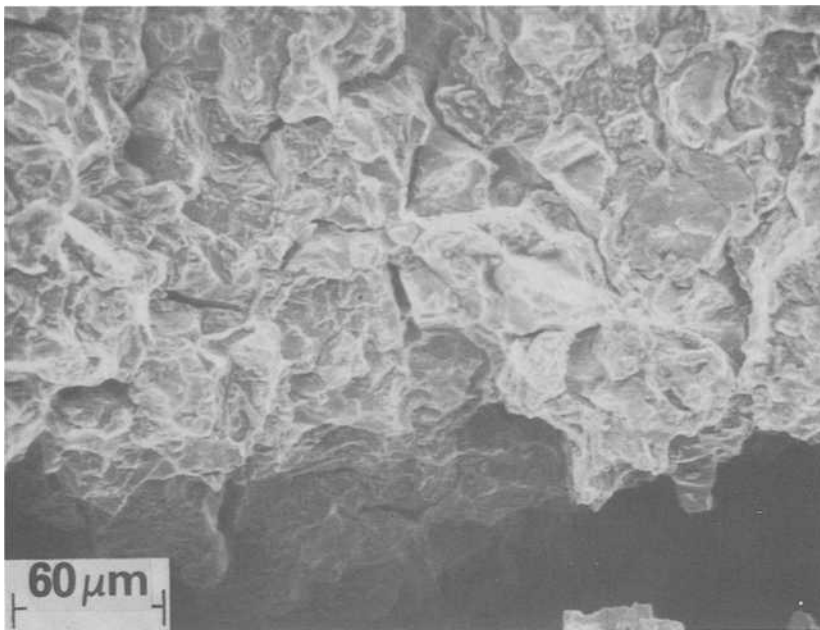


FIG. 14—HY-180 (9 nickel) EF-VAR specimen, as received. The fatigue-SCC interface shows secondary cracks, IG separation, and cleavage.

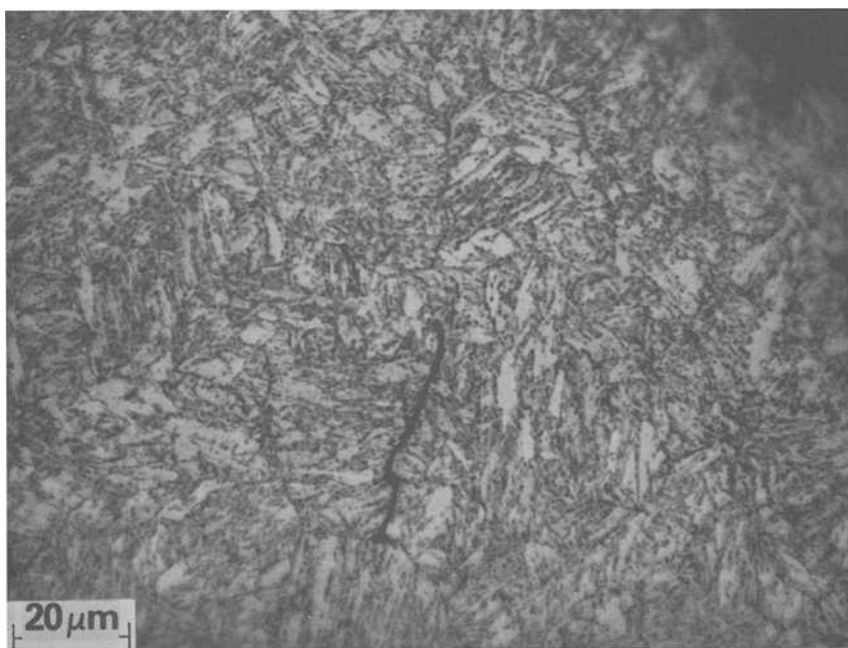


FIG. 15—HY-180 (9 nickel) EF-VAR specimen, as received; with tempered martensite with a heavy precipitation of carbides. These are discontinuous intergranular and transgranular microcracks. The specimen is etched with 2 percent nital.



FIG. 16—HY-180 (9 nickel) EF-VAR specimen, tempered, with heavily tempered martensite and bainite. The specimen is etched with 2 percent nital.

In general, the results obtained from the HY-130 specimens indicate that a significant decrease in SCC resistance as measured by the stress-intensity factor, K_{Isc} , occurs with the transition in the microfracture process from MVC to cleavage, IG fracture, or both.

In the HY-180 series, both the 10 nickel VIM-VAR and the 9 nickel EF-VAR specimens tested in the as-received condition exhibited IG fracture and cleavage in the SCC area, although the K_{Isc} value for the 10 nickel specimen was substantially higher. Tempering the VIM-VAR specimen to the 875-MPa (130-ksi) yield-strength level resulted in a change in the SCC fracture mode from IG separation and cleavage to MVC and, as with the HY-130 specimens, a higher K_{Isc} value.

In the EF-VAR heat-treated specimen, although a comparable change in SCC fracture mode from IG separation and cleavage to MVC was noted, the K_{Isc} value was actually lower. This is probably attributable to the much lower yield strength to which this specimen had been tempered. In both the VIM-VAR and EF-VAR specimens, tempering served to reduce the amount of crack macrobranching.

Of the two HY-130 steels tested as received, the homogeneous, tempered martensitic microstructure developed by the dual process EF-ESR method offered the better resistance to SCC. Further improvement in SCC resistance at a comparable yield strength appears to be made possible by tempering premium quality HY-180 (10 nickel) VIM-VAR plate to the 875-MPa (130-ksi) yield-strength level.

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Fractographic and Microstructural Analysis of Stress-Corrosion Cracking of ASTM A533 Grade B Class 1 Plate and ASTM A508 Class 2 Forging in Pressurized Reactor-Grade Water at 93°C

REFERENCE: Provenzano, V., Törrönen, K., Sturm, D., and Cullen, W. H., "Fractographic and Microstructural Analysis of Stress-Corrosion Cracking of ASTM A533 Grade B Class 1 Plate and ASTM A508 Class 2 Forging in Pressurized Reactor-Grade Water at 93°C," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 70-85.

ABSTRACT: Stress-corrosion cracking (SCC) studies were conducted in the following commonly used pressure vessel steels: ASTM A533 Grade B Class 1 (A533-B-1) plate and a fabricated forging that is equivalent in chemistry and thermomechanical processing to American-made ASTM A508 Class 2 (A508-2) forgings. The purpose of these studies was to determine the response of the materials in a simulated pressurized-water reactor environment. Round tensile specimens were bolt-loaded to 75 to 80 percent of yield and left in the water at 93°C for 2000 h. At the end of this time period, the specimens were taken out of the water, unloaded, and examined by scanning electron microscopy (SEM) and by energy-dispersive X-ray analysis. The specimen cut from A533-B-1 plate did not have any cracks as a result of the SCC tests. Several cracks, some of which were visible with the unaided eye, had developed in the specimen that was cut from the A508-2 forging. The polished and etched sections of the specimen that showed SCC, when examined with the SEM, revealed the presence of many microcracks starting from the outside surface and propagating along either inclusion-matrix or carbide-matrix interfaces; the corresponding microstructure was mostly pearlitic. The microstructure of the specimen that did not crack was bainitic; it had fewer inclusions and carbide particles, and no microcracks were observed. When the fracture surfaces of the larger cracks were examined by SEM, they revealed a cleavage-like failure propagating along inclusion bands. X-ray image scans and energy spectra of these bands showed that they were silicate and manganese-sulfide inclusions.

A hydrogen-assisted cracking model has been proposed to explain the experimental results on A508-2 forging. Produced by a cathodic reaction and aided by the stress

¹ Physicist and mechanical engineer, respectively, Thermostructural Materials Branch, Naval Research Laboratory, Washington, D.C. 20375.

² Head, Metals Laboratory, Technical Research Centre of Finland, Espoo, Finland.

³ Mechanical engineer, Staatliche Materialprüfungsanstalt (MPA), Universität Stuttgart, Stuttgart, West Germany.

fields, hydrogen diffuses ahead of the crack tip to the inclusion sites. This causes a preferential decohesion at the inclusion—matrix interfaces and subsequent cracking along inclusion bands.

The absence of stress-corrosion cracking in A533-B-1 plate tested under identical experimental conditions is due mainly to fewer inclusion and carbide particles and to the more refined bainitic microstructure of this steel. This type of microstructure is less susceptible than the mainly pearlitic microstructure found in A508-2 forging to hydrogen-assisted cracking.

KEY WORDS: stress-corrosion cracking, pressure vessel steels, fractography, fractographic features, energy-dispersive X-ray analysis, inclusion particles, hydrogen embrittlement, reactor-grade water, materials, materials science

Primary pressure vessels for water-cooled nuclear reactors are fabricated from low-alloy steels such as ASTM A533 Grade B Class 1 plate (A533-B-1) and ASTM A508 Class 2 (A508-2) forging. The combinations of sustained and cyclic loads can give rise to the phenomena of stress-corrosion cracking (SCC), corrosion fatigue, or both.⁴ Stress-corrosion cracking is known to be a problem in high-strength and the austenitic steels when exposed to certain aggressive environments [1–3].⁵ Recently, however, limited data have become available on low-alloy steels which show that an aqueous environment at temperatures close to 100°C can promote SCC in these materials [4, 5]. Other data concern SCC failure of steam turbines in conventional power plants [6, 7].

Lately, especially in connection with the light-water reactor safety program, a great deal of attention has been given to the potential problems of corrosion fatigue occurring in the low-alloy steels exposed to a pressurized water environment [8]. However, little research has been done in the area of stress-corrosion cracking occurring in these steels when exposed to similar environments. The present SCC study of A533-B-1 plate and A508-2 forging and the attendant fractographic and microstructural analyses are being conducted to assess the magnitude of the problem. The study developed when a stress-corrosion cracking component appeared to be present, under certain test conditions, in A508-2 forging fatigue tested in reactor-grade water at high temperatures [8].

In the present work, two bolt-loaded tensile specimens, one of A533-B-1 plate and the other of A508-2 forging, exposed to a reactor-grade water at 93°C for 2000 h, were examined by scanning electron microscopy (SEM) and by energy dispersive X-ray analysis. The basic aims of the fractographic and microstructural analyses were to search for correlations and relationships between the fractographic features and microstructural components, on one hand, and the SCC behavior of the two steels on the other.

⁴ Stress-corrosion cracking is the cracking process that is caused by the joint action of a mechanical load and an aggressive environment, whereas corrosion fatigue is the crack growth resulting from cyclic stresses and an aggressive environment.

⁵ The italic numbers in brackets refer to the list of references appended to this paper.

Materials and Test Specimens

The materials used in this investigation were ASTM A533 Grade B Class 1 plate and ASTM A508 Class 2 forging pressure vessel steels. The chemical compositions for both steels are given in Table 1. A533-B-1 plate, a high-shelf [high energy absorbed in a Charpy V-notch (CVN) test in the upper-shelf temperature region], low-sulfur content steel, was obtained from the Heavy Section Steel Technology (HSST) program; the A508-2 forging, of foreign fabrication, came from a large ~ 200 -mg (200-ton) block intended for a flange ring for a reactor pressure vessel. The forging, however, had been rejected because nondestructive and metallographic examinations had revealed the presence of cracks at the center and macrosegregation, that is, an excessive amount of inclusions, throughout the forging block. These two pressure vessel steels were chosen for the SCC tests because they constitute a best case–worst case study. The intention was to examine the SCC response of materials of different qualities in a simulated reactor-water environment at 93°C—one material of relatively high quality (the A533-B-1 plate) and the other of relatively low quality (the A508-2 forging). The A508-2 tensile specimen used for the SCC tests was cut along the through-thickness direction, from a section of the forging that was free of cracks but had excessive segregation. Round tensile specimens were machined from both steels, with the following dimensions: 32 mm (1.25 in.) along the gage section and 9.5 mm (0.375 in.) in diameter. Prior to the SCC testing, the specimens were examined optically to be sure that they were free of machining flaws.

Experimental Procedure

The specimens were bolt-loaded, along the tensile axis, to about 85 to 90 percent of their respective yield strengths using a 53 400-N (12 000-lb) capacity tensile machine. The specimens were loaded in air at room temperature, and the applied loads were measured by means of strain gages attached to the specimens. To avoid introducing large shear components on the specimens, the following loading procedure was employed. The specimen was loaded to 85 to 90 percent of its yield strength with the tensile machine, and the strain gage reading was recorded for this stress level. The room temperature yield strength was 557 N/mm^2 (80.2 ksi) for the A533-B-1 plate and 367 N/mm^2 (53.2 ksi) for the A508-2 forging. Afterwards, the tensile load on the specimen was reduced to about 70 percent of the yield strength. The bolts on the loading assembly were tightened while the specimen was still under tension. The specimen was taken out of the tensile machines when the bolt-loading produced a strain-gage reading equivalent to 85 to 90 percent of the yield strength. To be sure that bolt-loading *per se* did not produce cracks in the specimens, after the first loading, the specimens were unloaded and examined once again with an optical microscope. Then, the specimens were loaded a second time to the previous stress level (85 to 90 percent of the yield stress), using the procedure just described. Computing the loads for, and applying them at, room temperature

did not introduce any significant errors in the actual levels realized for the tests at the higher temperature. Further, the test jig was also made from A508 forgings, thus eliminating electrochemical reactions and ensuring that the (zero) differences in thermal expansion and elastic modulus did not produce any artificial load changes upon the change in temperature from 27 to 93°C. This was verified by the fact that the difference in the strain-gage readings between room temperature and 93°C was the same in both the unstrained and strained conditions.

Before the SCC tests, any stress relaxation on either the loading assembly or the specimen that might be caused by the temperature difference between the loading temperature (room temperature) and the test temperature (93°C) was measured by leaving the bolt-loaded specimens for a number of days inside an oven kept at 93°C. The specimens were taken out of the oven when it became apparent that all stress relaxation had stopped. The maximum stress relaxation measured this way was about 10 percent of the room temperature yield strength. That is, the final loading on the specimens was about 75 to 80 percent of the room temperature yield strength, which is, for all practical purposes, the same as the 93°C yield stress value.

The SCC tests were conducted using a closed environmental chamber known as the "water pot." This chamber is primarily used for fatigue studies on A508-2 pressure-vessel steel in connection with the light-water reactor safety program carried on at the Naval Research Laboratory (NRL) for the U.S. Nuclear Regulatory Commission (NRC). The water pot holds about 6 litres of reactor-grade water at 93°C at a pressure close to 1 atm. The water in the chamber was circulated at about 1 litre per hour. The reactor-grade water used in the environmental chamber was high-purity water containing 1000 ppm boron as boric acid and 1 ppm lithium as lithium hydroxide. The total impurity content of other elements was less than 1 ppm. The specific conductance of the reactor-grade water was 16 to 20 mho/cm after exposure in the water pot, and the pH approached 5.8 from an initial value of 6.7 to 6.8. The dissolved oxygen level was held as low as possible (0 to 2 ppb) by continuous deoxygenation of the water. Additional details concerning this device may be found elsewhere [8]. The loaded specimens were placed in the environmental chamber and left there for 2000 h. After this time period, the specimens were removed from the water pot, unloaded, and examined by microscopic techniques.

The specimens were sectioned lengthwise for metallographic specimen preparation and subsequent microstructural analyses. One half of one specimen was broken in liquid nitrogen, and the fracture area was examined with a scanning electron microscope and with an energy-dispersive X-ray analyzer. The metallographic sections were ground and polished by conventional techniques. Before the polishing, the sections were nickel-plated for edge retention. The polished sections were etched in 1 percent nital solution for about one-half min and then examined by SEM, using an International Scientific Instruments (ISI) Super II instrument.

An SEM examination of the metallographic sections provided information on microstructural components. Phase identification was obtained by energy-dispersive X-ray analysis employing an Advanced Metals Research (AMR) 1000 SEM equipped with EDAX Model 707B dispersive-energy X-ray analyzer. Fractographic examination was carried out using two different scanning electron microscopes: a Coates and Welter field emission microscope and an ISI Super II microscope. Both electron microscopes were operated at an accelerating voltage of 20 kV.

Results and Discussion

When the A508-2 specimen was taken out of the water pot and unloaded, seven separate cracks were present along the specimen gage section. These cracks were clearly visible with the unaided eye, and their circumferences ranged in length from 1.5 mm (0.060 in.) to 9 mm (0.360 in.); the depth of the largest crack was about 3 mm (0.12 in.). No cracks were seen in the A533-B-1 specimen, which had been tested under identical experimental conditions. The photographs in Fig. 1 show the appearance of the A508 tensile specimen. These photographs show the entire specimen (Fig. 1a), the largest of the seven cracks (Fig. 1b), and a smaller crack (Fig. 1c). As can be seen from the photographs, the cracks are oriented perpendicular to the direction of the applied stress. Some other smaller cracks, found also on the gage section of the A508-2 specimen, are shown by the SEM photomicrographs in Fig. 2.

The SEM photomicrographs in Fig. 3 show typical features of the microstructure of A533-B-1 plate and A508-2 forging materials; these photomicrographs were taken from the polished and etched sections of the two specimens. The microstructure of the A508-2 forging (Fig. 3a) shows several inclusions and coarse carbides. On the other hand, the microstructure of the A533-B-1 plate (Fig. 3b) shows smaller carbides and fewer inclusion particles. An additional microstructural evaluation of the two steels showed that the microstructure of the A508-2 forging was ferritic-pearlitic with small amounts of bainite, whereas that of A533-B-1 plate was completely bainitic. Figure 4 is a montage of SEM photomicrographs of a polished and etched section of the A508-2 specimen showing one of the many microcracks that were present in this specimen. The microcrack started from the outside surface and propagated along the inclusion-matrix material interface. As can be seen in the photomicrographs, decohesion also occurred along the carbide-matrix interface. The SEM photomicrographs presented in Fig. 5 are higher magnification photomicrographs of the crack-tip areas of the cracks shown in Fig. 1b and Fig. 2a and of the microcrack in Fig. 4. The crack tips shown in this figure are of two different characters. Figure 5a shows the tip of a developed crack, which, consequently, has been blunted by plastic deformations. Figure 5b, instead, is the crack tip of a microcrack—that is, it represents the tip of a crack not developed—therefore, both the crack and its tip are tight and sharp. The important point to observe in Fig. 5 is the presence of small voids in the vicinity of both

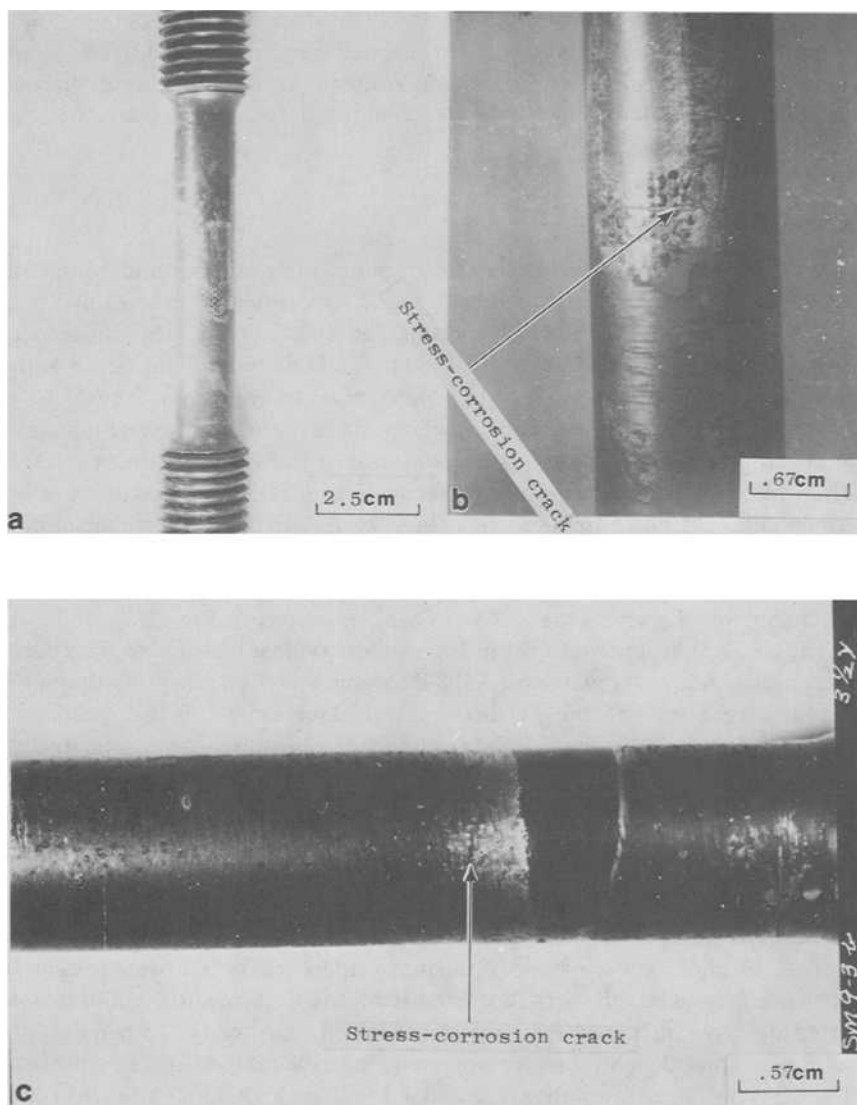


FIG. 1—Optical photographs of A508-2 tensile specimens used in the SCC testing. Photograph (a) shows the entire specimen; photographs (b) and (c) show some of the larger cracks that developed on the gage section following exposure to reactor-grade water at 93°C for 2000 h.

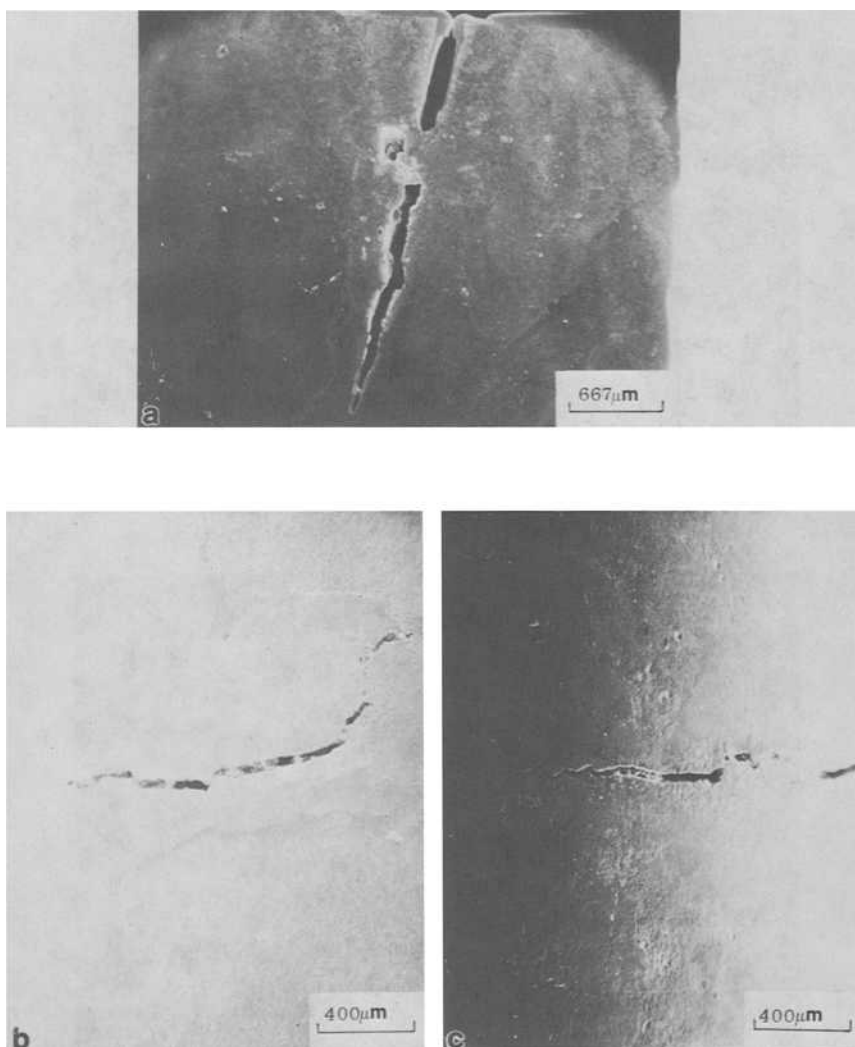


FIG. 2—Scanning electron photomicrographs of gage and the transverse gage sections of an A508-2 tensile specimen. Prior to SEM examination, the transverse section was metallographically polished and etched in 1 percent nital solution, the gage section was ultrasonically cleaned in alcohol and acetone. (a) shows the depth of the largest crack; (b) and (c) show two smaller cracks that developed during the SCC tests.

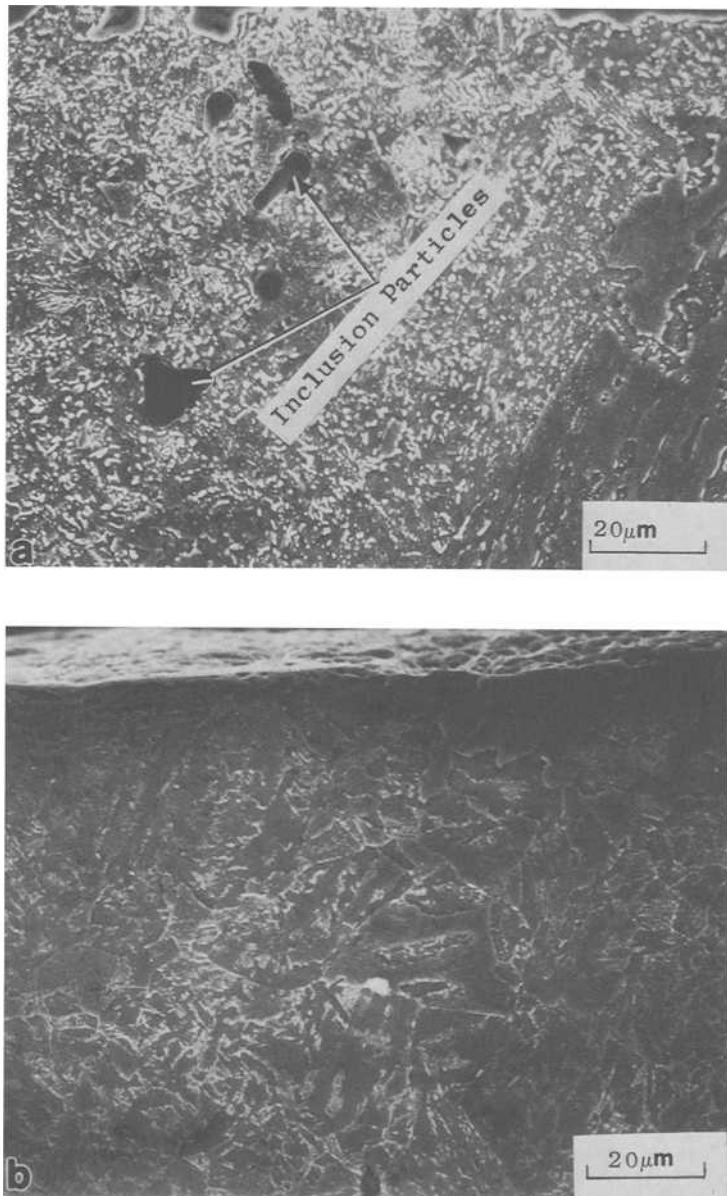


FIG. 3—Scanning electron photomicrographs of A508-2 forging and of A533-B-1 plate specimens. The photomicrographs were taken from the transverse sections of the specimens. The microstructure of A508-2 (a) is characterized by large amounts of carbide and inclusion particles; the microstructure of A533-B-1 (b) has fewer carbide particles and is almost free of inclusions.

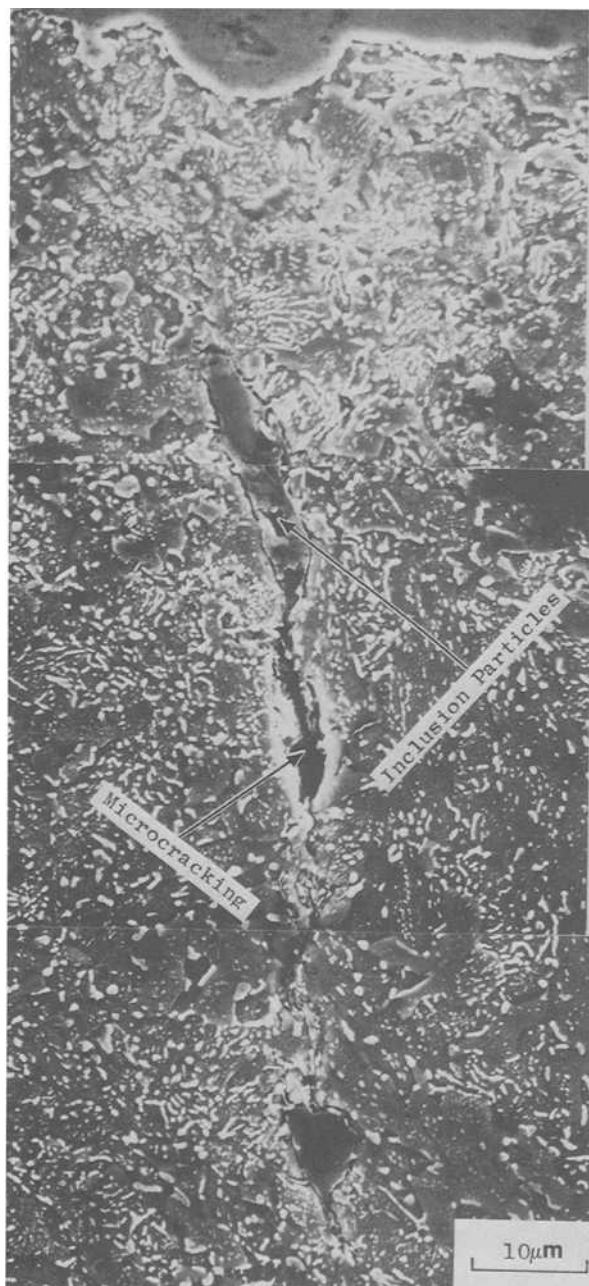


FIG. 4—A montage of SEM photomicrographs of a transverse section of an A508-2 specimen. The montage shows one of many microcracks found on the transverse sections after the SCC test. The microcrack originated at the outside surface and propagated along the inclusion-matrix and carbide-matrix interfaces.

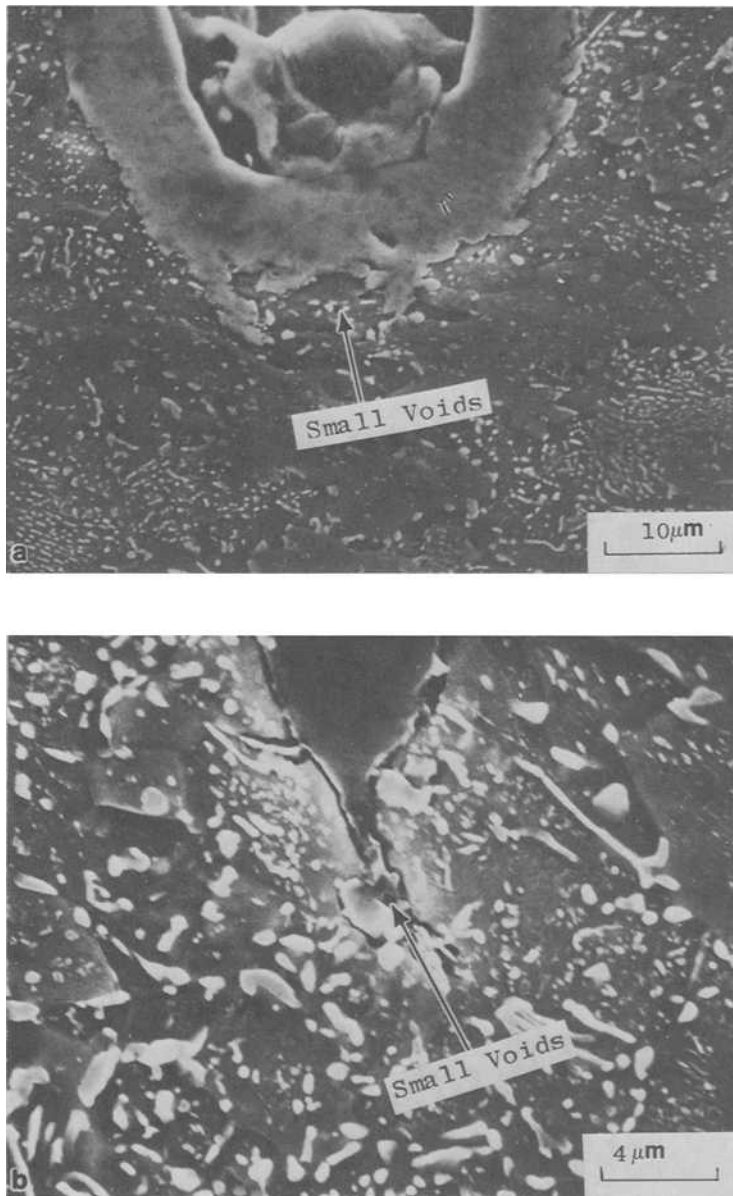


FIG. 5—Scanning electron photomicrographs of transverse sections of an A508-2 tensile specimen. Photomicrograph (a) is an enlarged view of the crack tip shown in Figs. 1b and 2a; photomicrograph (b) is an enlarged view of the tip of the microcrack shown in Fig. 4. The micrographs show the presence of small voids near the tips of both cracks.

crack tips. These voids are similar to ones observed in a parallel study of the fatigue crack growth resistance of A508-2 forging steel in reactor-grade water at temperatures up to 288°C (550°F) [8]. In that study it was suggested that the small voids are the sites to which hydrogen can most easily diffuse and be trapped.

The significant fractographic features observed on the stress corrosion area in one of the several cracks found in the A508-2 specimen are illustrated in the SEM photomicrograph presented in Fig. 6. Figure 6a is a low-magnification photomicrograph that gives a general view of the stress-corrosion area. The failure mode appears to be cleavage-like, and inclusion particles are clearly visible on the fracture surface. The inclusions are quite evident in the adjacent photomicrographs, which are of higher magnification (Fig. 6b and c). The inclusion particles have an elongated shape and are quite numerous. Figure 6d is the energy-dispersive X-ray spectrum of a small region in the stress-corrosion area near an inclusion particle. The spectrum, besides the expected peaks of iron and chromium, reveals the presence of manganese, silicon, and sulfur peaks and a small chlorine peak. The spectrum indicates that the inclusion particles are manganese sulfide (MnS) and silicate inclusions. The X-ray image scans presented in Fig. 7 refer to manganese and sulfur; similar image scans were obtained for the silicate inclusions.

To be sure that the SCC observed on the A508-2 forging was not the result of pure mechanical effects as is, for example, creep cracking, a control test in a hydrogen environment was conducted on the A508-2 forging. The same loading procedure and stress level used for the other two tests were employed in this control test. The bolt-loaded tensile specimen was exposed to pure hydrogen at ambient pressure for 2000 h at 93°C. After this time, the specimen was unloaded and examined. Optical examination showed that this specimen did not develop any cracks during SCC testing in gaseous hydrogen. This finding seems to suggest that SCC of A508-2 forging in reactor-grade water at 93°C is the result of an interaction between certain microstructural components and the aqueous environment.

Environment-Assisted Cracking Model

A hydrogen-assisted cracking model has been developed to explain the results described here. When the test specimen is exposed to a pressurized reactor-grade water at 93°C, an oxide layer, which consists mainly of hematite, is formed on the surface. Hematite is not protective, thus it allows further corrosion reactions to occur. Due to the presence of manganese sulfide and silicate inclusions, the prevailing electrochemical conditions initiate a localized corrosion attack on the metal matrix around the inclusions [9, 10], and a decrease of pH occurs [11, 12]. Also, the solubility of MnS even in pure water allows the formation of a sufficient concentration of hydrogen sulfide (HS^-) ions and sulfide (S^{2-}) ions. It is known that the sulfide ion promotes the entry of hydrogen

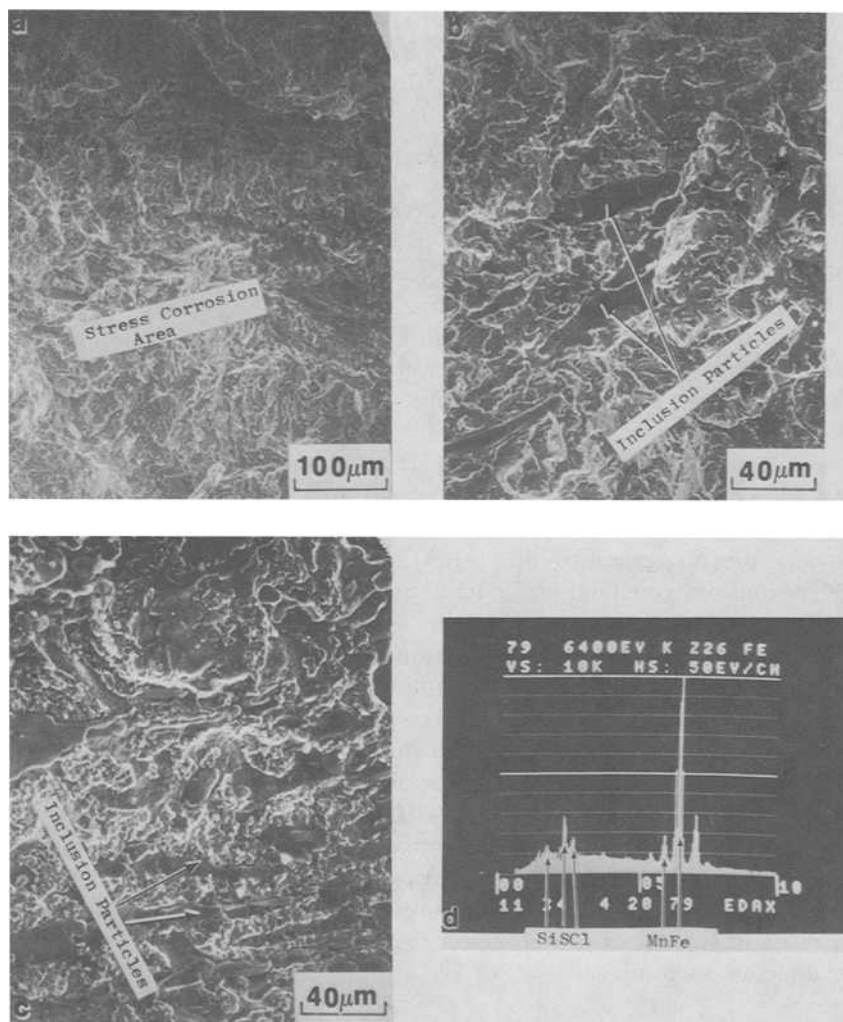


FIG. 6—Scanning electron photomicrographs (a, b, and c), and the energy-dispersion X-ray spectrum (d) taken from the stress-corrosion crack of an A508-2 tensile specimen. This stress-corrosion crack became accessible for SEM examination when one half of the specimen was broken at liquid nitrogen temperature along one of the several cracks that had developed on the gage sections. Photomicrographs (a) through (c) show cleavage-like fracture propagation along the inclusion bands. The X-ray spectrum shows that the bands are silicate and manganese sulfide inclusions.

into steel under conditions of electrochemical corrosion or cathodic charging [13]. The hydrolizing of the MnS inclusions therefore accelerates the corrosion reaction and the hydrogen evolution [11, 14]. Further enhancement of the corrosion attack may be caused by the migration of the chlorine ions into the crevice [9]. The energy-dispersive X-ray spectrum shown in Fig. 6d provides support for this hypothesis.

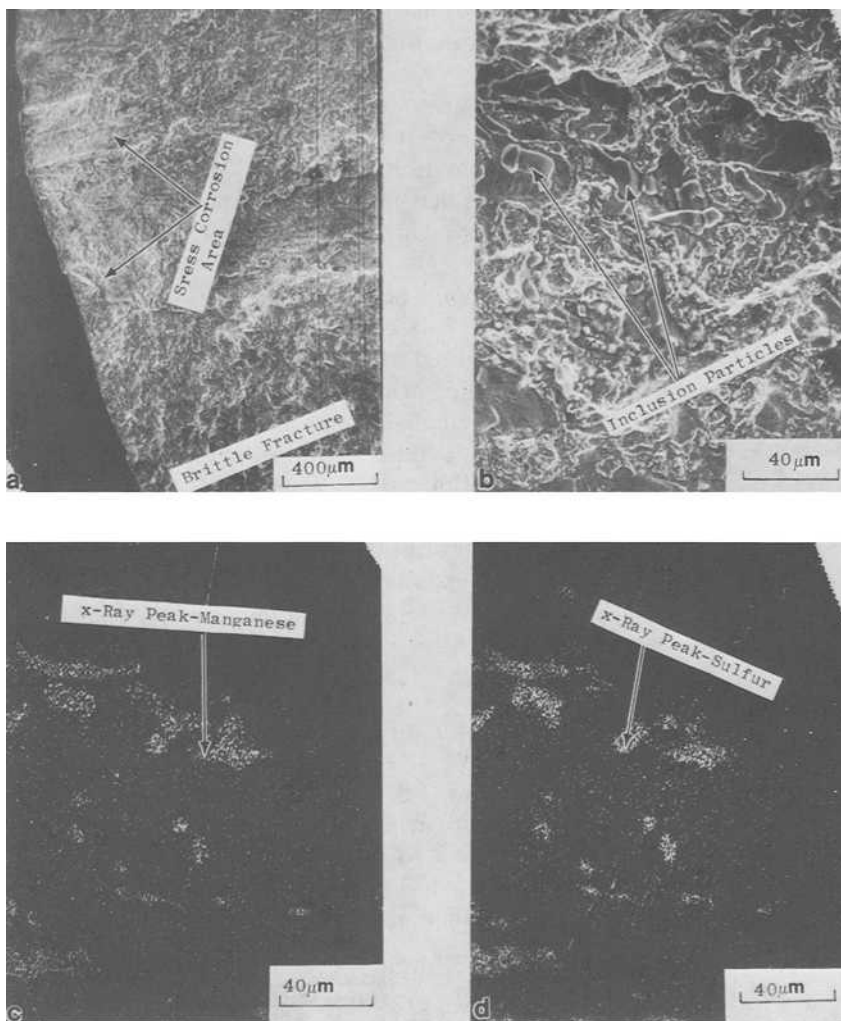


FIG. 7—Stress-corrosion area (a and b) and corresponding energy-dispersive X-ray image scans of an A508-2 specimen. The stress-corrosion area is the same one shown in Fig. 6. The X-ray image scans refer to manganese and sulfur; similar image scans were obtained for the silicates.

Hydrogen is generated and subsequently absorbed into the metal due to the local cathodic reactions and hydrolysis of water. The absorbed hydrogen diffuses along the stress gradient produced by the propagating crack and the inclusions under the applied stress, and hydrogen becomes trapped at the inclusion sites [13]. The presence of hydrogen at the interface lowers the bond strength between the inclusions and the matrix, as well as the carbides, which causes the subsequent separation of the inclusions and the carbides from the

matrix [15, 16]. This is clearly illustrated in Figs. 4 and 5 which show voids around inclusions and carbides close to the crack tip. This sufficiently increases the local stress so that, with the assistance of hydrogen, crack propagation can occur in the metal matrix, as evidenced by the cleavage-like features on the fracture surface. The absence of stress-corrosion cracking in A533-B-1 plate steel tested under identical conditions is due mainly to smaller amounts of inclusion and also to the more refined bainitic microstructure, which is less susceptible to hydrogen-assisted cracking than the mainly ferritic-pearlitic microstructure of A508-2 forging.

In closing, the following points should be made. First, SCC was induced in a low-quality, substandard steel. It is possible that the cracking was the direct result of detrimental effects that caused the material to be rejected and does not represent an inherent susceptibility of low-alloy steels to SCC in a pressurized water-reactor environment. Second, the importance of the environment-assisted cracking model resides in its uniqueness rather than the fact that it has been employed successfully in explaining the SCC test results in pressure vessel steels, namely, in A533-B-1 plate and A508-2 forging. Finally, to separate the effects of microstructure from those of the inclusion particles, it would be necessary to heat treat the A508-2 material to a fully bainitic condition. But such a heat treatment is beyond the scope of the present work.

Summary and Conclusions

Fractographic and microstructural studies by scanning electron microscopy (SEM) and by energy-dispersive X-ray analysis have been conducted on two pressure vessel steels: ASTM A533-B-1 plate and ASTM A508-2 forging tested for stress-corrosion cracking in a reactor-grade water at 93°C. The specimens, round tensile bars, bolt-loaded to about 75 to 80 percent of their respective yield strengths, were exposed to the water environment for 2000 h. The significant observations from this study may be summarized as follows:

1. The specimen from the A508-2 forging developed several cracks along the gage section, some of which were clearly visible to the unaided eye. The specimen from the A533-B-1 plate did not develop cracks.
2. Examination of the polished and etched sections of both specimens revealed that the A508-2 forging had large amounts of inclusions and coarse carbide particles, and its microstructure was mostly ferritic-pearlitic. In addition, many microcracks were observed that started from the outside surfaces and propagated preferentially along either the inclusion—matrix or the carbide—matrix material interface. The microstructure of A533-B-1 plate was mostly bainitic, had less carbide particles, and was almost free of inclusions. Finally, no microcracks were seen in this specimen.
3. Subsequent X-ray scans of the inclusions showed them to be silicate and manganese sulfide inclusions.

4. Fractographic examination of the stress-corrosion area of one of the cracks found in the A508-2 specimen showed a cleavage-like failure, with the crack propagating along inclusion bands.

5. A hydrogen-embrittlement model has been proposed to explain these results. For the specimen that cracked, the explanation is the following. Hydrogen is produced at the crack by a cathodic reaction between the water environment and the fresh metal surface. It diffuses into the plastic enclave just ahead of the crack tip by following the stress gradient caused by the propagating crack and the inclusions and is trapped along the inclusion—matrix interfaces. The presence of hydrogen at the interfaces causes local embrittlement by lowering the bond strength between the inclusions and the matrix. On the other hand, the presence of much fewer inclusions and carbide particles in the A533-B-1 plate, together with a more refined bainitic microstructure, makes this material immune to stress-corrosion cracking when exposed to reactor-grade water at 93°C.

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Hydrogen-Induced Brittle Fracture of Type 304L Austenitic Stainless Steel

REFERENCE: Caskey, G. R., Jr., "Hydrogen-Induced Brittle Fracture of Type 304L Austenitic Stainless Steel," *Fractography and Materials Science*, ASTM STP 733, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 86-97.

ABSTRACT: Type 304L stainless steel supersaturated with hydrogen gas fails in tension at 200 to 250 K by brittle fracture. Fracture surfaces exhibit isolated facets oriented 0 to 30 deg to the tensile axis. These facets have many of the characteristics of cleavage fracture. The threefold symmetry of Laue back-reflection photographs and trace analysis of deformation bands on the facets demonstrate that fracture is along {111} planes. The fracture paths were identified as coherent twin boundaries on polished sections through facets and on microcracks across interior grains. Although twin-boundary parting is not commonly observed in alloys with a face-centered cubic (FCC) lattice, these observations for Type 304L stainless steel and similar observations of Nitronic-40 and Tenelon demonstrate that parting is possible in austenitic steels under some circumstances.

KEY WORDS: fractography, fracture, stainless steel, metallography, hydrogen embrittlement, X-ray diffraction, materials science, materials

The effects of hydrogen on the deformation and fracture of Type 304L austenitic stainless steel have been studied extensively [1-11].² Several modes of hydrogen-assisted fracture (HAF) have been reported; such as, dimpled failure [3], quasi-cleavage [4], regular striations [9], intergranular fracture [4], and faceted fracture [5, 10, 11]. Investigation at the Savannah River Laboratory, Aiken, S.C., of the temperature and grain-size dependence on HAF in Type 304L stainless steel has shown that hydrogen damage is most severe at 200 to 250 K, and faceted fracture predominates at these temperatures [10, 11]. This paper describes the examination of facets in Type 304L stainless steel by scanning electron microscopy (SEM), X-ray diffraction, and optical microscopy. The author has concluded that the mode of formation of the facets is parting along coherent twin boundaries, a process similar to cleavage. A detailed mechanism has not been proposed. Twin parting is unusual in face-centered cubic (FCC) alloys; however, similar facets have been observed in

¹ Metallurgist, Savannah River Laboratory, E. I. du Pont de Nemours and Co., Aiken, S.C. 29801.

² The italic numbers in brackets refer to the list of references appended to this paper.

TABLE 1—Type 304L stainless steel chemical composition, weight percent.

C	Mn	P	S	Si	Cr	Ni	Mo
0.03	1.57	0.015	0.008	0.43	18.35	10.29	0.17

hydrogen-charged Tenelon³ and Nitronic-40⁴ and in stress-corrosion cracking (SCC) of Type 304 stainless steel in magnesium chloride MgCl_2 [12].

Experimental Results

The specimens came from bar stock of a single heat of consumable electrode, vacuum-melted steel with the composition given in Table 1. The average grain size of this steel was ASTM 11.4 ± 0.25 ($6.4 \pm 0.2 \mu\text{m}$). The grain size was estimated in accordance with the ASTM Method of Estimating the Average Grain Size of Metals (E 112-77), except that both twin and grain boundaries were counted. Coherent twin boundaries were included because they have many properties in common with grain boundaries, such as acting as barriers to dislocation motion and as dislocation sources during deformation [13, 14]. Specimens with ASTM grain sizes of ~ 5 ($\sim 80 \mu\text{m}$) and 1 ($\sim 300 \mu\text{m}$) were obtained by vacuum annealing for 24 h at 1270 and 1470 K, respectively.

Fractographic examination and supporting optical metallography and X-ray diffraction were performed on specimens cut from round, smooth bar-tensile specimens 4.8 mm in diameter, with a 25-mm gage length. Specimens were broken in tension at 200 to 250 K, the temperature region of greatest hydrogen damage [11]. As seen in Fig. 1, plastic strain to failure, $\epsilon_p = \ln A_0/A_f$, is a minimum at these temperatures. Also, note that there is no hydrogen damage evident in companion specimens tested at 78 K. All ductile fractures were by void coalescence, as shown in Fig. 2. The specimens selected for detailed examination were the most severely embrittled and displayed the most distinctive and clearly delineated facets, as seen in the example of Fig. 3. These facets have several distinctive features. They are not connected to one another, are planar and flat, frequently exhibit steps of 0.5 to 5 μm in height, always show from one to three sets of traces, never show river patterns, and have approximately the same linear dimension as the grain size.

X-ray Diffraction

By the Laue back-reflection X-ray diffraction technique, the fracture plane of the facets was established as a $\{111\}$ plane in the FCC austenite. A portion

³Tenelon is a trade name of U.S. Steel Co.

⁴Nitronic-40 is a trade name of Armco Inc.

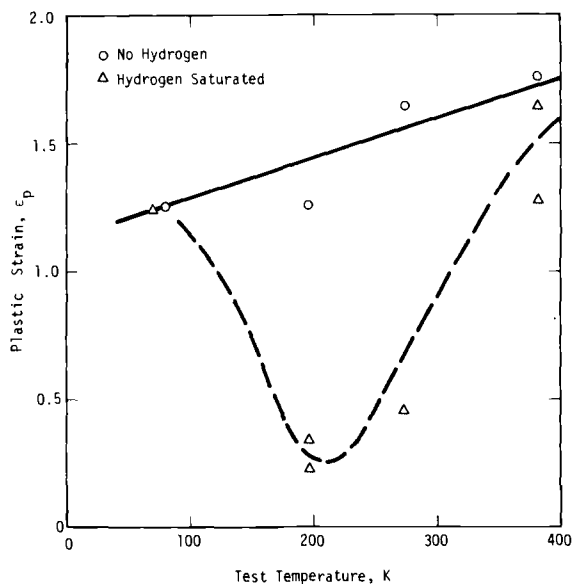


FIG. 1—Ductility minimum in hydrogen-charged Type 304L stainless steel.

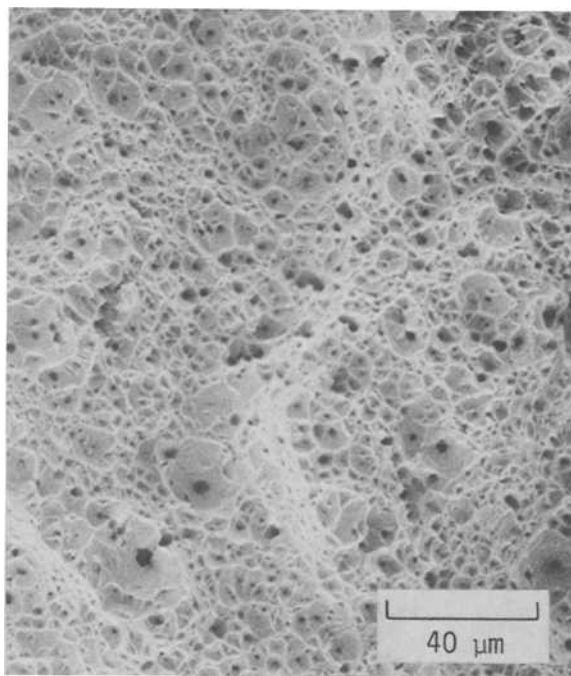


FIG. 2—An ordinary ductile fracture of Type 304L stainless steel.

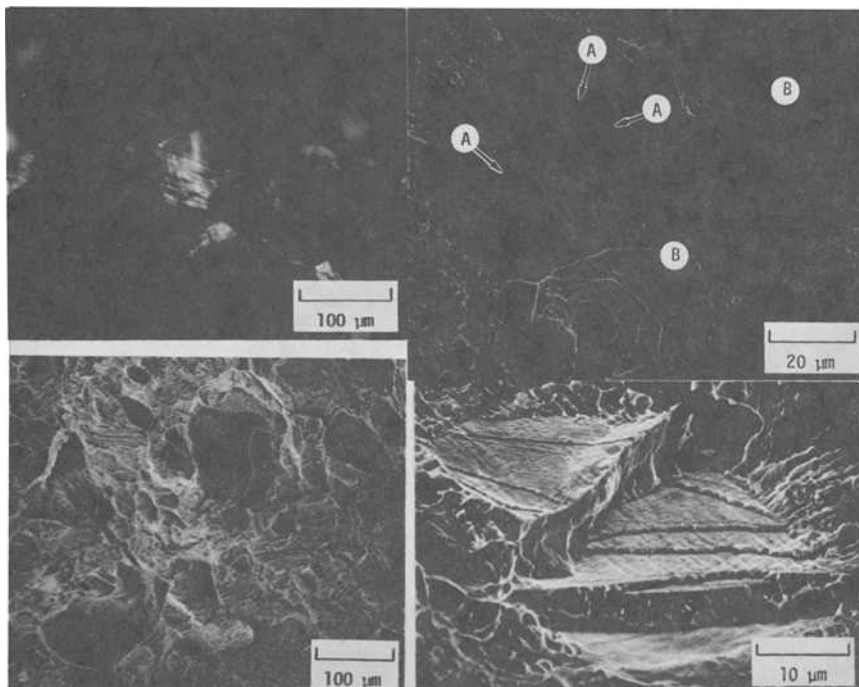


FIG. 3—General appearance of facets and surrounding matrix. The specimen was tested at 200 K. (Top, left) Optical photomicrograph; (bottom, left) scanning electron photomicrograph (not the same area as shown in top photomicrograph); (top, right) slip traces (A) and steps (B); (bottom, right) view of a facet at an angle.

of a fractured tensile specimen was mounted on a goniometer with the fracture face exposed. A large facet was selected and aligned by centering it in the cross hairs of a telescope that previously had been aligned with the center of the collimator (0.75-mm diameter) on the Laue camera. The goniometer was then adjusted visually to rotate the facet into a position perpendicular to the beam. Subsequent adjustments between Laue photographs were made as needed to center the diffraction pattern. Only specimens with the largest grain size ($\sim 300 \mu\text{m}$) had facets large enough to be examined readily by this technique.

The diffraction patterns, seen in Fig. 4, displayed two sets of Debye arcs each with a threefold axis of symmetry. Such patterns arise by reflection of characteristic radiation from symmetrically equivalent planes in the crystal when a symmetry axis is parallel to the incident beam. In this case, the beam is parallel to the $[111]$ axis and perpendicular to the (111) plane because of the threefold symmetry. The Debye arcs of the outer set have a Bragg angle of 21° and index as $\{331\}$ planes of austenite, given that the X-ray beam is perpendicular to a (111) plane. The angles between the centers of the Debye arcs

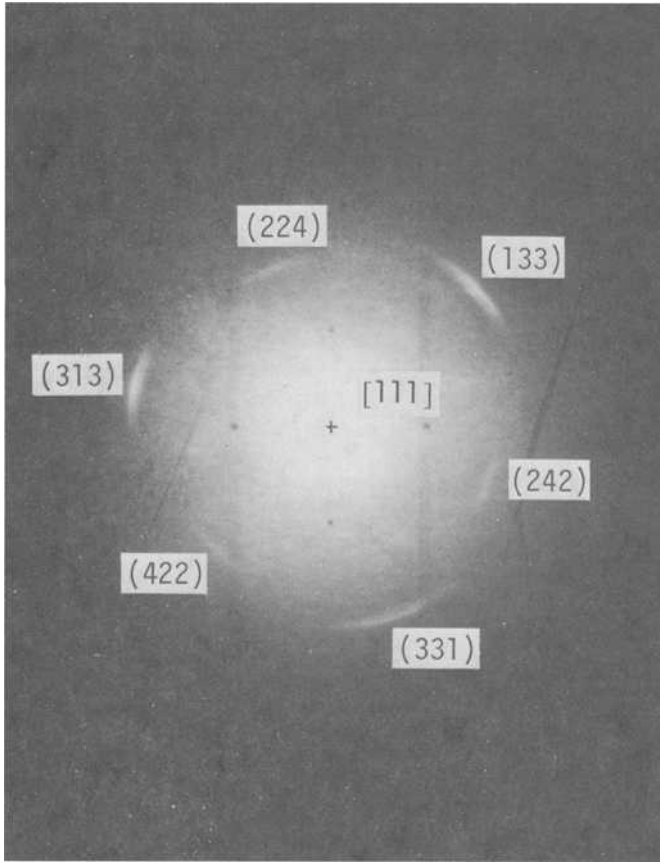


FIG. 4—Laue back-reflection pattern from a facet on the fracture of the specimen broken at 200 K. (Specimen-to-film distance = 3 cm.)

measure 36 deg, which corresponds closely to the calculated value of 38 deg. The Debye arcs of the inner set also have threefold symmetry but are rotated 60 deg from the outer set and may be indexed as $\{422\}$ planes of austenite, with a measured angle 19 deg from $[111]$, which is close to the calculated value 19 deg 28 min.

The calculated interplanar spacings are 0.825 and 0.73×10^{-10} m, assuming that they arise from copper $K\alpha$ and $K\beta$ characteristic radiation, respectively. These spacings are in close agreement with spacings of austenite for a lattice parameter of $a_0 \approx 3.589 \times 10^{-10}$ m as measured by wide-angle X-ray scattering (WAXS) on this bar stock. The calculated interplanar spacings do not match those of α' -martensite for either copper $K\alpha$ or copper $K\beta$ radiation. The measured lattice parameter for α' -martensite was 2.866×10^{-10} m.

Single-surface trace analysis by the technique described by Fong [15] agreed with the Laue X-ray pattern. The traces on the facet faces were presumed to arise by intersection of the deformation bands with the coherent twin boundary. Slip, ϵ -phase formation, and deformation twinning occur on $\{111\}$ planes. The bounding planes of α' -martensite laths also lie along $\{111\}$ planes in the austenite. Therefore, the poles of the traces should plot as $\langle 111 \rangle$ directions on a standard stereographic plot. Plots from several facets confirmed these assumptions.

The occurrence of short Debye arcs rather than Laue spots arises from non-uniform deformation or bending of the austenite lattice. Bending of the facet planes is evident in Fig. 3 (*bottom, right*).

Wide-angle X-ray scattering from the fracture faces indicated the presence of austenite with small amounts of martensite and ϵ -phase. The absence of diffraction by martensite and ϵ -phase in the Laue patterns of the facet could be attributed to too low an intensity because of the small volume of phase present or to the absence of these phases from the facet. Experimental data do not resolve this question.

Metallography

Metallographic examination of longitudinal sections of fractured and plastically deformed tensile specimens identified the facet planes as coherent boundaries of annealing twins that occur in all common austenitic stainless steels with frequencies of two to three twins per grain [16]. Microcracks were found predominantly along coherent twin boundaries throughout the plastically deformed regions of the specimens, as well as in the immediate vicinity of the fracture. The twin-boundary traces lie most commonly at an angle of 60 to 90 deg from the tensile axis, as in Fig. 5. In most cases, there is a distinct curvature of the deformation bands adjacent to the twin boundaries, which is indicative of higher dislocation densities in those regions.

Faces of the microcracks were inspected by sectioning specimens longitudinally and then inspecting them in the scanning electron microscope (SEM) to observe the crack face, or by bending the polished section to open the cracks. Markings identical to those on the facets of the fracture surface were observed, which indicates that facets and cracks form by the same mechanism. Figure 6 shows optical and SEM photographs of a crack on a polished and etched section with the crack face along a twin boundary. Other cracks, which were slightly below the polished surface before bending, also show markings identical to those of the facets (Fig. 7). Small microcracks ~ 0.5 to $2 \mu\text{m}$ in length are visible along the twin boundaries in Figs. 5 and 6 (*bottom*) and appear to form where slip traces intersect the twin boundary.

Slip traces, steps, and grooves on a facet plane on one half of a fractured specimen mate with the same feature on the opposite half of the specimen. A

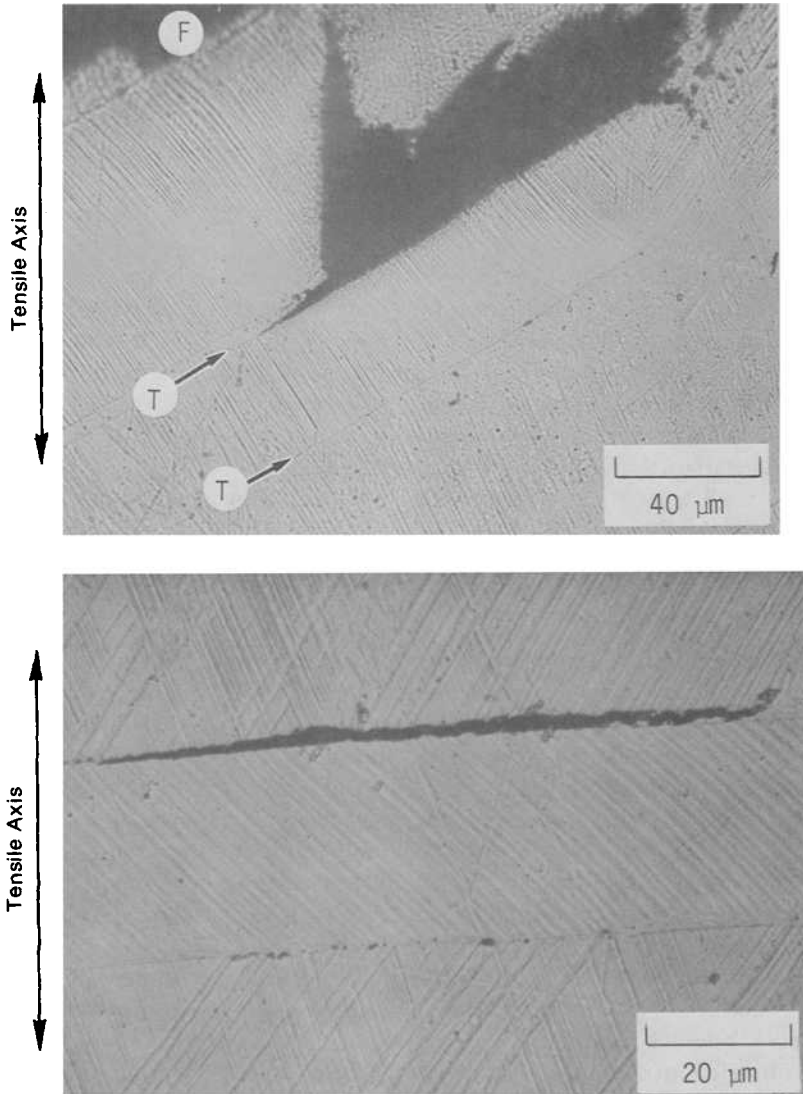


FIG. 5—Microcracks along coherent twin boundaries on the longitudinal section of the fractured specimen. (Top) Fracture (F) and twin boundaries (T).

step up on one face matches a step down on the other, and a projection of one face matches a hole on the matching face, as seen in Fig. 8. In no case did the facets have matching tear ridges that would identify the fracture mechanism as quasi-cleavage in the sense defined by Beachem [17].

Characteristics of the facets correspond to those attributed to cleavage facets: separation along a crystal plane, steps and other irregularities that

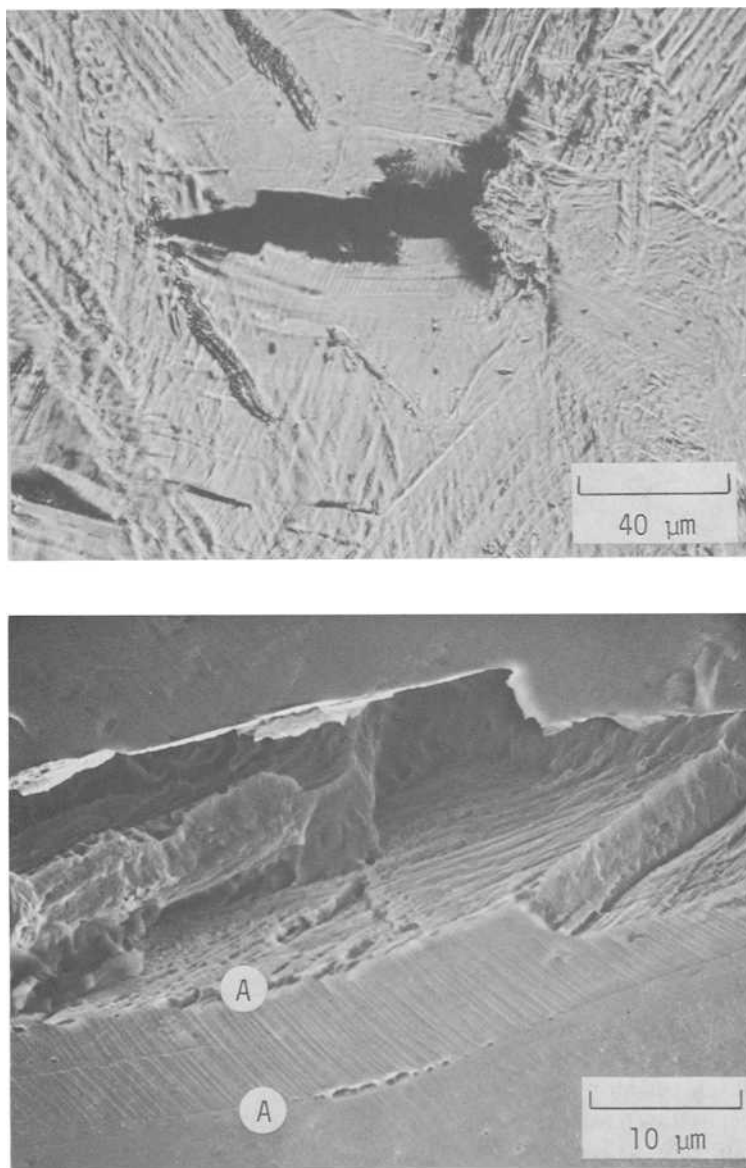


FIG. 6—An interior microcrack revealed by sectioning and bending of the fractured specimen: (top) longitudinal section, optical photomicrograph; (bottom) SEM of microcrack face and polished-face coherent twin boundaries (A).

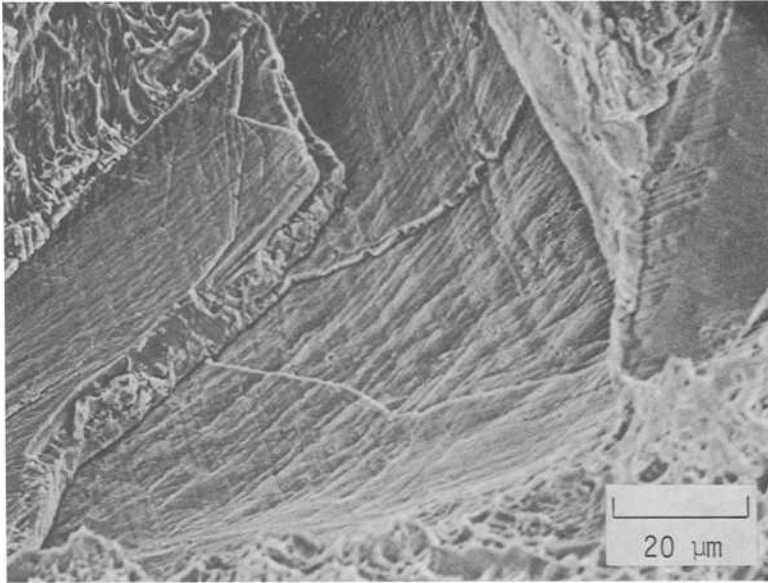


FIG. 7—Microcrack face from the interior of the specimen below the polished face.

mate across the cleavage plane, and facets only forming in crystals oriented correctly in relation to the applied stress system. However, river patterns, which are generally observed on cleavage facets, are absent in the present case. The absence of river patterns may be explained by the observation that their formation is linked to the presence of intersecting cleavage planes or to the intersection by twin boundaries of the operative cleavage plane. In the present case, the twin boundaries along which fracture takes place are generally parallel to one another in any given grain. For these reasons, fracture is better described as twin parting rather than cleavage.

Discussion

Coherent twin boundaries have been identified as a fracture path in alloys with an FCC lattice under widely differing circumstances. Wedge-type cracks have developed along twin boundaries during deformation of Type 304 stainless steel in slow tension at 920 K [18, 19]. Fatigue fracture of copper and high-purity nickel has been shown to favor twin interfaces [20]. Therefore, fracture along coherent twin boundaries occurs relatively easily and does not depend upon the presence of hydrogen. The stress or strain requirements for initiation of twin-boundary fracture may be modified by hydrogen, however, so that twin-boundary fracture occurs in a temperature region where only ductile fracture would ordinarily be observed.

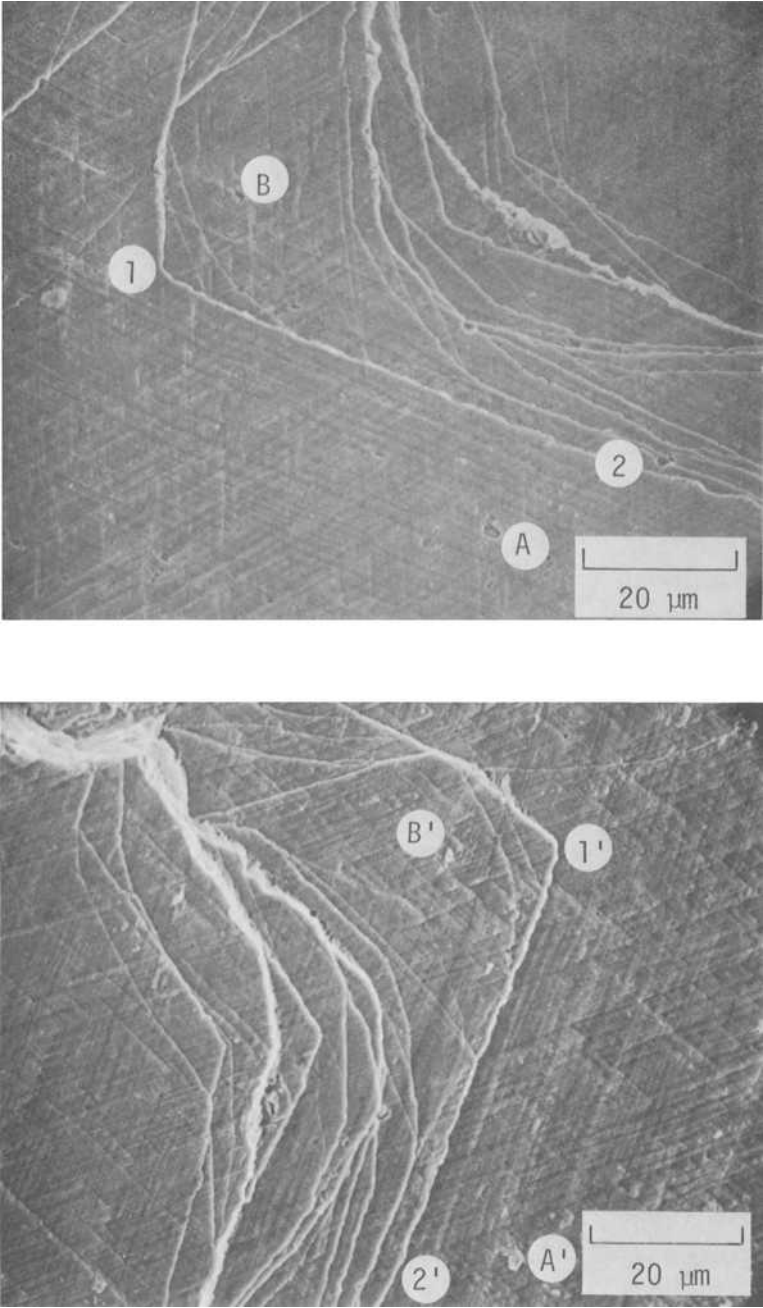


FIG. 8—*Matching faces of the fracture surface.*

The possibility of twin-boundary parting in austenitic stainless steel has been reported in stress-corrosion testing, although the path was not identified as a twin boundary. Faceted fracture similar to that in Fig. 3 was observed in stress-corrosion cracking of Type 304 stainless steel in boiling 42 percent MgCl_2 solution [12]. The fracture path was suggested to be along martensite-austenite interfaces. However, traces and steps on the facet surface suggest that the fracture path is along coherent twin boundaries, as in the HAF of Type 304L stainless steel. Supporting metallographic evidence that would confirm this suggestion was not presented. Therefore, faceted fracture may occur in austenitic steels under conditions in which hydrogen may not be present.

The accumulated evidence on hydrogen-assisted fracture of austenitic steels indicates that martensite is not necessary for twin-boundary parting. Facets are seen in Tenelon and Nitronic-40 under conditions in which martensite is believed to be absent [11]. Furthermore, martensite does not appear to be a sufficient condition for brittle fracture. The fracture of hydrogen-charged Type 304L stainless steel at 78 K is wholly ductile in spite of the fact that martensite forms during the cooling to 78 K as well as during subsequent deformation. Consequently, the role of martensite in HAF of austenitic steels is uncertain in spite of the known brittleness of martensite in comparison with austenite and the presence of strain-induced martensite in austenitic steels that are most severely embrittled by hydrogen.

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Effect of Temperature on the Fracture Toughness Behavior of Inconel X-750

REFERENCE: Mills, W. J., "Effect of Temperature on the Fracture Toughness Behavior of Inconel X-750," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 98-114.

ABSTRACT: The elastic-plastic J_{Ic} fracture toughness response of precipitation heat-treated Inconel X-750 was evaluated by the multispecimen resistance-curve (R -curve) technique at room temperature and elevated temperatures. The fracture toughness of this nickel-base superalloy was found to increase slightly under intermediate temperature conditions (427°C), but it decreased dramatically at the highest test temperature (649°C). Metallographic and electron fractographic examination of fracture surfaces revealed that the slight increase in J_{Ic} at intermediate temperatures was associated with an intergranular-to-transgranular fracture mechanism transition. At room temperature, crack extension occurred primarily by an intergranular dimple rupture mechanism attributed to microvoid coalescence along a grain-boundary denuded region. In the 427 to 538°C regime, the fracture surface was dominated by a transgranular morphology consisting of poorly defined dimples coupled with a rather flat, faceted appearance. At 649°C, intense heterogeneous slip initiated localized separation, which resulted in a very faceted fracture surface morphology and a severe degradation in fracture toughness.

KEY WORDS: fractures (materials), J -integral, nickel-base superalloys, elevated temperature tests, fractography, fracture mechanism transition, materials, materials science

Inconel² X-750 is a nickel-base superalloy strengthened by precipitation of $Ni_3[Al,Ti]$, termed gamma prime (γ'), and various metallic carbides. Like many superalloys, it possesses excellent corrosion and oxidation resistance, in addition to good tensile and creep behavior at elevated temperatures. As a result of these properties, this nickel-base alloy has been considered for application as a heat-resistant structural material in the nuclear and aerospace industries. Design and safety analyses for such components require a comprehen-

¹Senior engineer, Hanford Engineering Development Laboratory, Westinghouse Hanford Co., Richland, Wash. 99352.

²Inconel is a registered trade name of the International Nickel Co. General specifications for this material may be found in the ASTM Specification for Precipitation Hardening Nickel Alloy Bars, Forgings, and Forging Stock for High-Temperature Service [A 637-70 (1976)].

TABLE 1—*Chemical composition, percent by weight.^a*

Producer/Heat No.	C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	Co
Huntington/HT6716X	0.03	0.56	6.66	0.007	0.40	0.09	72.84	15.27	0.67	2.60	0.07

^a Melt process: air melt. Heat treatment: solution annealed at 1149°C for 2 h and air cooled; aged 24 h at 843°C and air cooled and aged 20 h at 704°C and air cooled.

sive understanding of the elevated temperature mechanical properties of this high-strength structural alloy. The tensile [1–4],³ stress rupture [1, 2, 5], and fatigue [6, 7] responses of Inconel X-750 have been evaluated at elevated temperatures; however, fracture toughness data have been generated only at room temperature and cryogenic temperatures [8]. Consequently, a fracture mechanics approach was employed in this study to evaluate the elevated temperature fracture behavior of Inconel X-750.

The ductile fracture response of this heat-resistant superalloy precludes the use of conventional linear-elastic fracture mechanics techniques. However, recent advances in the ductile fracture regime have been made with the elastic-plastic J_{Ic} fracture mechanics approach [9, 10], which extends the use of fracture mechanics to materials that exhibit appreciable plasticity prior to failure. Therefore, J_{Ic} fracture mechanics concepts were employed during the current investigation to characterize the ductile fracture toughness response of Inconel X-750 at temperatures ranging from 24 to 649°C. In addition, fracture surfaces were examined to relate operative fracture mechanisms to key microstructural features found in this nickel-base superalloy.

Experimental Procedure

Material

The 2.5-cm thick plate of Inconel X-750 used in this study was obtained from Huntington Heat HT6716X. The chemical composition for this heat is given in Table 1. The material was tested in the double-aged, precipitation-hardened condition:

- (a) solution annealed at 1149°C for 2 h and air cooled,
- (b) aged at 843°C for 24 h and air cooled, and then
- (c) aged at 704°C for 20 h and air cooled.

The tensile properties for heat-treated Inconel X-750 are summarized in Fig. 1. In general, the yield strength, σ_{ys} , was found to be relatively insensitive to temperature over the entire test temperature range, 24 to 649°C, whereas

³ The italic numbers in brackets refer to the list of references appended to this paper.

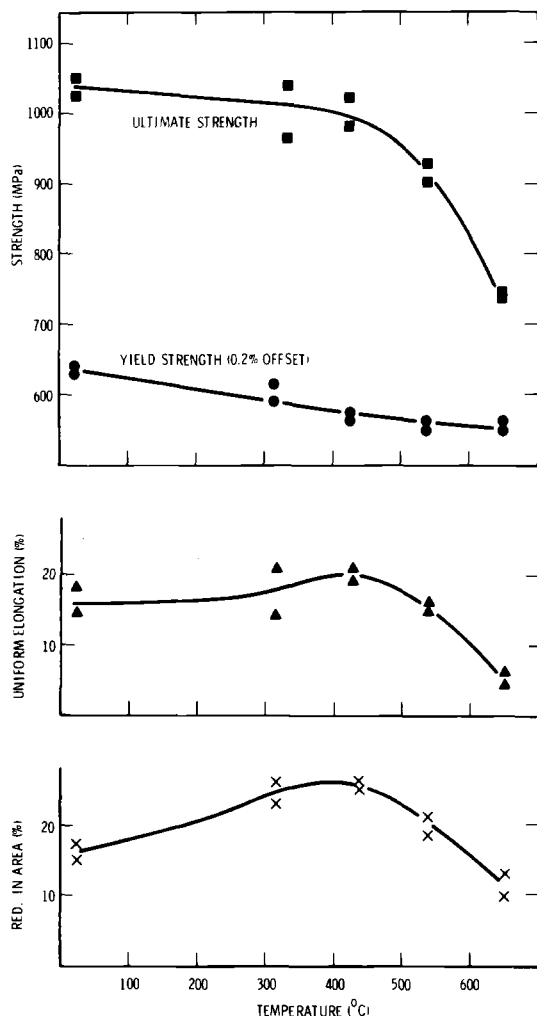


FIG. 1—The effect of temperature on the tensile properties of precipitation heat-treated Inconel X-750.

significant reductions in the ultimate strength, σ_{UTS} , and ductility levels were observed at higher temperatures.

The typical microstructure of the precipitation-hardened alloy is illustrated in Fig. 2. The double-aging heat treatment resulted in the formation of a duplex γ' precipitate structure in the grain interior, with coarse γ' particles appearing after the 843°C intermediate age and fine γ' particles forming during the final 704°C age. In addition, Fig. 2 (top) reveals the presence of an $M_{23}C_6$ carbide precipitate [11] along the grain boundaries, accompanied by a zone on either side of the boundary depleted in coarse γ' particles. Higher magnifica-

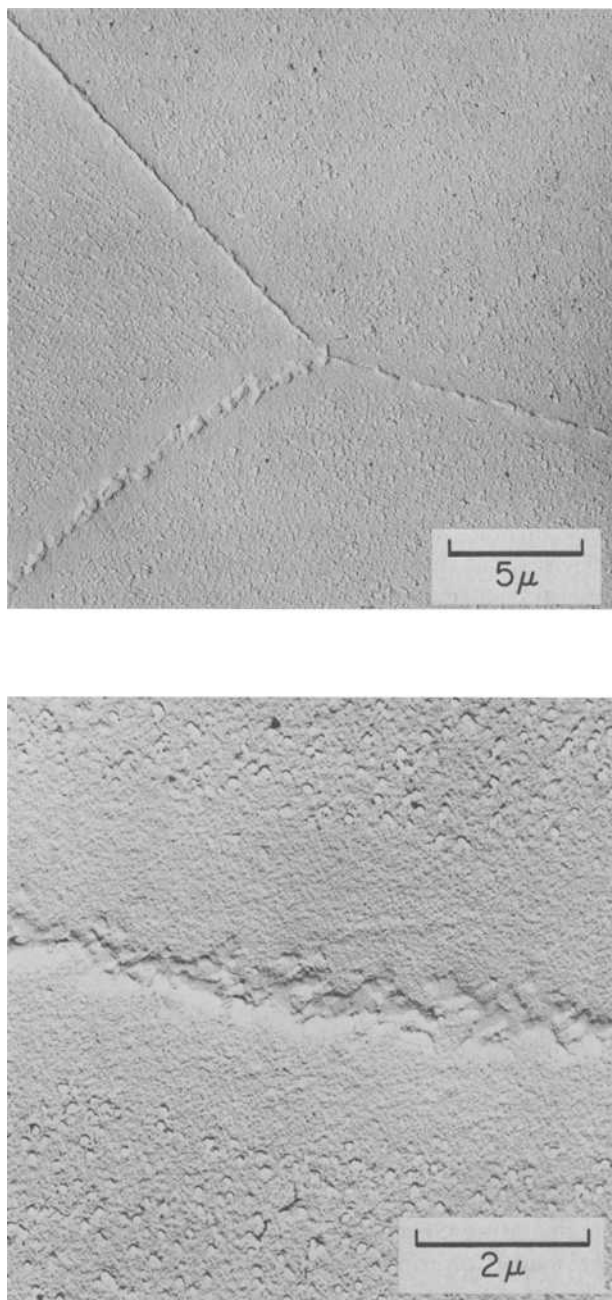


FIG. 2—Microstructure of the precipitation heat-treated Inconel X-750 illustrating (top) the duplex γ' precipitate morphology and $M_{23}C_6$ carbides decorating the grain boundary; (bottom) the presence of a zone on both sides of the grain boundary that is depleted in coarse γ' precipitates.

tion of the grain boundary region (Fig. 2, *bottom*) illustrates, however, that fine γ' particles had precipitated within this denuded zone. The net result is the presence of narrow zones along grain boundaries that are somewhat weaker, but in general more ductile, than the grain interior.

Fracture Toughness Testing

Fracture tests were performed on deeply cracked (fatigue precracked to $a/W > 0.65$ at a maximum stress intensity factor of $27.5 \text{ MPa } \sqrt{\text{m}}$) compact specimens with a thickness, B , of 1.47 cm and a width, W , of 2.93 cm [12]. All test specimens were oriented in the transverse (T-L) direction [12] and tested on an electrohydraulic closed-loop machine in stroke control (stroke rate of 0.25 mm/min) at room temperature, 427°C, 538°C, and 649°C. During the lower temperature fracture tests (24 to 538°C), displacements were measured on the load-line by a high-temperature displacement monitoring technique [13]. This method utilizes a rigid fixture, attached to the specimen directly over the loading pin holes, which translates the load-line displacement outside the loading clevises. The displacement is then monitored by two linear variable differential transformers (LVDT) attached to the outer portion of the rigid fixture and centered on the loading pin holes of the specimen. The output from each LVDT is summed in series to eliminate any misalignment phenomena. During each test, the load-line displacement was recorded continuously on an x-y recorder as a function of the load.

Since the maximum operating temperature for LVDTs is below 600°C, an alternative technique, similar to the test methods reported in Refs 14 and 15, was used to determine the load-line displacements at 649°C. This alternative test method utilizes the ram displacement, which measures both the specimen load-line displacement and an extraneous displacement associated with the compliance of the entire load train. The extraneous displacement was taken into account by loading an identical but unnotched test specimen at the desired test temperature and plotting a load-displacement calibration curve (see Fig. 3). At a given load, the extraneous displacement was subtracted from the original ram displacement, thereby yielding a corrected load versus load-line displacement plot as illustrated in Fig. 3. The corrected load-displacement curves were then used to compute values of J for the 649°C fracture tests.

In accordance with the recommended procedure for establishing J_{Ic} , outlined by Landes and Begley [16], the multispecimen R-curve technique was used to characterize the ductile fracture toughness behavior of Inconel X-750 at each test temperature. Specifically, a number of deeply cracked compact specimens were loaded to various displacements, producing different amounts of crack extension, Δa , and then unloaded. After unloading, each specimen was heat tinted, in order to discolor the crack-growth region, and subsequently broken open so that the amount of crack extension could be measured.

The value of J for each specimen was determined from the load versus load-line displacement curve by the following equation [17-19]

$$J = \frac{2A}{bB} \frac{(1 + \alpha)}{(1 + \alpha^2)} \quad (1)$$

where

A = area under load versus load-line displacement curve,

B = specimen thickness,

b = unbroken ligament size,

$\alpha = [(2a/b)^2 + 2(2a/b) + 2]^{1/2} - (2a/b + 1)$, and

a = crack length.

The R-curve was constructed by plotting values of J as a function of Δa ; J_{Ic} was then taken to be the value of J where a least-squares regression line through the crack-extension data points intersected the stretch zone line associated with crack-tip blunting prior to actual tearing of the material

$$J = 2\sigma_f (\Delta a) \quad (2)$$

where σ_f = flow strength = $1/2 (\sigma_{ys} + \sigma_{uts})$.

Finally, the specimen size requirement proposed by Landes and Begley [16] to ensure a valid J_{Ic} fracture toughness value

$$a, B, b \geq 25 \left(\frac{J}{\sigma_f} \right) \quad (3)$$

was met during the current investigation.

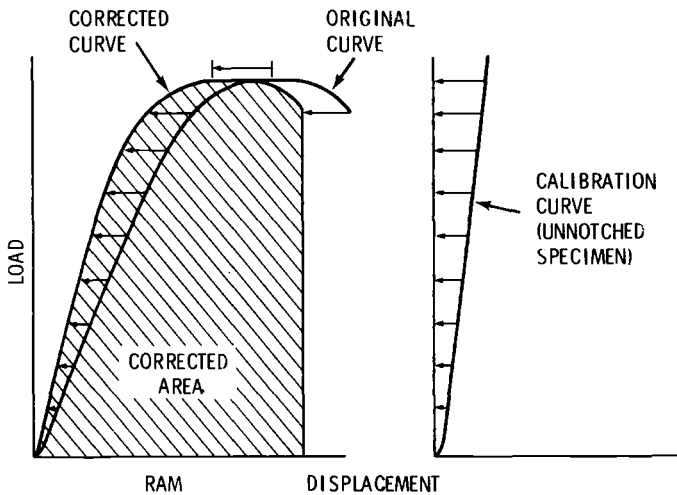


FIG. 3—Schematic illustration of the corrected load versus load-line displacement curve [14, 15] for the 649°C fracture toughness tests.

Metallographic and Fractographic Techniques

Metallographic specimens were prepared for optical microscopic examination by using standard metallographic techniques. To reveal the general microstructure, the specimens were electrolytically etched in an oxalic acid solution. The fracture surface appearance of this nickel-base alloy was characterized by fractographic examination of appropriate fracture toughness specimens on a scanning electron microscope operated at an accelerating potential of 15 kV.

Presentation and Discussion of Results

Individual R-curves for Inconel X-750 at room temperature, 427°C, 538°C, and 649°C are illustrated in Figs. 4 and 5. These results, summarized in Fig. 6, reveal that the J_{Ic} fracture toughness response of this superalloy increased slightly with a temperature increase from 24 to 427°C (increasing from 102 to

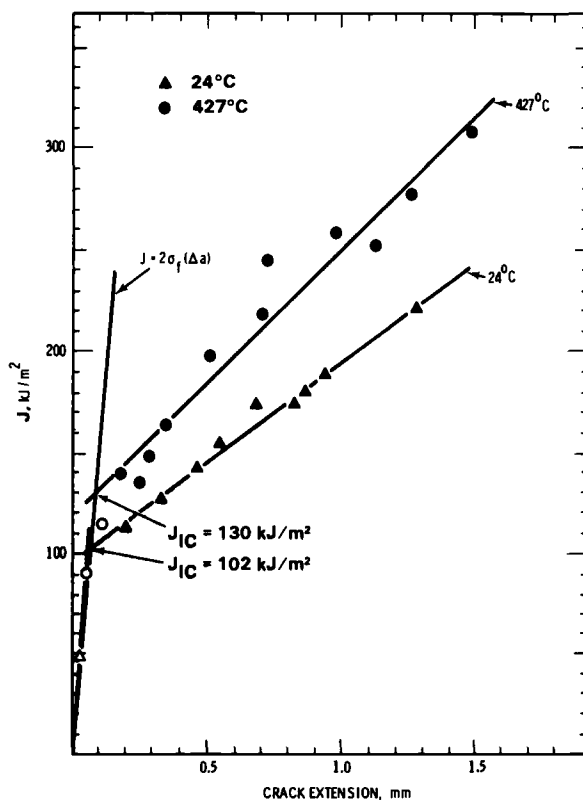


FIG. 4—R-curves for Inconel X-750 tested at room temperature and at 427°C. The open data points represent stretch zone formation.

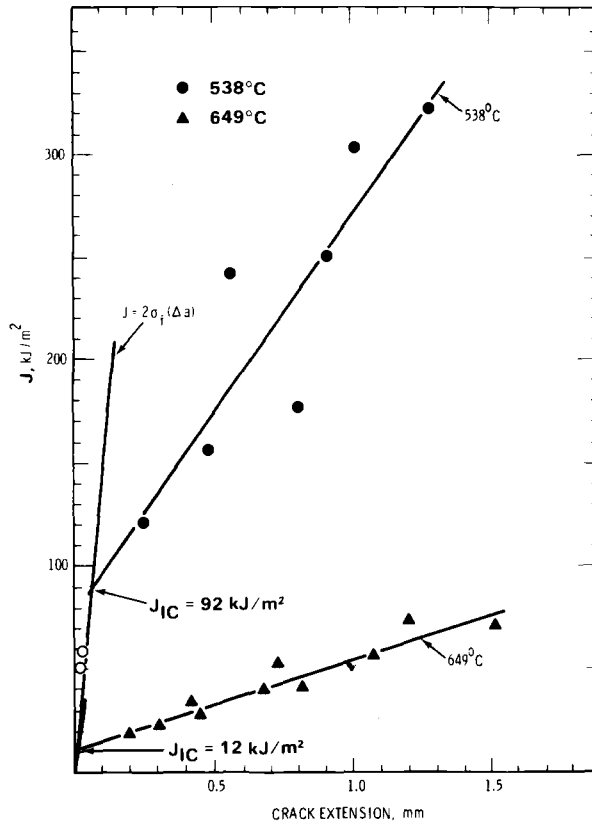


FIG. 5—*R*-curves for Inconel X-750 tested at 538°C and at 649°C. The open data points represent stretch zone formation.

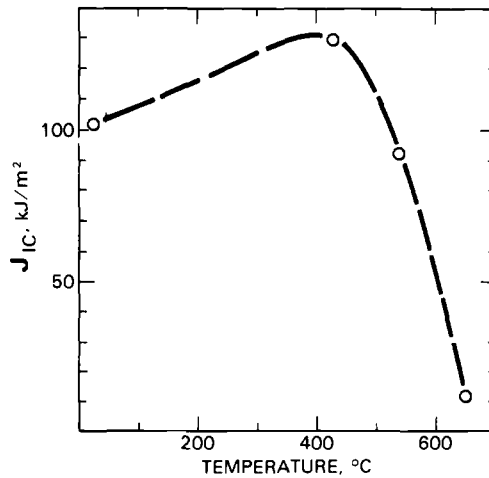


FIG. 6—Effect of temperature on the fracture toughness behavior of Inconel X-750.

130 kJ/m²) before decreasing to 92 kJ/m² at 538°C. At the highest test temperature (649°C), an order of magnitude reduction in toughness was observed as J_{Ic} dropped to 12 kJ/m². These trends in fracture toughness behavior were paralleled by variations in the ductility, as is shown in Fig. 1 (that is, the ductility levels reached a maximum at 427°C and then decreased markedly at 649°C). Furthermore, the fracture toughness and ductility responses were found to be associated with various fracture mechanisms operative over the test temperature range.

Fracture surfaces were examined in order to relate the J_{Ic} fracture toughness behavior to operative fracture mechanisms. At room temperature, crack propagation occurred primarily by an intergranular mechanism, illustrated by the metallographic fracture profile in Fig. 7. Electron fractographic examination revealed that the intergranular walls, illustrated in Fig. 8 (*top*) exhibited a rumpled appearance, which was attributed to a dimple-rupture network superimposed on the intergranular facets (Fig. 8, *bottom*). Similar intergranular dimple-rupture mechanisms, generally associated with coalescence of voids around grain-boundary precipitate particles, have been reported in the literature [3, 20–23].

In the Inconel X-750 alloy, this intergranular fracture mode is believed to result from microvoid coalescence along the grain-boundary denuded zone

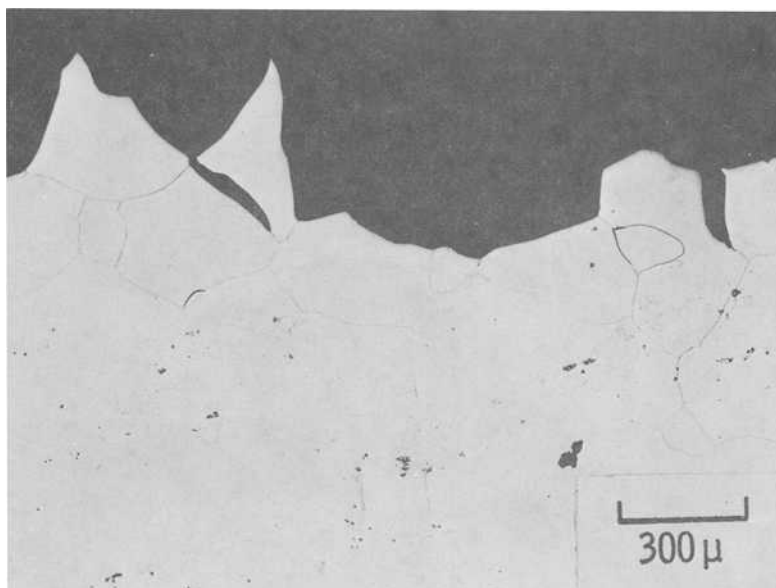


FIG. 7—Typical metallographic fracture profile at room temperature, revealing an intergranular fracture mode.

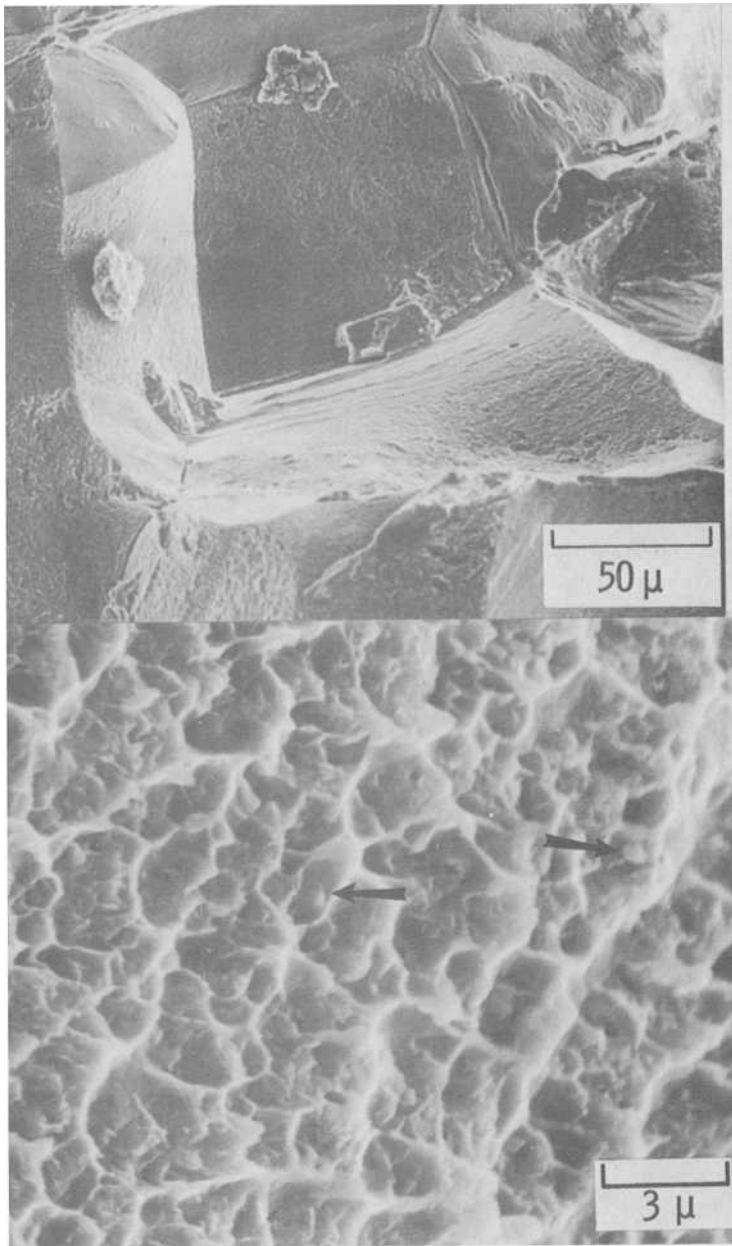


FIG. 8—Electron fractographs which reveal the fracture surface appearance at room temperature: (top) intergranular fracture morphology (note the rumpled appearance of the grain walls); (bottom) evidence of a dimple-rupture network superimposed on the intergranular walls.

(recall Fig. 2). Apparently, extensive plastic flow around the grain-boundary carbides leads to a stress concentration at the carbide-matrix interface, which results in a decohesion between the carbide particles and the nickel matrix. Figure 8 (*bottom*) illustrates the particles, believed to be $M_{23}C_6$ carbides, that initiated the microvoids (see the arrows). Subsequent growth and coalescence of these voids within the denuded region result in the observed intergranular dimple-rupture mechanism. Hence, the grain-boundary denuded zones provide a continuous path along which fracture occurs at room temperature.

At elevated temperatures, an intergranular-to-transgranular fracture mode transition was detected. As shown in Fig. 9 (*top*), the typical 427°C fracture surface consisted of relatively small intergranular dimple-rupture regions (denoted by the letter I) surrounded by a transgranular fracture morphology consisting of poorly defined dimple rupture and a rather flat, faceted appearance (Fig. 9, *bottom*) similar to the faceted fracture modes reported by Oblak et al [24] and Menon et al [25] in other nickel-base superalloys. The slight increase in ductility and fracture toughness properties observed under intermediate temperature conditions (Figs. 1 and 6) was attributed to the intergranular-to-transgranular fracture mechanism transition. Recall that crack extension at room temperature involved an intergranular dimple-rupture mechanism that resulted from microvoid coalescence along the weakened grain-boundary depleted zones. This denuded grain-boundary network then provided a continuous path along which the crack propagated under room temperature conditions, thereby causing the lower fracture resistance. By contrast, at 427°C, increased evidence of a transgranular fracture mode (Fig. 10) accounts for the superior ductility and fracture toughness.

At progressively higher temperatures (538 and 649°C), the fracture surfaces exhibited a limited amount of intergranular fracture (Fig. 10, *top*), coupled with a highly faceted appearance (Figs. 10, *bottom*, and 11), particularly at 649°C. These pronounced facets were reminiscent of a channel-fracture mechanism [4, 26, 27], which results from a confinement of slip on a narrow band of planes in such a way that the material literally slips apart. In a similar fashion, the highly faceted morphology observed in Inconel X-750 at elevated temperatures was also attributed to failure along dislocation channels formed by extensive heterogeneous slip. More specifically, deformation of most γ' precipitation-hardened alloys involves a planar-slip mechanism in which paired dislocations eventually shear both the nickel matrix and the γ' particles [24, 28–32]. After the initial dislocations cut through a region clearing a channel of sheared γ' particles, the effectiveness of these sheared precipitates as dislocation barriers is diminished and extensive dislocation activity is subsequently “channeled” through these zones. Consequently, these narrow dislocation channels are severely weakened by extensive dislocation activity so that the material literally slips apart (as illustrated schematically in Fig. 12). This results in the formation of a stepped or faceted fracture appearance, which is termed *channel fracture* [26, 27].



FIG. 9—Fractographs of the 427°C fracture surface: (top) intergranular dimple-rupture network (I) surrounded by a faceted transgranular morphology; (bottom) evidence of a rather flat, faceted appearance, coupled with poorly defined dimples.

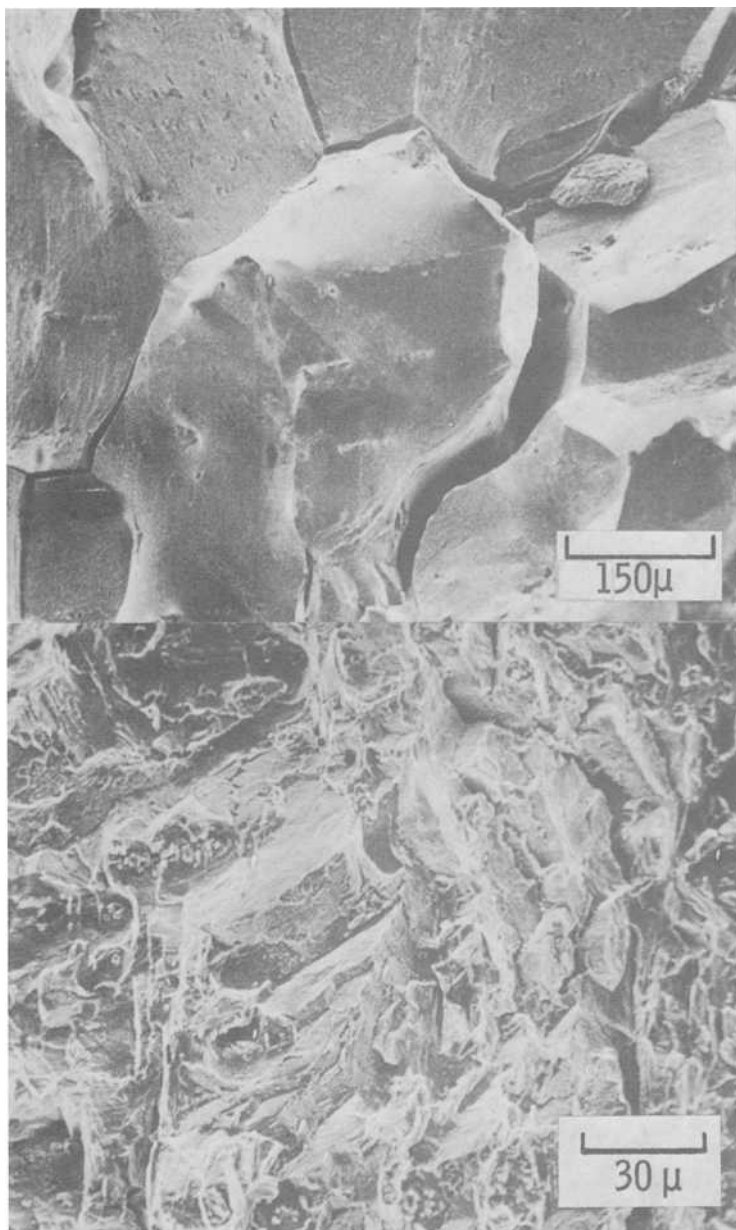


FIG. 10—Electron fractographs illustrating the fracture surface appearance at 538° C: (top) evidence of an intergranular dimple-rupture network; (bottom) faceted transgranular morphology, channel fracture.

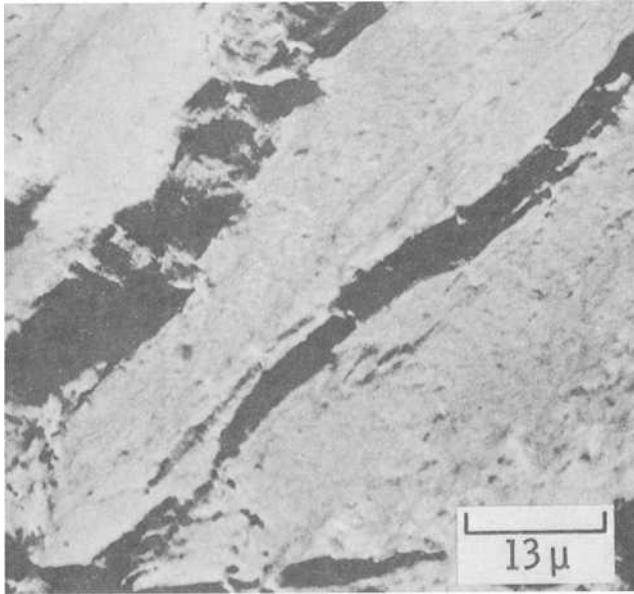


FIG. 11—Electron fractograph illustrating extensive channel fracture on the 649°C fracture surface.

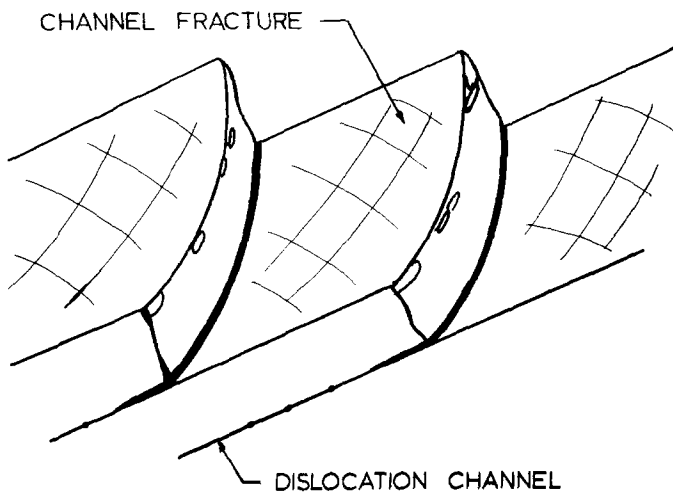


FIG. 12—Sketch of a model describing the channel fracture mechanism. The dislocations are concentrated along a narrow band of planes forming a dislocation channel. These zones are so weakened by extensive dislocation activity that material separation occurs along the dislocation channels.

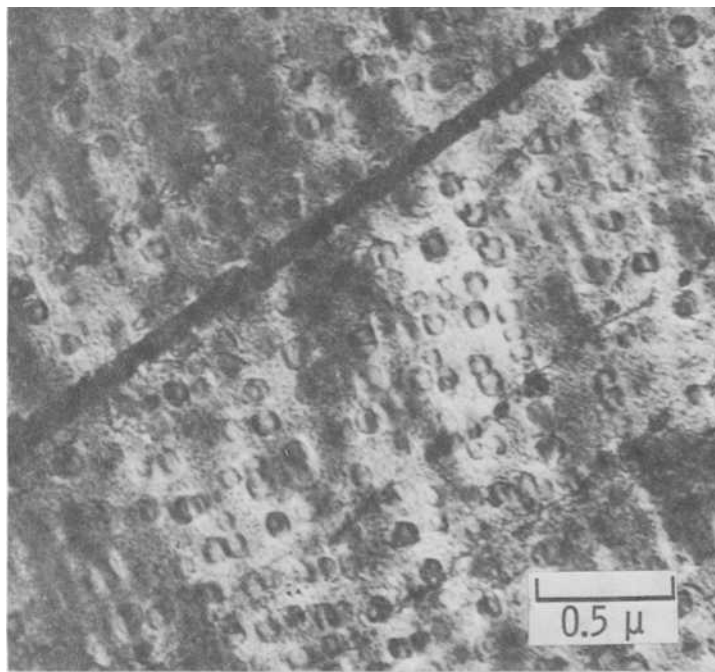


FIG. 13—A thin-foil electron photomicrograph of an Inconel X-750 tensile specimen tested at 649°C, which reveals well-defined dislocation channeling.

This proposed channel-fracture mechanism was supported by transmission electron microscopy (TEM) [4] of Inconel X-750 tensile specimens. At 649°C, this superalloy exhibited evidence of dislocation channeling along localized slip bands, while essentially no dislocation activity was observed in the surrounding matrix (as illustrated in Fig. 13). Severe weakening of these intense heterogeneous slip bands initiated localized separation, thereby resulting in the well-defined channel fracture. In addition, this localized failure along well-defined dislocation channels, coupled with the absence of a uniform dislocation substructure throughout the surrounding matrix, resulted in the poor ductility (Fig. 1) and dramatic reduction in J_{Ic} (Fig. 6) observed for Inconel X-750 at 649°C.

Conclusions

The room temperature and elevated temperature J_{Ic} fracture toughness response of Inconel X-750 has been characterized, and operative fracture mechanisms have been related to the J_{Ic} behavior. The following conclusions have been drawn:

1. The J_{Ic} fracture toughness of Inconel X-750 increased slightly at intermediate temperatures, but a marked reduction in J_{Ic} was observed at the higher test temperatures.
2. At room temperature, failure occurred by an intergranular dimple-rupture mechanism that was attributed to microvoid coalescence along the grain-boundary denuded zones.
3. The enhanced fracture resistance at intermediate temperatures resulted from an intergranular-to-transgranular fracture mechanism transition. The transgranular fracture morphology consisted of poorly defined dimples in addition to a flat, faceted appearance.
4. At the highest test temperature (649°C), intense dislocation channeling resulted in a very faceted fracture surface morphology. This localized failure along intense planar slip bands caused a dramatic reduction in J_{Ic} at 649°C.

Acknowledgments

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Microstructure and Fatigue

Correlation of Fractographic and Microstructural Features

REFERENCE: Steele, J. H., Jr., "Correlation of Fractographic and Microstructural Features," *Fractography and Materials Science*, ASTM STP 733, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 117-130.

ABSTRACT: One of the most important aspects of fractographic analysis is the relationship between the fracture surface microtopography and the underlying microstructure. This type of correlation requires comparison of the size, shape, and spatial arrangement of both fractographic and microstructural features and identification of the specific microstructural features that adjoin the fracture surface. Two examples of qualitative fractographic-microstructural analyses conducted with a scanning electron microscope in ferrous alloys are presented. The first example involves brittle cracking of oriented Fe-3Si sheet during cold rolling and the second low tensile ductility in a hot-rolled 0.47C-1.6Mn steel.

KEY WORDS: fractography, microstructure, scanning electron microscopy, mechanical twinning, cleavage, ferrous alloys, ferrite, pearlite, prior austenite grain size, microcracking, materials, materials science.

An important application of fractography in materials science is in the study of the role of microstructure in controlling fracture mechanisms. To develop an understanding of how microstructure controls fracture properties such as toughness or ductility, it is necessary to have correlations between fractographic and microstructural features. A variety of techniques have been applied to obtain these correlations, including the following: (1) metallographic sectioning to determine the microstructural features adjoining the fracture surface and thus evaluate the path of fracture in relation to the microstructure. This technique, which has been applied by a great many investigators, dates back to Martens in the 1870s, as indicated in Ref. 1;² (2) metallographic sectioning of specimens deformed to various levels of plastic strain less than fracture, as discussed in detail by Van Stone and Cox [2]; (3) direct etching of the fracture surface to reveal the underlying microstructural constituents, as was used effectively by Park and Bernstein [3] to identify pearlite colonies beneath cleavage facets in eutectoid steel; (4) quantitative measure-

¹Senior research metallurgist, Armco, Inc., Research Center, Middletown, Ohio 45043.

²The italic numbers in brackets refer to the list of references appended to this paper.

ments of microstructural parameters such as volume fraction, particle spacing, and grain size for correlation with fracture properties (the much cited paper by Edelson and Baldwin [4] on the effect of second-phase particles on ductility provides an excellent example of this approach); (5) the combined use of these techniques, as has been described by Almond et al [5], who used simultaneous viewing of fractographic and microstructural features along the edge of a section through the fracture surface to obtain correlations between river patterns and pearlite lamellae.

Application of the scanning electron microscope (SEM) for correlating fractographic and microstructural features has recently been described by Steele and Lentz [6]. In the two examples presented, these authors demonstrated that fractographic features can be more readily interpreted when microstructural observations at the same magnification as the fractographs are obtained with the SEM. In addition, nickel plating and metallographic sectioning of fracture surfaces were shown to provide sufficient edge retention to allow identification of the microscopic fracture path. As an example, crack propagation after hydrogen charging of a quenched and tempered steel was shown to occur along subboundaries. Details of the microstructure smaller than $1\text{ }\mu\text{m}$ (1000 nm) can thus be observed and related to the fracture mechanisms by using the SEM in conjunction with the various techniques cited in Refs 1-5.

This paper will present two additional examples of qualitative fractographic-microstructural correlations obtained using the SEM. These illustrate how the details of the microtopography of fracture surfaces can be interpreted from microstructural observations and how these interpretations can lead to a description of the failure mechanism. The examples represent problems encountered with fracture phenomena and properties rather than with failure analyses. The first problem involved the formation during cold rolling of brittle cracks several centimeters in length in oriented (cube-on-edge texture) Fe-3Si sheet. The second involved low tensile ductility in MT-6³ specimens of hot-rolled 0.47C-1.6Mn steel.

These two examples will be described separately in the following self-contained sections, each of which represents a different microstructure and failure mechanism.

Cracking of Oriented Fe-3Si Sheet During Cold Rolling

A fractographic-microstructural examination of the cause of transgranular cracking of oriented 0.42 mm (0.0165-in.) Fe-3Si sheet during cold rolling was conducted. The sheet material consisted of very large grains, ranging in size from 5 to 10 mm, which had a strong cube-on-edge texture (that is, $\langle 100 \rangle \parallel$ to rolling direction and $\{110\} \parallel$ to the sheet surface). The orientations of the indi-

³ Modified T-6 tensile specimen. American Society of Mechanical Engineers Boiler and Pressure Vessel Code, Section 9, Fig. Q6a.

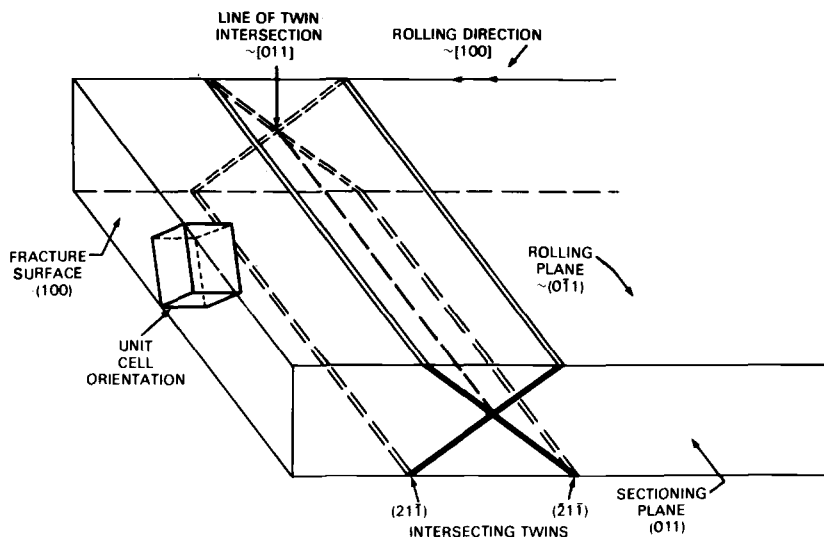


FIG. 1—A schematic diagram showing the relative orientations of the fracture surface, sectioning plane, rolling plane, and potential intersecting twins in an ideal cube-on-edge texture sheet material.

vidual grains were within approximately ± 5 deg from the ideal texture, which is indicated by the diagram in Fig. 1. A photograph of the appearance of the cracks and the grain structure is shown in Fig. 2a. Macroscopic examination suggested that the cracks had been formed by brittle crack propagation along planes that were essentially perpendicular to the rolling direction. In addition, parallel lines, whose orientations varied in different grains, could be observed on the surface of the rolled sheet. These crystallographic markings were also very nearly perpendicular to the rolling direction, as can be seen in Fig. 2b. An SEM photomicrograph of these surface markings is provided in Fig. 2c. As will be discussed, metallographic examination showed that these markings were a result of mechanical twins intersecting the surface of the sheet.

Fractographic Observations

Scanning electron microscope photomicrographs illustrating the fractographic features observed on the cracks are presented in Fig. 3. Tongues, which form during cleavage crack propagation [7], were observed on some grains, as is indicated in Fig. 3a. River patterns were present on the fracture surfaces of many grains. An example of these river patterns where a cleavage crack crosses a grain boundary is shown in Fig. 3b. Features marked as twins in Fig. 3a and d, which extend over large distances, were also quite common on the fracture surfaces. These features (which were confirmed to be a result of mechanical twins with metallographic sections) were oriented parallel to the edges of the tongues (when tongues were present), as can be observed in Fig.

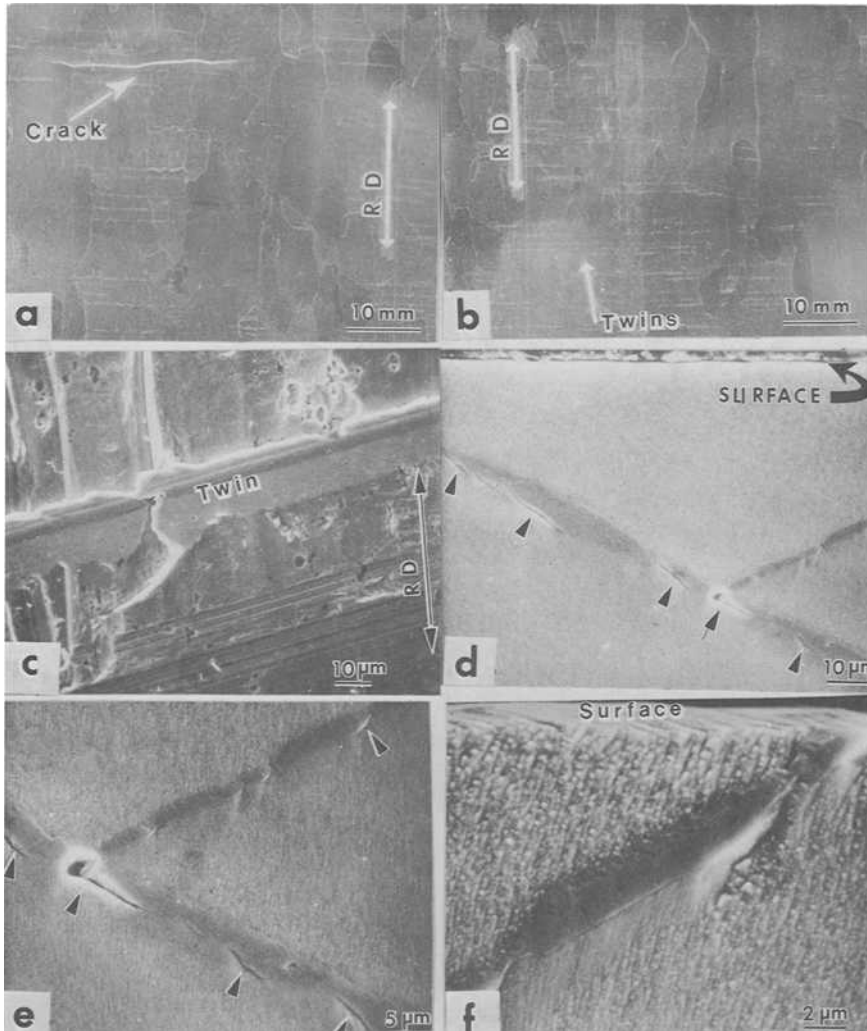


FIG. 2—Photomicrograph showing the following: (a) a photograph of crack and mechanical twin markings; (b) a photograph of mechanical twin markings on sheet; (c) an SEM photomicrograph of sheet surface twin marking; (d) an SEM photomicrograph of a polished and etched section with mechanical twin intersections; (e) an enlargement of d; (f) an SEM photomicrograph of a twin intersecting sheet surface.

3a. There also appears to be a tendency for cleavage cracks to exhibit a propagation direction outward from the twins, as indicated by the converging of the “steps” marked in Fig. 3d. Side cracking along the twin boundary can also be observed in Fig. 3d. The fracture surfaces, however, were confined completely to {100}-type cleavage planes and there was no evidence of propagation along twin boundaries, as has been observed by Hull [8].

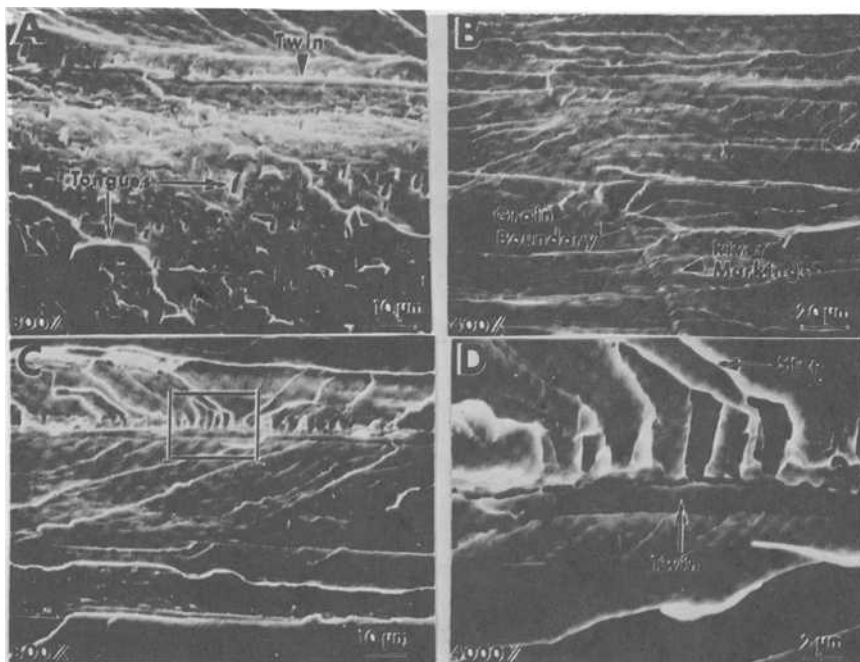


FIG. 3—Scanning electron microscope fractographs showing: (a) tongues and $\{112\}$ -type mechanical twins; (b) river markings at a low-angle grain boundary; (c) a $\{112\}$ mechanical twin intersection with fracture surface; (d) an enlargement of c with cleavage markings indicating crack propagation away from the twin.

Microstructural Observations

The presence of mechanical twins in the microstructure and adjacent to the fracture surfaces was confirmed by metallographic examination on longitudinal sections. Twins and twin intersections such as those shown in Fig. 2d, e, and f were observed. In addition, evidence of microcracking at twin intersections was found. The appearance of these cracks, indicated in Fig. 2d and e, is similar to that of the twin-matrix interface cracking reported by Hull [8] and the “crack embryo” shown by Priestner [9]. Microcracks also appear to be present along the twin boundaries marked by the arrows in Fig. 2d and e.

The fracture surface features and the microstructure are compared in Fig. 4. Twins intersecting the fracture surface can be seen, as well as microcracks along the twin boundaries. The presence of the grain boundary in Fig. 4b allows the topography along the bottom of Fig. 4a to be interpreted as resulting from cleavage through a different grain.

The angles between the sheet surface and the mechanical twins were measured for several twins and found to be approximately 27 deg. Based upon the ideal crystallographic texture, this angle, which is schematically illustrated in

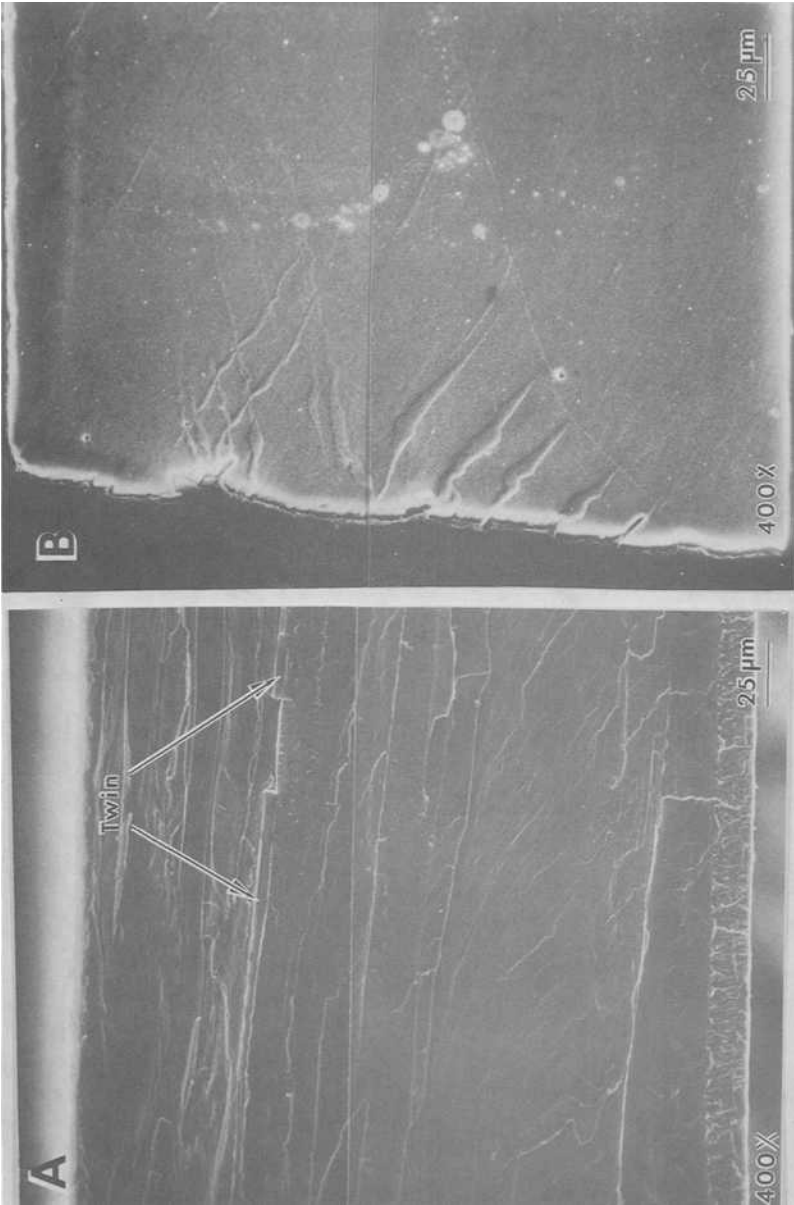


FIG. 4—Comparison of fractographic features with mechanical twins found adjacent to the fracture surface: (a) SEM fractograph; (b) SEM photomicrograph showing mechanical twins adjacent to the fracture surface.

Fig. 1, should be 35.26 deg. This suggests that the sheet may have undergone a thickness reduction of approximately 16 percent after the twin formation in order to provide the deformation required to change the twin angles from 35 to 27 deg. The flattened appearance of the lines of intersection of the twins with the sheet surface also indicate that some rolling deformation occurred after the twin formation.

Discussion of Failure Mechanism

The observations indicate that brittle cleavage cracks are initiated at microcracks present along mechanical twin intersections and boundaries. Twins intersecting the {100} cleavage facets along $\langle 110 \rangle$ directions were present on all the crack surfaces examined. In addition, mechanical twins and twin intersections were observed within the microstructure both adjoining and independent of the fracture surfaces. Microcracks along twin interfaces and at twin intersections were shown to be present within the microstructure in regions where cleavage had not been initiated.

Observations of the angular arrangements of twins and the morphology of their intersections with the sheet surface suggest that twinning occurred during the compressive deformation associated with thickness reduction. This suggests that sheet tension in the rolling direction was sufficient to induce cleavage propagation from the microcracks.

Conclusions

1. Cracks formed in oriented Fe-3Si sheet during cold rolling were found to be caused by microcracks formed at the boundaries and intersections of mechanical twins.
2. Mechanical twinning appears to have occurred during compressive deformation of the sheet, with cleavage crack formation initiated subsequently by tension applied in the rolling direction.

Low-Ductility Tensile Failures in an as-Hot-Rolled High-Carbon (0.47 percent)-Manganese (1.6 percent) Steel

As-hot-rolled steel exhibiting low tensile ductility was examined to evaluate the role of microstructure in the fracture process. A chemical analysis typical for the alloy with tensile properties is listed in Tables 1 and 2. The microstructure consisted of thin proeutectoid ferrite layers formed along prior austenite grain boundaries and pearlite colonies that filled the interior of the prior austenite grains (see Fig. 5b). A very coarse prior austenite grain size ($\sim$ ASTM No. 1), which was developed during processing of the ingots into billets, was suspected of being a contributing factor to the low tensile ductility.

TABLE 1—*Chemical analysis of an as-hot-rolled high-carbon (0.47 percent)–magnesium (1.6 percent) steel.*

Elements	Percent
C	0.47
Mn	1.56
Si	0.19
S	0.026
P	0.009
Cu	0.014
Mo	0.008
Cr	0.005
N	0.0049
O	0.0013
Al	
total	0.010
solution	0.008
Ca	0.003
Sn	0.004
Ni	0.02
V	0.006
Ti	0.008

Fractographic Observations

Figure 5 illustrates the appearance of the fracture surface on the MT-6 tensile specimens that exhibited low ductility. In addition to the small shear lip around the outer edge, two distinct zones are observed: (1) a central region roughly circular, having the dull gray appearance of ductile failure; and (2) a speckled (sparkling to the eye) region, which appeared to have undergone brittle fracture. Figure 6 shows SEM photomicrographs of the central region, which exhibits planar arrays of microvoids, as indicated by the enlargement presented as Fig. 6*b*. The SEM photomicrographs included as Fig. 6*c* and *d* illustrate the cleavage characteristics present in the brittle region.

Microstructural Observations

Nickel plating of the fracture surface allowed metallographic sections to be prepared with sufficient edge retention for SEM identification of the microstructural features adjacent to the fracture surface. In the central region, the path of fracture was identified as occurring through the proeutectoid ferrite layers, indicated in Fig. 7*a* and *b*. Some manganous sulfide (MnS) inclusions, with orientations essentially perpendicular to the fracture surface, were observed. These inclusions did not appear to affect the fracture process. The

TABLE 2—Tensile data for the alloy analyzed in Table 1.

0.5% yield strength	468 MPa (67.8 ksi)
Ultimate tensile strength	779 MPa (112.9 ksi)
Ductility (elongation)	15%

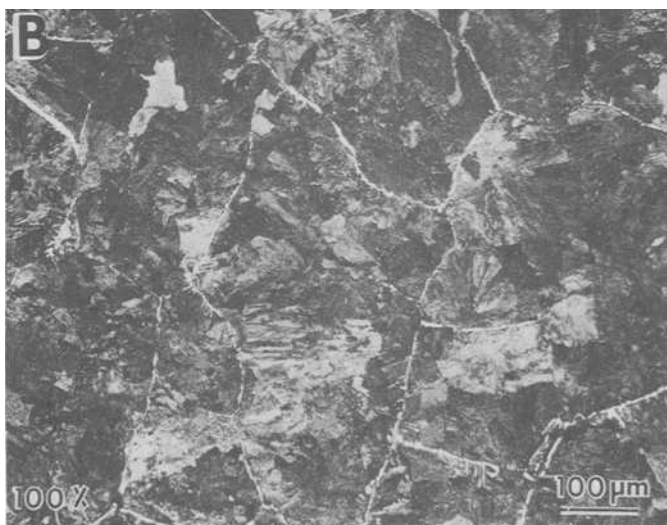
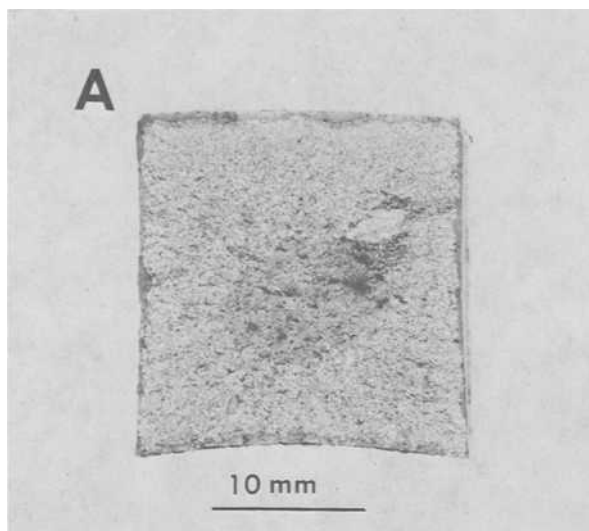


FIG. 5—(a) Photograph of a fractured MT-6 tensile specimen; (b) an optical photomicrograph showing a typical microstructure, consisting of thin proeutectoid ferrite layers along prior austenite grain boundaries with pearlite.

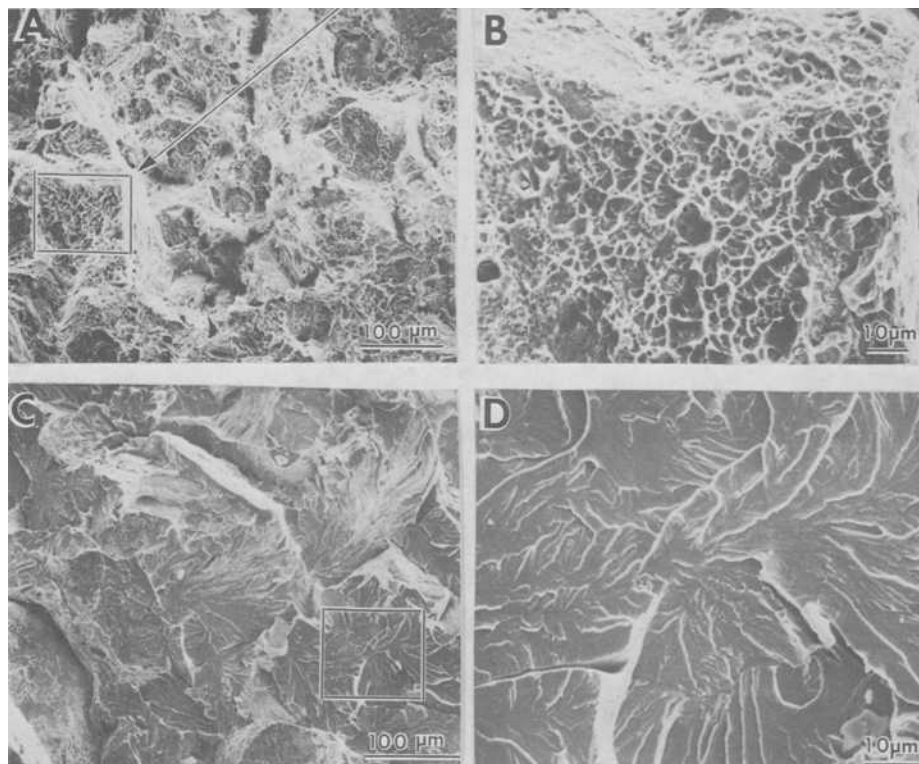


FIG. 6—Scanning electron microscope fractographs of an MT-6 tensile specimen: (a) the central region; (b) an enlargement of the area marked in a showing planar arrays of microvoids; (c) the outer region; (d) enlargement of the area marked in c showing cleavage markings.

fracture path in the brittle region is through the pearlite colonies, as shown in Fig. 7c and d.

Examination of the microstructure below the central region revealed the presence of cracks and microvoids along the proeutectoid ferrite layers. These features are illustrated in Fig. 8. Individual microvoids shown at high magnification can be observed in Fig. 8a. Linking up of the microvoids produces cracks along the thin ferrite layers, such as those indicated in Fig. 8b and c. These cracks tend to occur more frequently along ferrite layers inclined at angles of nearly 45 deg to the tensile axis. Linking up of these cracks produces larger voids, such as those illustrated in Fig. 8d.

Some deformation within the pearlite colonies was observed in the form of kinking [10]. This phenomenon, which appears to be the result of deformation banding within a pearlite colony, is illustrated in Fig. 9c. Microcracking was also observed to occur to a small degree along some of the kinks and also within some colonies, as shown in Fig. 9a and b. A ductile fracture surface

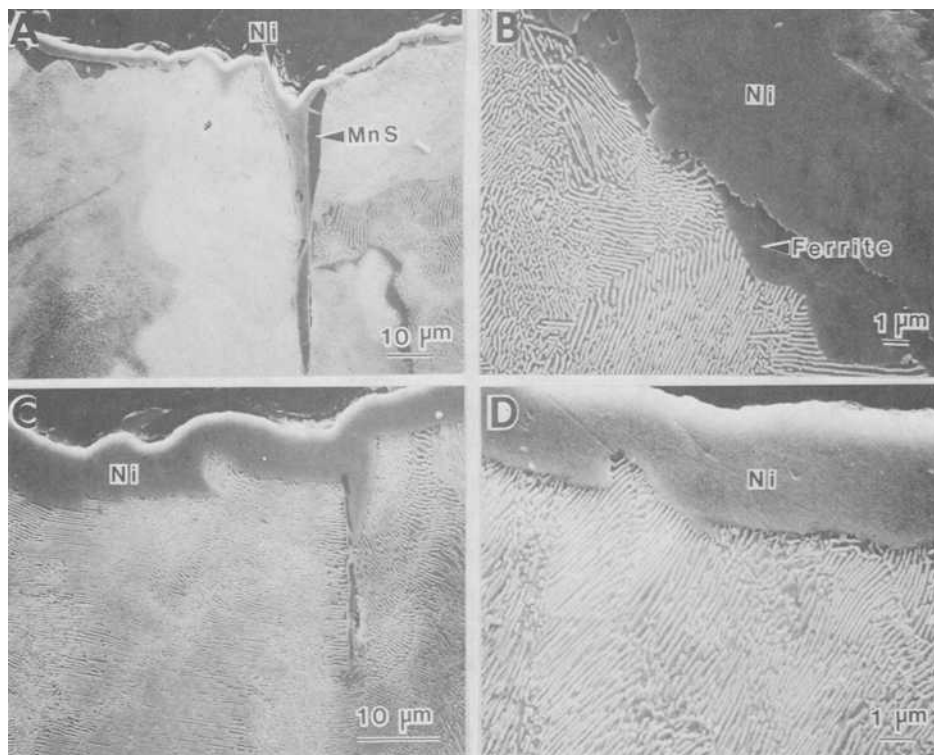


FIG. 7—Scanning electron photomicrographs illustrating the microstructure adjacent to the fracture surface of an MT-6 specimen: (a) the central region at low magnification; (b) the central region showing proeutectoid ferrite along the fracture path; (c) the outer region at low magnification; (d) the outer region showing brittle crack propagation across pearlite colonies.

produced on a sheet Charpy specimen cut from the microstructure showed fractographic features that appeared to have resulted from microcracking within the pearlite colonies. Absence of these fractographic features on the MT-6 specimens indicates that pearlite deformation and microcracking does not play a major role in the low-ductility fractures.

Discussion of the Failure Mechanism

The observations indicate that localized plastic flow occurs within the proeutectoid ferrite layers leading to the formation of planar arrays of microvoids along regions coincident with the prior austenite grain-boundary facets. Growth and linking of these planar ductile cracks occurs primarily within the central portion of the MT-6 specimens. This process eventually leads to the formation of a crack that exceeds the critical size for brittle (that is, cleavage) propagation. At this stage, the specimen will fail with very little additional

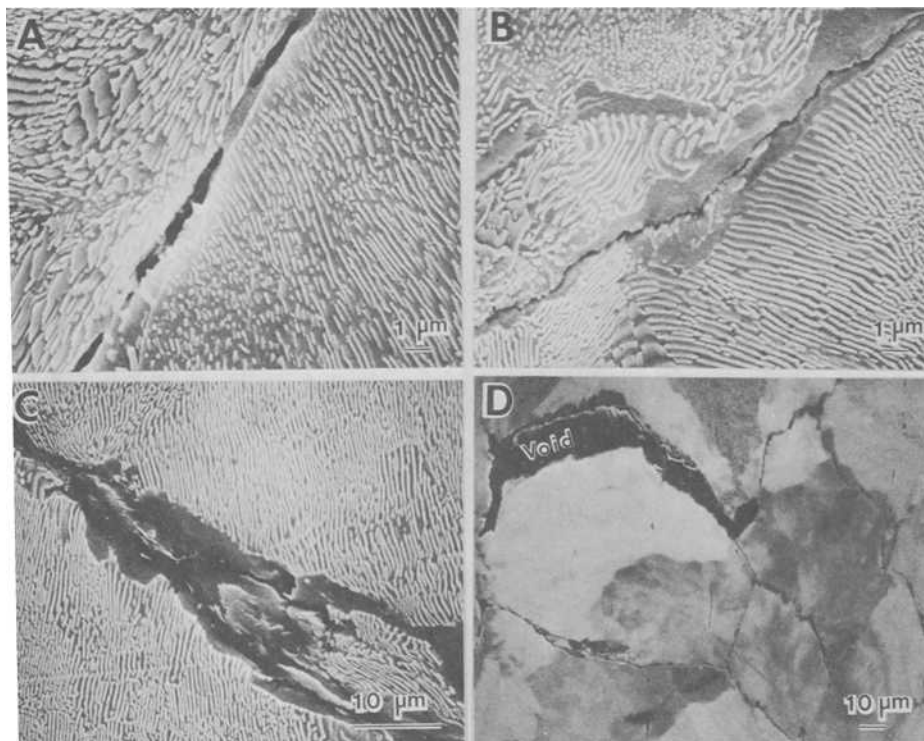


FIG. 8—Scanning electron photomicrographs showing microvoid formation, cracking, and large voids within the microstructure below the central region: (a) small microvoids in a proeutectoid ferrite layer; (b) a crack through the proeutectoid ferrite layer; (c) a crack along the proeutectoid ferrite layer; (d) a large void opened up along the ferrite layer.

bulk elongation. Thus, the tensile ductility will be controlled primarily by the amount of localized plastic flow.

Failure resulting from localized slip on narrow soft layers at grain boundaries, such as that demonstrated in the steel microstructure, has recently been described by Hornbogen [11] and by Hornbogen and Gräf [12]. This mechanism occurs because ferrite yield strengths are approximately one fourth that for pearlite, and the ferrite is distributed as a thin layer at the prior austenite grain boundaries. The effect of grain size was shown by Hornbogen and Gräf [12] to be

$$\epsilon_f = \epsilon_{f_i} (t/D)$$

where ϵ_f = the bulk true fracture strain; ϵ_{f_i} = the localized true fracture strain in the soft zone; and t/D is the zone-thickness to grain-size ratio. Thus, by halving the grain size (ASTM No. 1 to No. 3), the bulk elongation should be changed from 0.15 to 0.30, thereby eliminating the problem. It is also likely

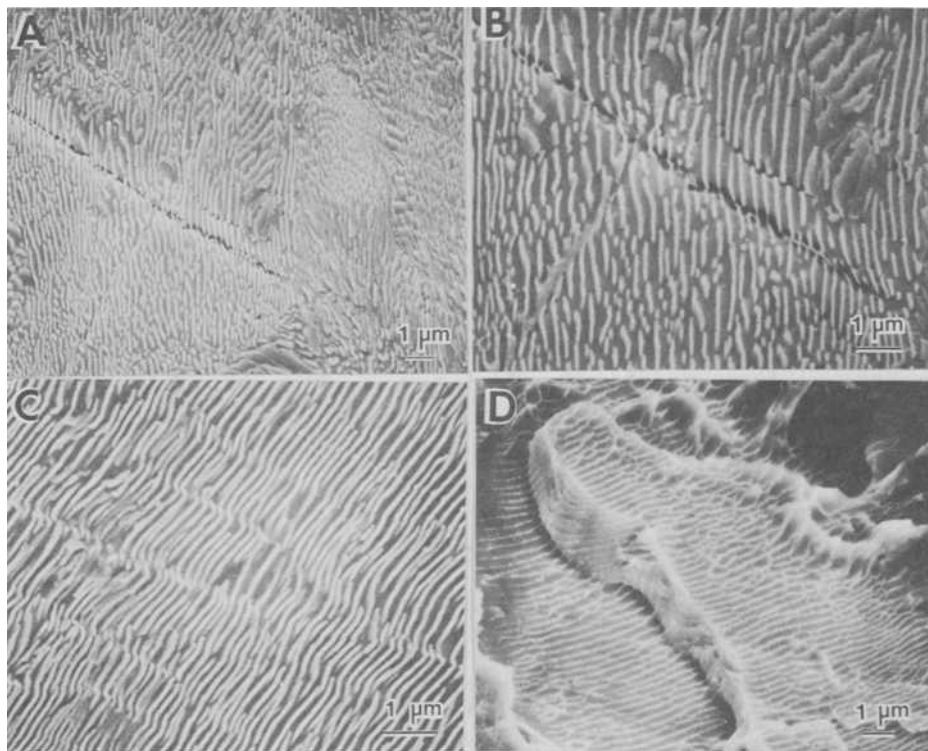


FIG. 9—Scanning electron photomicrographs (a, b, and c) and a fractograph (d) illustrating deformation effects observed in the pearlite below the central region: (a and b) a crack within the pearlite colony; (c) kinking within the pearlite colony; (d) a fractograph of a sheet Charpy-type specimen cut from the hot-rolled material.

that this mechanism would not control the fracture process, since the typical bulk ductile rupture process leads to elongations of approximately 0.3 or less.

Conclusions

1. The failure mechanism for the hot-rolled tensile specimens has been described in terms of localized plastic flow along proeutectoid ferrite layers, leading to the formation in the central region of a crack that exceeds the critical size for cleavage propagation.

2. Decreasing the prior austenite grain size by a factor of 2 (ASTM No. 1 to No. 3) would eliminate the low-ductility problem.

Summary

Fractographic-microstructural correlations have been presented for two examples that involve different microstructures and failure mechanisms in

steels. The importance of these correlations in understanding the role of microstructure in the failure mechanisms was illustrated in the discussion for each example. Scanning electron microscope examination, particularly of the microstructure adjoining the fracture surface, was shown to be extremely valuable since it allows comparisons of microstructural features and fractographic features at equivalent magnifications. The SEM was also shown to be quite useful in the identification of microvoids and microcracks within the microstructure.

Acknowledgment

The assistance of Andy Golembiewski in the metallographic preparation of specimens is gratefully acknowledged.

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Fractography of Laser Welds

REFERENCE: Metzbower, E. A. and Moon, D. W., "Fractography of Laser Welds," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 131–149.

ABSTRACT: A series of high-strength alloys has been laser-beam welded in thicknesses of up to 1.27 cm (0.5 in.). These welds in steels, a titanium alloy, and an aluminum alloy have been tested for their mechanical properties, fracture toughnesses, and hardness. The microstructures of these weldments have been determined in the base plate, heat-affected zone, and fusion area. The fractographic features for each alloy have been determined and correlated with the weldment's properties and structures.

KEY WORDS: fractography, microstructure, high-strength steels, aluminum alloy, titanium alloy, laser-beam welds, fracture toughness, mechanical properties, materials, materials science

New and innovative techniques for joining materials, especially metals, are in constant demand in the fabricating community. The ability to join metals is only one area of concern in the overall development of a process. The properties of the weldments are equally important. This paper will discuss the mechanical properties, fracture toughnesses, hardness values, microstructures, and fractographic features of a series of laser-beam weldments. These weldments were fabricated in 1.27-cm (0.5-in.) thick plates of steels (HY-80 and HY-130), a titanium alloy, and an aluminum alloy. Correlations between the fractographic features, microstructures, and properties and the laser-beam welding process will be made.

Both the collimation and the coherency of the laser beam contribute to its ability $[I]^2$ to be focused to the size of a small spot with a high-energy density. When the laser impinges upon the surface of a metal, part of the energy is absorbed and part of it is reflected. The photon energy that is absorbed heats the surface, and melting occurs. If this energy is sufficiently high, "keyholing" occurs; that is, a small bore of metal is melted through the thickness of the plate. A column of vapor is produced in this hole. This vapor column is surrounded by a liquid pool, and, as this column moves along the plate, the metal on the

¹Supervisory metallurgist and metallurgist, respectively, Naval Research Laboratory, Washington, D.C. 20375.

²The italic numbers in brackets refer to the list of references appended to this paper.

advancing side of the hole, throughout its depth, is melted. The molten metal flows along the sides of the hole and solidifies at the back. The vapor column is stabilized by means of a balance between the energy density of the laser beam and the translational speed of the work piece.

Laser-beam welds have depth-to-width ratios typically greater than 4 to 1. Narrow heat-affected zones (HAZ) and minimal distortion are the results of the low-heat input per unit length of weldment. Another result of the low-heat input is the very fast cooling rate of the fusion zone. Fusion zone purification [2], resulting from a decrease in the inclusion size and a modification of the number and shape of inclusions, is another consequence of laser-beam welding.

Process and Properties

The nominal compositions of the alloys used in this study are given in Table 1. Their mechanical properties are listed in Table 2. These values are average properties or minimum requirements, as given in the *Welding Handbook* [3]. The values for the fracture toughnesses as measured by the dynamic tear test are from ratio analysis diagrams [4] (plots of fracture toughness versus yield strength) and extrapolated to 1.27-cm (0.5-in.) section sizes. The laser-beam welding parameters used to fabricate these 1.27-cm (0.5-in.) thick plates are given in Table 3. The mechanical properties of these weldments are listed in Table 4. The mechanical properties of each weldment were measured using a standard ASTM round specimen 6.4 mm (0.252 in.) in diameter. The weld was transverse to the loading direction. A 25-mm (1.0-in.) gage length was used to determine the percentage of elongation. The fracture toughness was measured on a subsized, 12-mm (0.5-in.) thick, dynamic tear (DT) specimen that was notched in the weld. The fracture toughness was measured at room temperature. In order to be concise and complete, each of the laser-beam weldments will be discussed in turn.

HY-80 Laser Welds

A hardness traverse across the HAZ and the fusion zone of the HY-80 laser-beam weldment is shown in Fig. 1. These hardness values were obtained on a polished and etched structure using a diamond pyramid microhardness indenter with a 300-g load. These hardness values were converted to HRC values. The quenched and tempered martensitic microstructure of the HY-80 base plate is shown in Fig. 2. In the fusion zone, the microstructure transforms to an untempered martensite with some bainite (Fig. 3). The HAZ consists predominantly of untempered martensite and a little bainite. Since the fusion zone and the HAZ are considerably harder than the base plate, the tensile specimen becomes a composite specimen with a hard central area (the fusion zone and HAZ) and softer areas (base plate) on both sides. Yielding and eventually fracture occur in the softer region, that is, the base plate. The DT speci-

TABLE 1—*Nominal composition of base plates.*

Alloy	Fe	C	Cr	Mo	Ta	Ni	Al	Ti	Nb	Mg	Mn
HY-80	balance	0.18	1.68	0.45	...	3.0	...	...	...	...	0.30
HY-130	balance	0.11	0.55	0.55	...	5.0	...	...	...	...	0.8
Al 5456	...	...	0.12	...	...	...	balance	...	...	5.10	0.8
Ti 6211	...	...	...	0.8	1.0	...	6.0	balance	2.0	...	...

TABLE 2—*Nominal mechanical properties of base plates.*

Alloy	σ_{ys} , MPa (ksi)	σ_{ult} , MPa (ksi)	Elongation, percent	Fracture Toughness, N · m (ft · lb)
HY-80	580 (85)	725 (105)	14	470 (350)
HY-130	890 (130)	...	14	675 (500)
Al 5456	255 (37)	350 (51)	16	120 (90)
Ti 6211	790 (115)	860 (125)	15	200 (150)

TABLE 3—*Laser-beam welding parameters.*

Material	Power, kW	Speed, mm/s (in./min)	Heat Input, kJ/mm (kJ/in.)
HY-80	10.6	12.7 (30)	0.83 (21.2)
HY-130	11	12.7 (30)	0.87 (22.0)
Al 5456	11.0	14.8 (35)	0.74 (18.9)
Ti 6211	8.0	27.5 (65)	0.29 (7.4)

TABLE 4—*Mechanical properties of laser welds.*

Alloy	σ_{ys} , MPa (ksi)	σ_{ult} , MPa (ksi)	Elongation, percent	Reduction in Area, percent	Fracture Toughness, N · m (ft · lb)
HY-80	627 (91)	752 (109)	23.5	75.8	76 (56)
HY-130	952 (138)	987 (143)	18.5	68.4	707 (530)
Al 5456	193 (28)	289 (42)	9.2	16.9	116 (86)
Ti 6211	758 (110)	855 (124)	11.5	31.7	249 (184)

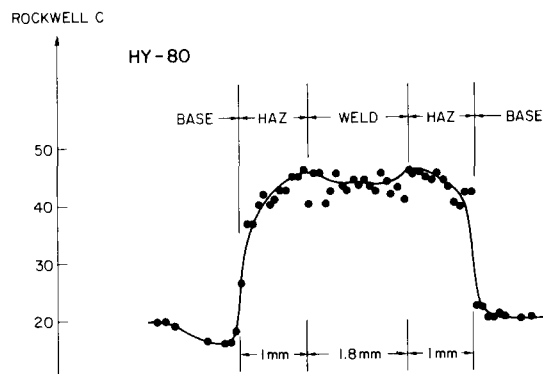


FIG. 1—Hardness traverse across the fusion zone of HY-80.

mens are notched in the weldment, and their values reflect the hard, brittle untempered martensitic structure of the fusion zone. Typically, HY-80 fracture toughness values are greater than $470 \text{ N} \cdot \text{m}$ ($350 \text{ ft} \cdot \text{lb}$), whereas HY-80 weldments generally exceed $470 \text{ N} \cdot \text{m}$ ($350 \text{ ft} \cdot \text{lb}$). A fractographic examination of the DT surface indicated a considerable amount of solidification cracking (Fig. 4) along the center line of the weldment. Energy dispersive X-ray

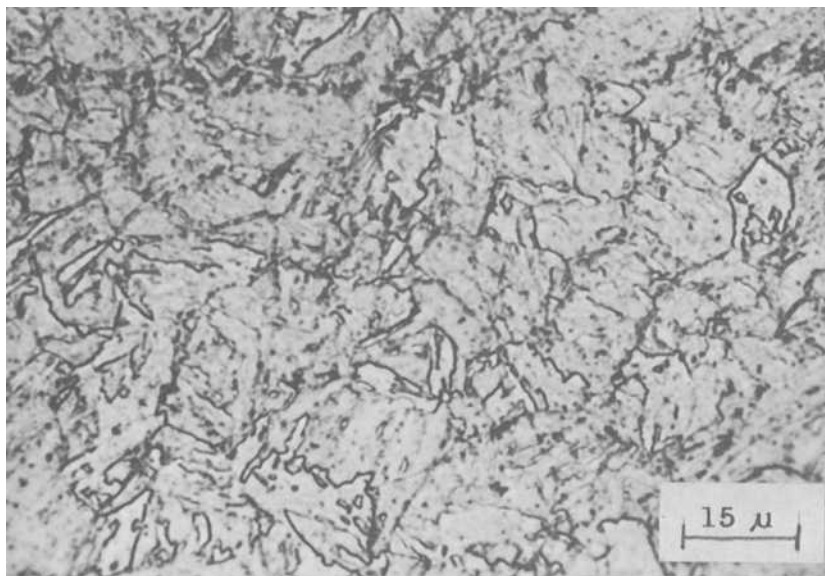


FIG. 2—Martensitic microstructure of HY-80 plate, etched in 1 percent nital. The hardness is 20.0 HRC.



FIG. 3—*Untempered martensite and bainite in the fusion zone of the HY-80 laser-beam weldment, etched in 1 percent nital. The hardness is 44.0 HRC.*

analysis of these areas indicated a considerable increase in silicon. There was also some evidence of scattered porosity on these fractures.

HY-130 Laser Welds

The hardness traverse across the HAZ and the fusion zone of the HY-130 laser-beam weld is shown in Fig. 5. The base plate microstructure consists of a quenched and tempered martensitic structure and is essentially the same as the microstructure of the HY-80 base plate. The fusion zone of the HY-130 laser-beam weldment consists of martensite and bainite (Fig. 6). Again, the microstructure of the HAZ is very similar to that of the fusion zone; however, the bainite that exists in the fine-grain region near the fusion boundary disappears as one approaches the coarse-grain region nearer the base plate, where it is replaced by ferrite. The room-temperature fracture toughness of these weldments is extremely good at 707 N · m (530 ft · lb) and compares favorably to the base plate toughness of 675 N · m (500 ft · lb). Fractographic evidence indicates that in the “pop-in” region separation occurs by microvoid coalescence (Fig. 7). After the crack leaves the pop-in region, which is in the fusion zone, it takes off at an angle of about 45 deg to the fusion zone and propagates through the base plate. This is an indication that the fusion zone is tougher than the base plate. The quenched and untempered structure observed in the

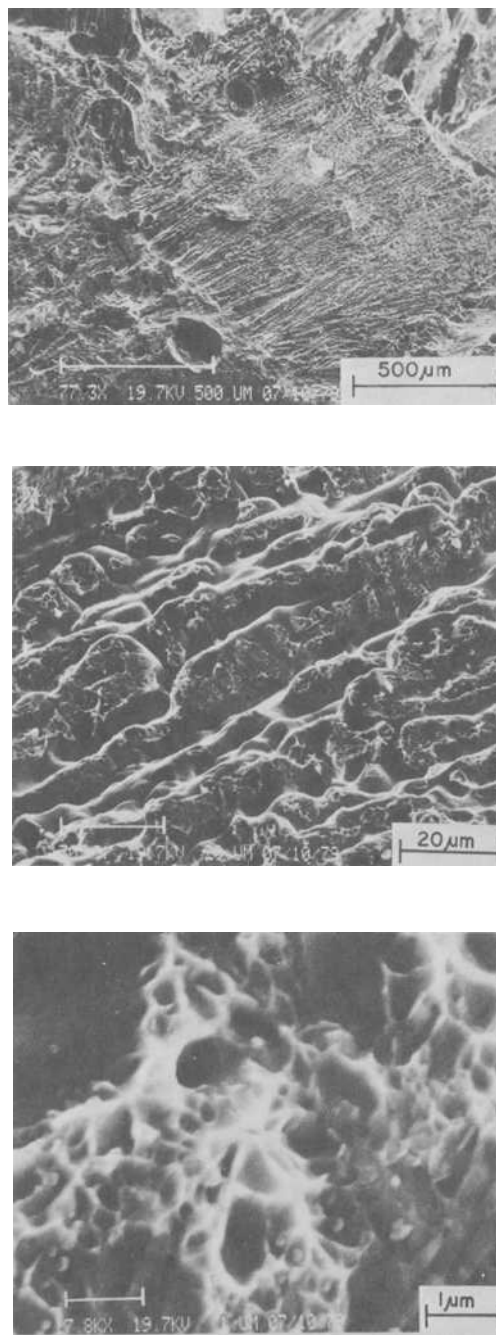


FIG. 4—Solidification cracking on the fracture surface of the HY-80 laser-beam weldment.

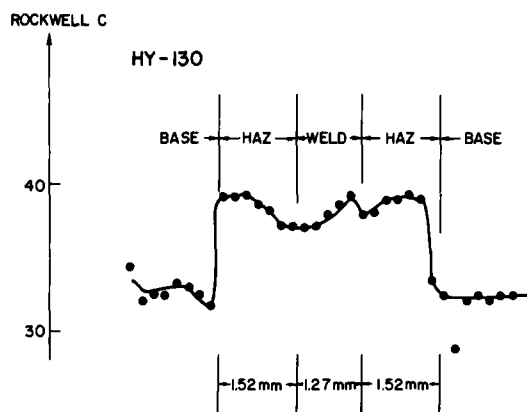


FIG. 5—Hardness traverse across the fusion zone of HY-130.

fusion zone is reflected by the lower temperature fracture toughness measurements, which are shown graphically in Fig. 8. In the laser-beam weldments tested at -18°C (0°F) or below, the fracture separation process both in the pop-in region and throughout the rest of the fracture was predominantly cleavage (Fig. 9). By tempering these weldments at 590°C (1100°F) and testing them at -18°C (0°F), the fracture toughness was increased to $625 \text{ N} \cdot \text{m}$ ($475 \text{ ft} \cdot \text{lb}$). The fracture separation process in these specimens was predominantly microvoid coalescence with some cleavage.

Aluminum 5456 Laser Welds

The hardness traverse across the HAZ and the fusion zone of a laser-beam weld of aluminum alloy 5456 is shown in Fig. 10. The softening that occurs in the HAZ and fusion zone is a typical result of applying heat to a strain-hardened aluminum alloy. A result of the high-energy density of the laser beam is the high temperature of the vapor in the keyhole of the weld. This high temperature causes the evaporation of the elements with low boiling points. In the case of aluminum alloy 5456, this results in a decrease in the magnesium content of the fusion zone by approximately 10 percent, as seen in Fig. 11, which is a trace of the concentration of magnesium and aluminum across the fusion zone of the weldment. A redistribution of the small second phases, which give the alloy its strength and toughness, also occurs as a result of the heat input. The microstructure of the base plate is shown in Fig. 12. The large black particles are magnesium silicide (Mg_2Si); and the gray particles are $(\text{Fe}, \text{Mn})\text{Al}_6$ complex. The elongated structure of the second phases in the base plate indicates the rolling direction and implies the strain-hardened characteristics of the alloy. In the fusion zone (Fig. 13), the particles are no longer elongated but have been melted and redistributed. This is seen in Fig. 14, which

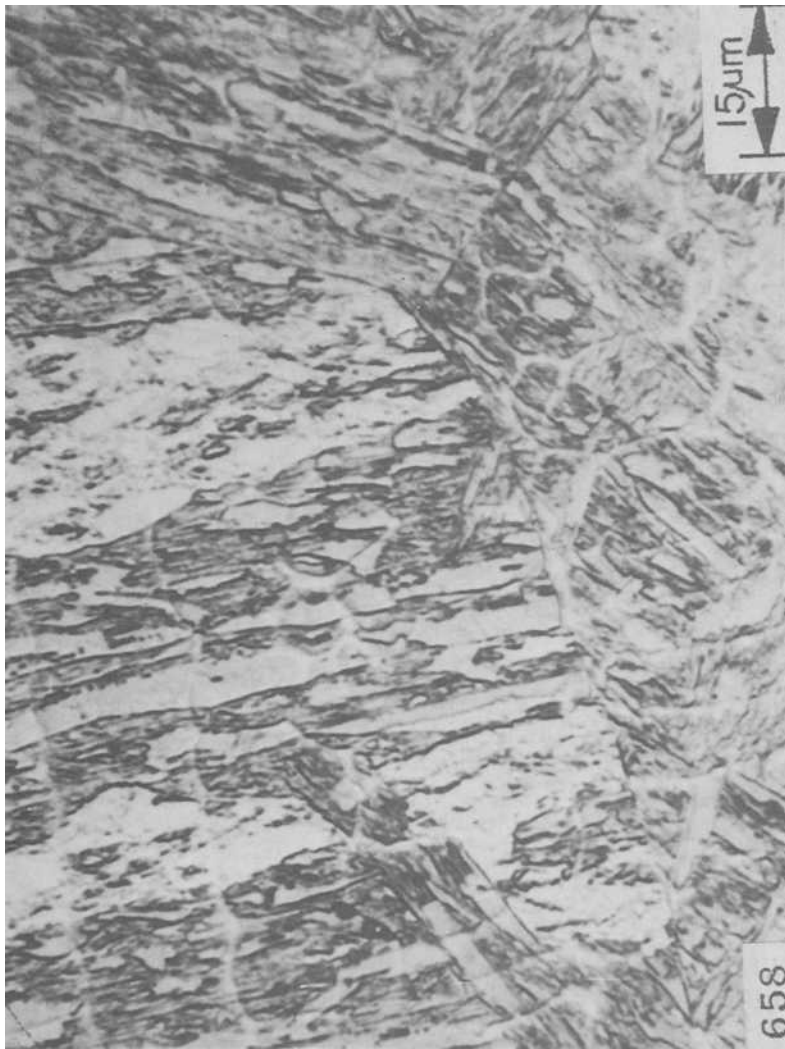


FIG. 6—Untempered martensite and bainite in the fusion zone of the HY-130 laser-beam weldment, etched in 1 percent nital. The hardness is 38.0 HRC.

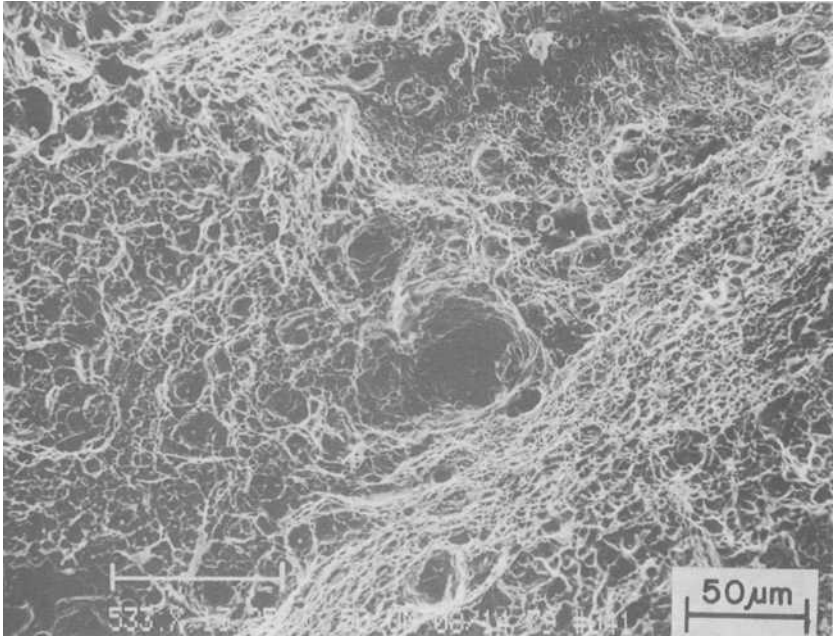


FIG. 7—Microvoid coalescence in "pop-in" region of the DT specimen of the HY-130 laser-beam weldment.

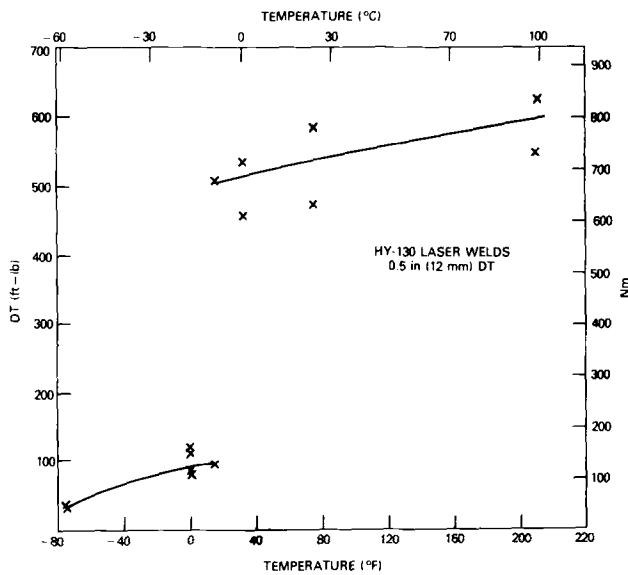


FIG. 8—Dynamic tear values measured as a function of temperature for laser-beam weldments of HY-130.

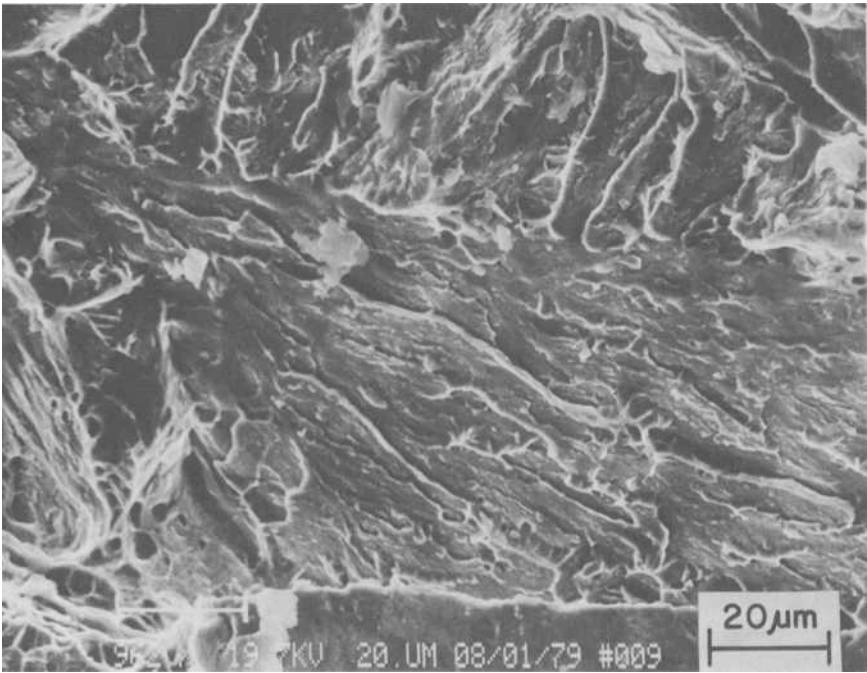


FIG. 9—Cleavage in the pop-in region of a DT specimen of HY-130 laser-beam weldment.

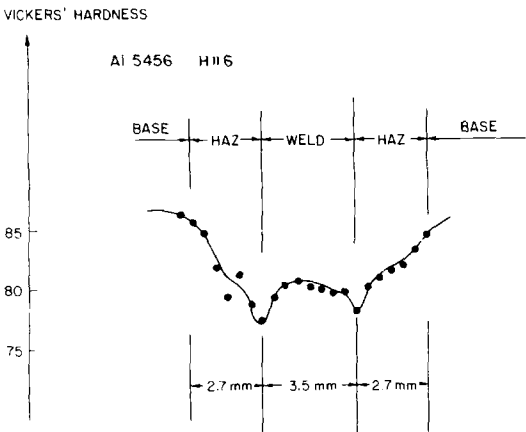


FIG. 10—Hardness traverse across the fusion zone of an aluminum alloy 5456 laser-beam weldment.

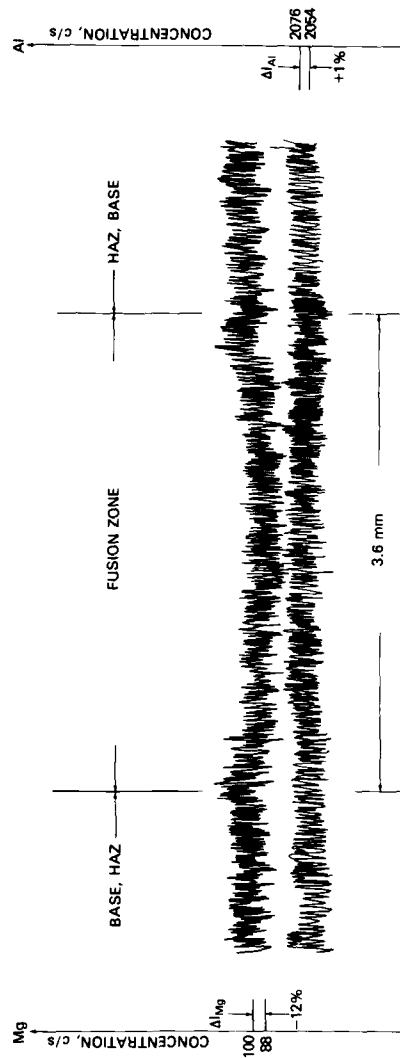


FIG. 11—Composition of magnesium/aluminum across the fusion zone of a laser-beam weldment of aluminum alloy 5456.

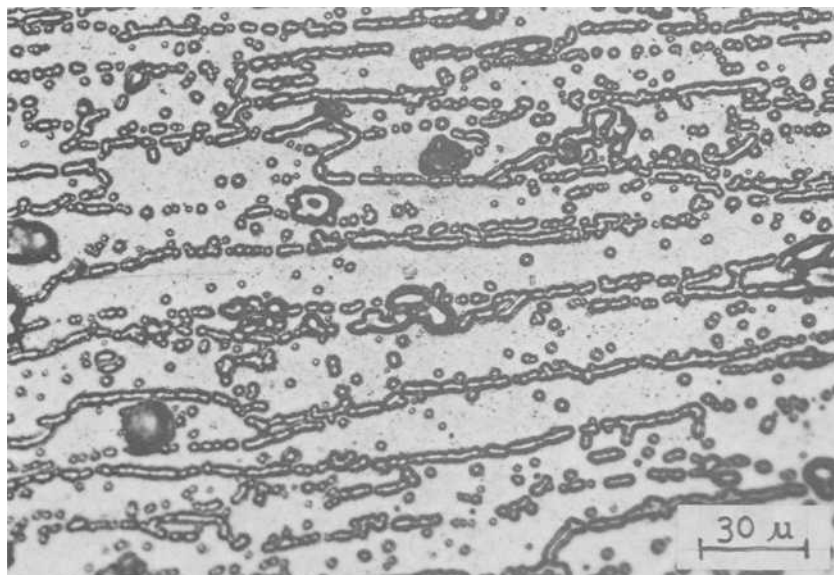


FIG. 12—Microstructure of aluminum alloy 5456 plate showing elongated α aluminum and a fine dispersion of Mg_2Al_3 . It is etched in A-2 solution of the Knuth system. The hardness is 87 VHN.

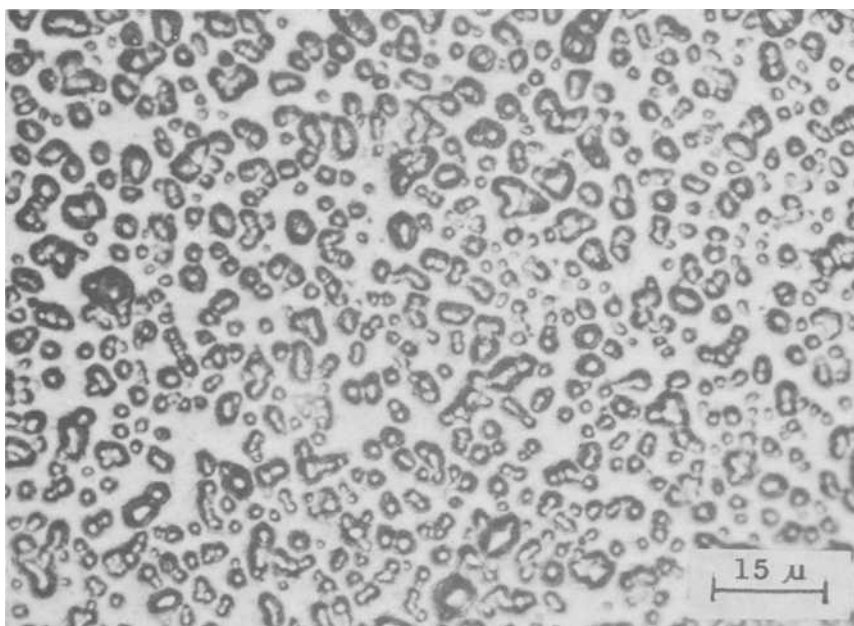


FIG. 13—Microstructure of the fusion zone of laser-beam weldments of aluminum alloy 5456 showing fine-grained α aluminum. It is etched in A-2 solution of the Knuth system. The hardness is 80 VHN.

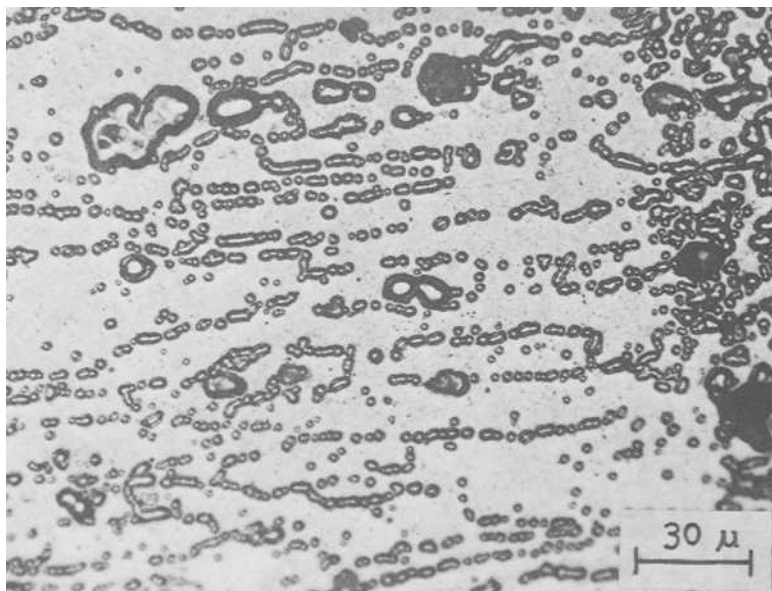


FIG. 14—Microstructure at the fusion zone boundary of aluminum alloy 5456 laser-beam weldment, etched in A-2 solution of the Knuth system.

depicts the fusion boundary. On the left is the fusion zone with its redistributed particle size and distribution; on the right is the HAZ with its elongated particles.

In laser-beam welds of aluminum alloys, a considerable amount of porosity is present. This porosity reduces the net section size in the fusion zone and contributes to fracture occurring in this region. These pores, in which the solidification structure can often be seen, are surrounded by microvoid coalescence, which is the fracture separation process (Fig. 15). This porosity causes the decreases in both the ultimate strength and the elongation of these welds. The fracture surface of the DT specimens also shows a considerable amount of porosity. Despite this porosity, the DT values are reasonably good and, when scaled up to values found in 2.54-cm (1-in.) thick specimens, compare quite favorably with them.

Titanium Alloy Laser Welds

The hardness traverse across the fusion zone and the HAZ of the titanium alloy laser-beam weldment is shown in Fig. 16. The scatter in the hardness data results from the small difference in hardnesses of the primary microstructural components: α , α' (martensitic α), Widmanstätten α , and β . The microstructure of the base plate is shown in Fig. 17, and this basket-weave appearance is characteristic of the Widmanstätten structure. The beta surrounds the acicular alpha-phase. In the fusion zone (Fig. 18) the basket-weave appear-

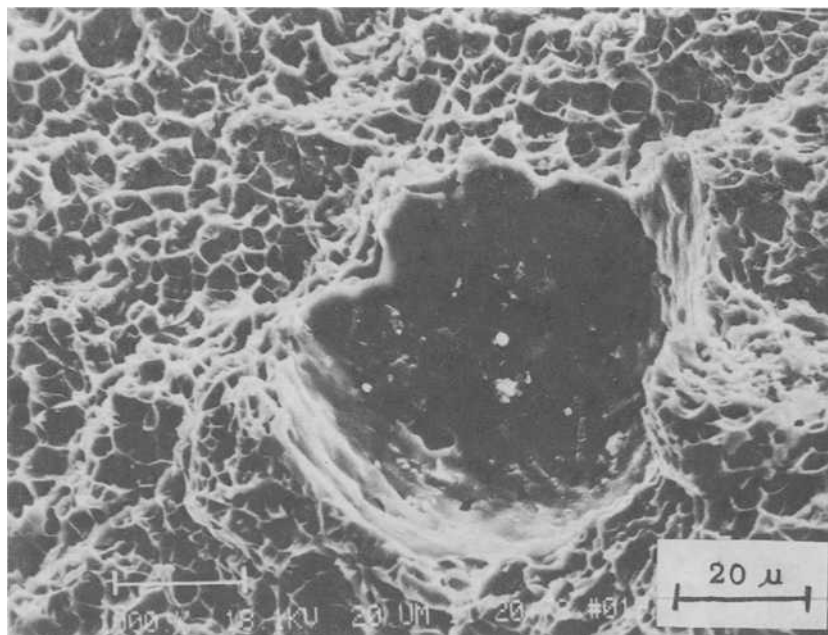


FIG. 15—Porosity surrounded by microvoids on the fracture surface of a DT specimen of a laser-beam weldment of aluminum alloy 5456.

ance has been replaced by the needlelike structure of martensitic alpha, surrounded by beta, and the boundaries of the elongated beta grains present prior to cooling. The heat-affected zone again shows the primary alpha, Widmanstätten alpha, and beta, although the basket-weave appearance is not as prevalent as it is in the base plate (Fig. 19). The mechanical properties of the laser beam weldments of this titanium alloy are excellent (Table 4) even though the

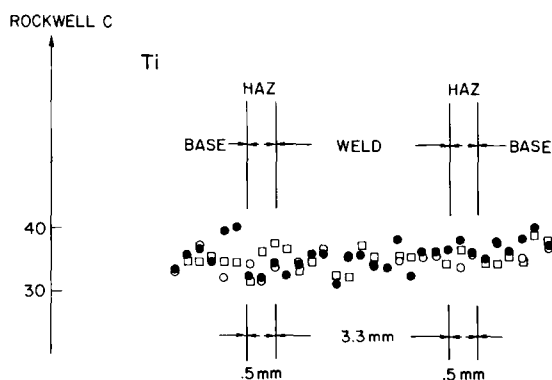


FIG. 16—Hardness traverse across the fusion zone of a laser-beam weldment of the titanium alloy.

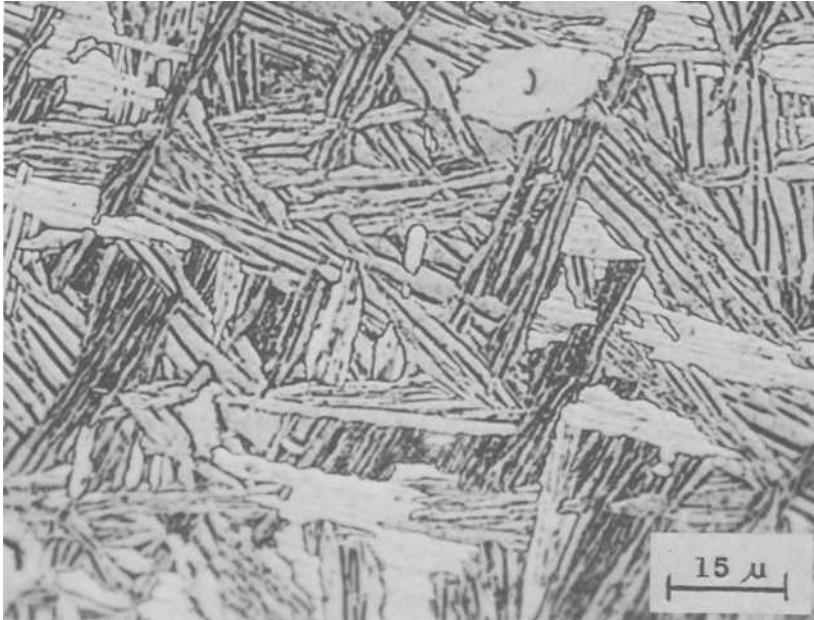


FIG. 17—Primary α , Widmanstätten α , and β in the titanium alloy, with a hardness of 32 to 40 HRC, etched in Kellers.



FIG. 18—Martensitic α with some β and prior β -grain boundaries in the fusion zone of the laser-beam weldment of the titanium alloy. The hardness is 32 to 38 HRC. The material is etched in Kellers.

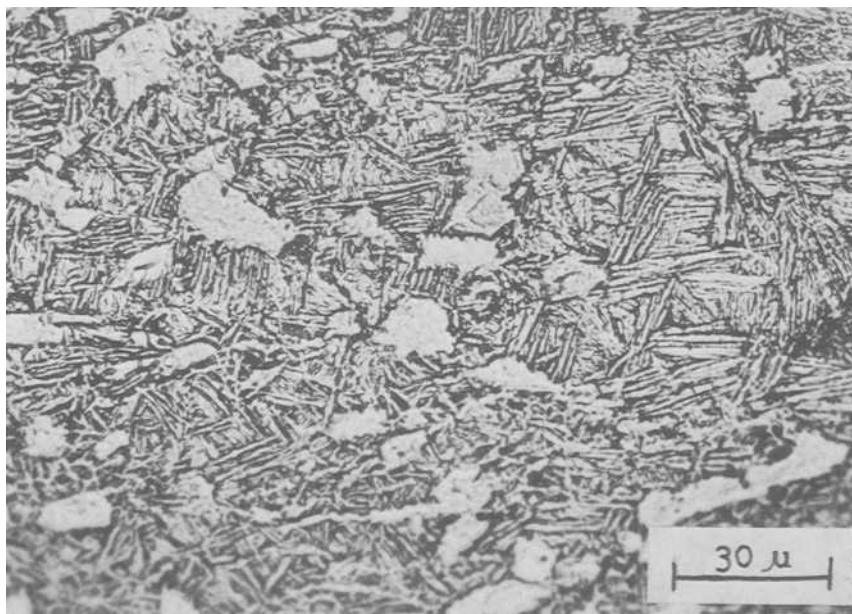


FIG. 19—Primary α , Widmanstätten α , and β in the HAZ of the laser-beam weldment of the titanium alloy, with a hardness of 32 to 37 HRC. It is etched in Kellers.

fracture surfaces of the DT specimens of this alloy showed a certain amount of porosity (Fig. 20). The fracture separation mechanism occurred entirely by microvoid coalescence.

Discussion

The properties and structures of the laser-beam weldments stated in the sections for each alloy are what one would expect to measure and determine on a metal part that had been subjected to a very intense, localized heat source for a short time. The microstructures and properties of laser-beam weldments are quite similar to those of electron-beam weldments. The microstructures that have been observed in the steels correlated well with the known transformation behavior of the alloys, yielding hard, untempered martensite and a decrease in the toughness within these zones. In the titanium alloy, the transformations that occur in the fusion zone upon laser-beam welding are the expected fast-cooling rate transformations in titanium alloys, that is, from basket-weave structures to acicular, even martensitic, structures. In the aluminum alloy, the low vaporization temperature of magnesium results in a modest but significant decrease in the percentage of magnesium measured in the fusion zone; but, more important, the size, shape, and distribution of the second phases— Mg_2Al_3 , Mg_2Si , and $(\text{Fe}, \text{Mn})\text{Al}_6$ —are altered drastically by the heat generated from the laser-beam welding process.

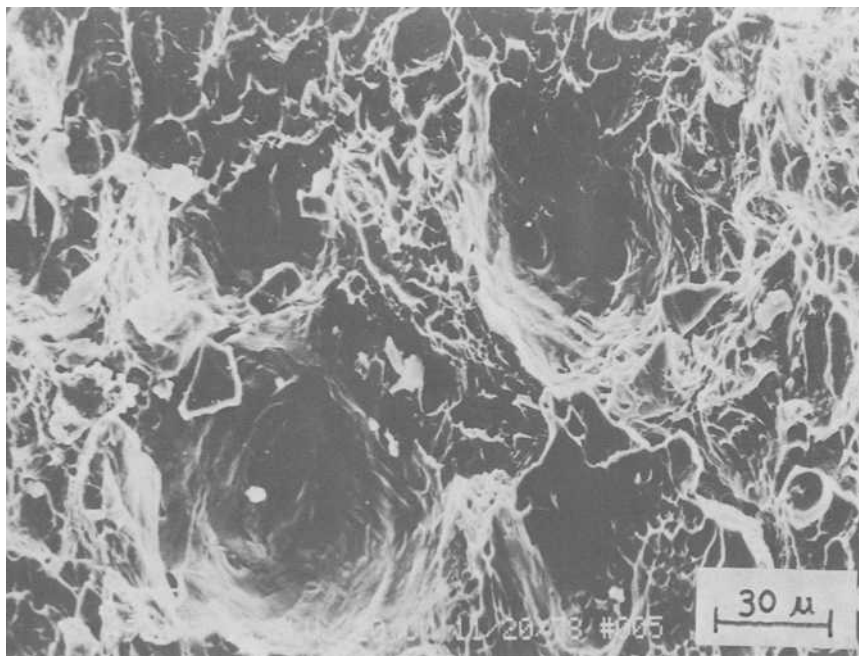


FIG. 20—Porosity in the fracture surface of the DT specimen of the laser-beam weldment of the titanium alloy.

In all of the laser-beam weldments, fusion-zone purification depends on the degree of cleanliness of the base metal, the vaporization temperature of the elements, and the geometrical morphology of the second-phase particles. Significant reductions in the inclusion content of the fusion zone of laser-beam weldments [5] have been found by counting the inclusions both in the fusion zone and in an equivalent area of the base metal. The ratio of inclusions in the fusion zone to inclusions in the base plate is 0.21 for HY-80, 0.28 for HY-130, 0.04 for aluminum alloy 5456, and 0.50 for the titanium alloy. Thus, fusion-zone purification results in a significant change in the fusion zone.

The hardness measurements in the fusion zone of the steels are a result of the untempered martensitic structures found in these alloys. The softening of the fusion zone in the aluminum alloy results from applying the intense heat source of the laser beam to a strain-hardened metal. The scatter in the hardness values of the titanium fusion zone results from the similar hardness values of the different phases found in this area.

The mechanical properties of all of the laser-beam weldments are considered to be good. Most of these specimens, with the exception of the aluminum alloy, broke in the base plate, which reflects the base plate properties. Since the fusion zone is harder than the base plate in these cases, we would expect the fractures to occur in the base plate. In the aluminum alloy, we have

a softening in the fusion zone and considerable evidence for porosity, both of which are factors in the occurrence of fracture in this area.

The toughness of HY-130 at room temperature is more than adequate. The high temperature at which the very sharp ductile-to-brittle transition occurs is a cause for concern. However, preliminary indications are that, by a judicious selection of either a postweld heat treatment or a time-delayed heat-treating pass with the laser beam, this transition temperature can be lowered considerably. The low toughness values of HY-80 result from solidification cracking, considerable evidence of which is observed on the fracture surfaces of these specimens. The titanium alloy has adequate fracture toughness despite the formation of porosity in the fusion zone. This porosity is readily understandable considering the high affinity to gases for titanium at elevated temperatures. The fracture toughness of the aluminum alloy reflects the softening of the fusion zone and the porosity, some of which always is found in aluminum welds. Despite this, the fracture toughness of this alloy scales remarkably well to a standard 2.54-cm (1-in.) dynamic tear specimen.

Transverse bend specimens were machined and tested in order to determine the soundness, weldability, and ductility of these laser-beam weldments. The radius of the bend is a function of the thickness of the specimen and the minimum required elongation. The transverse face-bend and root-bend specimens usually expose defects in the interior of the weld, whereas the transverse side-bend specimens expose defects in the interior of the weld and give an indication of the weld joint cross section. The ability of the laser-beam weldment to satisfy the bend test depended on the lack of undercut in the fusion zone. Without undercut in the toe of the weldment, the bend specimens satisfied the test criteria; with undercut, the specimens cracked and sometimes even fractured. Normally a test specimen would be without undercut. Longitudinal bend specimens, which are preferred for ductility evaluations since they strain the whole joint equally, were not tested because of a lack of laser-beam weldments.

Conclusions

The results of this experimental program indicate that laser-beam welding of these alloys is a viable technique, although not without its difficulties. These difficulties should be overcome in the near future by judicious experiments.

Acknowledgments

Some of the laser-beam weldments were fabricated under contract by United Technologies Research Center, and the work of Conrad Banas and his staff is gratefully acknowledged. Other welds were made at the Naval Research Laboratory, Washington, D.C. The assistance of E. R. Pierpoint, M. E. Hammer, and R. F. Beigel in fabricating these welds was most beneficial. E. R. Pierpoint and M. E. Hammer carried out the testing and fractographic analysis.

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Fractographic Comparison of Plane-Strain Fracture Toughness, Instrumented Precracked Charpy, and Slow-Bend Precracked Charpy Tests on a Quenched and Tempered AISI 4340 Steel

REFERENCE: Datta, K. P. and Wood, W. E., "Fractographic Comparison of Plane-Strain Fracture Toughness, Instrumented Precracked Charpy, and Slow-Bend Precracked Charpy Tests on a Quenched and Tempered AISI 4340 Steel," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 150-165.

ABSTRACT: Toughness testing of a quenched and tempered AISI 4340 steel has been carried out by precracked Charpy (instrumented and slow-bend) and fracture toughness test techniques. Results show that the toughness values, as a function of heat treatment, follow similar trends for identical heat-treatment schedules in each type of testing. Fractographic observations also indicate the same fracture initiation mode in all the tests for identical heat treatments, including an intergranular fracture mode associated with the temper embrittlement of the steel in question. It indicates that, in this particular application, mechanical testing variables such as specimen dimensions, crack length, crack length to width ratio, and strain rate have no effect on the fracture initiation process.

KEY WORDS: fractography, toughness, heat treatment, AISI 4340 steel, materials, materials science, mechanical testing variables

Toughness is an important mechanical property for the engineering application of any structural material. However, in recent years, evaluation of toughness has undergone considerable change. From an essentially empirical test such as the Charpy [1],³ sophisticated tests such as fracture toughness [2], K_{Ic} , based on linear elastic fracture mechanics, have evolved. Simultaneously, severe restrictions on specimen dimensions such as thickness, crack length, a , and the ratio of crack length to width, a/W , have limited its practical applica-

¹Department of Metallurgical Engineering and Materials Science, University of Kentucky, Lexington, Ky. 40506.

²Chairman, Department of Materials Science, Oregon Graduate Center, Beaverton, Ore. 97006.

³The italic numbers in brackets refer to the list of references appended to this paper.

TABLE 1—Chemical composition of AISI 4340 steel, by weight percent.

C	Mn	Si	Cr	Ni	Mo	Cu	S	P	V	Fe
0.40	0.60	0.32	0.69	1.87	0.20	0.16	0.015	0.015	...	balance

bility. In fact, for an AISI 4340 steel, the volume of one compact tension K_{Ic} specimen is equivalent to that of twelve Charpy specimens, as used in this investigation. This ratio is much higher for a medium-strength or low-strength material. In order to overcome these limitations, tests such as the J-integral [3], crack-opening displacement (COD) [4], and equivalent energy toughness [5] tests have been developed based on nonlinear elastic fracture mechanics. The Charpy test itself has been instrumented [6] so as to obtain an approximate fracture toughness value.⁴ Special fixtures [7,8] have been designed to test Charpy specimens under slow-bend conditions and obtain a toughness value in a strain rate approaching that of K_{Ic} testing. Various statistical correlations have been proposed between Charpy V-notch energy values and K_{Ic} [9, 10]. However, all such methods are based on either a macroscopic continuum approach or a statistical correlation, and they do not consider the microscopic process of fracture, which should have a direct bearing on the fracture toughness value. Furthermore, processing modifications in ultrahigh strength steel such as AISI 4340 may induce large variations in K_{Ic} without affecting Charpy V-notch energy values [11]. This paper examines the microscopic fracture initiation of a quenched and tempered AISI 4340 steel under instrumental precracked Charpy, slow-bend precracked Charpy, and fracture toughness tests and tries to correlate the toughness values from the different tests with the microscopic fracture processes.

Experimental Procedure

The material in the present study was AISI 4340 steel. The chemical composition of the steel in weight percent is shown in Table 1.

Specimens were austenitized in a vertical tube in argon atmosphere for 1 h at 1200°C and directly quenched in an agitated oil. All tempering was carried out in a neutral salt bath for 1 h. The dimensions of the specimens are given in Fig. 1. All the specimens were tested in the longitudinal directions (L-T for K_{Ic} and L-TS for Charpy) according to ASTM procedures [2]. The instrumented Charpy testing was done on a Dynatup system developed by Effects Technology, Santa Barbara, Calif. A special fixture (shown in Fig. 2) was designed to test Charpy specimens under slow-bend conditions. Scanning electron mi-

⁴Toughness obtained from instrumented and slow-bend Charpy tests is known as apparent toughness, since the specimen dimensions do not conform to ASTM standards, although the method of calculation is the same as that of a fracture toughness specimen.

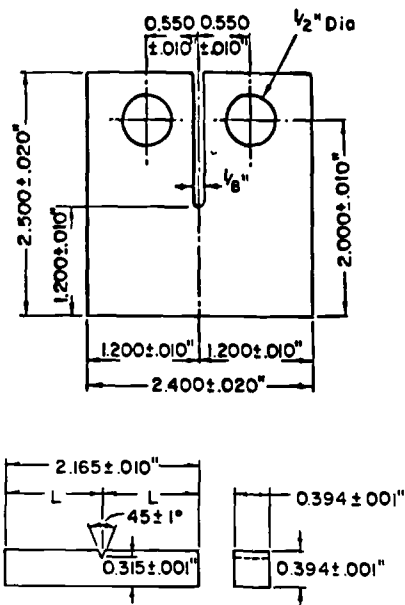


FIG. 1—(Top) Dimensions of a fracture toughness specimen. (Bottom) Dimensions of a Charpy specimen (1 in. = 2.54 cm).

scopy was carried out using a Japan Electron Optics Laboratory Co. scanning electron microscope at 25 kV (secondary electron voltage). For each specimen, the region adjacent to the fatigue precrack was examined, since this is the region of crack initiation in all types of specimens. In order to preserve the fracture surface from oxidation or corrosion, the fracture surface was coated with Dem-Kote spray. Before examining the fracture surface, the spray was removed with acetone.

Results

The instrumented precracked Charpy test data on apparent toughness⁴ as a function of tempering temperature are given in Table 2. Tempering temperatures up to 280°C were studied. The corresponding results are shown in Fig. 3. Note that for the high-temperature austenitization case (1200°C), the toughness increased at first as the tempering temperature increased, but at a tempering temperature of 280°C, there was a substantial drop in the toughness value. However, in the as-quenched condition the toughness of the low-temperature austenitized specimen was substantially lower than that of the high-temperature austenitized specimen.

The fracture toughness data as a function of tempering temperature are given in Table 3 and Fig. 4. Here also the two previously mentioned trends are

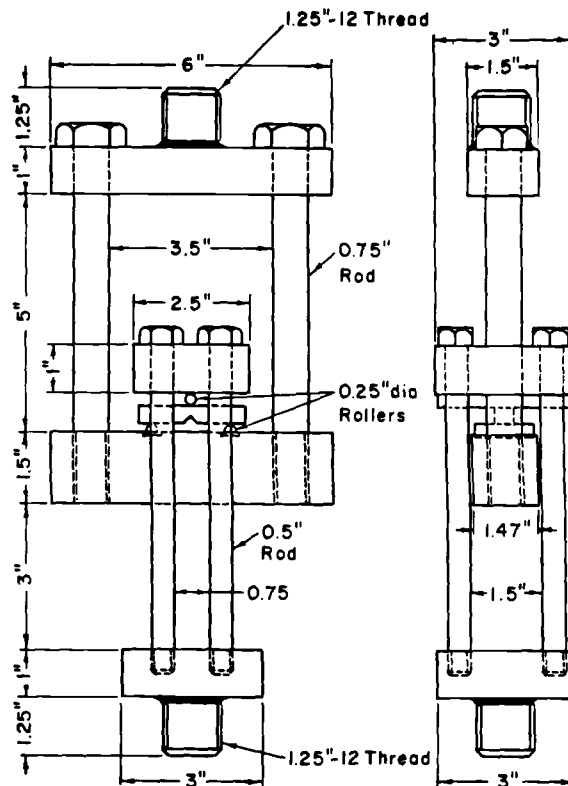


FIG. 2—Testing fixture for a slow-bend Charpy specimen (1 in. = 2.54 cm).

 TABLE 2—Room-temperature instrumented precracked Charpy testing data for AISI 4340 steel.^a

Austenitizing Treatment, deg C/1 h	Tempering Temperature, deg C/1 h	a/W	K_{APP}	
			ksi $\sqrt{\text{in.}}$	MPa $\sqrt{\text{m}}$
1200	AQ ^b	0.3	63.72	69.96
	175	0.25	93.40	102.55
	280	0.23	59.95	65.83
870	AQ	0.3	39.4	43.26
	175	0.23	65.23	71.62
	280	0.24	73.8	81.03

^a Results are based on an average of two specimens for each heat treatment.

^b AQ = oil quenched after austenitization and tested in the as-quenched condition.

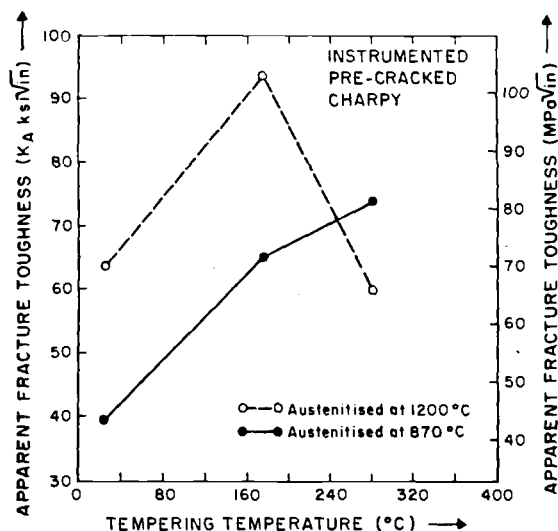


FIG. 3—Effect of the tempering temperature on the toughness of AISI 4340 steel given different heat treatments by instrumented Charpy testing.

noticeable; that is, there was a substantial drop in toughness for the high-temperature austenitized specimen at a tempering temperature of 280°C, whereas no similar drop occurred for the low-temperature austenitized specimen. Also, in the as-quenched condition, the toughness of the high-temperature austenitized specimen was substantially higher than that of the low-temperature austenitized specimen.

Table 4 and Fig. 5 show the slow-bend precracked Charpy data as a function of tempering temperatures. The results were identical, as in the previous two cases.

In summary, the toughness increased initially as the temperature increased, and then decreased substantially for the high-temperature austenitized case in all types of testing. Also, the as-quenched toughness of the high-temperature

TABLE 3—Room-temperature fracture toughness data for AISI 4340 steel.^a

Austenitizing Treatment, deg C/1 h	Tempering Temperature, deg C/1 h	<i>a</i> / <i>W</i>	<i>K_{Ic}</i>	
			ksi √in.	MPa √m
1200	AQ ^b	0.53	66.6	73.13
	175	0.52	86.25	94.70
	280	0.5	62.1	68.19
870	AQ	0.54	38.15	41.89
	175	0.51	59.8	65.66
	280	0.52	76.3	83.77

^a Results are based on an average of two specimens for each heat treatment.

^b AQ = oil quenched after austenitization and tested in the as-quenched condition.

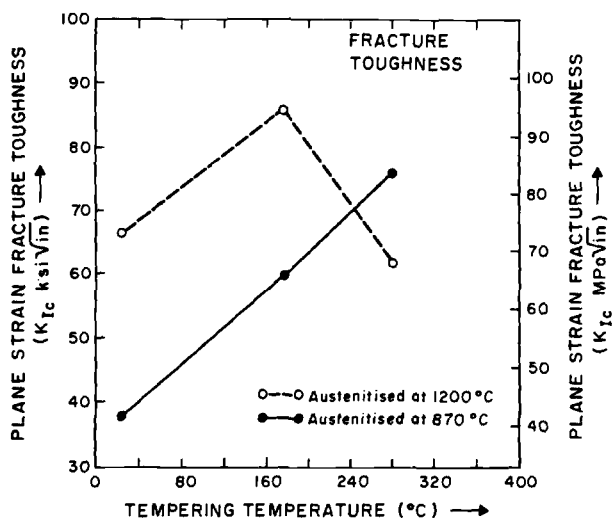


FIG. 4—Effect of the tempering temperature on the toughness of AISI 4340 steel given different heat treatments by fracture toughness testing.

austenitized specimen was considerably higher than that of the low-temperature austenitized specimen in each type of testing.

Microscopy

Figures 6 through 9 show the fracture morphology of precracked Charpy (instrumented and slow-bend) and fracture toughness specimens given different tempering treatment after high-temperature austenitization. As-quenched specimens and specimens tempered at 175 and 280°C were examined. The fracture mode underwent a continuous change as the tempering temperature changed from the as-quenched to the 280°C-temper condition, and it changed in the same way in each type of testing.

TABLE 4—Room-temperature slow-bend precracked Charpy testing data for AISI 4340 steel.^a

Austenitizing Treatment, deg C/1 h	Tempering Temperature, deg C/1 h	a/W	K _{APP}	
			ksi √in.	MPa √m
1200	AQ ^b	0.3	64.55	70.88
	175	0.27	90.21	99.05
	280	0.3	52.88	58.06
870	AQ	0.3	33.45	36.73
	175	0.29	53.2	58.41
	280	0.3	56.6	62.15

^a Results are based on an average of two specimens for each heat treatment.

^b AQ = oil quenched after austenitization and tested in the as-quenched condition.

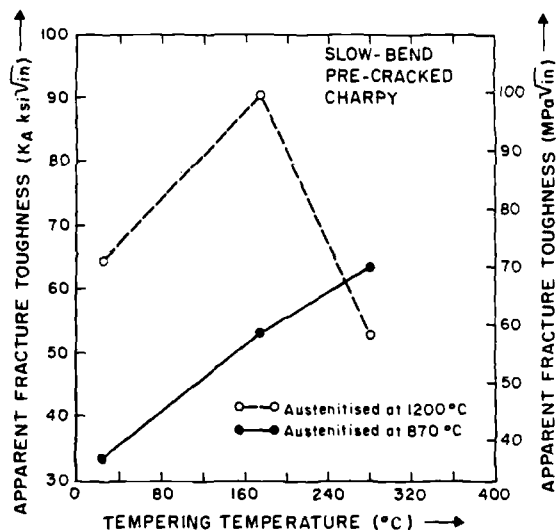


FIG. 5—Effect of the tempering temperature on the toughness of AISI 4340 steel given different heat treatments by slow-bend Charpy testing.

Fractographs from one specimen may be divided into three areas: (1) the fatigue precrack zone, (2) the fracture initiation zone, and (3) the fracture propagation zone, as illustrated in Fig. 6. In all subsequent pictures only the fracture initiation zone is shown, since that has a direct correspondence to the toughness value. Fractographs from the as-quenched specimen exhibited an uneven fracture at low magnification. However, at high magnification it was observed that the fracture initiation was by microvoid coalescence (Figs. 6b, 8a, and 9a). The fracture morphology of specimens tested after tempering at 175°C, which showed a high toughness value, exhibited a dimpled structure typical of ductile rupture (Figs. 7a, 8b, and 9b). On the other hand, the fractographs obtained from specimens tempered at 280°C showed an intergranular brittle fracture (Figs. 7b, 8c, and 9c). The surface, however, contained some regions with tearing marks.

Figure 10 shows the fracture morphology of as-quenched AISI 4340 steel after austenitization at 870°C, tested under various specimen configurations. All the specimens examined showed a quasi-cleavage/intergranular fracture morphology.

Thus, as in toughness results, the fracture morphology is the same in each specimen configuration for identical heat treatment.

Discussion

Changes in toughness data can be brought about by either microstructural features or by specimen dimension (that is, plane stress versus plane strain) at

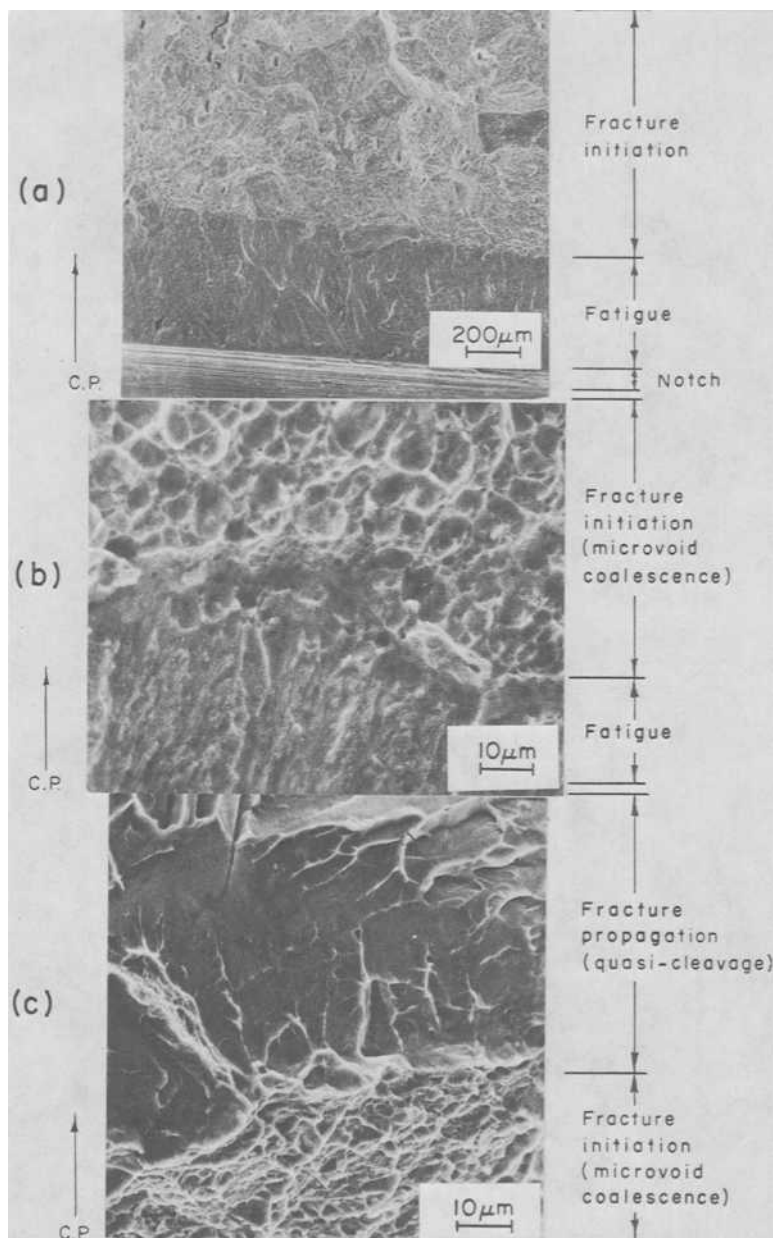


FIG. 6—Scanning electron microscope photomicrographs of as-quenched instrumented Charpy specimens austenitized at 1200°C: (a) notch, fatigue, and fracture initiation zones at low magnification; (b) at high magnification the fatigue zone changing into the microvoid coalescence zone; (c) at high magnification, the microvoid coalescence zone (fracture initiation) changing into the cleavage/quasi-cleavage zone (fracture propagation).

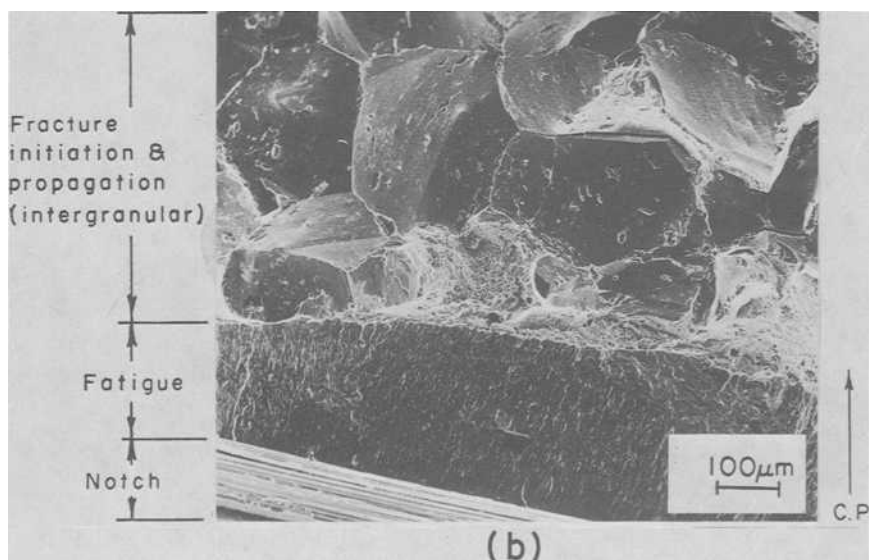
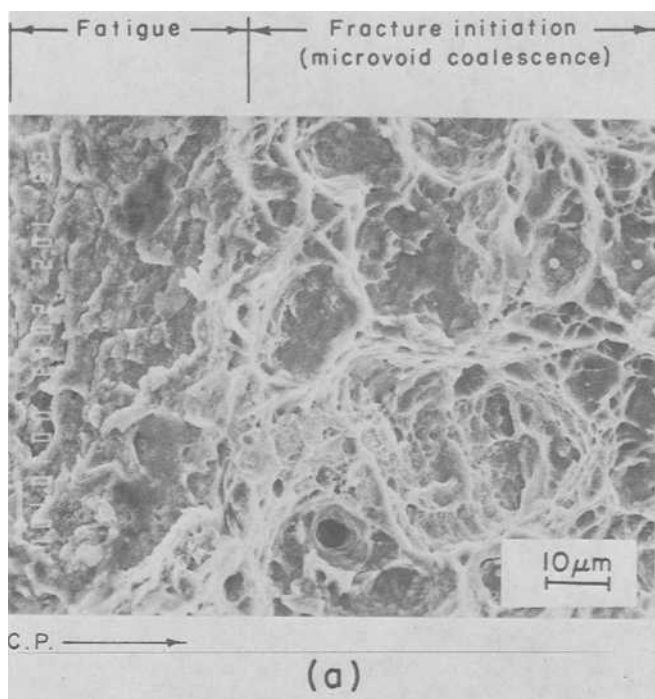


FIG. 7—Scanning electron microscope photomicrographs of quenched and tempered instrumented Charpy specimens austenitized at 1200°C: (a) tempered at 175°C, the fatigue zone changing into the microvoid coalescence zone; (b) tempered at 280°C, the notch, fatigue, and fracture initiation zones (intergranular).

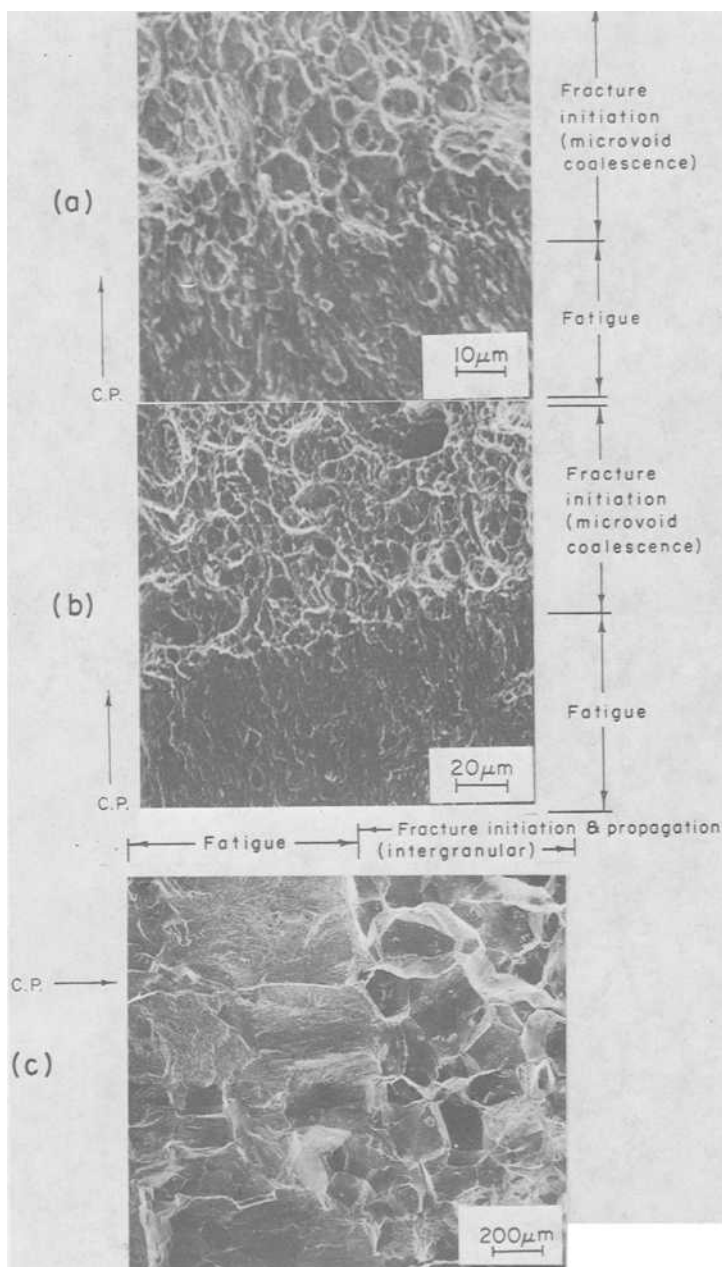


FIG. 8—Scanning electron microscope photomicrographs of quenched and tempered fracture toughness specimens austenitized at 1200°C : (a) the as-quenched fatigue zone changing into the microvoid coalescence zone; (b) tempered at 175°C , the fatigue zone changing into the microvoid coalescence zone; (c) tempered at 280°C , the fatigue and fracture initiation zones (intergranular).

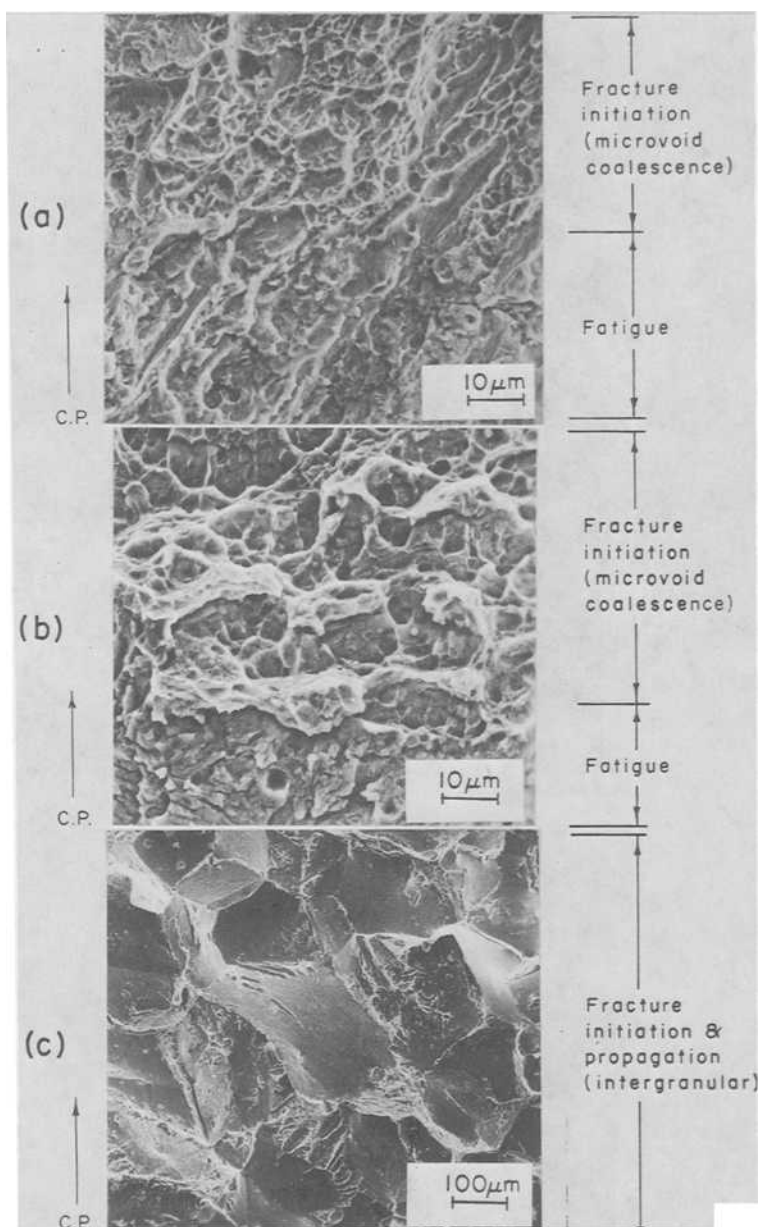


FIG. 9—Scanning electron microscope photomicrographs of quenched and tempered slow-bend Charpy specimens austenitized at 1200°C: (a) as-quenched, the fatigue zone changing into the microvoid coalescence zone; (b) tempered at 175°C, the fatigue zone changing into the microvoid coalescence zone; (c) tempered at 280°C, the intergranular fracture initiation and propagation zone.

a particular strain rate and temperature. The results in this investigation clearly demonstrate that changes in K_A data are due to changes in fracture modes, which in turn, are caused by microstructural variations.

Utilization of fracture mechanics principles, which are based on a continuum model rather than a physical model [12], results in severe specimen restrictions. It would generally be thought more "realistic" to represent the deformation, rupture, cracking, or cleavage of a real crystal as processes occurring in the lattice, that is, the physical model. But for quantitative reasons, the physical model is beset by severe mathematical difficulties. For the most part, these are avoided at the first opportunity by changing over to a continuum model. It may be borne in mind, however, that even though the most important zone is at the crack tip, this separation is induced by forces transmitted from a zone that is very remote, and this field of force can be well represented by the continuum model. We have thus an essential interaction between the two zones.

The continuum model is the basis of fracture mechanics principles, and that has resulted in quite a few restrictions on the specimen dimensions. First, the crack length [13]: the restriction on crack length is treated with reference to the distribution of the tensile stress, σ_{11} , ahead of a crack, as given by

$$\sigma_{11} = \frac{K_I}{\sqrt{2\pi r}} + \dots \text{series} \quad (1)$$

where r is the distance ahead of the tip. K_I is calculated as a single-valued parameter only as r tends toward zero. As r increases from zero, the stress will be described less and less accurately by a single value for K , because the extra terms in Eq 1 begin to contribute, as shown in Fig. 11. Hence, it must be ensured that fracture events are limited to a region ahead of the crack, which is small compared with other specimen dimensions. The specification of the size of such a region is one of many value judgments made in fracture toughness testing. Normally, this size is taken to be equal to one fiftieth of the crack lengths. Thus

$$r_{ly} = \frac{1}{6\pi} \left(\frac{K_{Ic}}{\sigma_{ys}} \right)^2 \leq 0.02 a \dots \quad (2)$$

where r_{ly} = the plane-strain plastic zone size, and σ_{ys} = the yield strength. In our study for a $K_{Ic} = 82.35 \text{ MPa } \sqrt{\text{m}}$ (75 ksi $\sqrt{\text{in.}}$) and $\sigma_{ys} = 1379 \text{ MPa}$ (200 ksi), $r_{ly}/a \approx 0.0075$ for the fracture toughness specimen and $r_{ly}/a \approx 0.06$ for the Charpy specimen. Thus, we see that the precracked Charpy specimens do not meet the crack-length criterion as prescribed in fracture toughness testing.

The second restriction is on the width of the specimen ligament, $W-a$ [14]. If $W-a$ is too small, compared with the size of the plastic zone at fracture, the

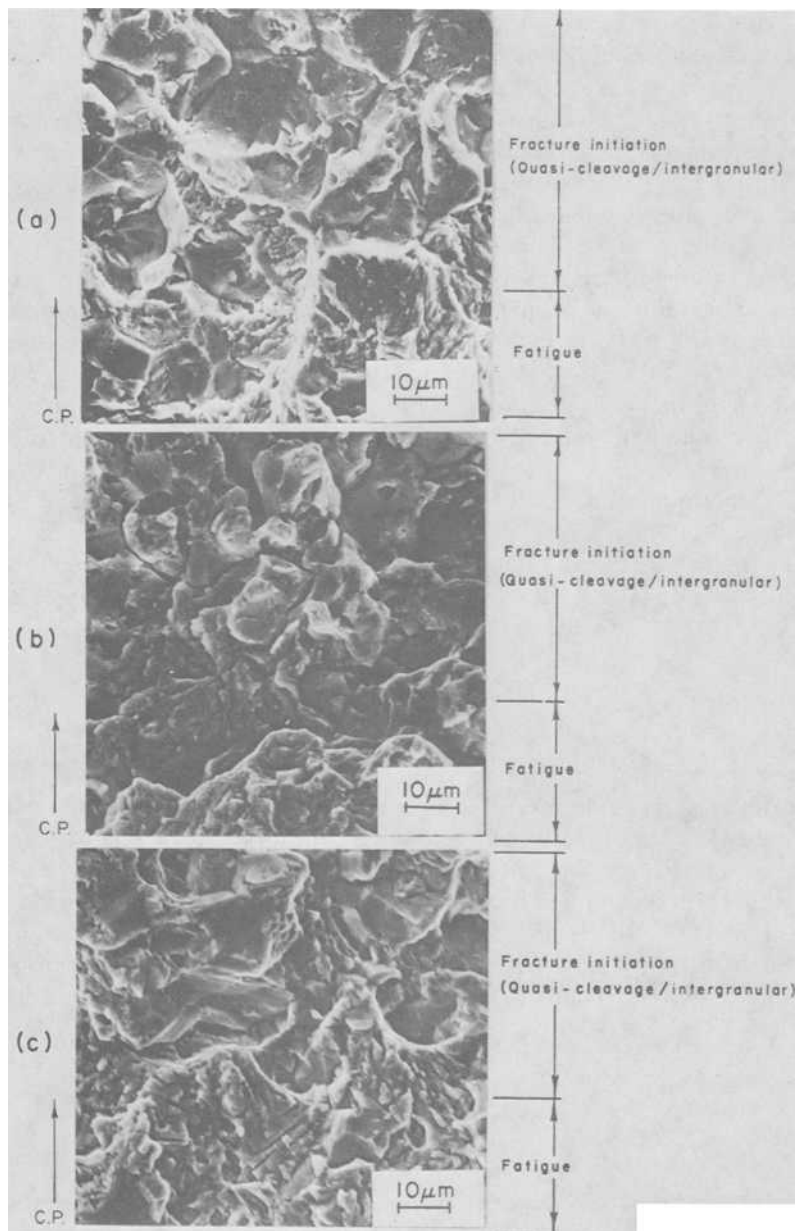
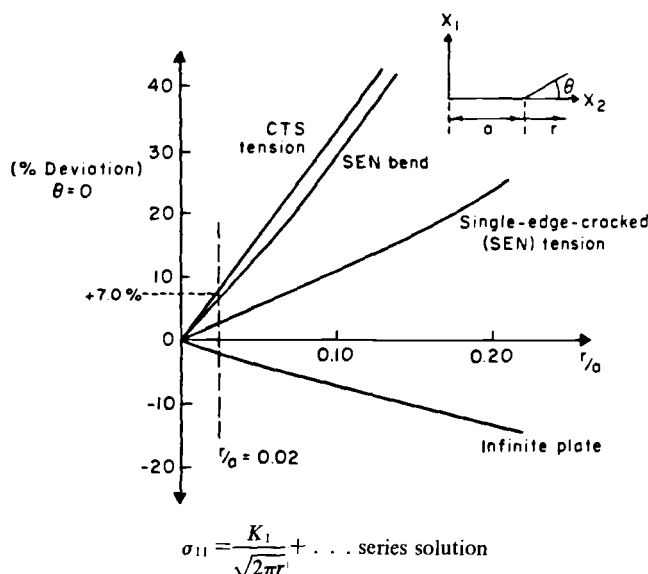


FIG. 10—Scanning electron microscope photomicrographs of as-quenched Type 4340 steel austenitized at 870°C: (a) instrumented Charpy test, (b) fracture toughness test, (c) slow-bend Charpy test. All tests show a quasi-cleavage/intergranular fracture initiation mode.



σ_{11} = longitudinal stress ahead of the crack tip; r = distance from the crack tip. K_I is a single-valued parameter as r tends toward zero.

FIG. 11—Deviation from the ideal stress field [13]. Variation between the crack-tip stress calculated from a single-valued stress-intensity factor, $\sigma_{11}(K)$, and that calculated from the full series, σ_{11} , expressed as the percentage

$$100 \frac{(\sigma_{11}(K) - \sigma_{11})}{\sigma_{11}} \theta = 0$$

and plotted versus the distance ahead of the crack tip [13].

quasi-elastic solution for stress intensity is invalid, because the closeness of the stress-free boundaries significantly affects the crack-tip stress field. The restriction on a/W , according to ASTM procedures, is that $a/W = 0.45$ to 0.55 . In our study a/W for fracture toughness specimens ≈ 0.5 , and for Charpy specimens $a/W = 0.3$. Thus, Charpy specimens also failed to meet the ligament criterion.

The third requirement is the specimen thickness [15], that is, $B \geq 2.5 (K_{Ic} / \sigma_{ys})^2$. Fortunately, for an ultrahigh-strength steel such as AISI 4340, the thickness restriction does not pose too great a problem. Still, assuming a $\sigma_{ys} \approx 1379$ MPa (200 ksi), Charpy specimens meet the valid criterion with regard to thickness only for $K_{Ic} 86.74 \text{ MPa } \sqrt{\text{m}}$ ($< 79 \text{ ksi } \sqrt{\text{in.}}$). In spite of a higher K_{Ic} value than this in some of our results, the fracture mechanisms are still found to be identical.

Another difference in the instrumented Charpy test and the fracture toughness test is the strain rate, (that is, from dynamic condition to static condition). Results for a semi-killed steel [16] show that an order of magnitude increase in loading rate is required to decrease K_{Ic} by 10 percent. The data in this regard

are scanty, since very large test pieces cannot usually be tested. In our studies both toughness and fracture mechanisms remain identical in spite of the change in loading rate by an order of magnitude of 5 to 6. However, the yield strength of AISI 4340 steel is also strain-rate insensitive.

Thus, in spite of the wide variations in mechanical parameters, the toughness values and fracture mechanisms (quasi-cleavage, microvoid coalescence, or intergranular fracture, which are the most commonly observed) remain identical in this particular class of materials (that is, low-alloy ultrahigh-strength steel, such as AISI 4340). This fact points to the dominance of microstructural variables controlling fracture mechanisms over the mechanical variables on the overall evaluation of toughness. This also points to the fact that a precracked Charpy test, in this particular application, can successfully withstand competition from more scientific fracture toughness tests with regard to both toughness and fracture mechanisms.

Conclusions

1. Toughness values remain identical for identical heat-treatment schedules in all types of testing for AISI 4340 steel.

2. As with toughness, the fracture mechanisms (either quasi-cleavage, microvoid coalescence, or intergranular fracture) remain identical in all three types of testing.

3. The specimen restrictions, such as crack length, ligament, and thickness, in this particular application do not affect either the toughness or the fracture mechanisms. Hence the precracked Charpy test (either instrumented or slow-bend) can successfully withstand competition from more costly fracture toughness tests for this class of materials.

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Fractographic Characterization of the Effect of Inclusions on Fatigue Crack Propagation

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ABSTRACT: The fracture surfaces of fatigue crack propagation (FCP) test specimens of ASTM plate steel Grades A516-70, A533B-1, and A514F are characterized primarily by using the scanning electron microscope. The evaluation involved two plates of each grade with different inclusion structures. These had been produced by conventional steel-making techniques or by calcium treatment. The FCP tests were performed in the six possible testing orientations for each material and quality level. In each grade, the Type II manganese sulfide and alumina inclusions associated with the conventional quality plate resulted in faster FCP growth rates, particularly in the through-thickness testing orientations (S-T, S-L). Furthermore, the group nature of the occurrence of these inclusions was found to be the primary influence on FCP behavior. In addition, these inclusion formations were found to give greater anisotropy of FCP behavior for the steels of the higher yield strength, principally because of the fracture mode of the material between inclusions of a particular formation. For all steel grades, the calcium treatment led to improved levels and isotropy of FCP resistance.

KEY WORDS: fatigue crack propagation, inclusions, fractography, steels, anisotropy, calcium treatment, desulfurization, yield strength effects, materials, materials science

Nonmetallic inclusions can have a very dramatic effect on the properties and behavior of steel. Inclusions have been found to increase the susceptibility of steel to lamellar tearing on welding [1]² and to affect its formability [2]. However, the effect of inclusions on specific mechanical properties also has been of great interest. In steels, inclusions have been found in general to have an adverse effect on tensile ductility [1,3-5], impact and fracture toughness [3-8], fatigue endurance limit [7,9], and fatigue crack propagation [5-7]. In each instance, fractographic characterization has been found to be extremely valuable in determining the influence of the inclusions on the property

¹Senior research engineer, Lukens Steel Co., Coatesville, Pa. 19320.

²The italic numbers in brackets refer to the list of references appended to this paper.

[1,3-11]. Fatigue crack propagation has been of particular interest because of its well-established historical association with fractography.

The indigenous nonmetallic inclusions in steel precipitate as discrete phases during solidification of molten steel, such as sulfides and oxides. Their composition, size, shape, and distribution are a result of a number of variables, the most important being the steel-making practice that is used [3-5]. Aluminum-killed plate steels that are produced by conventional electric furnace practices (CON) contain Type II manganese sulfide (MnS) inclusion patches that are elongated by hot rolling. In addition, clusters of alumina inclusions, called galaxies, are present in CON steels, and, although the individual alumina (Al_2O_3) inclusions are not deformed on hot rolling, they are spread out and elongated as a group. Both of these inclusion formations affect not only the level but also the isotropy of properties of CON steels. Calcium treatment (CaT) minimizes the formation of both of these inclusion groups through desulfurization (0.010 percent maximum sulfur) and inclusion shape control. The resulting inclusions are Type III MnS, which are more compact and uniformly distributed than the Type II inclusions. The calcium-modified inclusions that also remain are resistant to deformation on hot rolling. Thus CaT steels tend to have an improved level and isotropy of properties [3-8]. In this paper, steels produced by both CON and CaT practices will be evaluated.

The effect of inclusions on the FCP of certain plate steels has already been established [5-7,11]. In this paper previous FCP results, as well as new data, will be examined for three plate steel grades: Grades A516-70, A533B Class 1, and A514F. Fractographic analysis of the FCP test specimens will then be used to provide further insight into the interaction of the fatigue crack front with the steel inclusions.

FCP Results

The three plate steel grades that were evaluated, Grades A516-70, A533B-1, and A514F, have minimum 0.2 percent offset yield strength minimum requirements of 263 MPa (38 ksi), 345 MPa (50 ksi), and 690 MPa (100 ksi), respectively. The identification, chemistries, and other information on the test materials are given in Table 1. Typical microstructures of each steel grade are given in Fig. 1. The CON and CaT quality levels were examined for each plate steel grade. All the plates in this study were given approximately the same amount of reduction by hot rolling. The Grade A514F plates were produced from continuous cast slabs, whereas the Grades A516 and A533B steels were rolled from ingots. Table 2 shows the effects of inclusion control on the level and isotropy of certain selected basic mechanical properties. In particular, the significant effect on the level and isotropy of the Charpy-V-notch upper shelf energy (CVN-USE) is evident.

The FCP test results for the grades A516 and A533B plates were reported previously [6,7,11], but they will also be presented here for ease of discussion.

TABLE 1.—*Test materials.*^a

Grade	Plate Thickness		Chemistry, weight percent											
	Quality	mm (in.)	C	Mn	P	S	Si	Cu	Ni	Cr	Mo	V	Al	Ca, ppm
A516-70	CON	190 (7.5)	0.23	0.98	0.013	0.013	0.25	0.13	0.14	0.08	0.03	0.002	0.039	6
	CaT	254 (10)	0.24	1.09	0.014	0.002	0.19	0.11	0.20	0.06	0.05	0.002	0.038	43
A533B-1	CON	165 (6.5)	0.18	1.41	0.008	0.009	0.23	0.13	0.48	0.11	0.48	. . .	0.045	4
	CaT	190 (7.5)	0.20	1.27	0.012	0.009	0.18	0.11	0.61	0.08	0.55	. . .	0.046	20
A514F	CON	57 (2.25)	0.17	0.88	0.009	0.014	0.27	0.22	0.91	0.57	0.52	0.054	0.022	4
	CaT	57 (2.25)	0.16	0.86	0.011	0.006	0.26	0.19	0.95	0.63	0.45	0.046	0.045	30

^a Key to abbreviations:

CON = conventional electric furnace heat.

CaT = calcium-treated electric furnace heat.

The *heat treatments* used are as follows:

A516-70—normalized 899°C (1650°F) and air cooled.

A533B-1—899°C (1650°F) water-quenched, tempered 671°C (1240°F), air cooled, stress-relieved 593°C (1100°F), furnace cooled.

A514F—899°C (1650°F) water-quenched, tempered 621°C (1150°F), water-quenched, stress-relieved 593°C (1100°F), air cooled. (Note: It is normally not recommended that this grade of steel be stress relieved, however, due to the effect of residual stresses on FCP testing, stress relieving was required for these plates.)

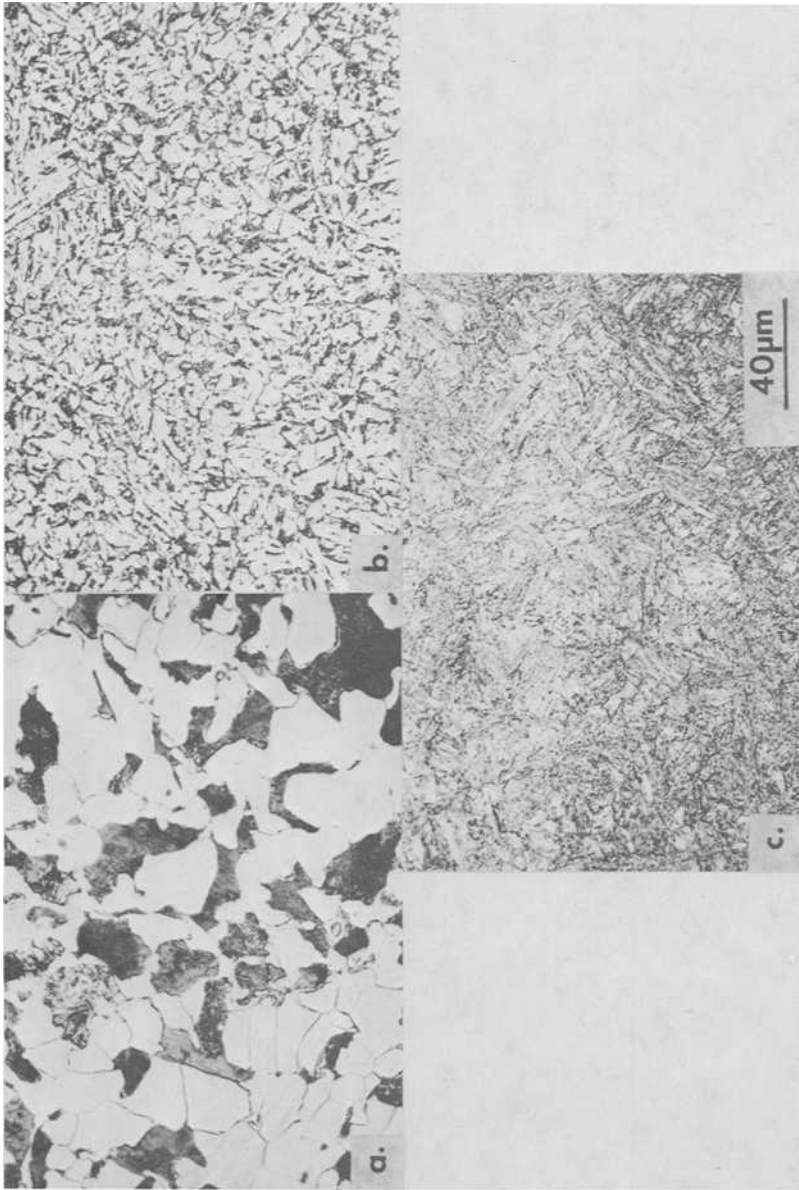


FIG. 1—Typical microstructures of tested grades at test locations of interest: (a) Grade A516-70 (ferritic-pearlitic), (b) Grade A533B Class 1 (bainitic), and (c) Grade A514F (martensitic).

TABLE 2—Selected basic mechanical properties.^a

Grade	Quality	0.2% Yield Strength, MPa			Percent Reduction of Area			Charpy-V-Notch, Upper Shelf Energy, joules						
		L	T	S	L	T	S	LS	LT	TS	TL	ST	SL	
A516-70	CON	254	253	261	64.6	63.7	44.2	123	119	123	118	66	64	
	CaT	301	265	293	67.1	63.8	57.2	165	172	153	160	126	122	
A533B-1	CON	444	436	439	71.4	57.5	20.3	203	172	115	115	58	54	
	CaT	494	494	496	72.8	70.2	66.9	186	191	172	174	130	127	
A514F	CON	786	792	745	70.9	54.1	8.2	129	111	61	56	19	16	
	CaT	799	784	781	71.8	64.9	51.8	152	148	96	89	50	46	

^aConversions: ksi = MPa $\times$ 0.145; ft $\cdot$ lb = joules $\times$ 0.737.

Test materials from these plates were taken at locations centered 51 mm (2 in.) from the plate surfaces. The Grade A514F FCP test results were obtained by following accepted ASTM procedures using testing and analysis procedures that had been reported previously [7]. Test specimens for the Grade A514F plates were centered on the center line of the plates. For all the six plates that were examined, FCP tests were performed in the six major testing orientations, as shown schematically in Fig. 2. Therefore, the FCP results can be used to compare both the CON and CaT quality levels for each steel grade and also to establish the anisotropy of FCP behavior within the quality level of each steel.

The plots of $\log \Delta K$ (range of stress-intensity factor) versus $\log da/dN$ (FCP growth rate) for the six plates that were characterized are summarized in Fig. 3. The best-fit straight lines determined using regression analysis to fit the Paris FCP equation (Eq 1) are presented [12]

$$\frac{da}{dN} = C_0 \Delta K^n \dots (n, C_0 \text{ are material constants}) \dots \quad (1)$$

The anisotropy of FCP in the CON quality steels for all three plate steel grades is readily apparent in Fig. 3. This anisotropy also appears to be more extensive with increasing strength levels. The values of n in Eq 1 for the Grade A516 steels ranged between 3.2 and 3.5 for the CON and 3.0 and 3.2 for the CaT steel. For Grade A533B steels the values were from 2.8 to 3.7 for the CON and

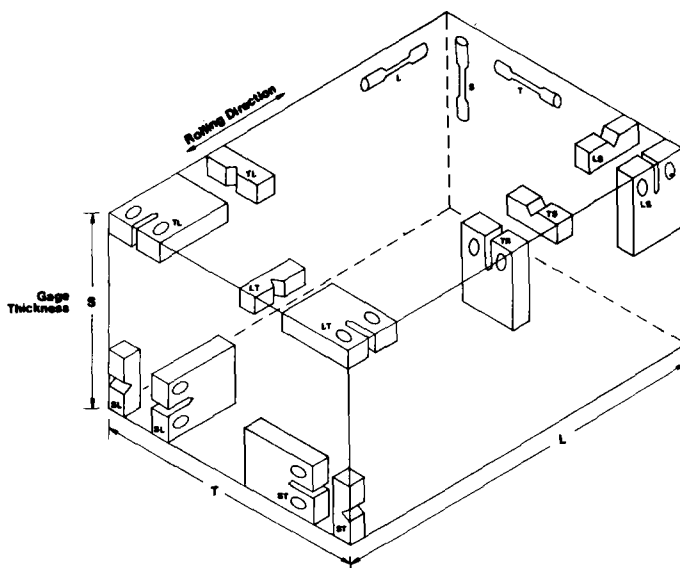


FIG. 2—Schematic drawing showing the specimen orientations used in this investigation. The actual test locations are described in the text.

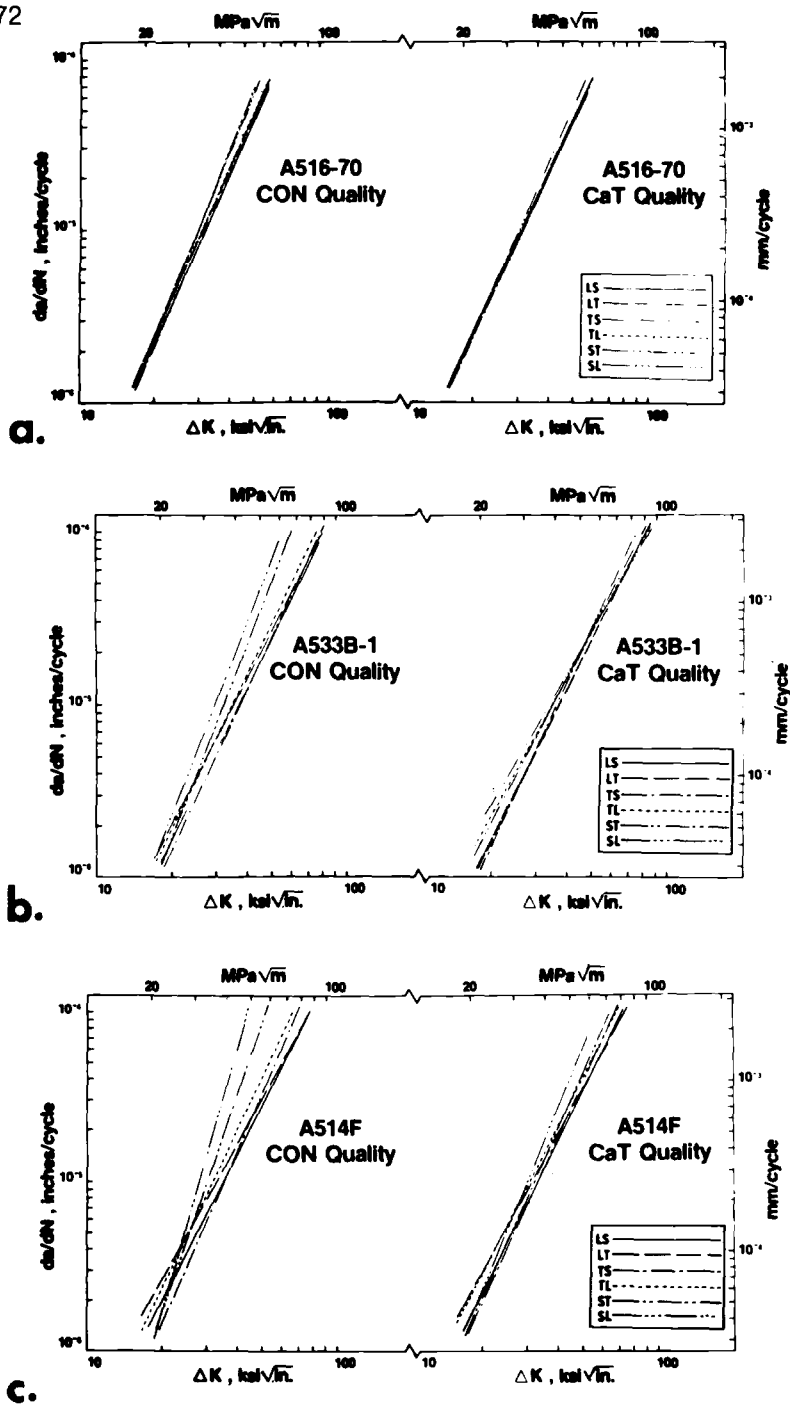


FIG. 3—Fatigue crack growth rate versus the range of stress-intensity factor best-fit lines for (a) A516 Grade 70, (b) A533 Grade B Class 1, (c) A514 Grade F. For each steel grade, results for both the CON and CaT quality levels are shown.

2.5 to 3.2 for the CaT steel. For Grade A514F the ranges were from 2.7 to 5.2 for the CON and 2.7 to 3.5 for the CaT steel. Although use of these n values alone does not describe completely the FCP behavior, these values do indicate the differences in the levels of anisotropy for the various steels.

The differences in the CON steels are greatest at the higher ΔK levels, where the through-thickness testing orientations (S-T, S-L) show faster growth rates than the other testing orientations. Also, in some instances it appears that there is a crossover in behavior because the orientations with faster da/dN levels at high ΔK have relatively slower da/dN values at the lowest ΔK levels. Furthermore, the anisotropy at all ΔK levels is reduced significantly for the CaT steels, particularly for the higher strength Grade A514F steel. This improved isotropy of the higher strength CaT steels is demonstrated more dramatically in Fig. 4. The effect of the quality level on the FCP behavior of these steels certainly is not as dramatic as the effect on other mechanical properties. For example, the level and isotropy of the CVN-USE is improved extensively for all these steels by CaT, which indicates the greater sensitivity of CVN-USE to inclusions.

It has been reported elsewhere that inclusions can increase the FCP growth rate in aluminum alloys [13-15] and steels [16-19], lead to retardation in aluminum alloys [20] or steels [9], and either retard or accelerate the FCP growth rate, as is reported here, depending on the ΔK level in aluminum alloys [21] or steels [6, 11, 22]. The work summarized here suggests that there is an additional variable contributing to the interaction of inclusions with the fatigue crack front, that is, the strength properties of the material. Fractographic examina-

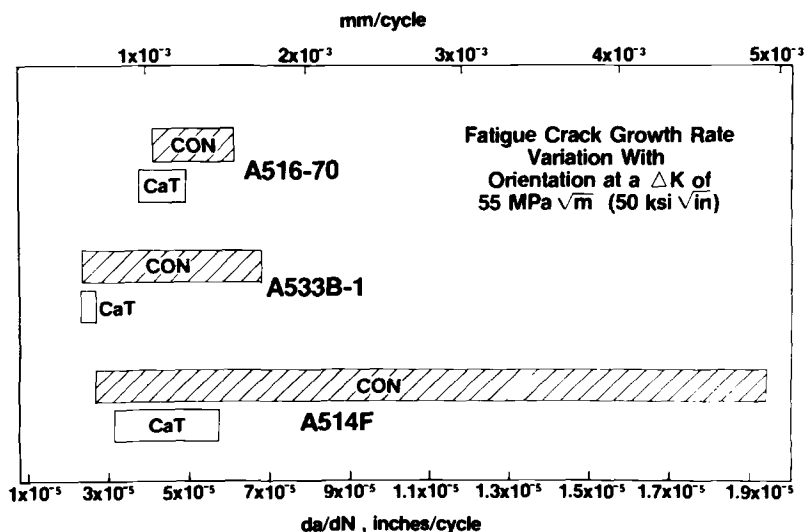


FIG. 4—Comparison of fatigue crack growth rate variation with orientation for the six plates at a ΔK level of 55 MPa $\sqrt{m}$ (50 ksi $\sqrt{in.}$).

tion of the FCP specimen fractures will provide further insight into this phenomenon.

Fractographic Characterization

The FCP fracture surfaces for the CON quality steel for all three steel grades showed more general surface roughness to the unaided eye than the CaT fractures. Although for all the FCP specimens there was increasing fracture surface roughness with increasing ΔK level, this relationship was most evident for the CON steels. Fractographic examination established that this increased roughness in the CON steels was a result of the interaction of the FCP crack front with the inclusion formations. The scanning electron microscope (SEM) was used principally in this investigation, and, except where noted, the fractures were examined at or near 0-deg tilt angle.

Through-Thickness Testing Orientations

As noted previously in Figs. 3 and 4, the CON steels displayed an apparent influence of inclusions in both the level of FCP growth rate and the anisotropy exhibited when compared with the respective CaT steels. The major influence on FCP behavior is particularly noted in the through-thickness testing orientations (S-T, S-L). On the CON quality steel, S-T and S-L fracture surfaces of all three plate steel grades, Type II MnS patches, and alumina galaxies were prominently displayed. This is indicated in Fig. 5. Their presence on the fracture surface increased with increasing ΔK level until, in the overload fracture of the specimen, inclusion formations almost covered the complete highly terraced fracture surface. The individual MnS inclusions in the Type II MnS patches may take on any of three forms, as shown in Fig. 5a-c. The MnS inclusions may be highly elongated or flattened and pancaked in appearance, or they may be small and round as a result of homogenization [23]. The Al_2O_3 galaxies are also readily identified as a collection of smooth inclusions. The orientations of both inclusion formations results from the hot rolling given the plate, as mentioned previously.

These inclusion formations are found on depressions or plateaus on the fracture surface and result in the terraced appearance of S-T and S-L fractures (see, for example, Fig. 5e). Figure 5f is a metallographic cross section of an S-L-oriented fracture that shows the MnS inclusions associated with a depression. These groups result in the faster FCP growth rates in these orientations for the CON steels because they act as local planes of weakness in the structure. Decohesion between the individual inclusions of a formation and the matrix takes place quite easily. However, the matrix material between the inclusions of a formation fractures by a conventional fatigue striation mechanism at low ΔK levels and by ductile tearing at high ΔK levels [11]. There is a variation in this behavior with the yield strength of the material, which will be discussed in a later section. Furthermore, the larger plastic zone size at higher

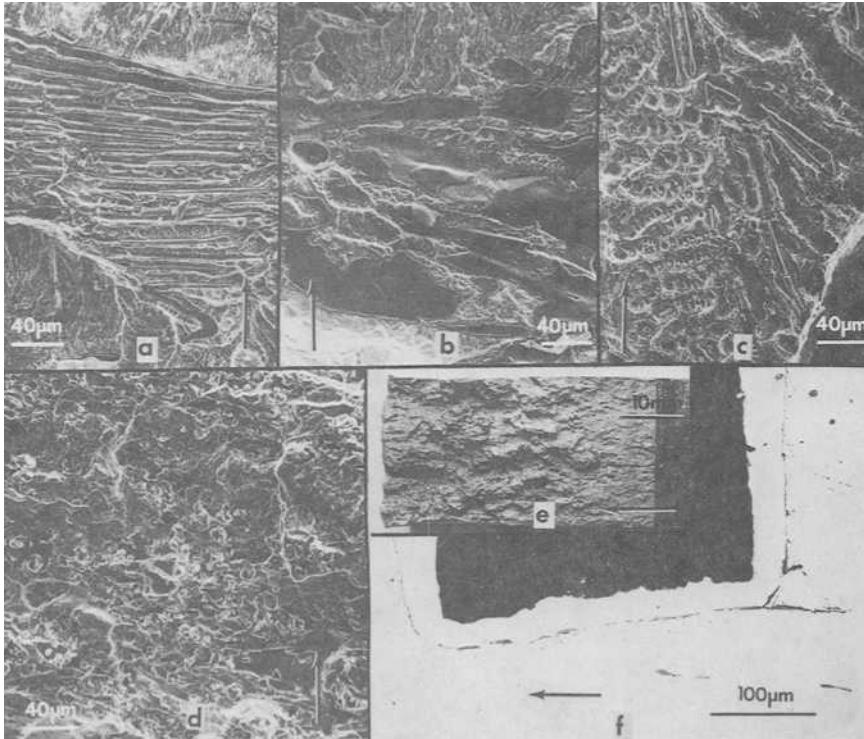


FIG. 5—Studies of CON quality and S-T and S-L-oriented Grade A533B FCP fractures showing (a) elongated, (b) flattened and pancaked, and (c) homogenized Type II MnS inclusion formations. Al_2O_3 alumina galaxies are given in (d) [(a), (b), and (d) at ΔK of $25 \text{ MPa}\sqrt{m}$ ($23 \text{ ksi}\sqrt{in.}$) and (c) at $58 \text{ MPa}\sqrt{m}$ ($53 \text{ ksi}\sqrt{in.}$)]. The terraced appearance of the S-L-oriented specimen is exhibited in (e) and (f). The direction of the macroscopic FCP is indicated by arrows.

ΔK levels gives individual portions of the crack front a larger volume of influence in which to find a plane of weakness (inclusion formation). Thus, when the complete inclusion formation fractures at the high ΔK levels, a greater effect is displayed on the overall FCP growth rate at the upper portion of the da/dN - ΔK curve.

On the S-T and S-L fracture surfaces of the CaT steels, very little inclusion interaction was found. This resulted in much smoother appearing fractures macroscopically than in the CON steels. The inclusions that were discovered in the CaT steels were normally well dispersed and smaller than those in the CON steels. Type III MnS inclusions were found only on the Grade A533B CaT fractures because of its higher sulfur level. Normally, Type III MnS inclusions are not found in CaT steels with very low sulfur levels [3]. These Type III MnS inclusions in Grade A533B were more compact and not associated in large groups, as shown in Fig. 6a. The remaining inclusions in all three steels were round, calcium-modified, duplex inclusions consisting of sulfide and

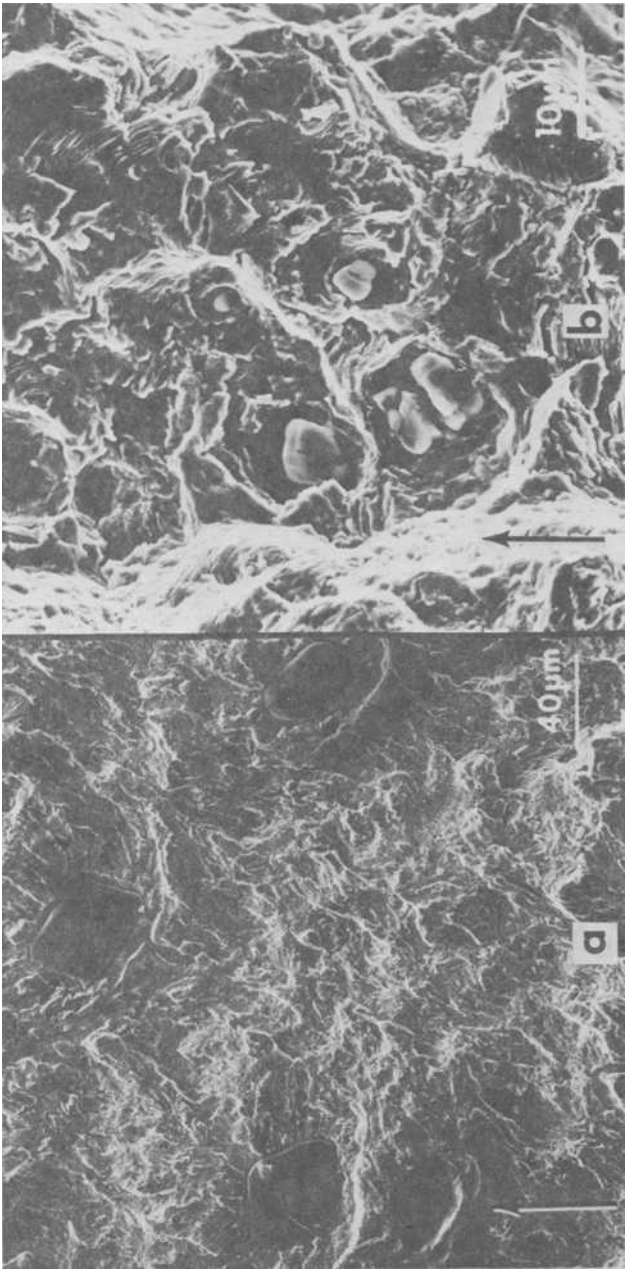


FIG. 6.—Inclusions on CaT-quality S-L-oriented FCP fractures, showing (a) Type III MnS in Grade A533B [ΔK of 26 MPa $\sqrt{m}$ (24 ksi $\sqrt{in.}$)] and (b) calcium-modified inclusions in Grade A516-70 [ΔK of 43 MPa $\sqrt{m}$ (39 ksi $\sqrt{in.}$)]. The direction of the macroscopic FCP is indicated by arrows.

aluminate phases. An example is shown in Fig. 6b. Not only the decrease in the size and number of inclusions but also the minimization of inclusion formations is responsible for the improved FCP behavior in the S-T and S-L orientations for the CaT steels.

Other Testing Orientations

The testing orientations in which the fatigue crack is propagating into the plate (L-S and T-S) also exhibited a unique interaction of the fatigue crack with the inclusion formations in the CON steels. Protrusions that were perpendicular to the direction of FCP were noted on the T-S and L-S fracture surfaces of the CON steels of all three steel grades. These protrusions have been found to result from the inclusion formations (Type II MnS and Al_2O_3) acting as crack deflectors [6, 11]. Examination of these protrusions in the SEM revealed that one or the other of these inclusion formations was present on the vertical face of the protrusion. This vertical nature of the protrusion was also confirmed using SEM stereo pairs. For example, Fig. 7 shows this behavior for both MnS and Al_2O_3 inclusion formations. The protrusions are shown in Fig. 7a on a fracture. Figure 7b shows a metallographic cross section displaying the MnS inclusions on a protrusion. Figure 7c and d examines a protrusion using the SEM. These protrusions have been shown [6, 11] to slow up the crack front because the crack front has to grow around the formation, or the formation is blunting the crack tip, or both. This accounts for the somewhat slower FCP growth rates for the L-S and T-S orientations for the CON steels at low to moderate ΔK levels. The influence of yield strength on this behavior will be discussed in a later section.

There was some appearance of inclusions on the T-S and L-S fractures of the CaT steels. However, once more there were no inclusion formations to affect the crack front, and thus there were no significant features or effects on the FCP behavior.

The standard transverse testing orientation (T-L) showed a further influence of the inclusion formations in the CON steels. The T-L fracture surfaces for the CON steels of all three steel grades showed "streaking" or elongated features, particularly at higher ΔK levels, in the direction of FCP, which also corresponds to the major direction of the hot rolling. This streaking was associated with inclusion formations that were present on the vertical face of long protrusions or depressions, which extended in the direction of FCP. This is shown in Fig. 8. This behavior did not appear to have a substantial effect on increasing the FCP growth rate for these steels in the T-L orientation. However, there is a small effect. It has been noted in steels that have seen significantly greater amounts of hot working, particularly in one direction, that the T-L direction can give faster FCP growth rates than the L-T [5, 17]. In addition, this influence has been found to increase with increased yield strength [5]. This will be discussed further in the next section.

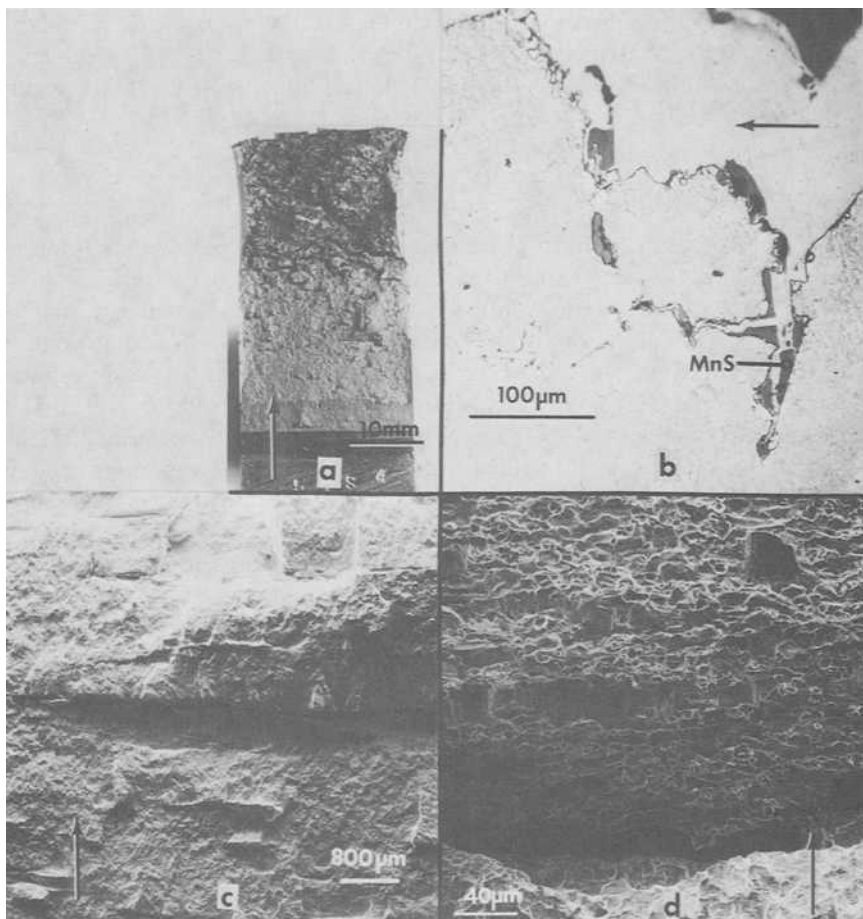


FIG. 7—Presentation of T-S orientation features for CON-quality Grade A533B FCP fracture showing protrusions associated with inclusion formations. Note in (b) a Type II MnS formation, in (d) an Al_2O_3 galaxy. A 28-deg tilt angle is used for (d) [(c) and (d) at ΔK of $40 \text{ MPa } \sqrt{m}$ ($36 \text{ ksi } \sqrt{in.}$)]. The direction of the macroscopic FCP is indicated by arrows.

As was the case for the T-S and L-S orientation, the T-L fractures for the CaT steels gave no indication of significant inclusion interaction. Also, in the L-T orientations for all of the steels, both CON and CaT, there were no significant macroscopic or SEM fracture features of note, which indicates that there was no major influence of inclusion structure in FCP behavior in this orientation.

The fracture surfaces of all the CaT steels and areas on the fracture surfaces of CON steels not near inclusion formations showed primarily ductile fatigue striation formation as the mode of fracture; that is, when the fracture took place remote from any inclusion formation, the principal mode of fatigue frac-

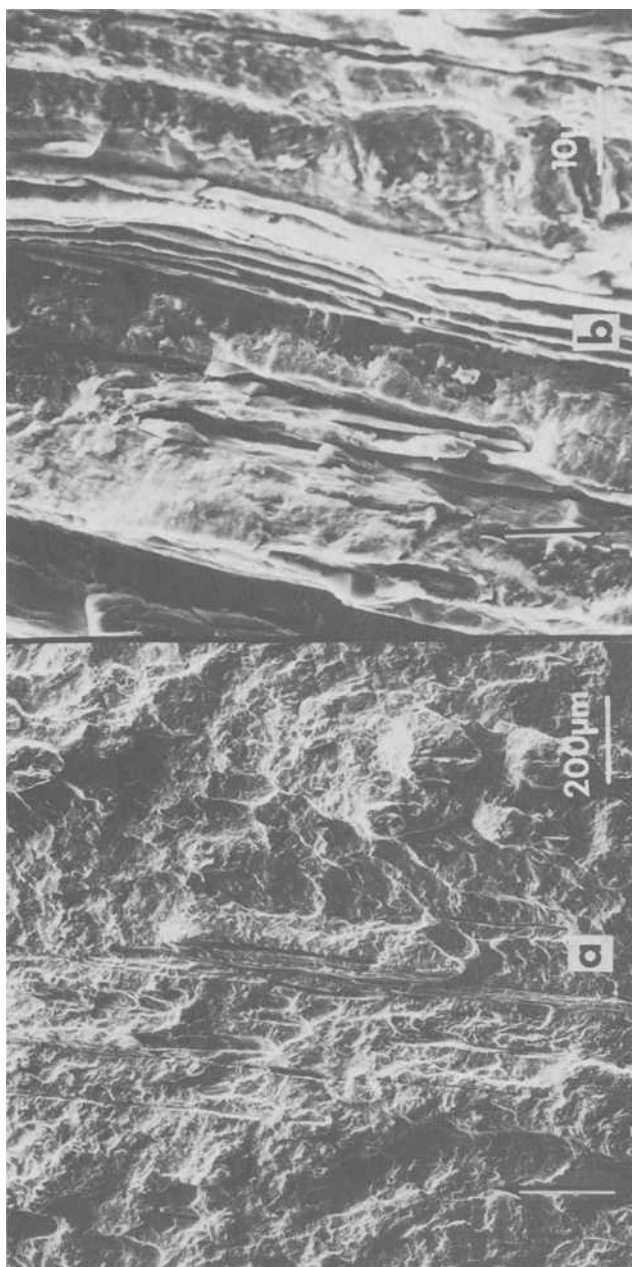


FIG. 8—Elongated features on the T-L orientation for CON-quality Grade A514F FCP fracture, showing long protrusion with Type II MnS [ΔK of 72 MPa $\sqrt{m}$ (65 ksi $\sqrt{in.}$)]. The direction of the macroscopic FCP is indicated by arrows.

ture was by ductile fatigue striation formation regardless of testing orientation or whether the steel was Grade A516 or A514F, CON or CaT quality, or at high or low ΔK levels. In addition, there was significant secondary cracking in these areas of fatigue striations. This is shown for examples of all three steels in Fig. 9.

Influence of Yield Strength

In the CON steels in the S-T and S-L, T-S and L-S, and T-L orientations, there appears to be an influence of the material strength level on the inclusion and FCP interaction. For example, the highest strength Grade A514F steel was found to have the greatest amount of FCP anisotropy among the CON steels. A method of quantifying this influence would be to establish a measure

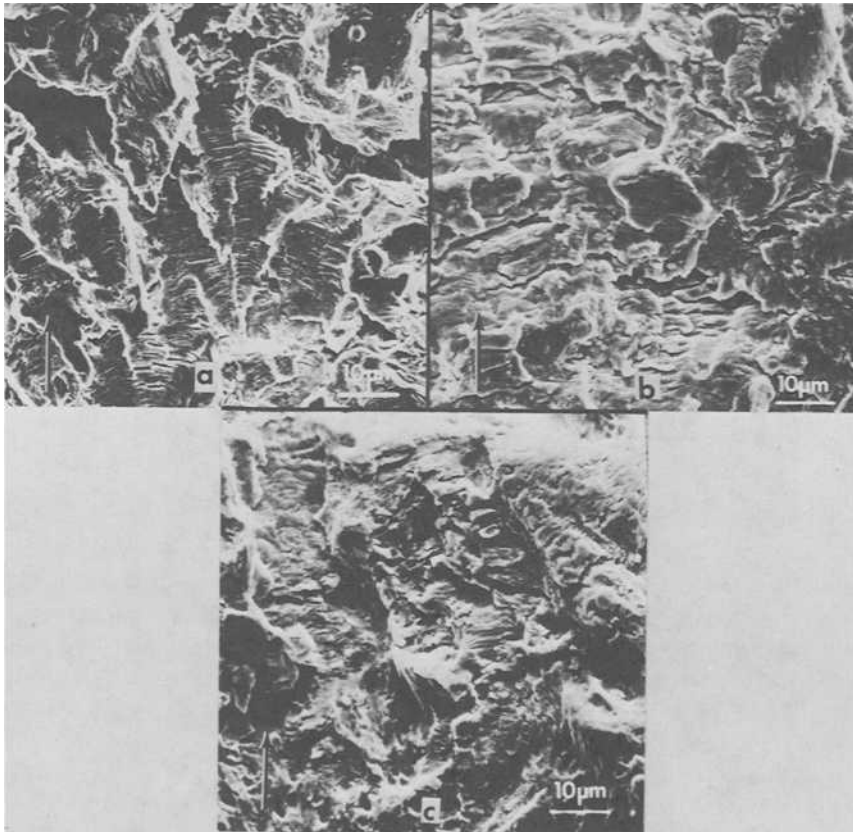


FIG. 9—Ductile fatigue striations and secondary cracking are present in areas remote from inclusions interaction: (a) Grade A516-70 CaT, S-L at ΔK of $43 \text{ MPa} \sqrt{\text{m}}$ ($39 \text{ ksi} \sqrt{\text{in.}}$); (b) Grade A533B, CON, L-T at ΔK of $46 \text{ MPa} \sqrt{\text{m}}$ ($42 \text{ ksi} \sqrt{\text{in.}}$); (c) Grade A514F, CON S-T at ΔK of $55 \text{ MPa} \sqrt{\text{m}}$ ($50 \text{ ksi} \sqrt{\text{in.}}$). The direction of the macroscopic FCP is indicated by arrows.

of the inclusion structure and determine whether a correlation exists between the specific inclusion measurement and the constants n and C_0 in Eq 1. This evaluation has been performed previously for Grades A516-70 and A533B-1 steels using quantitative image analysis [7, 11]. And, in fact, the higher sensitivity to inclusions of Grade A533B than of Grade A516 was established. A parameter that measures the average area of an inclusion on the metallographic cross section corresponding to the FCP fracture plane was found to provide the best correlation. However, these analyses could not quantify the group nature in which the inclusions exist and affect FCP behavior. Also, they could not account for the three-dimensional way in which the FCP crack front acts in the presence of these inclusion formations.

Furthermore, from the standpoint of comparing different steel grades and inclusion structures, other variables can also influence the inclusion structure besides the steel-making practice used [4]. For example, the amount of rolling reduction given the plate (from ingot or continuously cast slab to plate thickness), the relationship between the amount of rolling done in the longitudinal and in the transverse directions, and the ingot position (top versus bottom) of the plate all can influence the inclusion structure additionally. Fortunately, by studying the fracture surfaces in some detail a potential mechanism to explain the influence of yield strength was developed.

In all of the CON steel grades, it was noted that the individual inclusions separated from the steel matrix without much apparent difficulty. This indicates that the involvement of the inclusion formations themselves in the FCP process and the subsequent influence of yield strength appear to be due to the behavior of the steel matrix material between inclusions of a formation (inter-inclusion material). Examination of the fracture behavior of this interinclusion material in all three steels revealed differences between the steels.

Through-Thickness Testing Orientations

The S-L fractures of the CON steels of each of the three steel grades were examined at ΔK levels of 22 and 55 MPa $\sqrt{\text{m}}$ (20 and 50 ksi $\sqrt{\text{in.}}$). In the Grades A533B and A514F steels at the low ΔK level, there was fatigue damage by striation formation of the interinclusion material of an inclusion formation. At the higher ΔK level this interinclusion material was fractured by ductile fracture (microvoid coalescence). However, in the Grade A516 material there was fatigue fracture by striation formation of the interinclusion material at both ΔK levels. This comparison is demonstrated to some degree in Fig. 10 for Grades A516 and A514F. The fracture mode away from the inclusion formations in all three CON steels, at both ΔK levels, was still by fatigue striation formation, as mentioned previously and shown in Fig. 9c in particular. Furthermore, the fracture mode for all three steels in the CaT quality level at both ΔK levels was also by fatigue striation formation.

These results led to the following hypothesis regarding the FCP behavior of

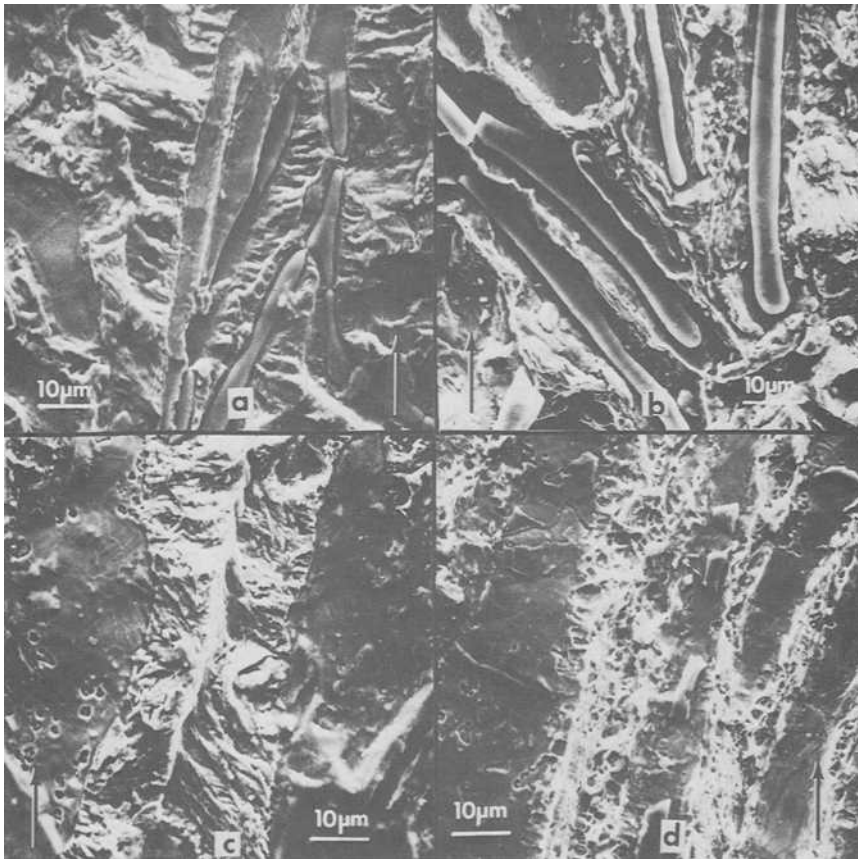


FIG. 10—Comparison of the fracture mode of interinclusion material of inclusion formation for S-L-oriented FCP fractures of Grade A516-70 at ΔK levels of (a) $22 \text{ MPa} \sqrt{\text{m}}$ ($20 \text{ ksi} \sqrt{\text{in.}}$), (b) $55 \text{ MPa} \sqrt{\text{m}}$ ($50 \text{ ksi} \sqrt{\text{in.}}$), and for Grade A514F at ΔK levels of (c) $22 \text{ MPa} \sqrt{\text{m}}$ ($20 \text{ ksi} \sqrt{\text{in.}}$) and (d) $55 \text{ MPa} \sqrt{\text{m}}$ ($50 \text{ ksi} \sqrt{\text{in.}}$).

these steels. At low ΔK levels, the fatigue cracks in all three CON steels approach an inclusion formation and propagate through the formation at a rate primarily controlled by the ductile fatigue striation mechanism that occurs at the interinclusion material (the inclusion–matrix interface offers very little resistance). Thus there is a small effect of inclusions at the low ΔK levels in the S-T and S-L orientation. At some higher ΔK level, a low-strength steel continues to have substantial propagation in the interinclusion material by the fatigue striation mechanism. At the same ΔK level, a higher strength steel appears to have “bursts” of ductile tearing, which allows more crack advance per cycle, and thus leads to faster overall FCP growth rates. This results because a higher strength steel tends to have matrix material of inherently lower toughness. Also, a higher-yield-strength steel requires a smaller plastic strain

to initiate voids at particles (carbides in this case), and thus fracture can occur at lower strains [24]. Therefore, these "bursts" of ductile fracture can occur at lower ΔK levels in a higher strength steel than in a lower strength steel, thus exposing the complete inclusion formation sooner and leading to faster FCP growth rates. Although these fractured inclusion formations do not completely cover the fatigue fracture surface, even at higher ΔK levels, their fracture gives local accelerations to the crack front. This leads to a general increase in growth rate for the main fatigue crack front.

The difference between the S-T and S-L orientations may also be explained by this mechanism. The S-L orientations showed faster growth rates because the interinclusion material between the elongated MnS inclusions can be fractured more easily with an S-L orientation than with an S-T one. In the S-L orientation, the effective fracture zone is bounded by the distance between two adjacent elongated MnS inclusions (which have easily separated from the matrix), whereas in the S-T case this zone may be as wide as a single elongated MnS inclusion. In both cases the depth of the effective fracture zone is controlled by the crack-tip plastic zone. Thus, the interinclusion material for the S-L-oriented specimen should fracture more easily and in greater amounts per cycle, which results in the faster FCP growth rates. This is demonstrated in Fig. 11.

Other Testing Orientations

A similar approach to that just described for the through-thickness specimens helps to explain the influence of yield strength in T-L-oriented specimens. In this case, the exposure of the complete inclusion formation does not have quite the same effect on the FCP growth rate. The pancake shape of the formation results in a larger effect on the S-T and S-L orientations, whereas the influence on the T-L orientation is due to the thickness or three-dimensional nature of the inclusion formations (thickness of the composite pancake).

An additional contribution to the phenomenon of a higher strength steel showing a greater sensitivity to inclusions is a result of such a steel's ability to maintain a higher degree of triaxial constraint at high ΔK levels. It has been established that increased constraint leads to faster void growth rates [25, 26], which contributes additionally to the ductile tearing of the interinclusion material of an inclusion formation in a higher-yield strength steel at the higher ΔK levels.

The increased retardation of FCP growth rates in the L-S and T-S orientations for a higher strength material is probably due to slightly different factors than those pertaining to the other orientations. A higher strength material will tend to have a smaller crack-tip plastic zone and crack-tip opening displacement at any given ΔK level. Thus, a specific inclusion formation will more effectively blunt the crack tip and will make it more difficult for the crack front

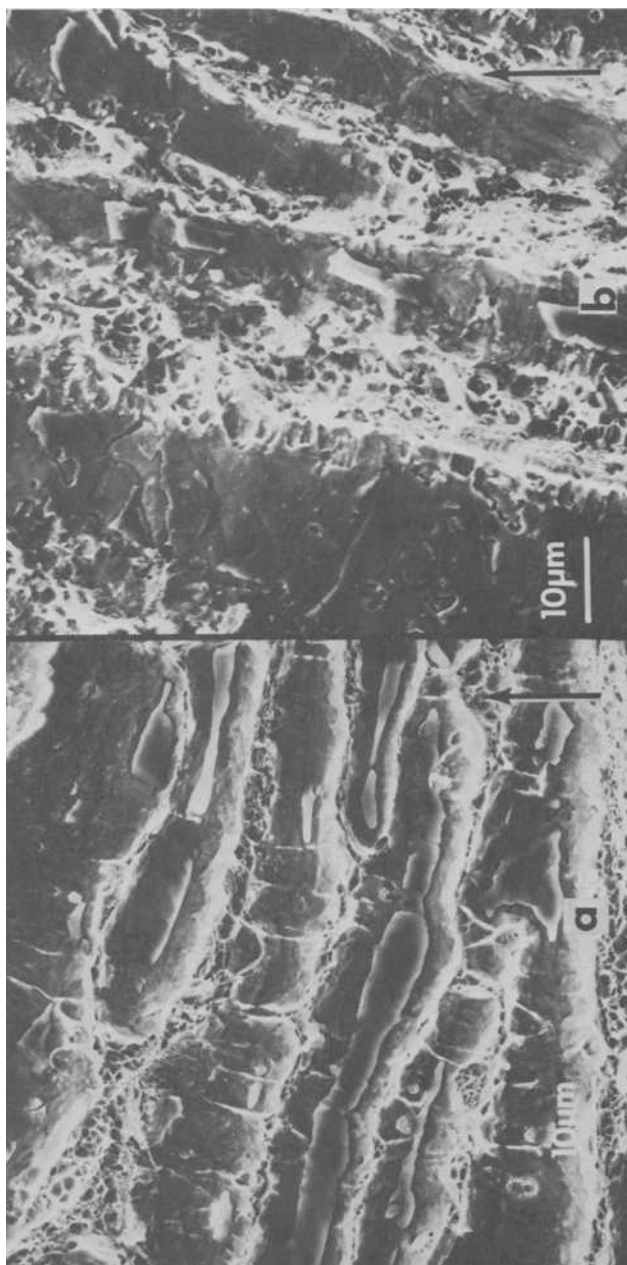


FIG. 11—Comparison of a ductile fracture of the interinclusion material in Type II MnS inclusion formation for (a) S-T and (b) S-L orientations for CON-quality Grade A514F FCP fractures [ΔK of 55 MPa $\sqrt{m}$ (50 ksi $\sqrt{in.}$)]. The direction of the macroscopic FCP is indicated by arrows.

to continue propagation around the formation. The low-strength steel already would have larger crack-tip dimensions and thus would not be as affected (blunted) by an inclusion formation of the same size. At higher ΔK levels, a high-strength steel will also have increased crack-tip dimensions, so that the formations can also be easily bypassed and will not influence the FCP growth rate in this fashion.

Conclusions

1. Calcium-treated (CaT) steels are generally found to have better resistance to fatigue crack propagation (FCP) than conventionally processed (CON) steels. The amount of improvement is found to increase with the yield strength level, that is, Grade A514F shows a greater enhancement than Grade A533B, which in turn shows more than Grade A516-70.

2. The improvement in FCP resistance is shown primarily at higher ΔK levels in the through-thickness testing orientation (S-T, S-L) for the CaT in comparison with the CON steels. Also, more anisotropy of FCP is exhibited in the CON steels.

3. The FCP differences between the CON and CaT steels are due to the presence of inclusion formations (Type II MnS and Al_2O_3 galaxies) in the CON steels. It is the inclusion formations rather than individual inclusions that are the principal cause of this phenomenon.

4. The influence of the yield strength level on the inclusion interaction with FCP appears to result from its effect on the mode of fracture of the steel matrix material between inclusions within a specific inclusion formation (interinclusion material).

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Effect of Compressive Loading on Fatigue Crack Growth Rate and Striation Spacing in Type 2219-T851 Aluminum Alloy

REFERENCE: Albertin, L. and Hudak, S. J., Jr., "Effect of Compressive Loading on Fatigue Crack Growth Rate and Striation Spacing in Type 2219-T851 Aluminum Alloy," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 187-201.

ABSTRACT: Macroscopic crack growth rates, da/dN , have been obtained as a function of the stress-intensity factor range, ΔK , for Type 2219-T851 aluminum in ambient air at load ratios, R , of 0.1 and -1 . Microscopic modes of crack extension were also determined by transmission electron fractography. Fatigue striation spacings, $\bar{S}$, compared well with da/dN when static fracture modes—primarily dimpled rupture—did not occur. These static fracture modes were more prevalent during tension-tension loading at $R = 0.1$ than during tension-compression loading at $R = -1$, and they contributed to higher growth rates at the positive load ratio. The relationship between striation spacing and $\Delta K/E$ (where E is the elastic modulus) in Type 2219-T851 aluminum was found to be similar to those for other engineering alloys. This relationship was observed to be much less sensitive to changes in the load ratio than the relationship between da/dN and ΔK .

KEY WORDS: fatigue, cracks, growth, fractography, striation, failures, analysis of structure, ratios, aluminum 2219, materials, materials science

It is now generally accepted that the crack-tip stress-intensity factor, defined by linear elasticity, provides a geometry-independent parameter that represents the mechanical driving force for fatigue crack growth. Over the past 10 years, since the initial contribution of Paris [1],² much crack growth rate data have been acquired which support the relationship between the amount of crack extension per cycle of fatigue, da/dN , and the range of the stress-intensity factor, ΔK . Furthermore, for most materials there exist da/dN regimes in which the growth rates are strongly dependent on the mean stress,

¹Senior engineer and senior engineer, respectively, Westinghouse Research and Development Center, Pittsburgh, Pa. 15235.

²The italic numbers in brackets refer to the list of references appended to this paper.

which is usually expressed in terms of the load ratio (R = minimum load/-maximum load). Thus, excluding temperature or environmental effects, the following general equation applies

$$da/dN = f(\Delta K, R) \quad (1)$$

Currently, there exists a multitude of formulations for the function, f , although most are limited to either a given material or a particular range of growth rates [2].

Parallel fractographic studies have focused on characterizing the micro-morphology of crack growth in an attempt to understand how cracks actually advance under fatigue loading.

Forsyth [3] and Pelloux [4] showed a direct correlation between the incidence of striations on the fracture surface and the number of fatigue cycles, and they related the size of the striation to the amplitude of the applied load. Hertzberg [5] and Bates [6] correlated the fatigue striation spacings in the observable range of 0.025 to 2.5 $\mu\text{m}/\text{cycle}$ to the macroscopic growth rate on various materials.

Although the macroscopic crack growth rates, expressed as a function of ΔK , differ for metals such as steel, titanium, and aluminum, their crack growth rates can be normalized to a large extent by dividing the stress-intensity range by the materials' respective moduli of elasticity [7]. Using this approach, Bates and Clark [6] showed that the size of the fatigue striations in several engineering metal alloys could be related to the range of the stress-intensity factor when normalized by the modulus of elasticity, as shown in Eq 2

$$\text{striation spacing, } S = 6 \left(\frac{\Delta K}{E} \right)^2 \quad (2)$$

In a similar fashion, Hahn [8] recently analyzed the data available on the fatigue crack propagation rates of a wide variety of steels and concluded that the growth rates can be given by the expression

$$da/dN = 8 \left(\frac{\Delta K}{E} \right)^2 \quad (3)$$

Equations such as Eqs 2 and 3 are useful in failure analysis where fatigue striation measurements can be obtained from the fracture surface of the failed component. If the size of the fatigue striations and the modulus of elasticity of the material are known, it is possible to estimate the stress-intensity range. Although Eq 2 was developed from data obtained at $R = 0$, it was found by Bates [9] to be valid for Type 403 stainless steel tested under several positive load ratios (that is, positive mean stresses). Hertzberg [10] modified Eq 2 to account for the dependence of da/dN on R in Type 2024-T3 aluminum through the application of the crack closure concept. The striation spacing in Type

2024-T3 aluminum was shown to be a function of the effective stress-intensity level, as in the equation

$$S \approx 24 \left(\frac{\Delta K_{\text{eff}}}{E} \right)^2 \quad (4)$$

Using the effective rather than the applied stress-intensity range allowed a better correlation of data from several sources and a variety of load ratios. Since Frost [11] showed that different engineering materials possess different mean stress sensitivities, it is necessary to examine additional material for striation spacing relationships with mean stress.

Most crack growth rate studies and correlations between fatigue striation spacing and the stress intensity range are carried out with $R \geq 0$, that is, for zero-tension or tension-tension loading. The principal objective of this investigation was to determine how the crack growth rate is affected by a loading sequence in which the alternating stress changes from compression to tension, specifically for $R = -1$. For comparison purposes, we also included a test specimen in which the load ratio was nearly zero. We have chosen for this study the aluminum alloy 2219-T851—a widely used weldable aluminum alloy that most likely would produce fatigue striations observable in the transmission electron microscope. The purpose of the study was to determine the relationship between the fatigue striation spacing and $\Delta K/E$ and to establish whether the macroscopic fatigue crack growth rate in this aluminum alloy is directly related to the fatigue striation spacing.

Experimental Procedure

Material

The aluminum alloy 2219-T851 was supplied as an 81-mm thick plate. The chemical composition and mechanical properties are shown in Tables 1 and 2. Both the chemistry and mechanical properties are typical of commercially produced 80-mm thick plate, and the latter properties are uniform in the longitudinal and long transverse directions.

Fatigue Crack Growth Rate Measurement

All fatigue crack growth rate tests were conducted in accordance with the ASTM Test for Constant-Load-Amplitude Fatigue Crack Growth Rates Above 10^{-8} m/Cycle (E 647-78T) [12]. Both 50.8-mm-wide compact type (CT) specimens (Fig. 1) and 76-mm-wide center cracked tension (CCT) specimens (Fig. 2) were employed. All specimens were nominally 6.4 mm thick and contained cracks in the longitudinal-transverse (L-T) orientation. The CT specimen was used for tests at $R = 0.1$, and the CCT specimen for tests at $R = -1$. The CCT specimen was clamped using a bolt and keyway assembly to enable

TABLE 1—*Chemical composition of Type 2219-T851 plate, weight percent.*

Cu	Mn	Fe	Si	Zn	Ti	Mg	Al
6.28	0.25	0.25	0.088	0.025	0.051	0.003	balance

backlash-free tension-compression loading. Previous work has verified the suitability of this particular specimen and gripping fixture for tension-compression loading [13].

Stress-intensity factors were calculated using the expressions provided in ASTM Test E 647-78T [12]. The value of ΔK for the test at $R = -1$ was computed using only the positive portion of the loading cycles.

Replicate fatigue tests were conducted with servo-hydraulic test equipment operated under constant amplitude loading in load control. A sinusoidal waveform was used at a frequency of 5 Hz. The load accuracy at this frequency was better than 2 percent.

Fatigue precracks were extended at least 2.5 mm from the machined notch to preclude interactions between the notch and crack-tip stress-strain fields. All precracking was conducted at $R = 0.1$, and the final $K_{\max}$ values were 60 percent of those used during subsequent testing.

Crack length was measured visually on both sides of the specimen at intervals of 0.5 mm with the aid of scribed lines on polished specimen surfaces and a $\times 30$ traveling microscope. With this technique, crack length could be determined within 0.05 mm. The average crack length and corresponding elapsed fatigue cycles were subsequently analyzed, using a 7-point incremental polynomial technique [13, 14].

Fractographic Measurements

Fractographic examinations were conducted on each of the fatigue fractures using two-stage cellulose acetate-carbon replicas shadowed with platinum carbon. Grids were attached to the cellulose acetate strip at intervals of approximately 4 mm, as shown in Fig. 3, with a scale attached. The shadowed carbon replicas (grids) were then examined using an electron microscope.

TABLE 2—*Room temperature mechanical properties of Type 2219-T851 plate.*

Orientation	0.2% σ_{ys} , MPa	σ_{ult} , MPa	% Elongation, (in 50 mm)	% Red in Area
Longitudinal	358	455	8.5	19
Long transverse	352	455	8.2	19

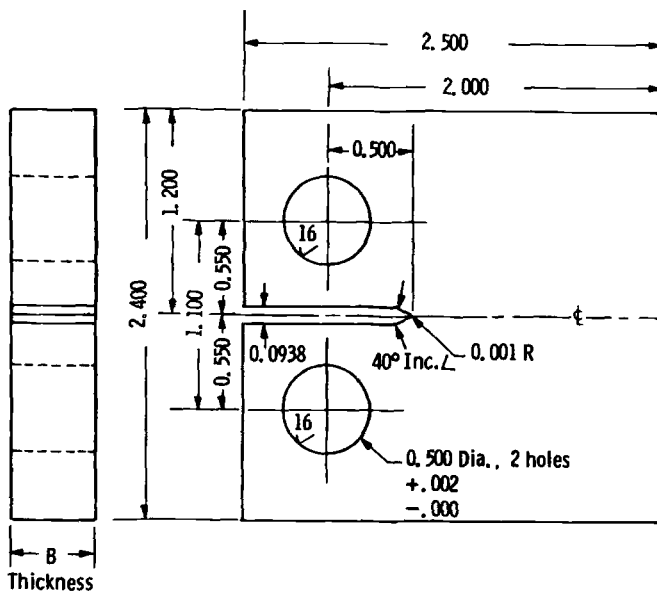


FIG. 1—A compact-type fatigue crack growth specimen for tension-tension (R greater than 0) testing. Note: the dimensions are in inches (1 in. = 2.54 cm).

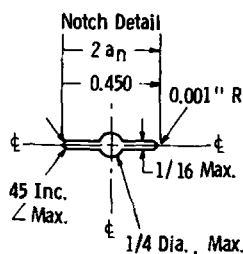
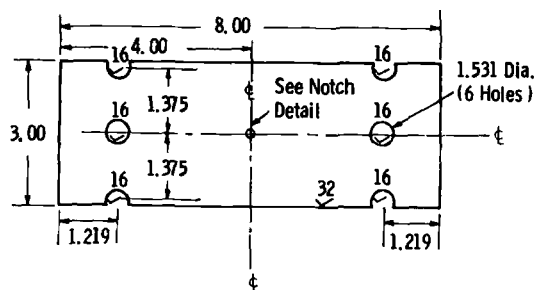


FIG. 2—A center-cracked tension (CCT) specimen for tension compression (R less than 0) fatigue testing. Note: the dimensions are in inches (1 in. = 2.54 cm).

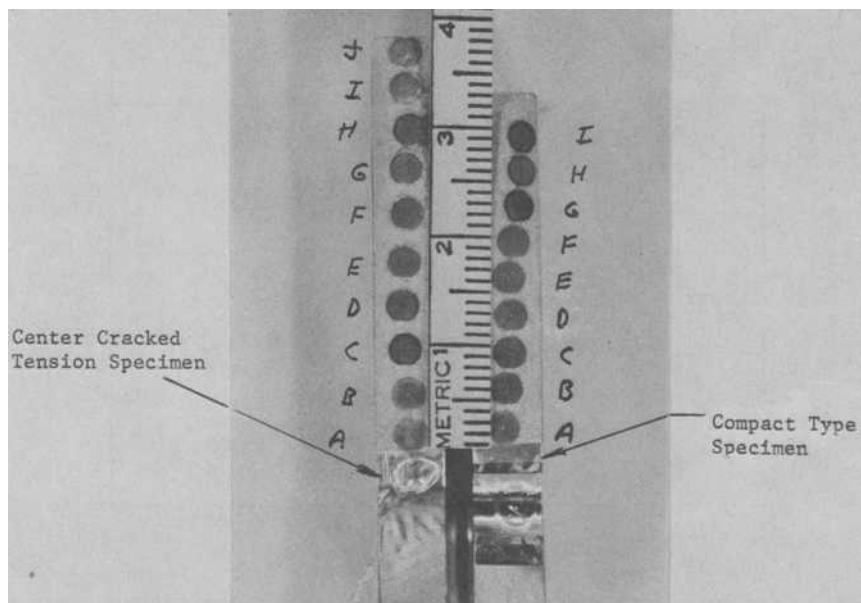


FIG. 3—Crack growth fracture mechanics specimens, showing typical grid locations.

Striated areas that appeared relatively flat and free of artifacts were selected and photographed so that the striation spacing could be measured. At least ten measurements of the striation spacing were made on each grid. The typical variation in these results was ± 50 percent.

Experimental Results and Discussion

Average striation spacings for several grid locations on specimens tested at $R = 0.1$ and $R = -1$ are given in Table 3. Corresponding values are also given for da/dN , nominal crack length, and $\Delta K/E$; the modulus of elasticity, E , is 72×10^4 MPa for Type 2219-T851 aluminum.

General Fractographic Observations

Typical fractographs illustrating general topographic features at various da/dN and ΔK values are provided in Figs. 4 through 6. Fracture surfaces at $R = -1$ contained regions whose primary features were obliterated by rubbing during the compression portion of the loading cycle. However, contrary to expectations, better defined striations were observed at $R = -1$ than at $R = 0.1$.

At growth rates below $0.025 \mu\text{m}/\text{cycle}$, the fractography consisted of cleavage-like facets with no detectable striations. This observation is in agreement with results on a variety of other structural alloys [15]. Nevertheless, since

TABLE 3—Fatigue crack growth rates and striation spacings versus ΔK in Type 2219-T851 aluminum plate tested at R-ratios of 0.1 and -1.^a

Crack Length, a , in.	$R = 0.1$ (CT specimen)				$R = -1$ (CT specimen)			
	ΔK_I , ksi $\sqrt{\text{in.}}$	$\Delta K/E_c$, $10^{-3} \sqrt{\text{in.}}$	da/dN , $\mu\text{in./cycle}$	Striation Spacing, $\mu\text{in.}$	Half Crack Length, a , in.	ΔK_I , ksi $\sqrt{\text{in.}}$	$\Delta K/E_c$, $10^{-3} \sqrt{\text{in.}}$	da/dN , $\mu\text{in./cycle}$
0.52	9.28	0.088	5.3	cleavage-like	0.43	11.4	1.075	5.0
0.72				4.6	0.57	13.7	1.294	8.3
0.87	11.3	1.064	11.5	13.2	0.62	14.5	1.368	10.3
1.05	14.6	1.377	26.0	12.2	0.87	19.7	1.856	23.0
1.37	28.0	2.615	1500.0	59.0	1.03	24.6	2.320	42.0
1.53				dimples (100%)	1.19	32.8	3.092	60.0

^a Metric conversion factors:1 ksi $\sqrt{\text{in.}} = 1.1 \text{ MPa}$ $E = 72 \times 10^6 \text{ MPa}$

1 in. = 25.4 mm

1 $\mu\text{in.} = 0.025 \text{ }\mu\text{m}$

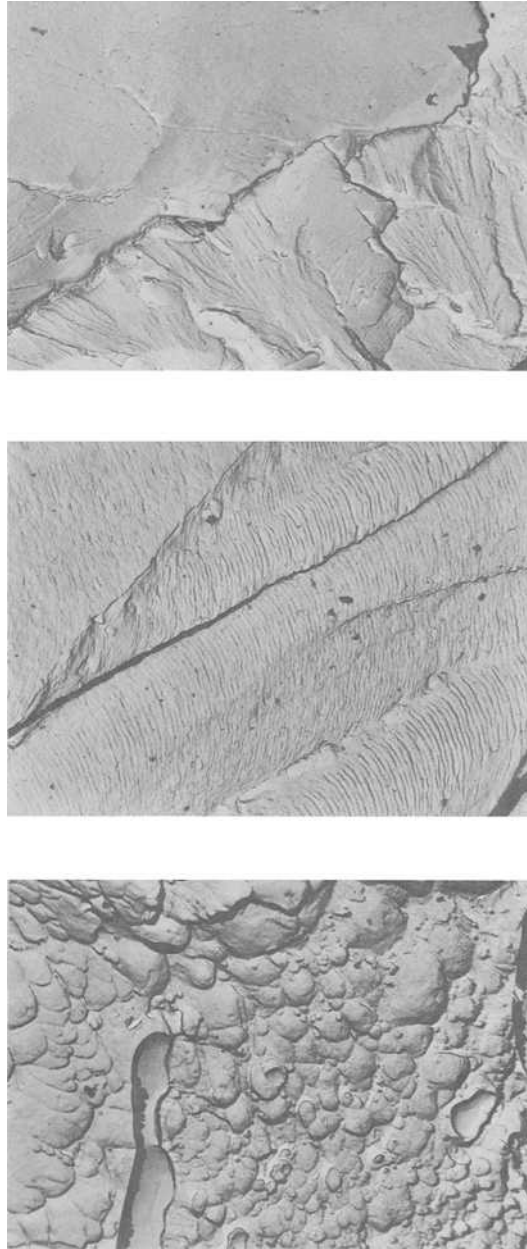


FIG. 4—Fracture features in Type 2219-T851 aluminum tested in fatigue at $R = 0.1$. (Top) Cleavage-like facets seen in the low fatigue crack growth regimen (da/dn less than $0.025 \mu\text{m}$ per cycle, magnification $\times 1000$); (middle) completely striated fracture at $\Delta K = 16 \text{ MPa} \sqrt{\text{m}}$ (da/dn larger than $66 \mu\text{m}$ per cycle, magnification $\times 2240$); (bottom) dimples rupture K greater than $33 \text{ MPa} \sqrt{\text{m}}$ (da/dn larger than $50 \mu\text{m}$ per cycle, magnification $\times 4640$).

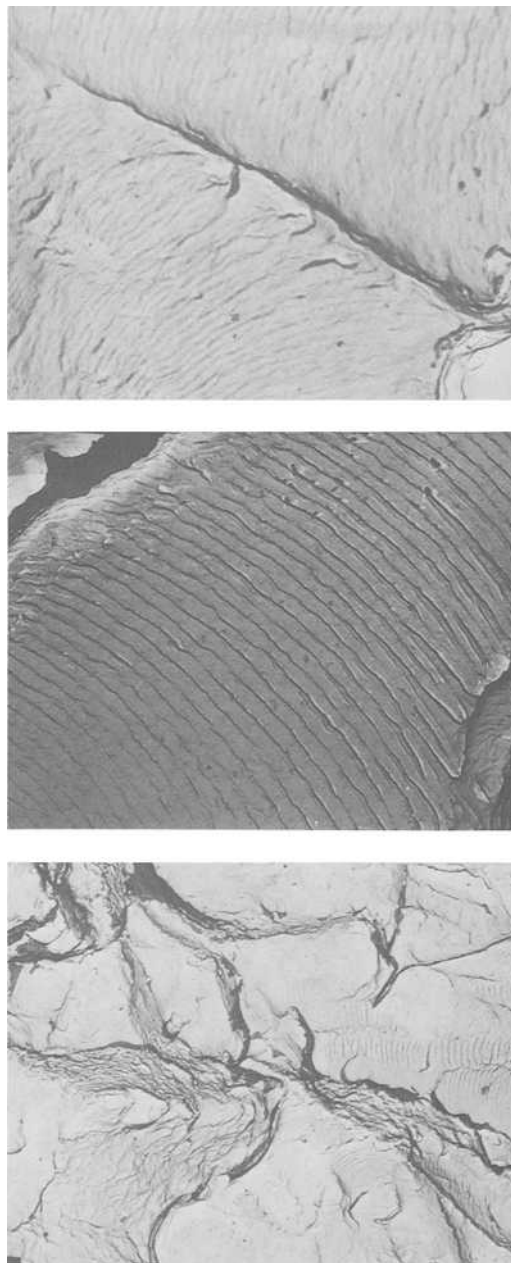


FIG. 5—Fracture features in Type 2219-T851 aluminum tested in fatigue at $R = -1$: (top) striated fracture at $\Delta K = 15 \text{ MPa}\sqrt{\text{m}}$ ($da/dn = 0.21 \mu\text{m}$ per cycle, magnification $\times 6000$); (middle) striated fracture at $\Delta K = 22 \text{ MPa}\sqrt{\text{m}}$ ($da/dn = 58 \mu\text{m}$ per cycle, magnification $\times 3600$); (bottom) mixed fracture consisting of dimples and striations at $K = 28 \text{ MPa}$ ($da/dn = 1.07 \mu\text{m}$ per cycle, magnification $\times 1000$).

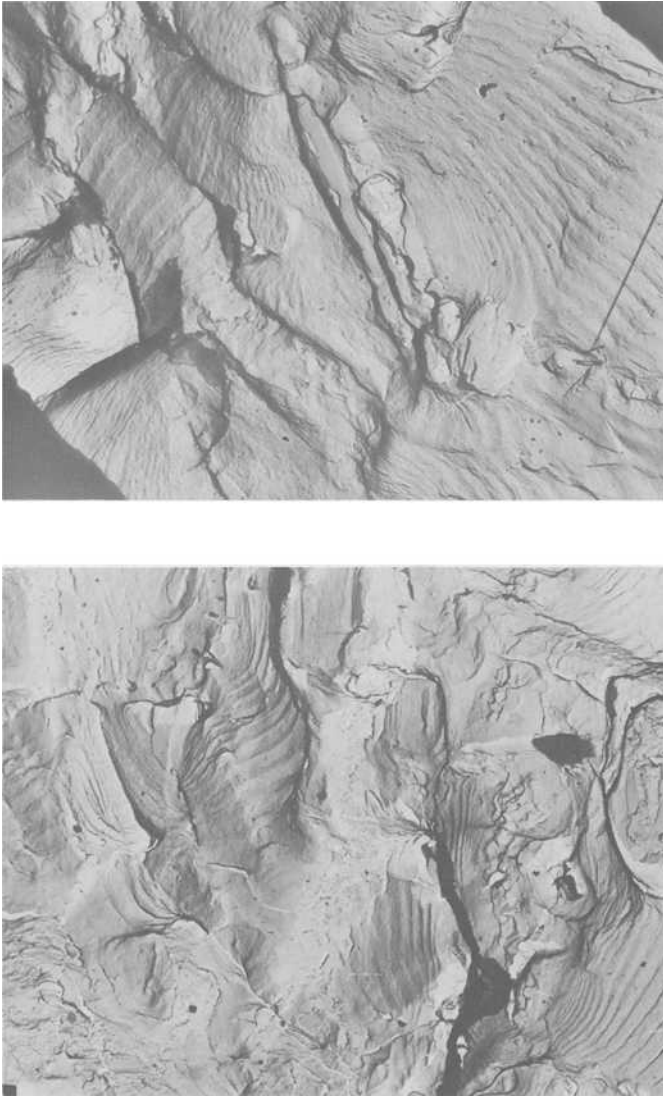


FIG. 6—Fracture features in Type 2219-T851 aluminum tested in fatigue at R values of 0.1 and -1 , showing the complex nature of crack growth in some areas: (top) $R = 0.1$, magnification $\times 1250$; (bottom) $R = -1$, magnification $\times 1500$.

$0.025\ \mu\text{m}$ is estimated to be near the resolution limit of fractographic measurements, it cannot be concluded that the striated growth mechanism is inoperative at these low growth rates.

As illustrated in Figs. 4 (*middle*) and 5 (*middle*), striations were clearly observable at higher growth rates and at both load ratios. Striations with increasing amounts of dimpled rupture became prominent as growth rates increased

(for an example, see Fig. 5 *bottom*). Although this trend occurred at both load ratios, growth at a given value of ΔK at $R = 0.1$ was accompanied by a significantly larger volume fraction of dimpled rupture than was growth at $R = -1$. In fact, growth rates above $50 \mu\text{m}/\text{cycle}$ at $R = 0.1$ corresponds to 100 percent dimpled rupture, as illustrated in Fig. 4 (*bottom*). This morphology is characteristic of failure under monotonic loading.

Figure 6 illustrates the complex nature of the microscopic cracking path and attests to the need for adequate sampling in order to obtain representative average striation spacings.

Comparison of Growth Rates and Striation Spacings

A comparison of the macroscopic fatigue crack growth rates, da/dN , and the microscopic striation spacings, S , is provided in Fig. 7. For the lower growth rates, relatively good agreement occurs between da/dN and S at both R values. As ΔK increases, the da/dN values become increasingly larger than S , particularly at $R = 0.1$. This divergence of microscopic and macroscopic results coincides with the development of increasing amounts of dimpled rupturing. The larger volume fraction of dimple rupturing, which occurs at $R = 0.1$, appears to be the cause of the higher growth rates at $R = 0.1$ than at $R = -1$.

Similar effects of static mode fracturing on fatigue crack growth under tension-tension loading have been reported in other materials, although the specific static mode that operates is dictated by the combination of material and test condition. For example, in mild steel growth rates are insensitive to mean stress when propagation is by ductile striations. However, growth rates became markedly sensitive to mean stress in the presence of static fracture modes [16]. Either dimple rupturing or cleavage can occur, depending upon whether fatiguing at high ΔK values is carried out above or below the ductile-brittle transition temperature, respectively [17]. Griffith et al [18] have also shown that growth rates in ferritic weld metal are sensitive to mean stress when propagation is enhanced by microvoid coalescence. The presence of intergranular cracking can cause similar effects, as has been demonstrated by Ritchie and Knott in a temper-embrittled low-alloy steel [19].

Thus, it appears that the increased sensitivity of fatigue crack growth rates to mean stress is due to the operation of a variety of static modes of subcritical cracking that supplement the classic fatigue-striated growth.³ These effects, to a large extent, are controlled by the fracture toughness of the materials, as is evidenced by the fact that high-strength, low-toughness aluminum alloys and steels exhibit significantly larger stress-ratio effects than do low-strength, high-toughness aluminum alloys and steels [13,20-26].

One interesting aspect of the current investigation is the influence of compressive loading on both the fatigue crack growth rates and the micromor-

³ However, it should also be noted that stress ratio effects at low and intermediate growth rates are likely to be controlled by completely different processes, as is discussed in Refs 13 and 21.

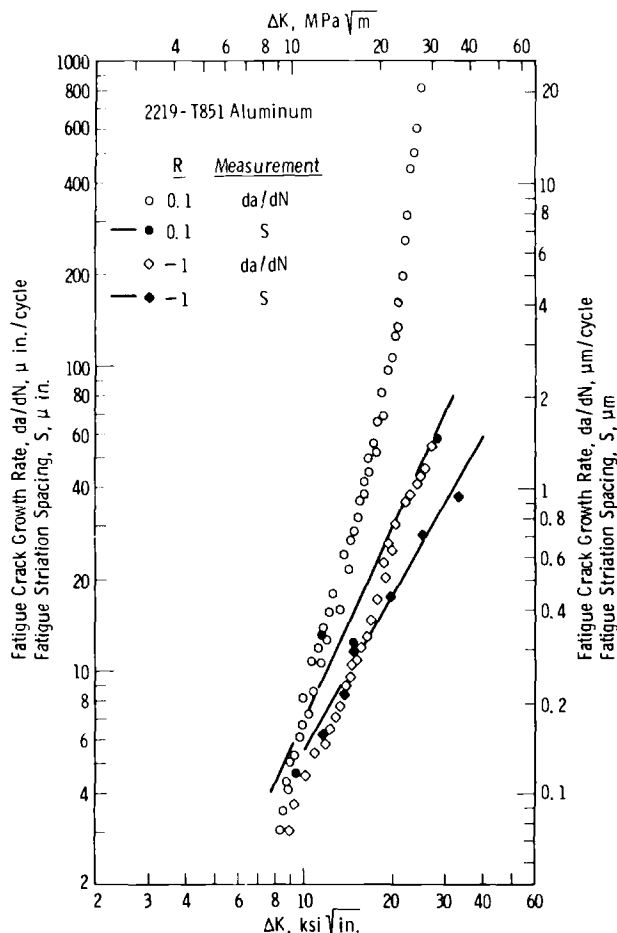


FIG. 7—Comparison of microscopic fatigue striations spacing and macroscopic fatigue crack growth rates in Type 2219-T851 aluminum at load ratios, R , of 0.1 and -1 .

phology of crack growth. At high ΔK values, the slower growth rates at $R = -1$ (Fig. 7) appear to be caused by a suppression of the static component of subcritical cracking, which is characterized by dimple rupturing. But even at intermediate and low ΔK values, where fatigue striations predominate, da/dN at $R = -1$ is less than that at $R = 0.1$. This behavior perhaps can be explained by the crack-closure model proposed by Elber [27]. According to Elber

$$da/dN = A(\Delta K_{\text{eff}})^n A (U\Delta K)^n \quad (5)$$

where the crack-closure parameter, U , is assumed to be dependent on R only and is empirically given by

$$U = 0.5 + 0.4R \quad (6)$$

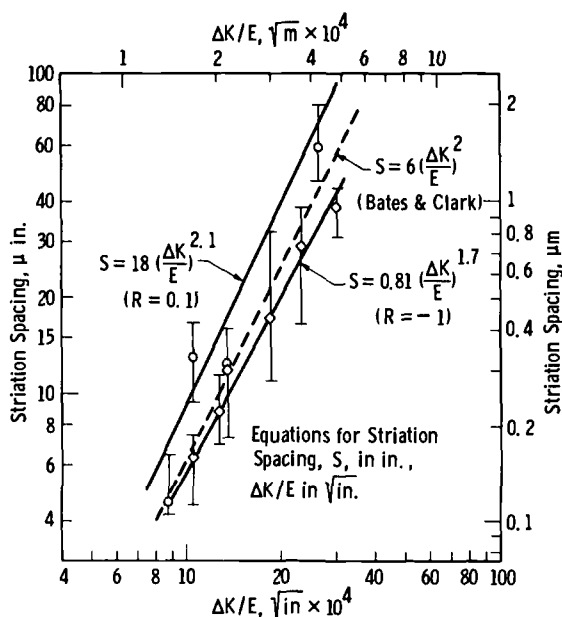


FIG. 8—Fatigue striations spacing as a function of $\Delta K/E$ for load ratios, R , of 0.1 and -1 in Type 2219-T851 aluminum.

The crack-closure parameter, U , definitely suggests an increase in da/dN with increasing R . It also predicts that da/dN at $R = -1$ will be less than that at $R = 0.1$, as was indeed observed. It should be kept in mind, however, that the simple Elber model cannot be applied directly to fatigue crack growth results since crack closure (the parameter U) depends not only on R , but also on $K_{\max}$ and the specimen thickness, as has been shown by Ungast, Shih, and Wei in a study on crack-closure effects in Type 2219-T851 aluminum alloy [28].

Correlation between Striation Spacing and $\Delta K/E$

Figure 8 gives empirical relationships between striation spacings and ΔK for $R = 0.1$ and $R = -1$. The results are expressed as a function of $\Delta K/E$ so that these relationships can be compared with those obtained for other materials. The equations and trend lines were derived using a regression analysis. The bands on each data point represent data scatter, which is given in terms of the range of measurements from ten microscopic locations, each at nominally the same crack length. Scatter in these terms varies from a factor of 1.5 to 3, with 2 being typical overall.

As indicated by the solid trend lines, striation spacings at $R = 0.1$ are generally larger than those at $R = -1$. This trend is qualitatively the same as that exhibited by the crack growth rates and is believed to be real, although it is admittedly small when considered in relation to data scatter.

Results from Bates and Clark [6] are also provided in Fig. 8 for comparison. This representation is purported to apply to a range of load ratios and, in fact, does provide a relatively good representation of results from both load ratios. Thus, in a practical sense when using striation spacing measurements to define applied ΔK values, the Bates-Clark relationship appears adequate, particularly in view of typical scatter in striation spacing measurements. For example, when a representative average striation spacing is determined, the results of Fig. 8 show that this relationship would enable ΔK to be defined within about ± 25 percent. However, further evaluations of this relationship are recommended for high positive load ratios in materials with fatigue crack growth rates that are markedly sensitive to load ratios.

Summary and Conclusions

1. In Type 2219-T851 aluminum at growth rates above $0.025 \mu\text{m}/\text{cycle}$, the presence of constant-amplitude compressive loading at $R = -1$ decreases the fatigue crack growth rate in relation to rates at $R = 0.1$.

2. At both $R = 0.1$ and $R = -1$ the microfractographic features in Type 2219-T851 alloy change systematically from cleavage-like facets to ductile striations to dimpled rupture as macroscopic growth rates increase.

3. Static modes of cracking, characterized by dimpled rupture, accompany striated fatigue crack growth at high ΔK (or $K_{\max}$) values and result in higher fatigue crack growth rates at $R = 0.1$ than at $R = -1$.

4. At growth rates above $0.025 \mu\text{m}/\text{cycle}$, a one-to-one correspondence exists between microscopic striation spacing and macroscopic fatigue crack growth rates, provided supplementary static cracking modes are absent.

5. The relationship between striation spacing and $\Delta K/E$ is much less sensitive to load ratio than is that between da/dN and ΔK .

6. The load ratio ($R = 0.1$ to $R = -1$) has a measurable effect on striation spacing, although this effect is small compared with the variability in striation spacing measurements. Thus, for practical application to failure analysis, the simple Bates-Clark expression, $S = 6 (\Delta K/E)^2$, which is independent of load ratio, is adequate. However, additional verification of the utility of this expression is needed for high positive load ratios.

Acknowledgments

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Correlation Between Fatigue Crack Growth Rate and Fatigue Striation Spacing in AISI 9310 (AMS 6265) Steel

REFERENCE: Au, J. J. and Ke, J. S., "Correlation Between Fatigue Crack Growth Rate and Fatigue Striation Spacing in AISI 9310 (AMS 6265) Steel," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 202-221.

ABSTRACT: Fatigue crack growth data were obtained for an AISI 9310 (AMS 6265) steel under various conditions. The effects of stress ratio, frequency, environment, and carbon content (strength) on both the fatigue fracture topography and the fatigue crack growth rate were studied. A good correlation was obtained between the fatigue striation spacing and the macroscopic crack growth rate for crack growth rates ranging from 10^{-4} to 10^{-3} mm/cycle.

KEY WORDS: steel, fatigue crack growth, fractography, fatigue striation spacing, macroscopic crack growth rate, stress ratio effect, frequency effect, environment (humidity) effect, carbon content (strength) effect, scanning electron microscope (SEM), materials, materials science

It has long been recognized that fatigue crack propagation is often the life-limiting factor for many structural materials. The concepts of linear fracture mechanics developed by Irwin [1]³ and the empirical relationships subsequently proposed [2-9] have been used successfully to approximate the fatigue life of a structure, based on its loading conditions and material characteristics. In this approach, either the crack-tip stress intensity factor, K , or the stress intensity range, ΔK , is used as the appropriate fracture mechanics parameter for characterizing the mechanical driving force for fatigue crack growth [2]. The empirical relationship most often used is the Paris equation

$$da/dN = C\Delta K^n \quad (1)$$

where a is the crack length, N is the number of elapsed cycles, and C and n are constants.

¹Senior metallurgist, Sundstrand Aviation Operations, Sundstrand Corp., Rockford, Ill. 61111.

²Specialist engineer, Boeing Wichita Co., Wichita, Kans. 67220.

³The italic numbers in brackets refer to the list of references appended to this paper.

Although this analytical approach is adequate from a design viewpoint, it does not give any information concerning the mechanism involved. The advent of electron fractography has revealed many features on fatigue fracture surfaces and has greatly increased our understanding of the mechanism of fatigue crack propagation [10–12]. The single most important characteristic pattern of a fatigue fracture surface is perhaps the so-called fatigue striations. Ryder [13] has demonstrated that these striations correspond numerically to the number of applied loading cycles.

Recently, the application of electron fractography for failure analysis has led to the realization that the cyclic loading conditions that cause component fatigue failures can be estimated by a quantitative correlation between fatigue striation spacing and known cyclic loading conditions. A technique for establishing this correlation is available through the use of linear fracture mechanics, as discussed previously. Thus, by establishing a relationship between the fatigue striation spacing and the macroscopic crack growth rate, da/dN , the loadings on the components can be estimated from Eq 1. Indeed, fairly good correlation between striation spacing and macroscopic crack growth rate has been obtained in aluminum alloys [11, 14–20], steels [21–27], and titanium alloys [28–30].

AISI 9310 steel has found wide application as a gear material in its carburized condition due to its high hardenability, low wear, high temper resistance, and good fracture toughness. The objective of this study was to develop data on the crack growth rate for this steel, to study the fractographic features observed on the fatigue surface, and to correlate the striation spacing with the macroscopic crack growth rate.

Experimental Procedures

The material used in the present study was a consumable-electrode vacuum-melted AISI 9310 steel (AMS 6265). The composition and room temperature tensile properties of this material are given in Tables 1 and 2. The specimens were heat treated to two different conditions. For one condition, the specimens were hardened and tempered to a hardness of 37 HRC (low-carbon, low-strength specimens). For the other condition, through-carburizing was done before the specimens were hardened and tempered to a hardness of 60 HRC (high-carbon, high-strength). The low-carbon specimens would be representative of the core structure of a normally case-carburized Type 9310 steel, whereas the high-carbon specimens would be representative of the case structure. Microstructures of the two types of specimens are illustrated in Fig. 1.

The specimen geometry and the testing procedure for the fatigue crack growth rate are described in the ASTM Tentative Test for Constant-Load-Amplitude Fatigue Crack Growth Rates Above 10^{-8} m/Cycle (E 647-78T). Compact tension (CT) specimens shown in Fig. 2, in the transverse (TS) orientation were used. These specimens had a half-height-to-width ratio of 0.6 and

TABLE 1—*Chemical composition of AISI 9310 steel, weight percent.*

C	Mn	P	S	Si	Cu	Ni	Cr	Mo	Fe
0.10	0.54	0.007	0.004	0.27	0.19	3.11	1.32	0.12	balance

nominal dimensions of 6.35 cm (2.5 in.) wide and 0.5 cm (0.2 in.) thick, with an initial notch 1.9 cm (0.75 in.) deep and 0.3175 cm (0.125 in.) wide.

Fatigue crack growth experiments were carried out with an MTS electro-hydraulic close-loop testing machine, which was equipped with a $\times 20$ traveling microscope for crack length measurements. Fatigue cracks were extended by constant load-amplitude sinusoidal fatigue cycling at selected frequencies, f , and load ratios (or stress ratios), R (R = minimum load/maximum load = minimum stress/maximum stress).

Environmental control was maintained by enclosing the specimens in a sealed chamber into which desiccant was placed for dry-air testing and through which water was circulated for wet-air testing.

Low-load, high-frequency fatigue precracking was used to create a sharp crack front about 0.32 cm (0.125 in.) deep and to move the crack tip out of any stresses induced during machining. At the end of the fatigue precracking, the crack length was recorded in preparation for the crack growth rate work. The cycle count and crack length were recorded at periodic intervals thereafter.

Fractographic examination was conducted by means of an Advanced Metals Research Corp. (AMR) 1000 scanning electron microscope (SEM). For each specimen, a sequence of photos was taken. Each photo was identified with the corresponding crack length and stress intensity range. The fatigue striations in each photo were counted, and the striation spacing was subsequently calculated.

Results and Discussion

Fractographic Observations

The general fracture surface appearance of a fatigue crack growth specimen is illustrated in Fig. 3. The fracture surface consisted of three zones; a pre-

TABLE 2—*Room temperature tensile properties of AISI 9310 steel.*

Tensile Strength, MPa ^a (ksi)	Yield Strength, MPa (ksi)	Elongation in 5.08 cm (2 in.), percent	Reduction of Area, percent
1220 (177)	930 (135)	17.6	64.1

^aOne MPa = 1 MN/m² = 0.145 ksi.

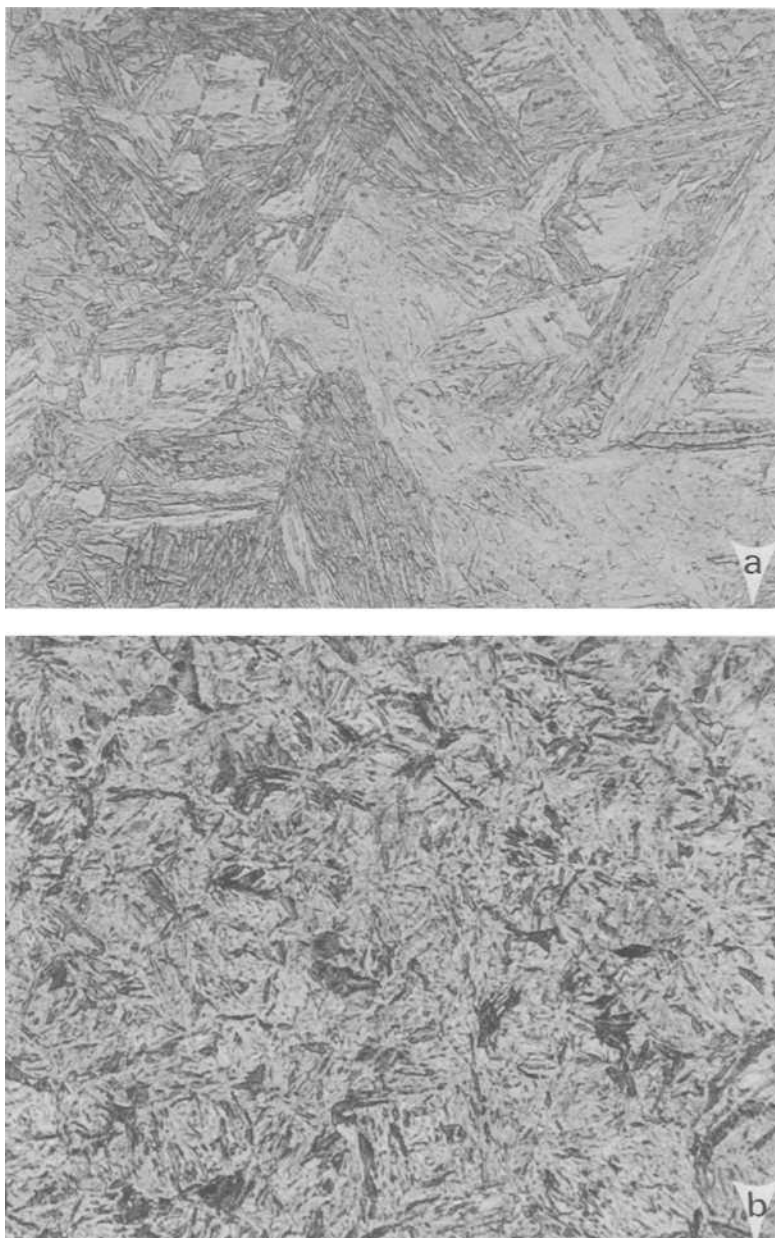


FIG. 1—Microstructure of AISI 9310 specimens in the noncarburized condition (a) and in the carburized condition (b). Etched with 3 percent nital, the magnification is $\times 1000$.

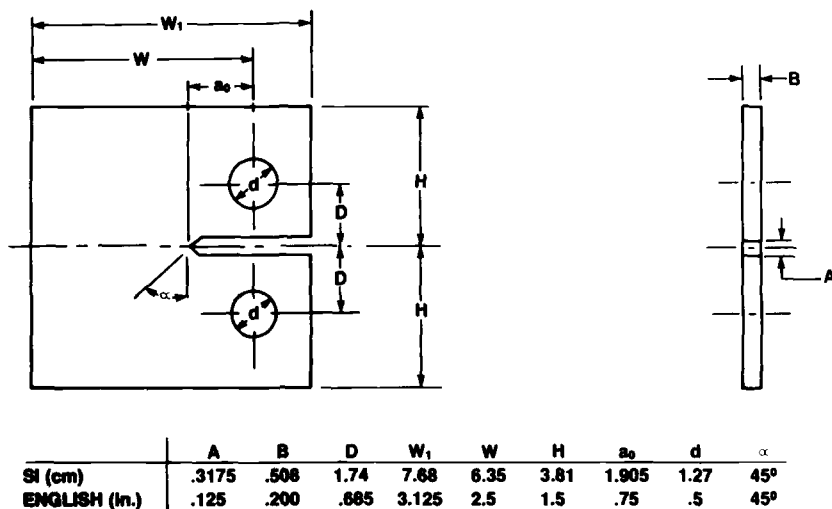


FIG. 2—Compact tension (CT) specimen for fatigue crack growth rate testing.

cracked fatigue zone, a fatigue crack growth zone, and a rapid fracture zone. The precracked fatigue zone and the fatigue crack growth zone exhibited similar smooth textures; subtle crack-arrest marking distinguished the two zones. The rapid fracture zone showed a fibrous texture, with a tensile fracture region (plane-strain mode) in the center and two slant fractures (plane-stress mode) at the edges. The fractographic examination was concentrated in the fatigue crack growth zone (F in Fig. 3). In particular, the effects on the fatigue fracture topography of stress intensity range, ΔK ; stress ratio, R ; frequency, f ; environment; and chemistry will be illustrated and discussed.

Stress Intensity Range, ΔK , Effect—At small values of ΔK , the area that was covered by fatigue striations was small. As shown in Fig. 4a, for example, only a small region, S, was covered by fatigue striations. Most of the fracture surface was covered by featherlike quasi-cleavage or quasi-fatigue [22] ridges. The chevron pattern formed by these ridges pointed back toward the crack origin. Fatigue striations observed in this region of small ΔK tended to be nonuniform, and correlation of the striation spacing with the macroscopic crack growth rate was poor.

In regions where ΔK values were large, extensive fatigue striations with secondary microcracks predominated. The fracture surface also showed a relatively smooth topography (see, for example, Figs. 4b and c, and 5a). With further increase in ΔK , the number and width of the secondary microcracks increased accordingly.

Stress Ratio, R , Effect—The only obvious effect of the stress ratio, R , on the fatigue fracture topography is on the amount of rubbing. As can be seen by

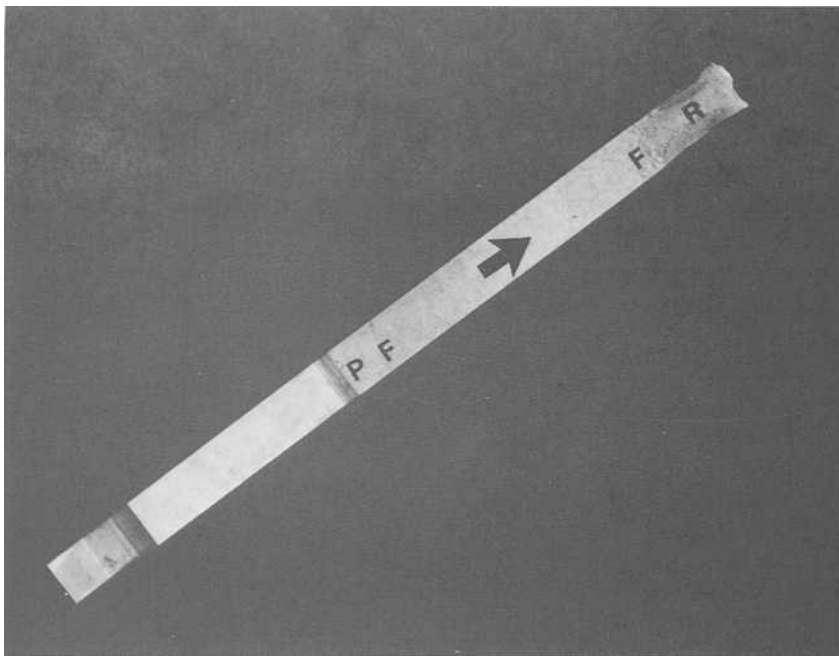


FIG. 3—Light macrograph showing the fracture surface of a typical fatigue crack growth specimen. P is the precracked zone, F is the fatigue crack growth zone, and R is the rapid fracture zone. (The arrow indicates the crack propagation direction). The magnification is $\times 1.4$.

comparing Fig. 4b and c, the smaller the R value, the greater the amount of rubbing on the fracture surface. Recently, a crack closure concept has been introduced by Elber [31] to explain the stress ratio effect observed in his fatigue crack growth experiments under constant-amplitude loading. Elber suggested that the crack is closed at the tip over the lower portion of the loading cycle and becomes open only after the applied stress exceeded a critical level. Using this concept, our observed R effect (increased rubbing with decreasing R) simply reflects a greater amount of crack closure during cyclic crack propagation at low R values.

Environmental Effect—The introduction of humidity into the testing environment promoted the formation of oxide on the fracture surface (see Fig. 4d and Fig. 5b). In a few isolated areas, corrosion pits were evident (Fig. 5c). It should be emphasized at this point that heavy oxidation and extensive rubbing provided great obstacles to our identification of fatigue striations on the fracture surface of specimens tested at low R values (0.05) in wet air. It is safe to assume that similar difficulties will be encountered in the analysis of parts that failed under corrosion fatigue.

Frequency Effect—The frequency effect was not tested under dry air, since no significant effect was expected from such a test. On the other hand, testing

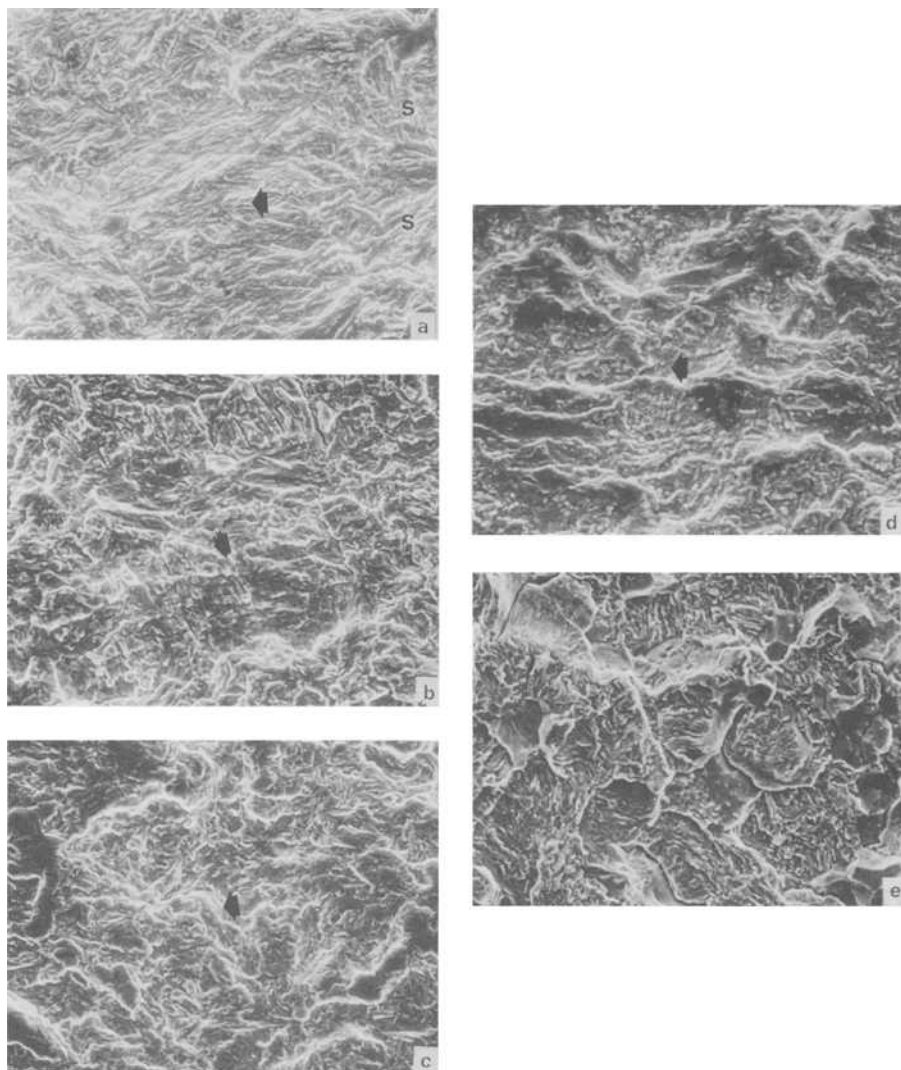


FIG. 4—Fracture topography of AISI 9310 fatigue crack growth specimens tested under various conditions: (a) noncarburized, $R = 0.05$, $f = 3.4$ Hz, dry air, $\Delta K = 9.14 \text{ MN m}^{-3/2}$; (b) noncarburized, $R = 0.5$, $f = 3.4$ Hz, dry air, $\Delta K = 29.6 \text{ MN m}^{-3/2}$; (c) noncarburized, $R = 0.05$, $f = 3.4$ Hz, dry air, $\Delta K = 23.63 \text{ MN m}^{-3/2}$; (d) noncarburized, $R = 0.05$, $f = 0.1$ Hz, wet air, $\Delta K = 20.27 \text{ MN m}^{-3/2}$; (e) carburized, $R = 0.05$, $f = 3.4$ Hz, dry air, $\Delta K = 14.45 \text{ MN m}^{-3/2}$. (The arrows indicate the crack propagation direction.) The magnification is $\times 510$.

under different loading frequencies in wet air did show some expected results. At a frequency of 0.1 Hz, brownish corrosion products (rust) were clearly visible on the fracture surface. At a frequency of 1.0 Hz, such rusting was absent, and the oxide film on the fracture surface was much thinner. This frequency dependency on rusting and oxidation is not surprising; it simply reflects the relative time of exposure to the humid environment during crack growth.

Carbon Content (Strength) Effect—Of the various effects we have discussed thus far, the carbon content (strength) had the most pronounced effect on the fatigue fracture appearance. Whereas the fractography of low-carbon, low-strength specimens showed relatively well-defined striations on a smooth topography, the high-carbon, high-strength specimens exhibited an intergranular fracture (Fig. 4e) with “brittle” striated facets on the face of the grains. Since no other fatigue striations could be found on the entire fracture surface, these “brittle” striated facets were considered to be the fatigue striations (Fig. 5d). The spacing between facets was then measured as the fatigue striation spacing.

It is apparent from Fig. 4e that the striated facets were not necessarily perpendicular to the macroscopic fracture path. Rather, these “striations” closely resembled the microstructure shown in Fig. 1b. This correlation of fatigue morphology with microstructure suggests that the local stress state and the local fracture path were controlled by the microstructure when both the carbon content and the yield strength were high. It is clear from the intergranular mode of fracture that there were weak links—namely, the grain boundaries—in the matrix. It is quite possible that similar weak links also existed within each grain.

The dependency of fracture mode on carbon content and yield strength has been studied systematically by Beachem [32]. In his work, only overload fractures were examined. The present work provides additional information on the dependency of fracture morphology on carbon content and yield strength in fatigue fractures. Beachem also found an increasing amount of intergranular rupture to be associated with increased carbon content, yield strength, or both in overload fractures. It is interesting to note that a similar trend was observed even in fatigue fractures.

Results of Fatigue Crack Growth Rate

The macroscopic fatigue crack growth rate was determined from the crack length, a , versus the elapsed cycles, N , by means of the secant or point-to-point technique. Thus, the crack growth rate, da/dN , is given by the equation

$$\left[\frac{da}{dN} \right]_{\bar{a}_i} = \frac{a_{i+1} - a_i}{N_{i+1} - N_i} \quad (2)$$

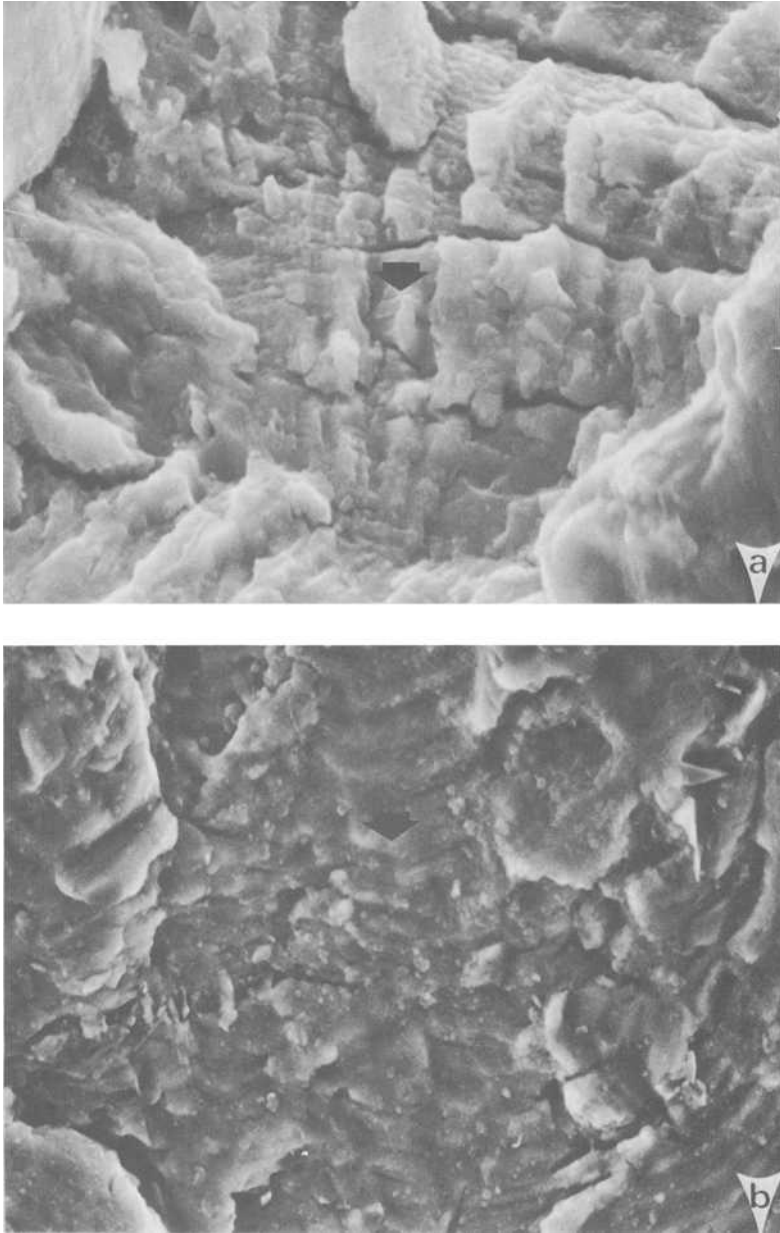


FIG. 5—Fatigue striations observed on AISI 9310 fatigue crack growth specimens tested under various conditions: (a) noncarburized, $R = 0.5$ or 0.05 , $f = 3.4$ Hz, dry air, $\Delta K = 23.06 \text{ MN m}^{-3/2}$, magnification $\times 11\,000$; (b) noncarburized, $R = 0.05$, $f = 1.0$ Hz, wet air, $\Delta K = 84.61 \text{ MN m}^{-3/2}$, magnification $\times 4300$; (c) noncarburized, $R = 0.05$, $f = 1.0$ Hz, wet air, $\Delta K = 71.42 \text{ MN m}^{-3/2}$, magnification $\times 4600$; (d) carburized, $R = 0.05$, $f = 3.4$ Hz, dry air, $\Delta K = 16.48 \text{ MN m}^{-3/2}$, magnification $\times 5700$. (The arrows indicate the crack propagation direction.)

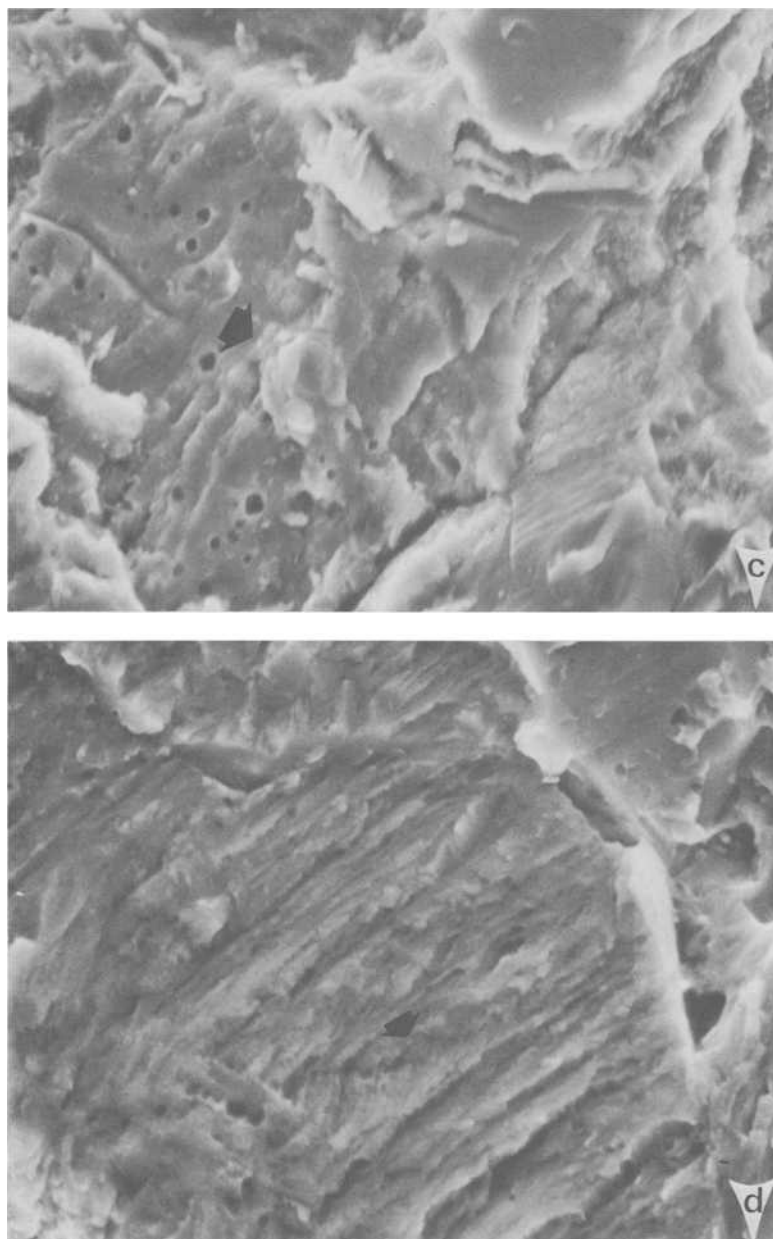


FIG. 5—Continued.

where the average crack length is

$$\bar{a}_i = \frac{1}{2} (a_{i+1} + a_i)$$

The stress intensity range, ΔK , corresponding to a given crack length, $\bar{a}_i$, was computed from ASTM Test E 647-78T

$$\Delta K(a_i) = \frac{\Delta P(2 + \alpha)}{BW^{1/2}(1 - \alpha)^{3/2}} (0.886 + 4.64\alpha - 13.32\alpha^2 + 14.72\alpha^3 - 5.6\alpha^4) \quad (3)$$

where

$$\bar{\alpha} = a_i/W,$$

B = specimen thickness,

W = specimen width, and

$$P = P_{\max} - P_{\min}.$$

and where $P_{\max}$ and $P_{\min}$ are the maximum load and the minimum load, respectively, of the cyclic load applied.

With the macroscopic crack growth rate calculated from Eq 2 and the microscopic crack growth rate obtained from fatigue striation spacing measurements, the two types of crack growth data could be plotted against ΔK and fitted to the Paris expression, Eq 1. The fitting procedure involved adjusting the parameters C and n of Eq 1 by means of a least-squares-regression computer analysis. The computer analysis also calculated the standard deviation, S , which described the data dispersion about the best-fitted curve, and the tolerance limit which predicted that there was 95 percent confidence, γ , that 90 percent, P , of all future observations will be within this limit.

The resulting da/dN versus ΔK curves are shown in Figs. 6-10, together with the best-fitted values of C and n of Eq 1. It is clear that the fatigue striation data compared favorably with the macroscopic crack growth data. This means that cyclic stress conditions in actual failures can now be estimated by fatigue striation measurements through the use of empirical relations such as Eqs 1 and 3.

Figure 11 summarizes the effects of various test conditions on the fatigue crack growth of AISI 9310 steel. These effects will now be discussed separately.

Stress Ratio, R , Effect—For the same material condition and tested in dry air with 3.4 Hz frequency, the fatigue crack growth rate is faster when the R value is higher.

Environmental Effect—For the same material and under similar loading conditions, the fatigue crack growth rate is faster when tested in the wet-air environment than when tested in the dry-air environment. In these tests, two different frequencies, 1.0 Hz and 3.4 Hz, were used. It has been established, in

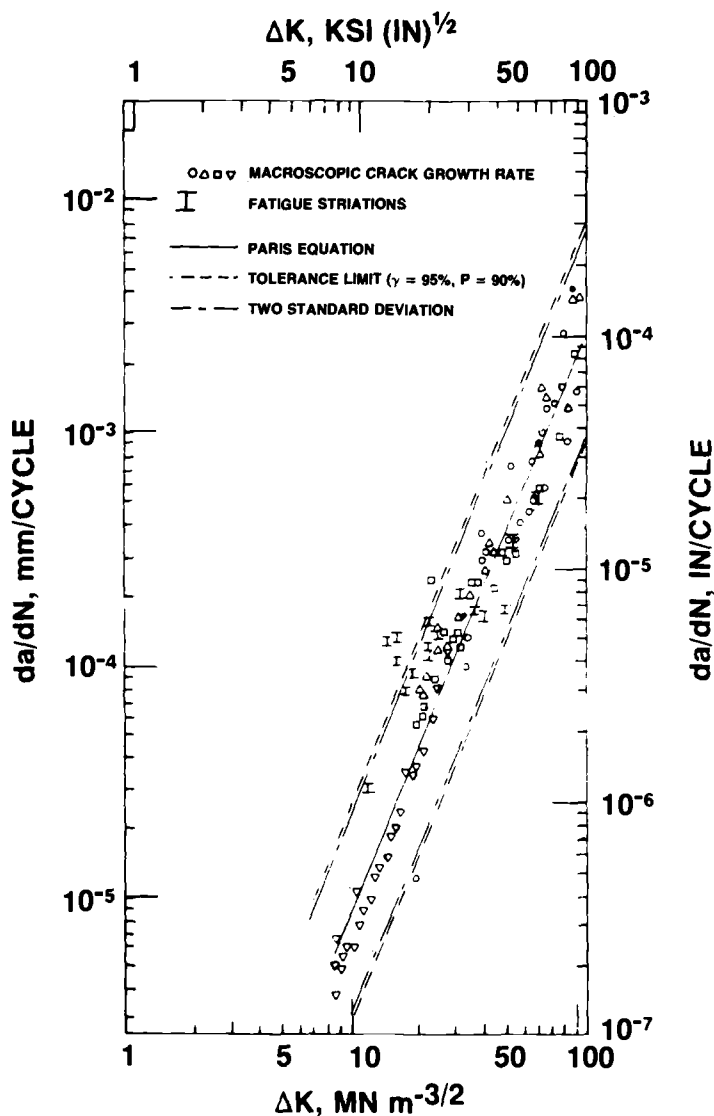


FIG. 6—Fatigue crack growth rate of noncarburized AISI 9310 as a function of stress intensity range when tested in dry air, 3.4 Hz, and $R = 0.05$. The fitted parameters are $C = 3.988 \times 10^{-8}$ mm/cycle, $n = 2.42$.

a previous investigation [33], that there was negligible difference in crack growth rate between the two frequencies used. The observed difference in crack growth rate could only be attributed to the effect of the environment.

Frequency Effect in Wet Air—When the frequency differed by about an order of magnitude ($f = 0.1$ Hz compared with $f = 1.0$ Hz), a small difference in crack growth rate could be detected. Thus, for the same material condition

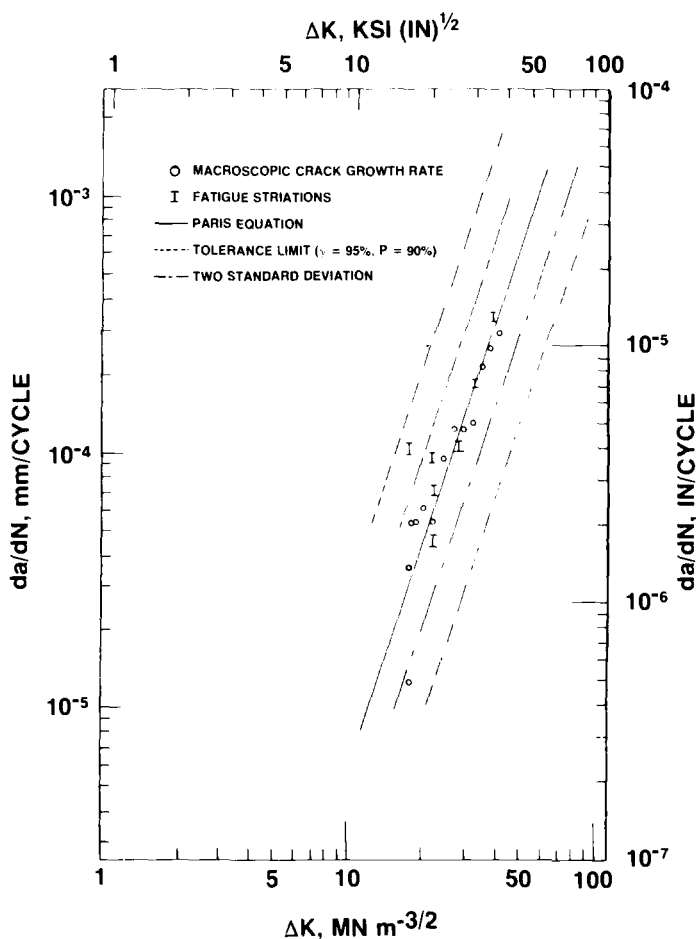


FIG. 7—Fatigue crack growth rate of noncarburized AISI 9310 as a function of stress intensity range when tested in dry air, 3.4 Hz, and $R = 0.5$. The fitted parameters are $C = 8.433 \times 10^{-9}$ mm/cycle, $n = 2.954$.

tested in wet air with $R = 0.05$, the fatigue crack growth rate increases when the frequency decreases.

Carbon Content (Strength) Effect—For the same loading conditions, the fatigue crack growth rate of high-carbon, high-strength AISI 9310 (characteristic of the case) is faster than that of low-carbon, low-strength AISI 9310 (characteristic of the core). This result is unaffected by the humidity level.

Correlation between Macroscopic Crack Growth Rate and Fatigue Striation Spacing

Figures 12 and 13 show the correlation between macroscopic crack growth rate and fatigue striation spacing. If the macroscopic crack growth rate is

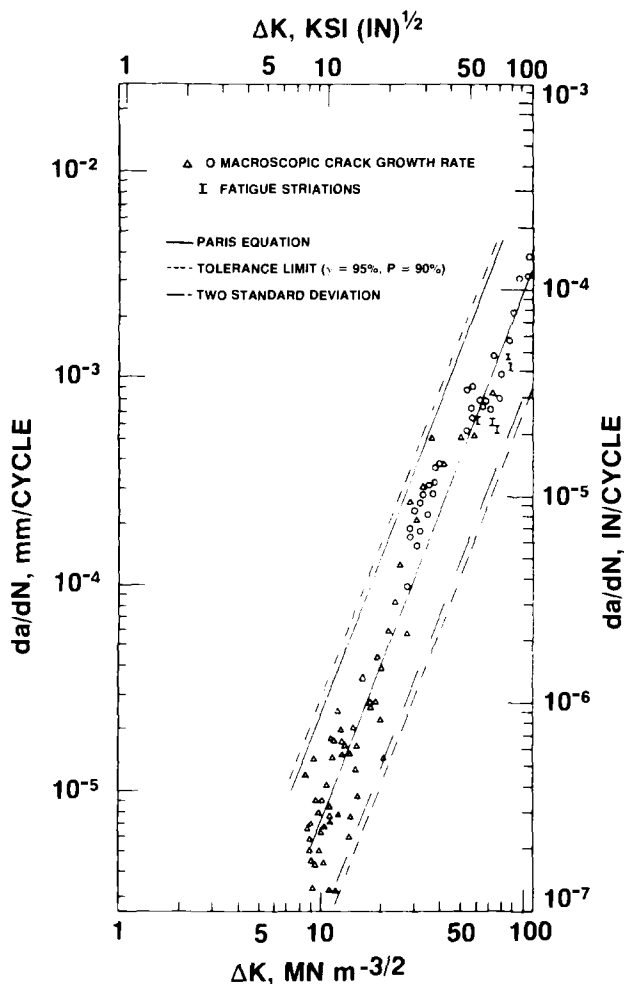


FIG. 8—Fatigue crack growth rate of noncarburized AISI 9310 as a function of stress intensity range when tested in wet air, 1.0 Hz, and $R = 0.05$. The fitted parameters are $C = 2.512 \times 10^{-8}$ mm/cycle, $n = 2.555$.

equal to the localized striation spacing, then the plots in Figs. 12 and 13 would yield straight lines inclined at 45 deg angles with the abscissa.

An examination of these two figures indicates that there was good correlation (dash lines) in the macroscopic crack growth rate range of about 10^{-4} to 10^{-3} mm/cycle. Below about 10^{-4} mm/cycle, the data points deviate increasingly from the 45-deg line. The reason for this deviation in the region of low crack growth rate is not clear. One possible reason is that striation spacing is nonuniform at low crack growth rates, as mentioned in the section on stress intensity range effect. Among the unevenly spaced striations, only the widely spaced ones could be resolved by the SEM.

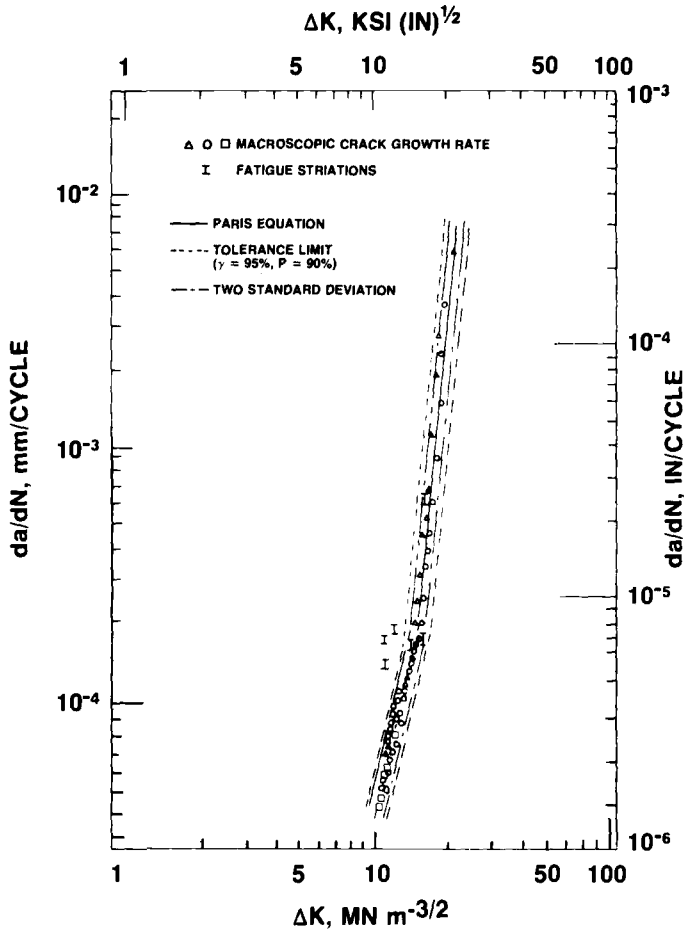


FIG.10—Fatigue crack growth rate of carburized ($C = 0.67$ percent) AISI 9310 when tested in dry air, 3.4 Hz, and $R = 0.05$. The fitted parameters are $C = 6.45 \times 10^{-17}$ mm/cycle, $n = 10.5$ ($\Delta K > 16.5 \text{ MN m}^{-3/2}$), $C = 1.18 \times 10^{-9}$ mm/cycle, $n = 4.367$ ($\Delta K < 16.5 \text{ MN m}^{-3/2}$).

ent. Within the scatter of the data, no such chemistry effect could be discerned, as shown in Fig. 13. In fact, Figs. 12 and 13 reveal that none of the test variables (stress ratio, humidity, frequency, and chemistry) had any significant effect on the correlation between the macroscopic crack growth rate and fatigue striation spacing. However, in view of the difficulties encountered in striation spacing measurements, as discussed in the section on fractographic observations, the lack of any effect should not be considered conclusive.

Conclusions

On the basis of experimental results obtained during this investigation on AISI 9310 (AMS 6265) steel, the following conclusions can be made:

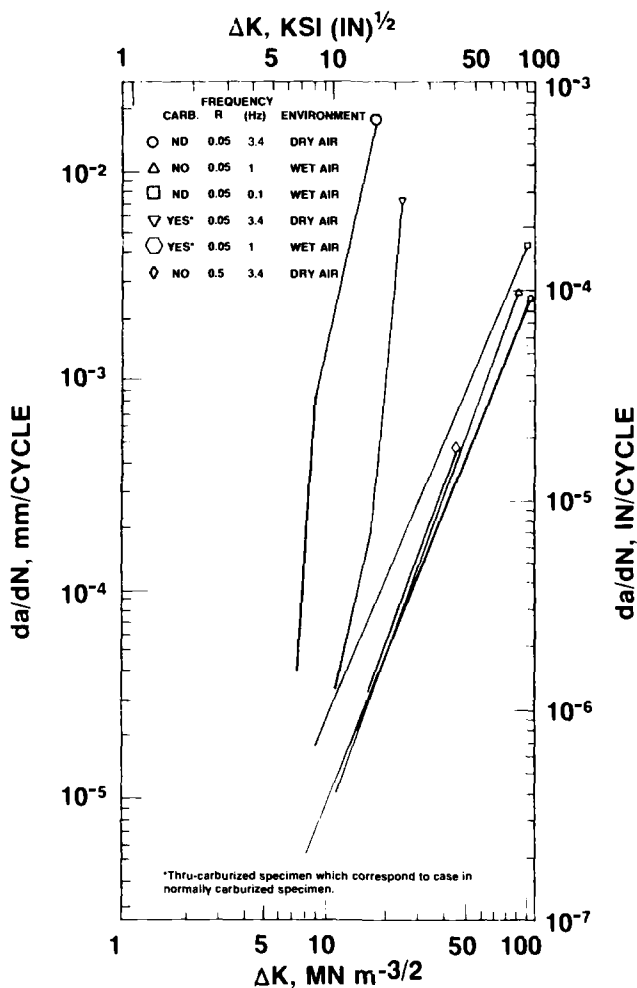


FIG. 11—Summary of fatigue crack growth data for AISI 9310 tested under various conditions.

1. There was good correlation between the macroscopic crack growth rate and fatigue striation spacing for crack growth rates ranging from 10^{-4} to 10^{-3} mm/cycle.

2. The correlation between the macroscopic crack growth rate and fatigue striation spacing was not affected by the stress ratio, frequency, environment, or carbon content (strength).

3. Decreasing the stress ratio tends to increase rubbing on the fatigue fracture surface and also to decrease the crack growth rate.

4. Humidity promotes oxidation and corrosion on the fracture surface while enhancing crack growth rate.

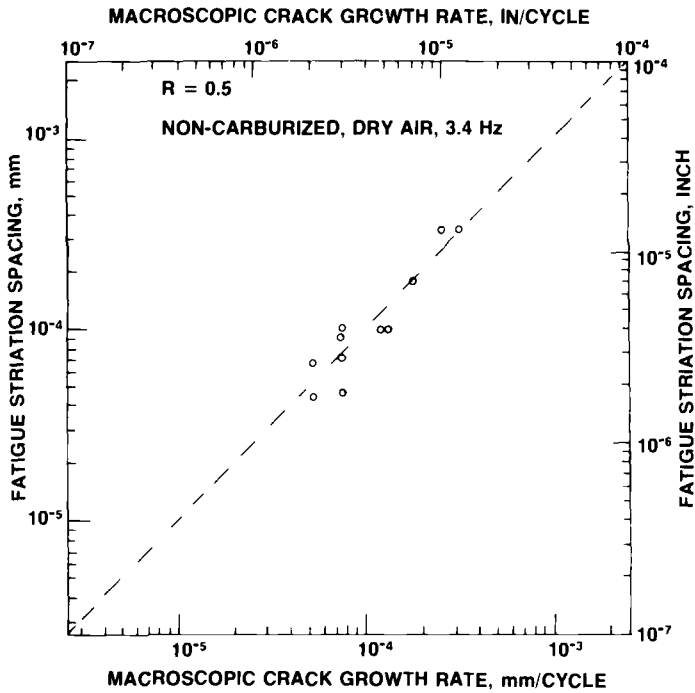


FIG. 12—Correlation of macroscopic crack growth rate with fatigue striation spacing for $R = 0.5$.

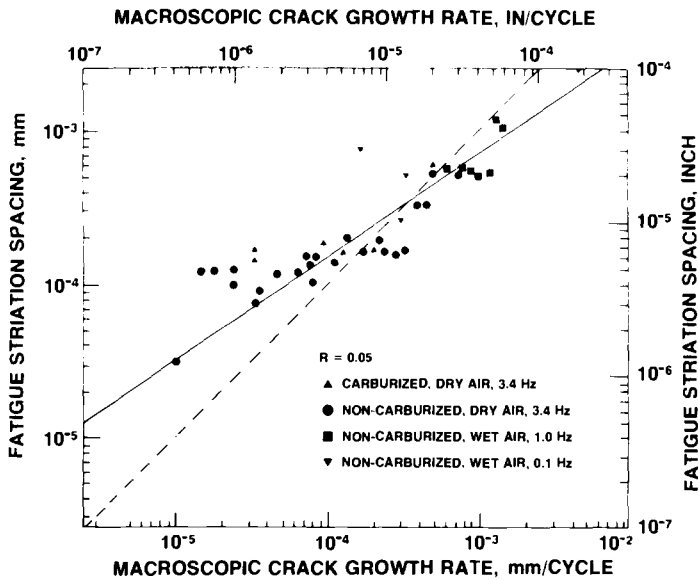


FIG. 13—Correlation of fatigue crack growth rate with fatigue striation spacing for $R = 0.05$.

5. When tested in wet air, low-loading frequency favors corrosion and oxidation while increasing the crack growth rate.

6. High-carbon, high-strength AISI 9310 and low-carbon, low-strength AISI 9310 showed markedly different crack growth behavior and fatigue fracture surface appearance; the crack growth rate of high-carbon, high-strength AISI 9310 is much faster.

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Nonmetallics and Composites

Strength Characterization and Nature of Crack Propagation in Ceramic Materials

REFERENCE: Govila, R. K., Beardmore, P., and Kinsman, K. R., "Strength Characterization and Nature of Crack Propagation in Ceramic Materials," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 225-245.

ABSTRACT: A detailed study characterizing the strength and nature of crack propagation has been made in several high-temperature structural ceramics, namely, hot-pressed silicon nitride (Si_3N_4), Refel silicon carbide (SiC), sialons, and lithium-aluminum-silicate (LAS) glass ceramic. Fracture phenomenology was studied fractographically as a function of flaw size, temperature, and loading rate. The temperature dependence of the fracture behavior was evaluated using uncracked and precracked specimens.

All the materials showed the same general characteristics of temperature dependence of both the fracture stress and crack-growth phenomena. The only variation between the different materials was the temperature regime in which the differing phenomena occurred. Two mechanistic regimes were manifest in the temperature dependence of the fracture stress. A region of fast fracture (catastrophic crack extension) existed up to a certain temperature, in which the mode of crack propagation was primarily transgranular. Above this temperature, the strength (fracture stress) decreased considerably due to the presence of subcritical or slow crack growth (SCG) prior to the final catastrophic fracture, and the extent of SCG increased with increasing temperature. Slow crack growth is a result of microplastic deformation, which occurred by grain-boundary sliding and grain separation, and the mode of crack propagation is exclusively intergranular.

The influence of the loading rate on SCG and fracture stress was investigated. Generally, at a given temperature, the extent of SCG decreased with increasing loading rate until, at a sufficiently high rate, catastrophic fracture occurred directly from the initial crack with no slow crack extension. Directly analogous observations were made in stress-rupture studies in which the extent of SCG decreased with increasing stress level.

KEY WORDS: fracture, fractography, silicon nitride (Si_3N_4), lithium-aluminum-silicate (LAS), Refel silicon carbide (SiC), silicon aluminum oxynitrides (sialons), crack propagation, slow crack growth, ceramics, materials, materials science

Polycrystalline ceramic materials such as hot-pressed silicon nitride (Si_3N_4), reaction-bonded silicon carbide (Refel SiC), silicon aluminum oxynitrides

¹Research scientist, principal staff scientist, and staff scientist, respectively, Scientific Research Staff, Ford Motor Co., Dearborn, Mich. 48121.

(Sialons), and lithium-aluminum-silicate (LAS) glass ceramics have received considerable attention as potential materials for structural applications in gas turbine engines because of good oxidation resistance, low coefficient of thermal expansion, and retention of high strength up to about 1000°C. However, the majority of ceramic materials in general have little tolerance for the incipient flaws that are present due to processing and fabrication. It is these random flaws that control the mechanical properties. In addition, in the majority of the structural ceramics, the presence of subcritical (or slow) crack growth (SCG)² at high temperatures ($\geq 1200^\circ\text{C}$, or even lower in the case of sialons and LAS) causes a time-dependent decrease in materials strength. In this paper, we report the results of a series of simple mechanical tests performed on precracked specimens in the aforementioned ceramic materials to measure the strength as a function of flaw size, temperature, and loading rate. In addition, an estimate of the size of strength-controlling flaws (or the inherent flaw size) of the as-received material, the temperature for the onset of SCG, and the nature of crack propagation during fast (catastrophic) failure and SCG have been determined. Fractography is the principal interpretative agent for the presence of SCG and for revealing the nature of crack propagation.

Materials

Hot-Pressed Silicon Nitride

The material used in this study was a nominally fully dense, hot-pressed silicon nitride commercially known as HS-130 made by Norton Co., which was obtained in the form of a billet (~ 150 mm) (6 in.) long by 150 mm (6 in.) wide by 25 mm (1 in.) thick.

Lithium-Aluminum-Silicate Glass Ceramics

The particular material used was a nominally fully dense LAS produced by Owen-Illinois under their code name C-140. It is a crystallized glass ceramic composed principally of β -spodumene and β -eucryptite structures together with some impurities that aid in the material processing. Typically, in such glass ceramics, crystallization is nominally though not entirely complete, and residual glass is located as a thin layer along much of the grain boundary surface.

Refel SiC

A commercially available polycrystalline ceramic composite material of alpha-silicon carbide and silicon, designated Refel by the producer, UKAEA,

² It should be pointed out that SCG can also occur at low temperatures (such as room temperature) in the presence of water or other environments in many ceramic materials and is controlled by different mechanisms. In this study, SCG, as influenced by the presence of high temperatures in air only, has been examined in detail.

Springfields, England, was evaluated. The material is made by a reaction-bonding process and is a fully dense material that contains about 10 percent free silicon. Detailed microstructural characterization of Refel SiC has been reported recently by Sawyer and Page [1].³

Sialon Materials

These materials are believed to be a solution of alumina (Al_2O_3) in Si_3N_4 , having the same structure as that of $\beta\text{-Si}_3\text{N}_4$. The sialon materials discussed herein were prepared by pressureless sintering and contained a mixture of 60 weight percent standard Advanced Materials Engineering (AME) Si_3N_4 and 40 weight percent Linde Al_2O_3 , together with yttria additions ranging from 3 to 16 weight percent. The sintering temperatures ranged from 1600 to 1700°C, and the times ranged from 4 to 12 h. Complete details regarding processing this material are given elsewhere [2,3].

Experimental Procedures and Techniques

Sample Preparation and Testing

Test specimens of dimensions ~ 25 mm (1 in.) long by 6 mm (1/4 in.) wide by 3 mm (1/8 in.) thick were machined from blocks of various materials. In those instances in which directionality due to hot-pressing is a factor to be considered, the specimens were machined so that the tensile face was perpendicular to the hot-pressing direction (that is, the "strong direction"). All faces were ground in a lengthwise direction using 220-grit diamond wheels, and the edges were chamfered to prevent notch effects. One surface (the tension face in bending) was carefully hand ground and wet polished to a 6- μm diamond finish for those specimens to be used for precracking by diamond indentation. The LAS specimens were then heated for 4 h in air at 200°C to remove any adsorbed water. Certain of the specimens from each group of materials were then precracked, using the indentation technique outlined briefly in the following section. Test specimens in both the as-received condition and the precracked condition were tested in four-point bending in an Instron testing machine at a constant crosshead speed of ~ 0.127 mm (0.005 in.) per min. The outer and inner loading edges of the silicon carbide testing fixture were spaced ~ 15 mm (0.6 in.) and ~ 5 mm (0.2 in.) apart, respectively. High-temperature (200 to 1400°C) bend tests were performed in air in a cylindrical muffle furnace (Centorr Associates) attached to the Instron machine head. In high-temperature tests, specimens were held at the test temperature for about 15 min in order to achieve equilibrium before the testing was started. No preload was applied on test specimens at either room temperature or high temperature. The fracture surfaces were examined by optical microscopy and scanning electron micro-

³The italic numbers in brackets refer to the list of references appended to this paper.

scopy (SEM). For LAS specimens, a flash coating of aluminum was found to enhance greatly the reflectivity without producing any distortion of the fractographic features.

Controlled Precracking of Specimens

Indenting these ceramic materials with a Vickers diamond pyramid or Knoop indenter resulted in the introduction of surface cracks of reproducible geometry and size controlled by the choice of indenter load. The technique has been described elsewhere [4–9] and is shown schematically in Fig. 1a. Typical cracks produced on the polished surfaces of test specimens of LAS and Si_3N_4 and corresponding fracture faces at 20°C are shown in Fig. 1b, c, d, and e, respectively. In the case of Refel SiC, some surface chipping occurred around the indentation site, and crack fronts are not clearly visible as in Si_3N_4 , LAS, and sialons. All indentations were made at room temperature.

Results and Discussion

Influence of Crack Size on Fracture Stress at Room Temperature

A series of specimens was prepared and precracked with microhardness indentation loads ranging from 200 to 4000 g. The variation of fracture stress with crack depth (as measured from the fracture face) for hot-pressed Si_3N_4 specimens tested at 20°C is shown in Fig. 2. Although there is considerable scatter in the data, a reasonably good linear relationship is evident, which correlates with the Griffith [10] criterion for brittle fracture. From such simple studies, an estimate of an important material parameter, namely the fracture energy, γ , of the material can be made as discussed elsewhere in detail for Si_3N_4 [11] and LAS [12]. From a practical point of view, perhaps the most useful application of the data shown in Fig. 2 is as a means of estimating the inherent flaw size of the as-received material. Extrapolation of the data in Fig. 2 to the fracture stress of uncracked specimens (shown as the approximate scatter band of materials strength) indicates that flaws of the order of 12 to 24 μm in depth are present in the as-received (uncracked) Si_3N_4 . Lange [13] and Mecholsky, Freiman and Rice [14] have also estimated the flaw size to be around 90 and 53 μm in a similar type of material, HS-130 Si_3N_4 , from their surface energy and mirror constant measurements, respectively. It is important to note that this can be taken only as a demonstration of principle. A body of evidence is accumulating which suggests that crack opening residual stresses can accompany hardness indentation-induced cracks in similar materials [5,6,9,15,16]. Although our own studies do not support the removal of residual stresses by annealing in air or vacuum in similar materials, the presence of such stress could influence significantly the absolute magnitude of the experimentally determined fracture energy. These data on Si_3N_4 are representative of the type of fracture data that can be obtained on other ceramic mate-

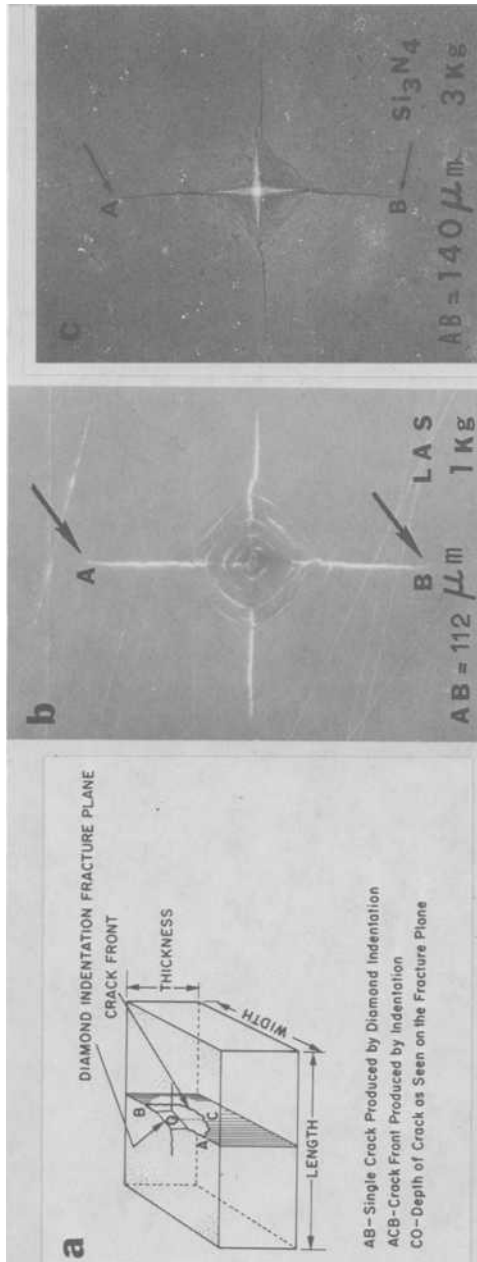


FIG. 1—(a) Schematic representation of crack geometry produced by microhardness indentation. (b) A typical crack produced on the polished surface of an LAS specimen using a diamond pyramid indenter with a 1000-g load. The picture is taken in polarized light. (c) A typical crack produced on the polished surface of a hot-pressed silicon nitride HS-130 specimen with a 3000-g indentation load. (d) A typical fracture surface of a precracked (1000-g indentation load) LAS specimen at 20°C. ACB is the original precracked region and CO is the depth of the crack. (e) A typical fracture surface of a precracked (3000-g indentation load) Si₃N₄ specimen at 20°C. The arrows identify the initial crack front boundary ACB.

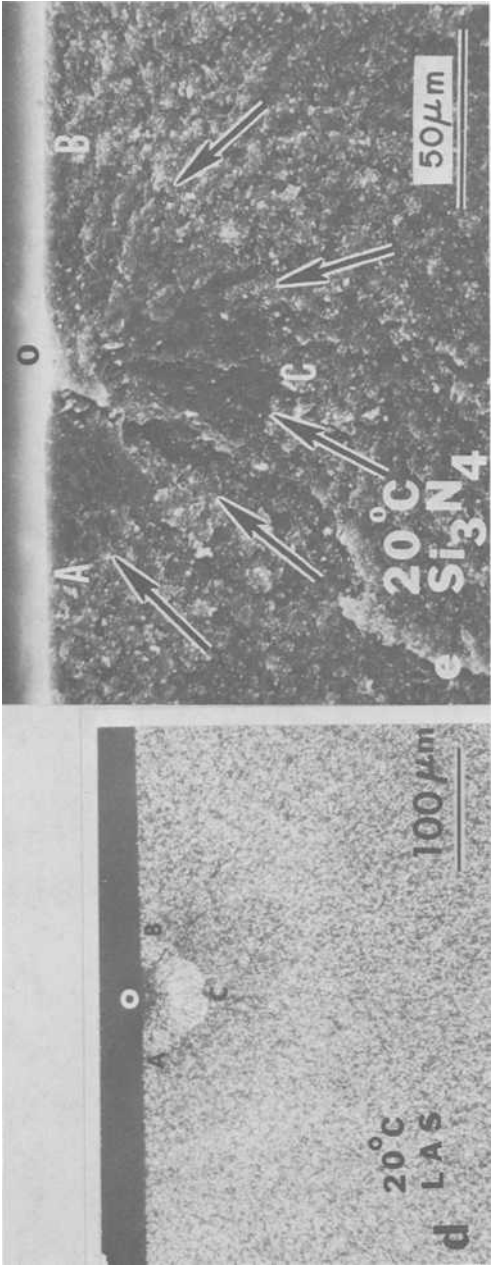


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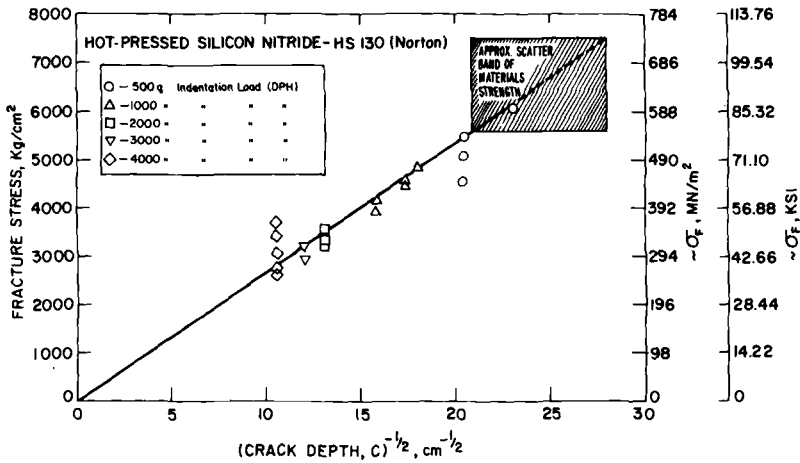


FIG. 2—Variations of fracture stress as a function of the inverse square root of the crack depth at room temperature for silicon nitride. Note that the data follow the simple Griffith [10] criterion (fracture-strength versus flaw-size relationship) for brittle fracture usually stated as, $\sigma_F \approx Y(E\gamma/c)^{1/2}$, where σ_F is the fracture stress, Y is a constant relating primarily to crack geometry (shape) and specimen dimensions, E is Young's modulus, c is the initial crack depth at which the fracture initiated, and γ is the fracture energy.

rials. For example, very similar data have been generated on LAS [12]. Consequently, the fracture dependence illustrated in Fig. 2 can be taken as generally representative of this semibrittle class of materials.

Effect of Temperature on Fracture Behavior

Basically three series of specimens were prepared for testing in the range 20 to 1400°C: a baseline series without any cracks, a second series precracked with a 4000-g (or 1000-g for LAS) indentation load, and a third series precracked with a 500-g (for LAS) indentation load. The temperature dependence of the fracture stress for all four ceramics, namely Si_3N_4 , LAS, Refel SiC, and sialons, is shown in Fig. 3a–d, respectively. In general, the fracture behavior is similar for all four materials and can be characterized by three regions: (1) the temperature-independent region, (2) the temperature-dependent region, and (3) the region of strength degradation due to subcritical crack growth (SCG); it is typically illustrated in Fig. 3a by Si_3N_4 .

Temperature-Independent Region—It is clear that σ_F for precracked specimens remains essentially constant and independent of temperature from 20 to about 1100°C for Si_3N_4 , and the upper limit of the temperature may vary from material to material. For example, in the case of LAS and sialons, it is about 700°C, whereas for Refel SiC it is about 800°C. The constancy of σ_F in this

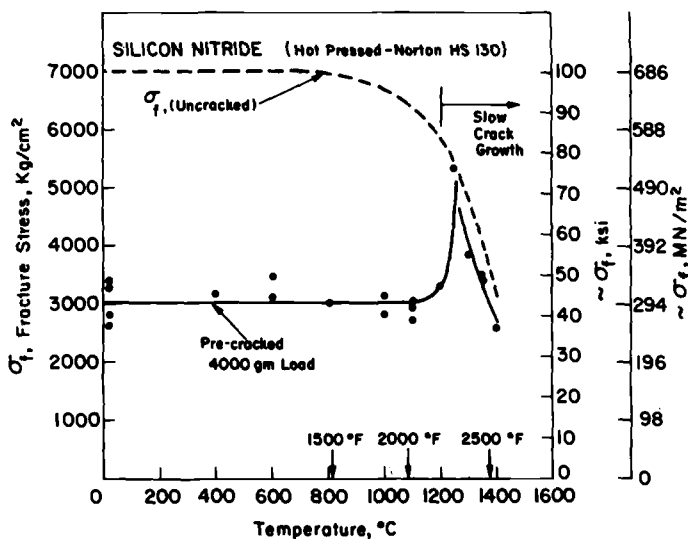
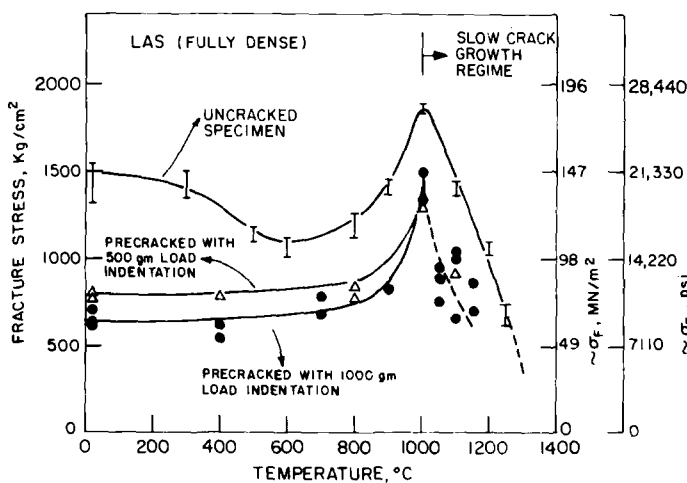
**a****b**

FIG. 3—Effect of temperature on the fracture stress of uncracked and precracked specimens of various ceramic materials. (a) hot-pressed silicon nitride. Precracked specimens contained a crack of about 90 μm depth. (b) LAS glass ceramic. Precracked specimens contained cracks of a constant depth of approximately 32 and 52 μm . (c) Refel SiC. (d) Yttria added sialons.

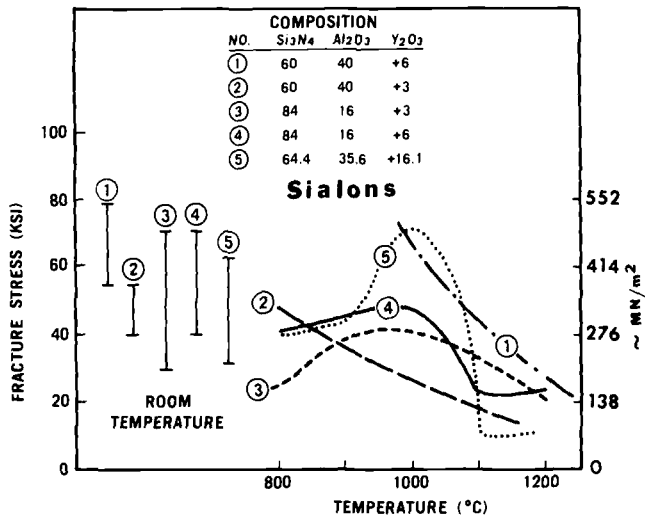
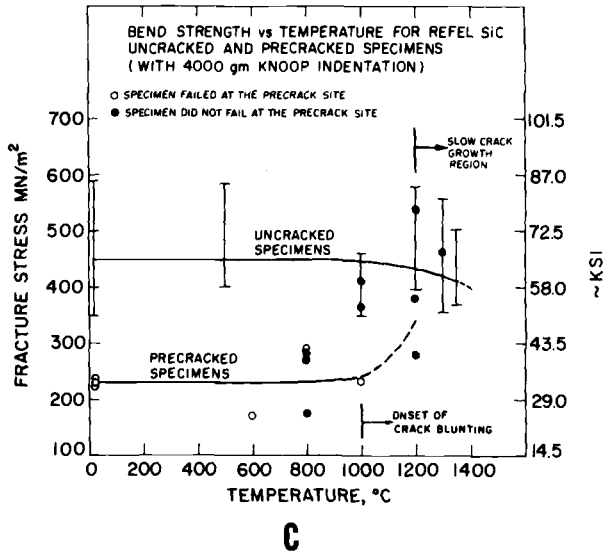


FIG. 3—Continued.

temperature range suggests that no change in the fracture mechanism occurs⁴ and indicates that there is little or no blunting of the crack tip by plastic deformation and that insignificant plastic deformation accompanied the fracture process. The mode of fracture during crack propagation can vary from material to material, with a mixed mode of fracture consisting of transgranular and intergranular crack growth for Si_3N_4 , Refel SiC, and sialons [11] and an exclusively transcrystalline cleavage for LAS [12]. It should be noted that in this region, catastrophic failure occurred directly from the initial microcrack for all materials. Specimens containing small flaws behaved in a similar fashion (Fig. 3b).

Temperature-Dependent Region—This region is characterized by a sudden increase in σ_F for precracked specimens, for example, above 1100°C leading to a maximum between 1200 and 1250°C for Si_3N_4 , (Fig. 3a), above 800°C leading to a maximum between 950 to 1000°C for LAS and sialons (Fig. 3b and d), respectively, and around 1200°C for Refel SiC (Fig. 3c). This sudden increase in σ_F leading to a peak value indicates that initial crack growth is more difficult as the temperature is raised. Since the crack path is confined to the grain boundary, the plasticity process at play is probably not a classical crack-tip blunting in the sense that crystal dislocation slip accommodation occurs, as is usually the case in single-crystal studies [4]. Rather, a displacement and general disruption of the continuity of the crack tip out of the emplaced crack due to viscous flow (softening of glassy phases) at the grain boundary is a more likely mechanism of crack blunting. Up to 1200°C for Si_3N_4 (or lower temperatures for LAS, Refel SiC and sialons), the fracture surface appearance was similar to that of the lower-temperature fractures (Fig. 1) and did not show any detectable presence of slow crack growth. Failure of precracked specimens occurred in a brittle (catastrophic) manner.

The crack blunting phenomenon is much more clearly displayed by Refel SiC and sialons. In Refel SiC, the precracked specimens did not break at the microcrack site at temperatures as low as 800°C, and at 1200°C the corresponding magnitude of σ_F for precracked specimens approached the magnitude of σ_F for as-received (uncracked) specimens (Fig. 3c). A high yttria-containing sialon displayed the crack blunting phenomenon in a classical fashion, as shown in Fig. 4a and b. The initial microcrack first widened prior to lengthening by means of a myriad of small branching cracks (possibly through the pores in the matrix), and the entire stressed tensile face became covered with slow-growing cracks before catastrophic failure.

Strength Degradation Due to Subcritical Crack Growth—The third region is characterized by a sudden decrease in strength (σ_F) for both as-received and precracked specimens caused by the presence of SCG. The temperature for the onset of SCG varies from material to material and is about 1200, 950, 1200 to

⁴Undoubtedly, many ceramic materials show significant variations in strength and changes in fracture mechanism for a given temperature due to environmental effects that are not examined in this study.

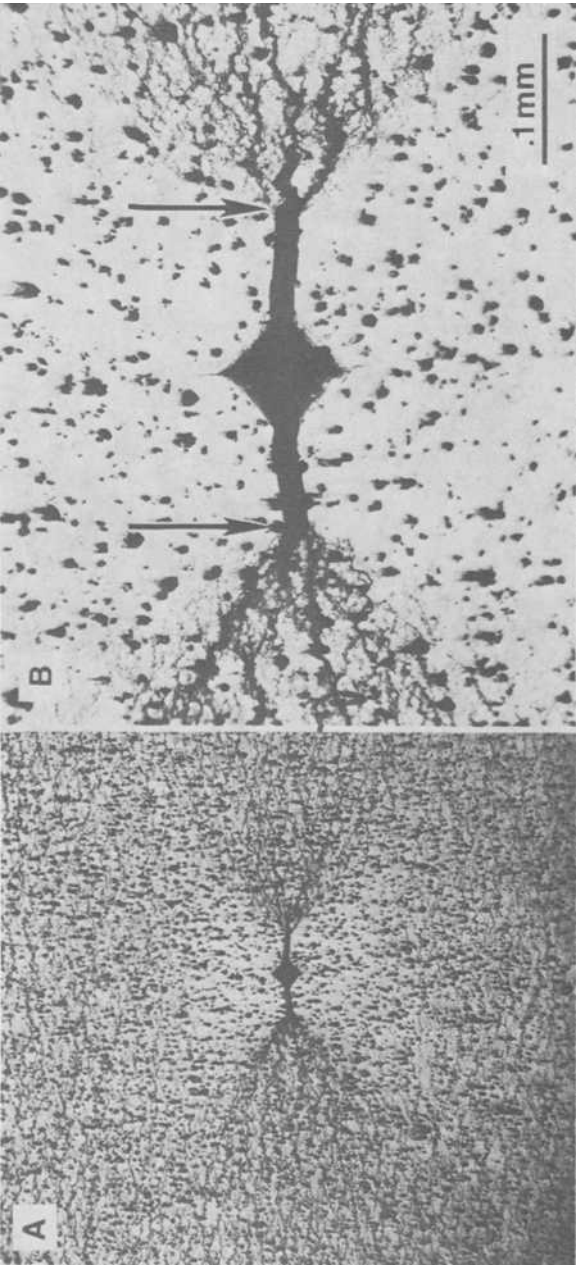


FIG. 4—Crack blunting through microcrack formation in a 64.4 percent Si_3N_4 + 35.6 percent Al_2O_3 + 16.1 percent Y_2O_3 sialon.

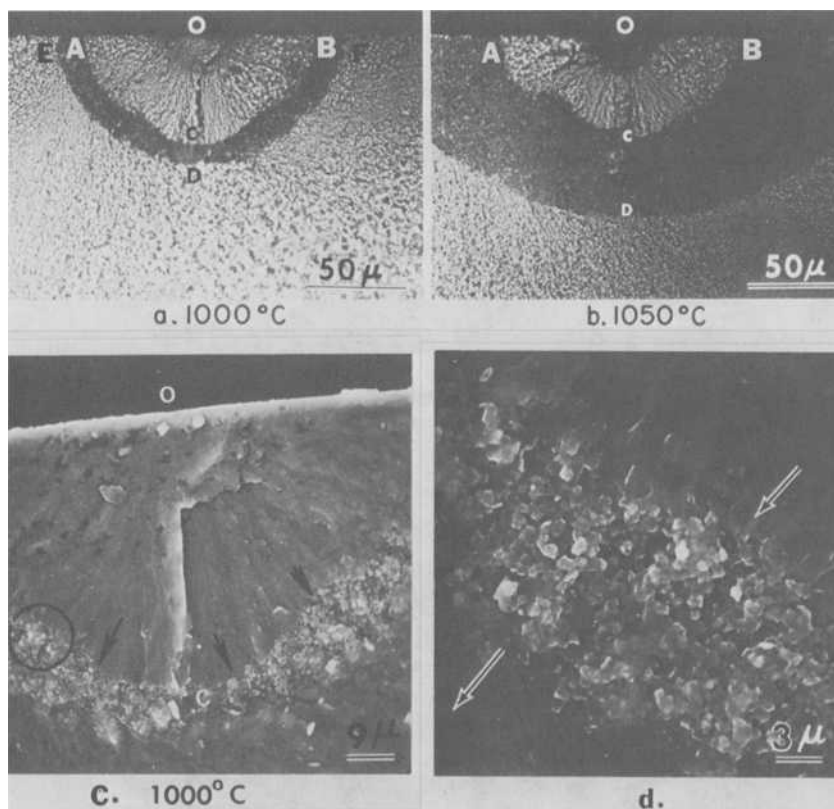


FIG. 5—(a–b) Typical fracture surfaces of LAS specimens precracked with a 1000-g load indentation and tested in four-point bending at 1000 and 1050°C. ACB is the initial crack. The dark band surrounding ACB is the slow crack growth region. (c–d) Structure and path of slow crack growth region as revealed in SEM [same specimen as viewed in (a)]. (c) Detailed view of precracked region, ACB; the arrows indicate the boundary of the initial crack front. (d) Circled area shown in (c) at a higher magnification. Note that the mode of crack propagation in the SCG region is completely intergranular. The photomicrograph also shows the transition from the intergranular crack growth to fast fracture. The arrows indicate the direction of crack propagation.

1300, and 900°C for Si_3N_4 , LAS, Refel SiC, and sialons, respectively. The nature and mode of crack propagation during SCG are entirely different from those observed in fast fracture, and they will be discussed for LAS.

The first signs of SCG in LAS specimens were observed on the fracture faces in tests made at 1000°C or higher (Fig. 5). The extent of SCG prior to catastrophic failure increased as the test temperature increased (compare Fig. 5a and b). The mode of crack propagation during SCG, revealed clearly by SEM (Fig. 5c and d), is entirely intergranular, whereas catastrophic failure occurs in a transgranular mode. The large differences in light scattering from the two types of fracture paths lead to clear demarcation of the SCG region (Fig. 5a and b) under conventional optical light examination. The transition from an

intergranular mode of SCG to a transgranular cleavage mode (or a mixed mode for nitrides) accompanying high-speed fracture indicates that a fine balance exists between the alternative crack paths. In high-speed fracture, plastic deformation integral to crack growth is minimized due to the very high strain rate in the vicinity of the crack tip, and the crack finds a transgranular path easier than a more deformation-accommodating grain boundary region. During SCG, the crack extends by plastic separation of what appears to behave as a ductile, low-viscosity grain-boundary glass phase, at stress levels considerably lower than that necessary to promote fast (or cleavage) fracture. When the stress intensity at the tip of the crack has increased sufficiently due to crack lengthening by SCG, a point is reached beyond which fast fracture is more favorable (the Griffith condition is satisfied), and a transition to high-speed catastrophic failure occurs (Fig. 5).

This behavior occurred in all four ceramics, although in Refel SiC, the SCG is not as clearly visible on the fracture face as it is in LAS, Si_3N_4 , and sialon. However, the SCG in Refel SiC can be seen on the polished tensile surface of a precracked specimen subjected to constant stress (just below the fracture stress level, Fig. 3c) at 1300°C (Fig. 6). Typical fracture surfaces showing SCG with increasing temperature are shown for Si_3N_4 (Fig. 7) and sialon (Fig. 8).

Furthermore, the temperature at which the two curves (uncracked and precracked specimens in Fig. 3) merge characterizes the importance of SCG and points out that failure is governed by the extent of SCG and not by the initial crack size.

Strain Rate and Stress Rupture Observations

The strain-rate sensitivity of SCG was determined in specimens containing a constant crack size (about 52 μm deep in the case of LAS and 90 μm deep for HS-130 Si_3N_4) and tested at a constant temperature in the SCG region, for example, 1100°C for LAS, as shown in Fig. 9, and 1300°C for Si_3N_4 [11]. Similar behavior was shown by sialons, but Refel SiC did not show clearly such qualitative behavior. Increasing the strain rate (or the machine head speed) makes the material more brittle and initially raises the fracture stress. In agreement with this view, the extent (or depth) of SCG decreased as the strain rate increased until it disappeared at a machine head speed of about 12.7 mm/min (0.5 in./min.), and fracture was instantly catastrophic from the original crack depth (CO in Fig. 9). The form of the rate dependence of fracture stress for precracked specimens in the temperature-strain-rate regime in which SCG is competitive is based on the nature of the plastic response localized at grain boundaries and was discussed fully for LAS earlier [12].

Thus far, it has been clearly demonstrated that SCG in hot-pressed Si_3N_4 , LAS, Refel SiC, and sialons starts at temperatures above 1200, 950, 1200, and 900 to 1000°C, respectively. To simulate practical applications, preliminary stress rupture tests were made in Si_3N_4 and LAS to determine the extent of

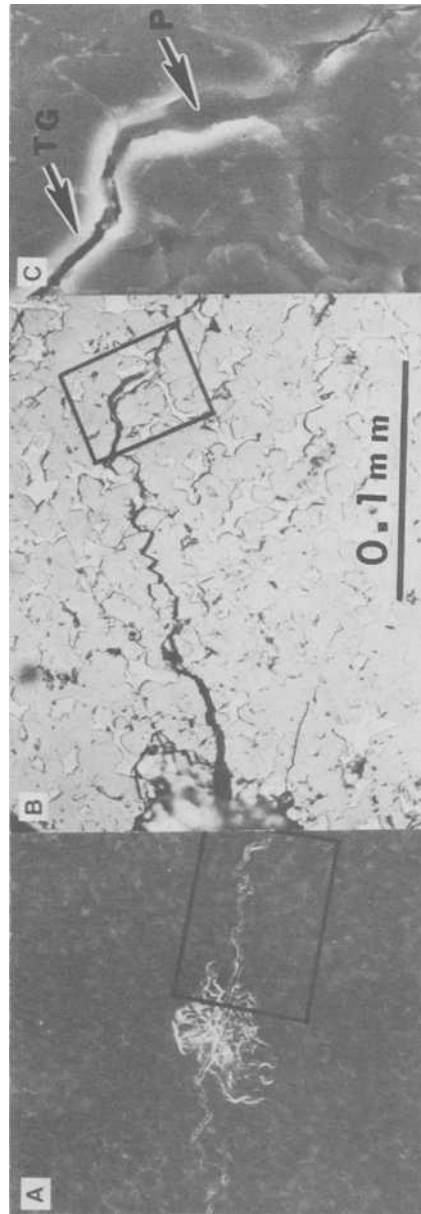


FIG. 6—(a) An SEM photomicrograph of crack extension in Refel SiC around an indented surface (Vickers diamond pyramid indenter with 4000-g load) under load at 1300°C. (magnification $\approx \times 100$). (b) Optical photomicrograph of the area indicated in (a) which illustrates the disposition of the silicon phase and the fracture path (magnification $\approx \times 320$). (c) A SEM photomicrograph of the area marked in (b). Plastic accommodation in silicon (P), as well as transgranular fracture of SiC grains (TG) (magnification $\approx \times 900$).

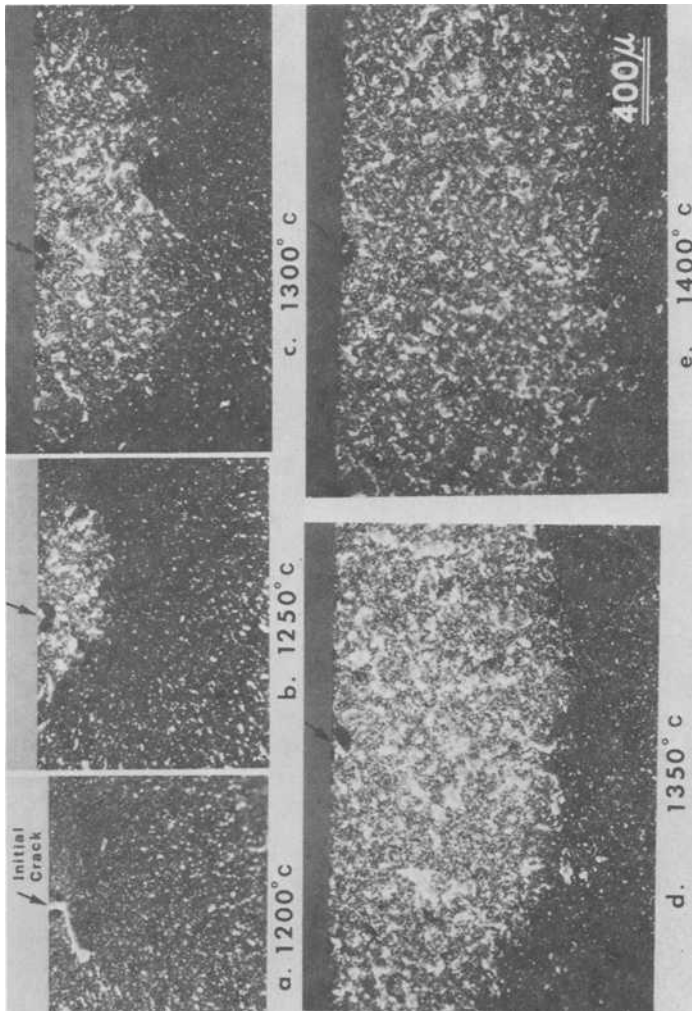


FIG. 7.—Typical fracture surfaces of precracked (crack depth $\sim 90 \mu\text{m}$) HS-130 Si_3N_4 specimens tested at a machine head speed of $\sim 0.127 \text{ mm/min}$ (0.005 in./min.) at various temperatures. The pictures were taken with plain polarized light. Note the absence of SCG at 1200°C and subsequent appearance of SCG surrounding the initial crack at 1250°C and higher temperatures. All photomicrographs were taken at the same magnification.

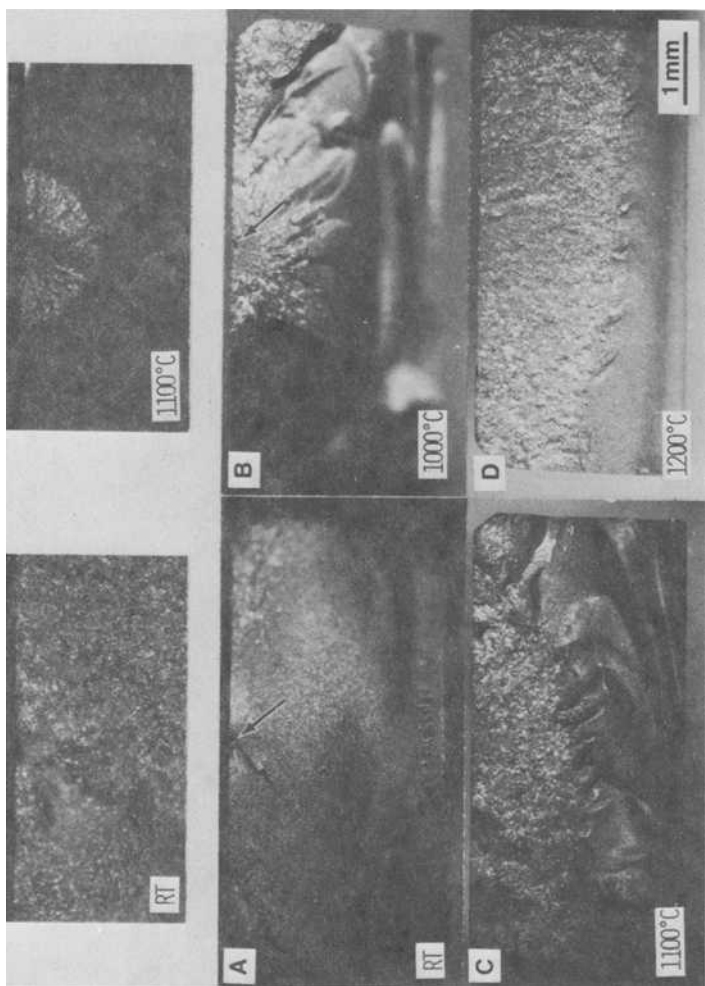


FIG. 8—Typical temperature dependence of the extent of SCG in a sialon material. The top insets are high magnification photomicrographs of the fracture utilizing the flaws indicated by the arrow (A-D). All photos were taken at the same magnification.

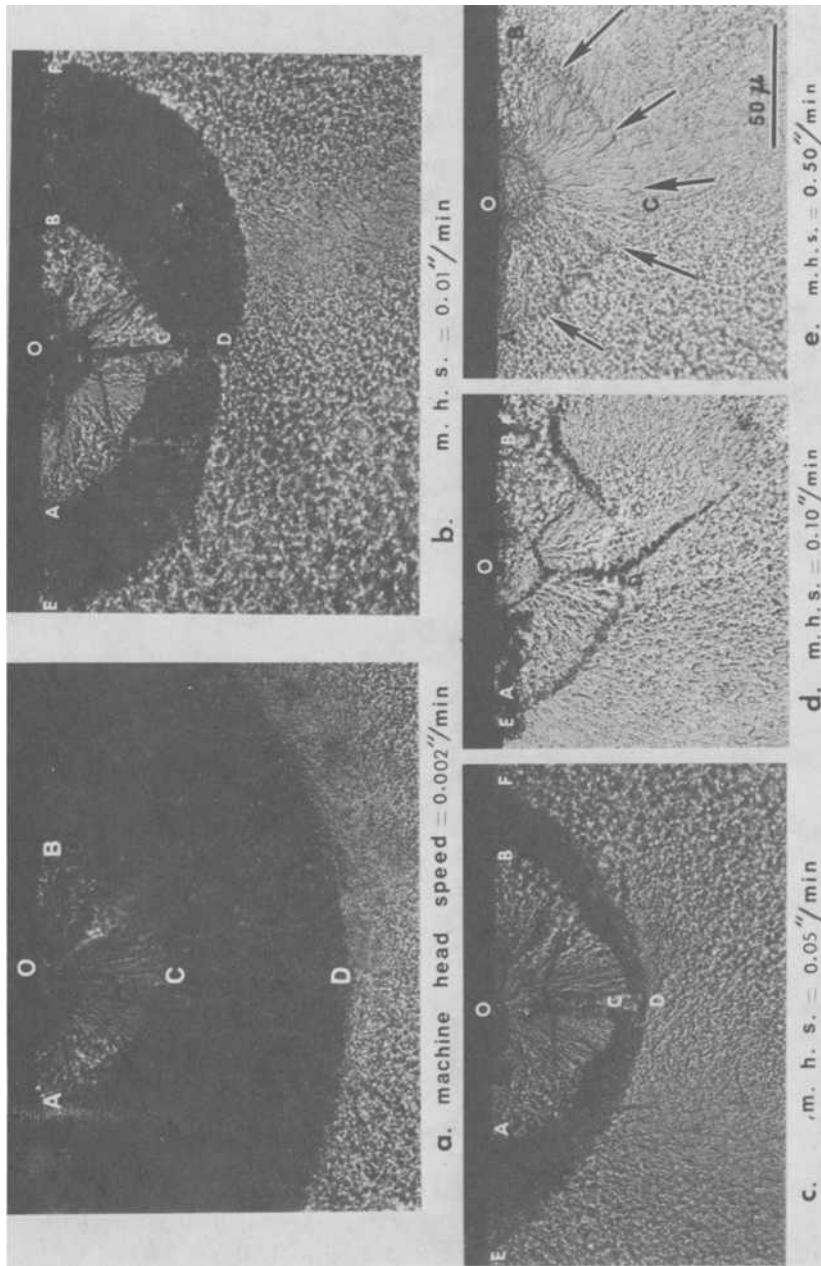


FIG. 9.—Successive stages in the fractographic appearance of precracked (1000-g indentation load) LAS specimens tested at 1100°C as a function of the machine head speed. All photomicrographs were taken at equal magnification.

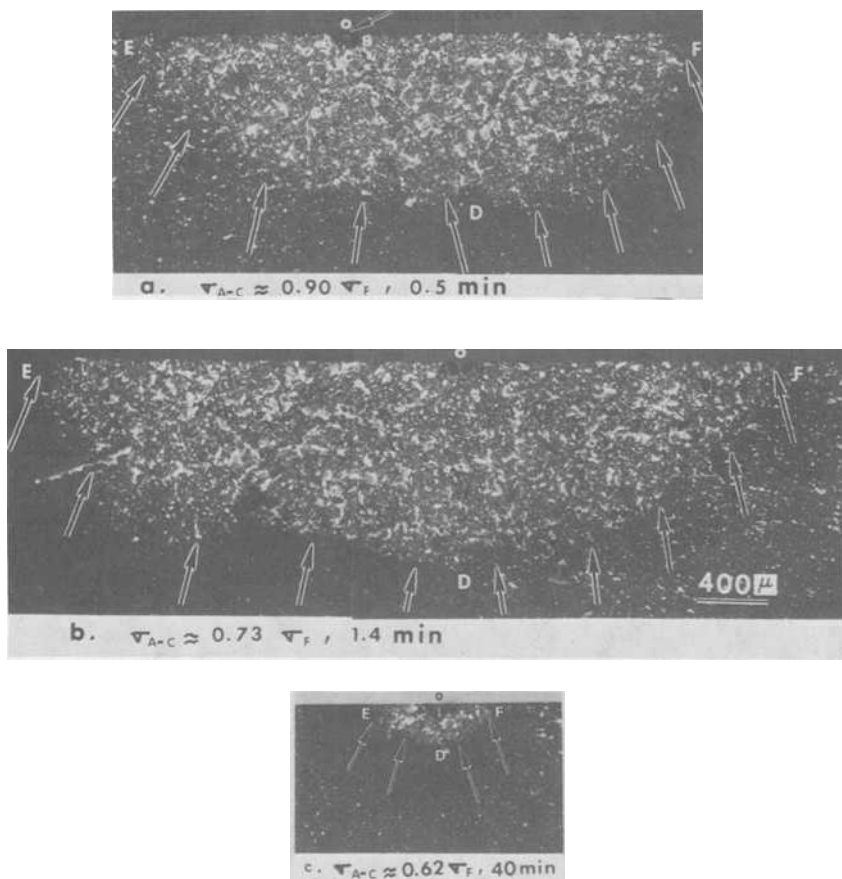


FIG. 10—Successive stages in the movement of the slow crack growth (SCG) region of HS-130 Si₃N₄ specimens, tested at varying stress levels at 1300°C. All specimens contained cracks of about 90 μm depth. All photomicrographs were taken with plain polarized light and equal magnification. (a) The specimen was loaded to a stress level of $\sim 0.90 \sigma_F$, which was kept constant. The specimen failed after 0.5 min. (b) The specimen was loaded to a stress level of $\sim 0.73 \sigma_F$, which was kept constant, and failed after 1.4 min. (c) The specimen was loaded to a stress level of $\sim 0.62 \sigma_F$, which was kept constant for 40 min., and was finally broken at 20°C to reveal the extent of SCG.

SCG when precracked specimens (constant initial crack size) were subjected at 1300 and 1000°C to a fraction of the fracture stress, as illustrated in Figs. 10 and 11, respectively. Examination of the photomicrographs indicates that, when the magnitude of the applied stress is very large ($\geq 0.9 \sigma_F$), rapid extension of the initial crack by SCG and catastrophic fracture occurred within a period of 0.5 to 2 min. At intermediate-stress levels (around $0.75 \sigma_F$), both the extent of SCG and time to failure increased substantially (Figs. 10b and 11b). Note that the growth of the initial crack in the depth direction (CO to DO) was much smaller than the growth along the edge, that is, transverse (AB to EF,

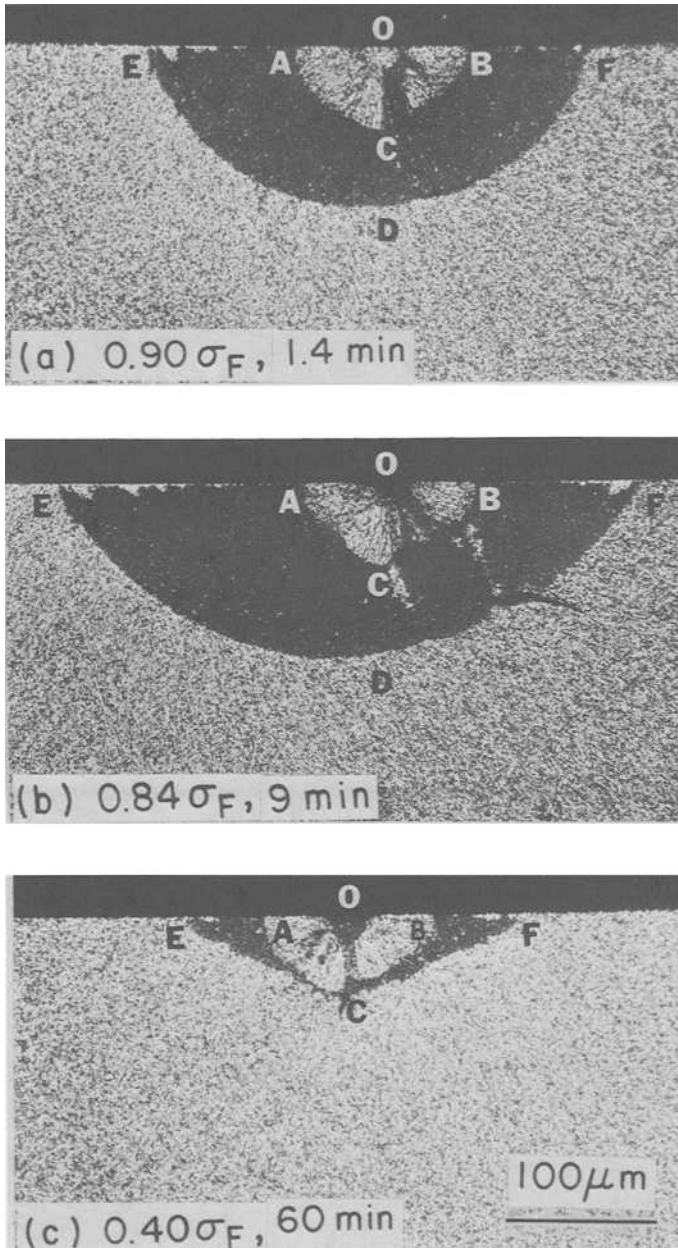


FIG. 11—Successive stages in the movement of the slow crack growth region of LAS specimens tested at varying stress levels at 1000°C . All the specimens were precracked with 1000-g load indentation. (a) The specimen was loaded to a stress level of $\sim 0.90 \sigma_F$, which was kept constant. The specimen fractured at the precracked site after 1.4 min. (b) The specimen was loaded to a stress level of $\sim 0.84 \sigma_F$, which was kept constant, and finally failed at the precracked site after 9 min. (c) The specimen was loaded to a stress level of $\sim 0.4 \sigma_F$, which was kept constant for 60 min., and finally was unloaded and subsequently broken at room temperature. All photomicrographs were taken at equal magnification.

Figs. 10b and 11b). This presumably is due to the stress distribution in a bend specimen in which the maximum stress in bending occurs along the outermost fibers on the tensile surface, that is, along the edge (*EOF*). At low-stress levels (around 0.4 to 0.6 σ_F), the extent of SCG decreased drastically while the time to failure increased as expected (Figs. 10c and 11c). In both cases, the specimens did not break even after sustaining the stress for 40 and 60 min for Si_3N_4 and LAS (Figs. 10c and 11c), respectively, and they were unloaded and broken at 20°C in order to reveal the extent of SCG. It can be inferred from these data that there exists a critical stress level below which no significant SCG will occur. To define precisely such a critical stress, long-term creep tests should be carried out.

Conclusions

The temperature dependence of fracture for precracked ceramics can be characterized in terms of three different stages of crack propagation, variation between the different ceramics is only in the temperature limits of the regions, and the same generic fracture processes always occurred.

All the materials examined exhibited a region of slow crack growth which was greatly influenced by strain rate and temperature effects.

In general, fast fracture was characterized by the presence of transgranular crack propagation, SCG was characterized exclusively by intergranular crack propagation.

The morphology of the particular fracture processes differs in the clarity found in the different materials. For example, in LAS all types of fracture are readily discernible; in Refel SiC, the different fracture mechanisms are extremely difficult to distinguish.

Acknowledgments

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Fractographic Analysis of Delayed Failure in Ceramics

REFERENCE: Mecholsky, J. J. and Freiman, S. W., "Fractographic Analysis of Delayed Failure in Ceramics," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 246-258.

ABSTRACT: Fracture surfaces of bars of glass and ceramics that failed after time under constant load were compared with those generated in rapid failure experiments. For each material, the relationship between the fracture stress, σ , and the fracture mirror radii, r ($\sigma r^{1/2} = \text{a constant}$), was the same for rapid and delayed failure. The ratio of the mirror radius to the critical flaw size for delayed failure is a constant equal to that found previously for rapid failure. However, the ratio of the fractured mirror radius to the initial flaw size increased with increasing time under load due to the growth of the fracture-initiating flaw from the original size to the "critical" size before catastrophic failure. Slow crack growth in several polycrystalline ceramics occurred primarily by intergranular fracture, whereas primarily transgranular fracture occurred during rapid crack propagation. Thus, the boundary between subcritical and catastrophic flaw growth can be quite distinct. The fracture mechanics analysis presented in this paper relates the time under load to the ratio of the mirror and the initial flaw dimensions. This analysis gives good agreement with most experimental observations.

KEY WORDS: fractography, quantitative fractography, delayed failure, fracture (materials), fracture mechanics, ceramics, brittle materials, mechanical properties, materials, materials science

Fracture surface features, such as the fracture-initiating flaw and fracture mirror boundaries, are related to the stress at fracture and to the fracture toughness parameter, K_{IC} , when failure occurs rapidly, as in strength tests [1-3].³ It will be shown that fractography can be used in analysis of delayed failure and that the fracture surface morphology in polycrystalline materials depends on crack growth rates. Slow crack growth in delayed failure will be discussed first for glasses; the effect of polycrystalline ceramic microstructure on flaw growth and development of surface features then will be described.

¹Member of technical staff, Sandia National Laboratories, Albuquerque, N. Mex. 87185.

²Ceramic Engineer, National Bureau of Standards, Washington, D.C. 20234.

³The italic numbers in brackets refer to the list of references appended to this paper.

Experimental Procedure

Delayed failure tests were performed [4,5] on bars (approximately 2 by 4 to 8 by 25 mm) of glass and polycrystalline ceramics using 4-point flexure with inner and outer spans of 7.6 and 22.9 mm, respectively. Loads were applied on eight test fixtures by gradually lowering the screw-operated platform supporting the appropriate weights until the desired stress was achieved; a micro-switch stopped an electric timer when failure occurred. The test atmosphere was controlled at $21 \pm 1^\circ\text{C}$ and 50 ± 5 percent relative humidity. The fracture surfaces were examined by optical and scanning electron microscopy; inner and outer mirror radii and flaw size measurements were made as described elsewhere [3].

Delayed Failure in Glasses

A schematic drawing of a typical fracture surface on which slow crack growth has occurred illustrates the important features of delayed failure (Fig. 1). The initial flaw (solid line, Fig. 1), produced by machining or handling, is observed on the fracture surface. The line of demarcation is usually easily seen because the stress state during testing is almost never the same as that which produced the flaw. The initial flaw grows subcritically under load to a size at which the stress intensity reaches a critical value, K_{Ic} . At this point, catastrophic failure occurs—the processes leading to mist, hackle, and crack branching follow. These processes occur at specific values of K_I , independent

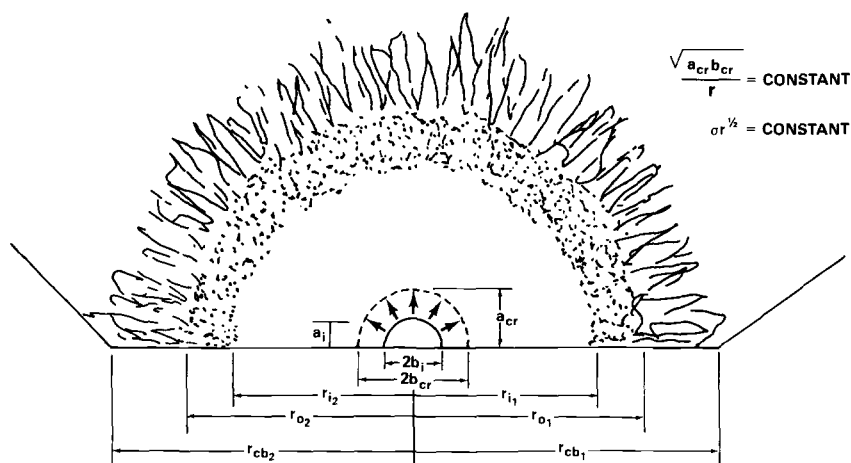


FIG. 1—Schematic drawing of a fracture surface of a brittle material subjected to a constant subcritical load, σ . The solid semielliptical line at the center represents the initial flaw size, and the dashed line represents the outline of the critical flaw size before catastrophic failure. The inner, r_1 , outer, r_2 , and crack-branching, r_{cb} , mirror radii are shown in the figure (the prime indicates that there may be unsymmetrical mirrors).

of the amount of subcritical crack growth. The radii from the origin to the boundaries between the mirror and mist regions (r_1 in Fig. 1), the mist and hackle regions, r_2 , and crack branching, r_{cb} , are related, respectively, to the stress, σ , at failure: $\sigma r_j^{1/2} = \text{a constant} = M_j$, where $j = 1, 2$, or cb . Since the transition from stable to unstable crack growth is continuous and smooth, there is no observable critical flaw boundary. The critical flaw size, c_f , can be calculated, however, from a fracture mechanics expression [6]

$$c_f = \frac{\Phi^2 K_{Ic}^2}{1.2\pi\sigma_a^2} \quad (1)$$

where σ_a is the applied stress (that is, $\sigma = \sigma_a$), and Φ is a geometric constant.

The fracture surface for glass that failed after approximately 200 h under a stress of 60 MPa is shown in Fig. 2. The mirror-to-flaw-size ratio of 80:1 compares with 13:1 observed for glasses tested rapidly to failure [1, 2]. This ratio difference between delayed and rapid failure can be explained by the subcritical crack growth of the initial flaw to the critical flaw size. A summary of data obtained for soda-lime-silica glass bars (Table 1) shows that the inner and outer mirror constants, M_1 and M_2 , respectively, are unaffected by the testing rate; however, the ratios of the inner and outer mirror radii to the initial flaw size are variable and appear to be a function of the time to failure, that is, $f(t_f)$. Because the mirror-to-critical-flaw-size ratios do remain the same as in rapid failure, fracture mechanics expressions based on subcritical crack growth can be used to determine the time under load.

A good representation of slow crack growth in brittle materials is given by Ref 7

$$V = V_0 \frac{K_I^n}{K_0^n} = AK_I^n \quad (2)$$

where V is the velocity of the crack, V_0 , A , and n are experimentally determined crack growth parameters, and K_0 is a normalizing factor usually taken as

TABLE 1—Fracture surface measurements for soda-lime-silica glass bars.

	Rapid Failure	Delayed Failure
M_1 (MN/m ^{3/2}) ^a	1.92	2.0 ± 0.1
M_2 (MN/m ^{3/2}) ^a	2.21	2.2 ± 0.1
r_1/c_f	12.5	$f(t)$ [4]
r_2/c_f	16.7	$f(t)$ [4]
r/c_f	12.5	11.6 ± 1.2
r_2/c_f	16.7	14.7 ± 1.6

^aFrom Eq 5, $M_j = \sigma_a r_j^{1/2}$.

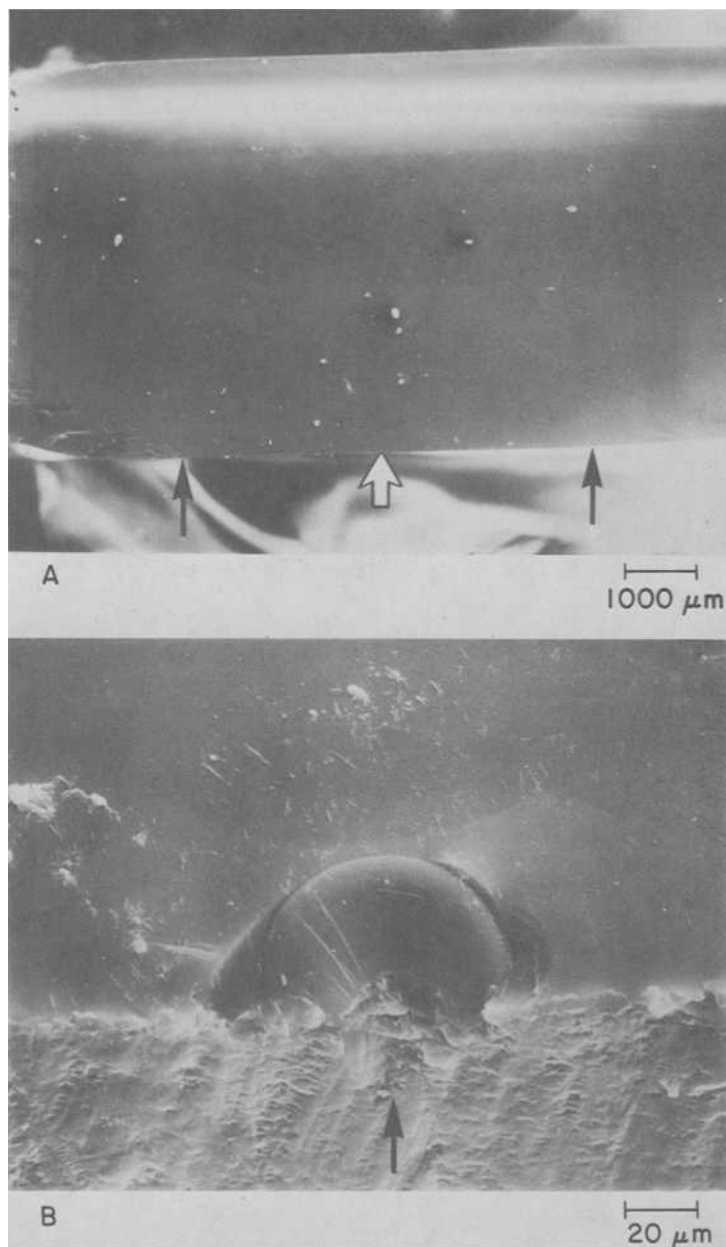


FIG. 2—Scanning electron photomicrograph of a typical soda-lime-silica bar fractured after 200 h at 60 MPa. The fracture origin is shown in (a) by the white arrow, and the beginning of the fracture mirror boundaries are shown by the black arrows. (b) is a higher magnification of the preexisting flaw, the smooth, approximately half-penny-shaped demarcation in the center of (b). The chip within the flaw (arrow) is probably the result of a grinding striation. The ratio of the inner mirror, r_1 , to the initial flaw size, $\sqrt{a_0}$, is 80, which indicates a relatively long time under load.

1 MPa/m^{1/2}. Since V is dc/dt , the failure time, t_f , can be determined through integration

$$t = \int_{c_i}^{c_f} \frac{dc}{V} \quad (3)$$

where c_i is the size of the initial flaw, and c_f is the size of the critical flaw. The result of this operation, using Eqs 1 and 2, is given as Eq 4 [4]

$$t_f = \frac{2}{A(n-2)} \left[\frac{(1.2\pi)^{1/2}}{\phi} \sigma_a c_i^{1/2} \right]^{-n} c_i \quad (4)$$

This failure time can be expressed as a function of the mirror dimensions by substituting the following into Eq 4

$$\sigma_a = M_j r_j^{-1/2} \quad (5)$$

where M_j is the mirror constant corresponding to r_j , and j is either 1, 2, or cb ; that is, r_1 , r_2 , and r_{cb} are the mirror-mist, mist-hackle, and crack-branching radii, respectively. The following expression is obtained

$$t_f = \frac{2}{A(n-2)} \left[\frac{(1.2\pi)^{1/2}}{\phi} M_j \left(\frac{c_i}{r_j} \right)^{1/2} \right]^{-n} c_i \quad (6)$$

Equations 4 and 6 relate the time to failure under constant stress to the initial flaw size and geometry (Eq 4) and the fracture mirror size (Eq 6), respectively. The data in Ref 4 are plotted in Fig. 3 based on the form suggested by Eq 4.

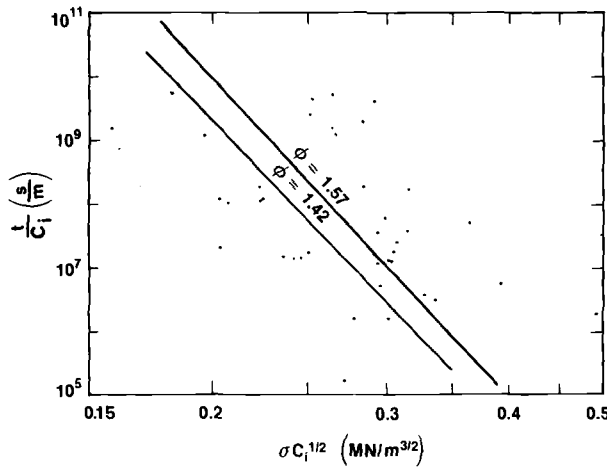


FIG. 3—Time to failure as a function of the applied stress, σ , and initial flaw size, $c_i = \sqrt{a_i b_i}$. The solid lines represent the calculated time to failure (Eq 4) for a semicircular flaw ($\phi = 1.57$) and a flaw with $a/b = 0.8$ ($\phi = 1.42$).

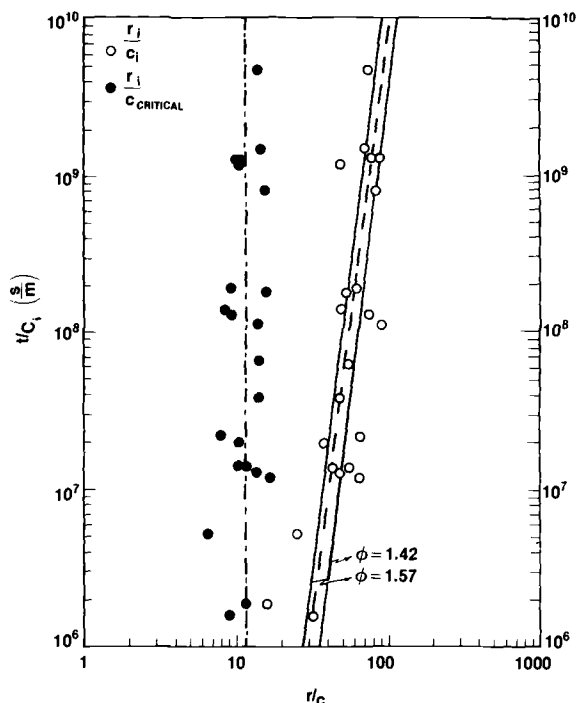


FIG. 4—Inner-mirror, r_i (r_i in Eq 5), to flaw-size ratio as a function of time under load; c_i is $\sqrt{a_i b_i}$ from Table 1, and the critical flaw size, c_i is determined from Eq 1. The solid lines represent Eq 6 for a semicircular flaw ($\phi = 1.57$) and one with $a/b \approx 0.8$ ($\phi = 1.42$). The dashed line is a least-squares fit to the data with a correlation coefficient of 0.72. The broken line represents the r_i/c_i value of 11.6 ± 1.2 from Table 1. It is drawn parallel to the ordinate as an aid to the eye. The least-squares fit has a slight positive slope.

The predicted curves were obtained by inserting values of n and A [8] into Eq 4. The two curves shown in Fig. 3 correspond to geometric factors of ϕ for a semicircular flaw (1.57) and for a flaw with an aspect ratio of 0.8 (1.42). Although the theoretical curves pass through the approximate center of the data, there is extensive scatter. A least-squares fit to the data had a correlation coefficient of 0.55.

Equation 4 is based on the assumption that the initial flaw lies in a plane perpendicular to the maximum applied stress, σ_a , which has been shown not necessarily to be the case [8]. Most flaws grow from an initial elliptical shape to a semicircular mirror; the shape change with time is not accounted for in the derivation of Eq 4.

Previous studies suggest that the fracture mirror is a measure of the stress at fracture even when the flaw is inclined at an angle to the plane of fracture [8,9]. The time-to-failure data were, therefore, plotted according to Eq 6 (Fig. 4) using all the data for which mirror-mist boundaries could be determined (22 points). The theoretical curves were obtained by inserting the values of frac-

ture mirror constants for soda-lime glass in Eq (6) along with the values of n and A . The dashed line is the least-squares fit to the data and has a correlation coefficient of 0.72. The critical flaw sizes calculated from Eq 1 are also shown. For every t/c_i point, there is a corresponding value of t/c_i for the same mirror radius. The best-fit line to the t/c_i data points corresponds to an r_i/c_i ratio of 11.6 ± 1.2 ,⁴ which is in good agreement with that obtained with rapid failure [1].

The correlation coefficient for a least-squares fit using only 22 of the data points in Fig. 3, corresponding to the data in Fig. 4, is the same (0.55) as it is using all the data in Fig. 3. Apparently, the scatter in c_i is compensated for by changes in r_i . The authors attribute this compensation to physical phenomena rather than to random scatter. The geometry and orientation of the flaw is different from that of the mirror.

As mentioned, the geometry at the initial flaw can be quite complicated and vary from specimen to specimen, whereas the geometry at the mirror boundary is usually the same for each specimen. Thus, the assumption that ϕ is 1.57 (for a semicircular shape) is probably true for Eq 6 but not for Eq 4. In addition, because of the shape of the flaw and its angle to the applied stress, σ_a , the stress at which crack propagation begins probably varies from the applied stress, depending on the angle of the flaw to σ_a . The mirror, on the other hand, is wholly contained within the fracture plane and the stress at formation is the applied stress. The formation of the mirror evidently compensates for any irregularities at the initial flaw.

The K_I at which catastrophic failure occurs does not depend on the previous crack growth history, as evidenced by the agreement in values of r_i/c_i for large variations in t_i . This evidence means that knowledge of r_i gives information regarding c_i that, combined with the observed c_i , allows the extent of the subcritical crack growth to be determined. Obviously, this analysis can only be performed after the specimen is broken and, thus, cannot be used for time-to-failure predictions. However, for in-service failures, when no other information is given, measuring the mirror and the initial flaw size provides a reasonable way of estimating the total time that the structure was subjected to load. This estimate is only good to approximately 2 orders of magnitude in time, but it is as good as any predictive capability that exists now [7]. An implied assumption⁵ is that the stress on the flaw was constant for this period.

Delayed Failure in Polycrystalline Ceramics

The fracture surface of magnesium fluoride (MgF_2) shown in Fig. 5 is an excellent example of delayed failure in polycrystalline ceramics. Three distinct regions of interest are shown in this figure: (1) The initial flaw is a crack ex-

⁴ 95 percent confidence limits.

⁵ This is not a necessary assumption. Analogous equations can be developed for the case of constant strain rate.

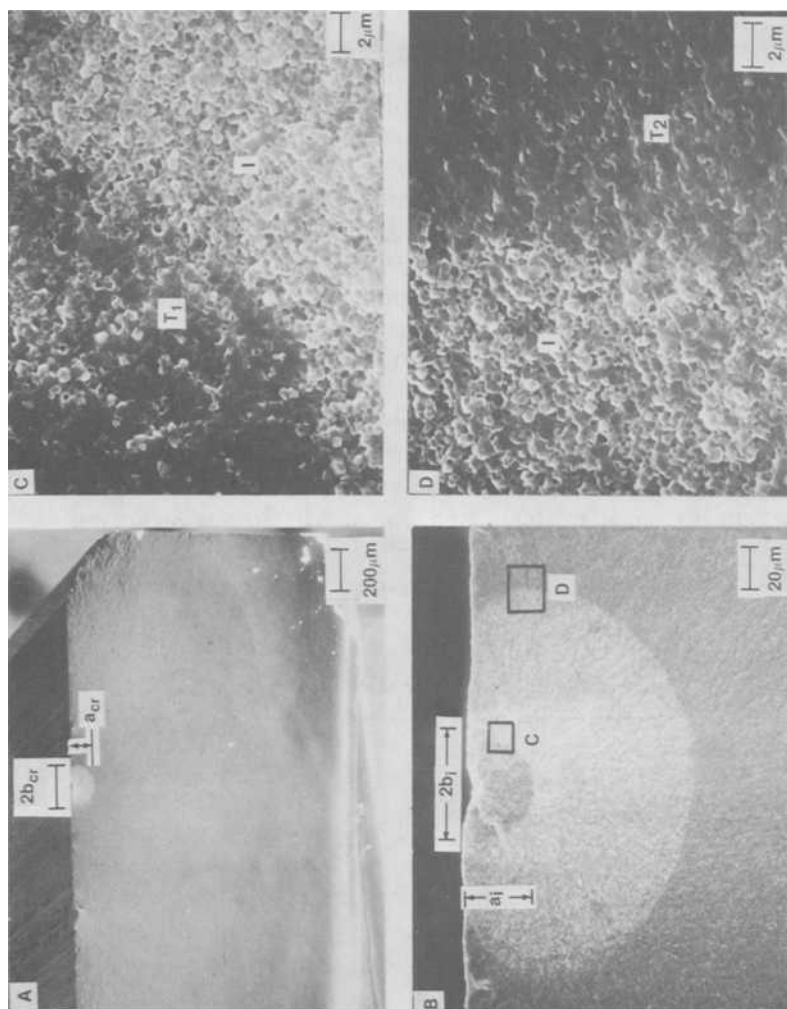


FIG. 5.—Scanning electron photomicrograph of the fracture surface of a MgF_2 bar failed after 2498 h at 88 MPa. The critical flaw dimensions are shown in (a), along with the mist and hackle. (b) shows a higher magnification of the initial flaw (a_i , b_i) and critical flaw. (c) is a magnification of the region at Location C in (b). Location T_1 indicates the primarily transgranular region within the initial flaw, and T_2 indicates the transition between the primarily intergranular nature of crack growth between the initial and critical flaw sizes. (d) shows the transition between the primarily intergranular region, T_2 , to the primarily transgranular region, T_1 , at the critical flaw boundary.

tending from the tensile surface; it probably occurred during machining and is primarily transgranular. (2) The light region surrounding the initial flaw (Fig. 5a) is a primarily intergranular region of fracture and corresponds to the distance over which subcritical crack growth occurred. (3) There is a primarily transgranular region outside the light region shown in Fig. 5c, wherein fast fracture took place. As shown in Table 2, calculations of critical flaw sizes (Eq 1) for the most part agreed with the size of the intergranular region. Equation 6 was applied to the MgF_2 data [10, 11] in order to predict failure times from the ratios of the mirror to the initial flaw size (Table 3). The reason for the disagreement in the measured and calculated failure times is not known. It can be speculated, however, that the slow crack growth parameters may be altered by long-term stress corrosion.

The authors previously observed delayed failure in chemically vapor-deposited (CVD) zinc selenide (ZnSe) [5]. An example of a delayed fracture surface of CVD ZnSe is shown in Fig. 6. Failure is characterized by an intergranular region surrounding the initial machining flaw (2 to 20 μm in size); fracture is transgranular over the remainder of the specimen. This intergranular region is attributed to subcritical crack growth. The boundary radius agrees quite well with critical flaw size calculations. In contrast, fracture surfaces of specimens failing during loading are entirely transgranular in nature, with the critical flaw contained within large grains [12]. Chemically vapor-deposited ZnSe is a relatively large-grained polycrystalline ceramic; the average grain size ranges from 50 to 70 μm , and grains of 300 to 500 μm are not uncommon. Large differences in the predicted times to failure depend on whether single-crystal or polycrystalline crack growth parameters are used in

TABLE 2—Comparison of calculations and observations for MgF_2 .

Specimen No.	Critical Flaw Sizes, c_f , μm		Failure Times, s	
	Observed ^a	Calculated ^b	Observed	Calculated ^c
1	>84	67	10	1×10^{-3}
2	>67	55	30	3×10^{-3}
3	58	48	360	8×10^{-2}
4	65	58	600	1×10^{-2}
5	90	62	1080	1×10^{-1}
6	71	78	1.3×10^4	1×10^{-1}
7	97	72	9×10^5	4×10^{-1}
8	100	47	3×10^6	2

^a $c_f = \sqrt{a_i b_i}$.

^b From Eq 1.

^c From Eq 6.

TABLE 3—Comparison of rapid and delayed failure in MgF_2 .

	Rapid Failure	Delayed Failure
$M_1 (\text{MN}/\text{m}^{3/2})^a$	1.8	1.9 ± 0.2
$M_2 (\text{MN}/\text{m}^{3/2})^a$	2.6	2.7 ± 0.3
M_{CB}		3.8 ± 0.4
r_1/c_f	5	4.7 ± 0.9
r_2/c_f	10	9.7 ± 0.7
r_1/c_i	5	$f(t) [10]$
r_2/c_i	10	$f(t) [10]$

^a From Eq 5, $M_f = \sigma_a r_f^{1/2}$.

the calculations made from Eq 4 [5]. Actual failure times fell between the two extremes but close to the values calculated from single-crystal parameters [5]. The authors hypothesize that delayed failure in this material is controlled by the crack growth resistance of the grain boundary. For ZnSe , this resistance is expected to be an intermediate value between the single-crystal and the polycrystalline values.

Because of the observations in MgF_2 and ZnSe , the authors were enticed to search for similar phenomena in other polycrystalline ceramics. As can be seen in Table 4, their observations indicate that slow crack growth occurs intergranularly for many ceramics, and rapid crack growth occurs transgranularly, intergranularly, or both. Observations of boron carbide (B_4C) fracture at room temperature show that slow crack growth occurs between the grains and that rapid failure occurs across the grains [13]. In contrast, silicon carbide exhibits almost no slow crack growth at room temperature. However, at high temperature, where subcritical crack growth does exist, the evidence shows that subcritical crack growth occurs intergranularly followed by transgranu-

TABLE 4—Fracture morphology-velocity relationships in ceramics.

Material	Rapid Fracture	Slow Crack Growth
MgF_2 (Kodak)	TG ^a	IG ^b
ZnSe (CVD)	TG	IG
Al_2O_3	IG + TG	IG
Si_3N_4 (Norton Co. 132)	IG + TG	IG
B_4C	TG	IG
SiC	TG	IG

^a TG = transgranular.

^b IG = intergranular.

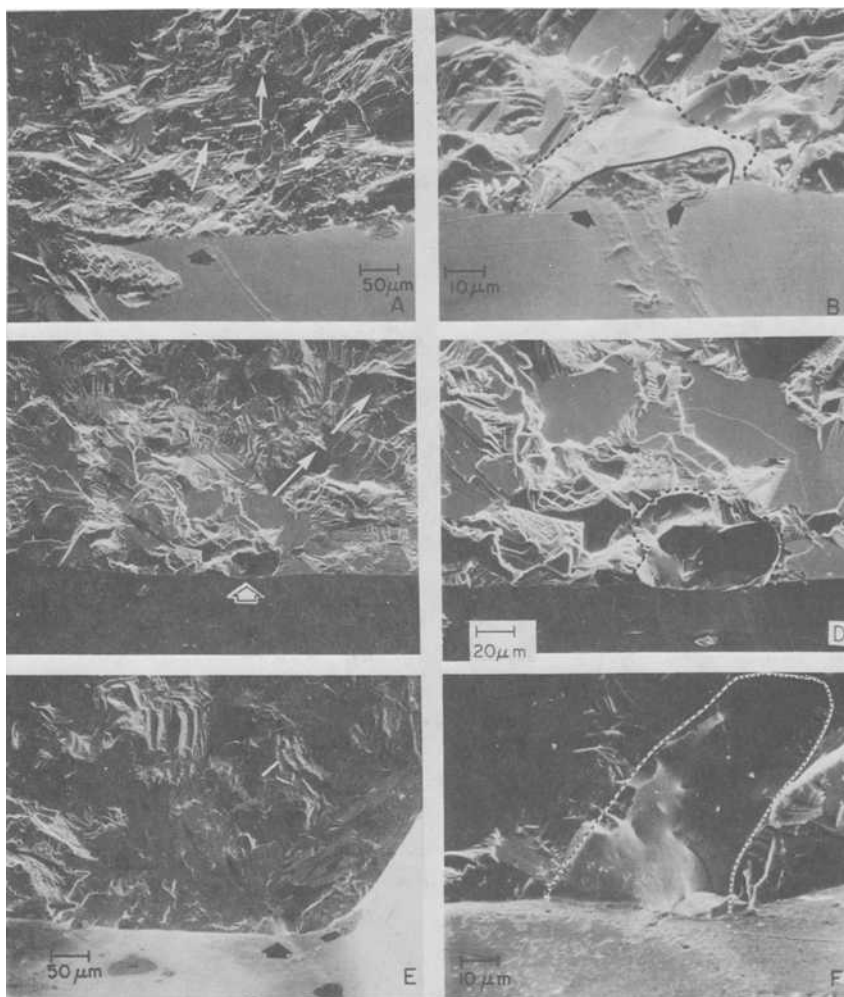


FIG. 6—Delayed failure surface for CVD ZnSe. (a and b) The specimen failed after 4000 s at 46.9 MPa. The fracture origin is found by tracing the "river" markings (arrows in a) back to their origin. Notice the initial flaw (solid line in b), which appears to result from two striations (arrows) that are probably the result of polishing damage. The intergranular region surrounding this area is outlined by the dashed line. The damage to the left of the origin (broad arrow) in (a) is due to postfracture damage. (c and d) The specimen failed at 55.8 MPa after 200 s. The "river" markings and cleavage steps (thin arrows in c) aid in determining the fracture origin (broad arrow in c). The intergranular region at the origin is outlined in (d). (e and f) The specimen failed after 1.6×10^4 s at 55.8 MPa. "River" markings are again used to determine the origin of failure (broad arrow in e). Notice the light striation in (e), which is probably the result of abrasive damage and leads to the initial defect, which in turn leads to failure under load. The approximate boundary of the intergranular region is outlined in (f).

lar rapid failure [14]. High-temperature intergranular subcritical crack growth has also been observed in hot-pressed silicon nitride (Si_3N_4) [15] and a glass ceramic [16]. Although intergranular, subcritical crack growth may not occur in all ceramics, it does occur in a large variety of polycrystalline ceramics. This behavior is attributed to an enhanced chemical potential of the grain boundary. This implies that more detailed study of the grain boundaries is needed to understand subcritical crack growth in polycrystalline ceramics.

Summary

Fracture surface analysis of glass under constant load can be used to estimate times to failure within 2 orders of magnitude. This accuracy is as good as any existing predictive capability [5,7]. Delayed failure calculations for polycrystalline ceramics appear to be less accurate. In both cases, the use of fracture mirror size data, rather than flaw size data alone, produces less scatter in estimates for time under load. Subcritical crack growth in a large number of polycrystalline ceramics occurs intergranularly.

Acknowledgment

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Multiple-Mist Regions on Glass Fracture Surfaces

REFERENCE: Abdel-Latif, A. I. A., Bradt, R. C., and Tressler, R. E., "Multiple-Mist Regions on Glass Fracture Surfaces," *Fractography and Materials Science*, ASTM STP 733, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 259-270.

ABSTRACT: Observations of the formation of multiple-mist regions on the fracture surfaces of soda-lime-silicate and fused silica glasses are reported. These regions occur only in high-strength specimens. The same empirical equation that is applicable to the basic fracture mirror-primary mist also describes secondary and tertiary mist regions, although the proportionality constants increase as the order of the mist region increases. Results suggest that these mist radii constants may be related in specific ratios, which decrease with successive mist regions. Criteria of mist-region formation are reviewed with respect to additional restrictions imposed by the consideration of multiple mists. The authors suggest that an energy criterion is most applicable, and a schematic diagram is proposed.

KEY WORDS: fractography, fracture mirrors, fracture mist, fused silica, glass, hackle lines, materials, materials science, mirror constants, soda-lime-silicate glass.

Postfailure examination of glass fractures has made an important contribution to the current level of understanding of glass failure. Many types of surface markings and crack patterns are characteristic of specific fracture conditions and can be used to describe the failure event. For example, in the failure of glass beverage containers, specific macrocrack patterns arise from different types or causes of failure, such as impact, internal pressure, and thermal shock. Some of these factors are discussed by Mould [1]³ and some by Watanabe [2], who very clearly describe these classic fracture patterns on a macro-scale. Lawn and Wilshaw [3] have very thoroughly described the formation of Hertzian cone cracks—the type commonly resulting when a small projectile, such as a "beebee," strikes a window. The description and analysis of these

¹ Supervisor, Materials Engineering, General Electric Co., Valley Forge Space Division, Philadelphia, Pa. 19101.

² Professor of Ceramic Science and Engineering and head, Department of Materials Science and Engineering, and associate professor of Ceramic Science and Engineering and Chairman, Ceramic Science Program, respectively, Pennsylvania State University, University Park, Pa. 16802.

³ The italic numbers in brackets refer to the list of references appended to this paper.

fracture patterns and surfaces, as well as many others, present interesting and challenging problems; however, in terms of analysis and experimentation, the fracture mirror formation problem completely overshadows all the others in expenditure of time and effort. Recently, Mecholsky and Freiman [4] extensively reviewed its relevance to crystalline ceramics, whereas Abdel-Latif et al [5] previously had summarized the literature on glasses. Numerous other papers on the topic exist and can be readily traced through the comprehensive bibliographies of Refs 4 and 5.

The term "fracture mirror" applies to the very flat, smooth, mirrorlike region immediately adjacent to the fracture origin or critical flaw. In the specific case of glass, this region is easily recognized, for when the fracture stress is high this region is generally in the shape of a semicircle, usually of a sufficient size to be seen by the unaided eye. It forms during the time period immediately following the critical condition of catastrophic failure and represents a region of rapidly accelerating crack growth. This smooth flat mirror region terminates with the appearance of a rough, or stippled region known as the mist. The mist region is concentric with the mirror and extends for only a short distance before giving way to a very rough area known as the hackle region. The hackle lines may be traced back to locate the critical flaw at the fracture origin, which lies near the center of the smooth mirror region. The formation sequence of these regions for a typical glass fracture is thus mirror to mist to hackle. If the strength of the glass object is sufficiently high, all of these regions will be in evidence; however, for many very low strength glass specimens, it is not uncommon for the mirror region to extend over the entire fracture surface.

If the fracture occurs under conditions of pure tension, then the boundaries between each of these concentric regions can be specified by a radius from the fracture origin. For example, the mirror-mist radius defines a semicircular line that separates the mirror and mist regions; similarly, the mist-hackle radius separates the mist and hackle regions. However, in the case of flexural or bending failures, frequently less symmetrical and less well-defined boundaries result [6]. What the criteria for the formation of all of these boundaries are has been a controversial issue that has not been completely resolved; however, the proposed possibilities include an empirical equation [7], velocity arguments [8], stress intensity [9], and energy balances [10]. In the opinion of these authors, the formation of these boundaries is an extremely difficult dynamic crack problem. It is, however, clearly a problem that must also address the formation of multiple-mist regions, for whatever the criterion for the formation of the initial mirror-mist boundary, it must be a recurring phenomenon when multiple-mist regions are formed on a single fracture surface.

There is practically no discussion of multiple-mist regions in the literature, despite their obvious appearance in the photomicrographs of at least four prominent articles on fracture mirror formation, including the studies of Sommer [10], Kerkhof and Richter [11], Kirchner [12], and Gulati, King, and Quan [13]. Multiple-mist regions are simply the recurrence of a second (sec-

ondary) or a second and third (tertiary) concentric mist region or band at increasing radii from the fracture origin, radii that substantially exceed the radius of the original mist region. These radii do not develop following a second mirror region; rather, they develop directly within the hackle and appear to nucleate additional hackle. As such, they must be specified as hackle-mist radii, as opposed to the mirror-mist radius of the initial, or primary, mist region. This paper is an initial report on the occurrence of multiple mists, both secondary and tertiary, in soda-lime-silicate glass and fused silica. It illustrates their correspondence to the same type of empirical equations that describe the primary mist and also suggests that they may be amenable to description by an energy-balance approach.

Experimental Procedure

Commercial soda-lime-silicate glass and fused silica rods $\approx 3.2 \times 10^{-3}$ m in diameter and about 1 m in length were utilized. Specimens about 5 cm in length were cut for four-point flexural tests. To produce tensile test specimens, the soda-lime-silicate glass was melted in a platinum crucible, drawn to fibers approximately 1×10^{-3} m in diameter, and annealed in air at 535°C.

The flexural strengths were measured at 25°C in water on a commercial testing machine (Instron, Canton, Mass.) over a 3.05-cm (1.2-in.) span, with an equidistant 1.02-cm (0.4-in.) span, center knife edges. For the flexural tests, the center portion of the rod was abraded with 600 grit silicon carbide (SiC) to ensure a high density of flaws at the maximum tensile stress location. In order to facilitate location of the mist regions and to preserve the specimens as well, a thin strip of Scotch tape was placed along the compression (top) surface of the test rods.

The fibers, about 15 cm in length, were tested in tension at room temperature in air. They were mounted through two hypodermic needles by beading the fibers at the ends and then using an epoxy cement to secure the glass fibers within the needles. The center portion of the fiber was then abraded with 600-grit SiC. A 5-cm section was tested in tension with the hypodermic needles seated in a free-aligning pivot-bearing fixture [14]. The fiber size and abrasion conditions were chosen to yield tensile strengths comparable to the flexural strengths, to allow their multiple-mist characteristics to be compared at the same strength levels.

Fracture surfaces were examined and measured both optically (a microscope fitted with a calibrated reticule) and by scanning electron microscopy (SEM). A large number of multiple-mist regions were actually observed; however, only those with a completely concentric appearance and obviously high symmetry about the fracture origin are reported in these results. This ensures a high degree of confidence in the reported fracture stresses as well as in each of the individual mist region radii. There is no difficulty in ascertaining a fracture mist region's radius in tensile specimens; however, flexural specimen mist re-

gions are often slightly elongated toward the neutral axis. Thus, in flexure, the mist region radii may be a function of the angle from the surface tangent to the neutral axis of the specimen. The mist region radii in flexure were always measured from the fracture origin or flaw to the intersection of the mist boundary and the specimen surface. The utilization of round rods as specimens, as opposed to square bars, facilitated the application of these symmetry judgments to the multiple-mist boundaries. It also facilitated the tensile measurements. It should be emphasized that applying these stringent conditions severely limits the number of data points that one can obtain, particularly for the flexural tests, where a valid data point (complete and symmetrical mist regions and a

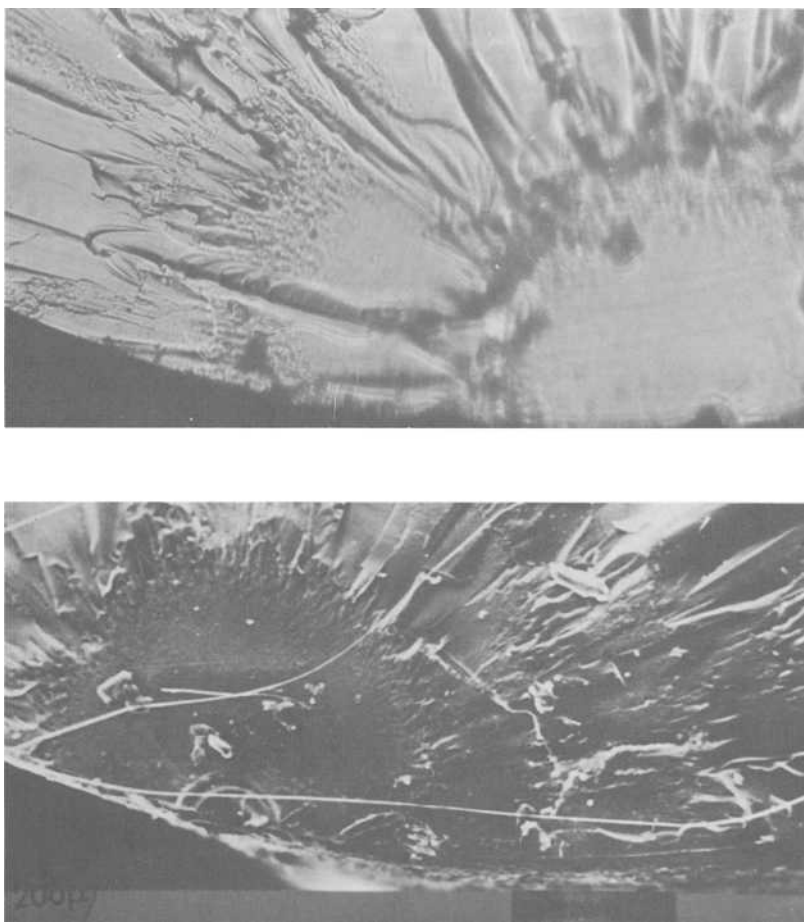


FIG. 1—The appearance of several multiple mists on soda-lime-silicate glass fracture surfaces. Optical photomicrograph (top) of a tensile specimen and a scanning photomicrograph of a flexural specimen (bottom).

fracture origin at the tensile surface directly beneath the center knife edge) is the exception rather than the rule. The individual data points were then plotted as σ_f versus $r^{-1/2}$, the classic fracture mirror plot, which intercepts the origin and has a slope equal to the "mirror" constant, "A," which might be better described as the mist constant or constants in this study.

Results and Discussion

Figure 1 illustrates the appearance of multiple-mist regions on actual fracture surfaces, whereas Fig. 2 schematically illustrates the idealized multiple-mist formation. Primary, secondary, and tertiary mists are depicted in both. Only a few of the large number of glass surfaces that were examined actually displayed these well-defined, symmetrical, complete multiple-mist regions. In these specimens, an obvious effect immediately following the development of a secondary or tertiary mist was evident in the intensification of the hackle

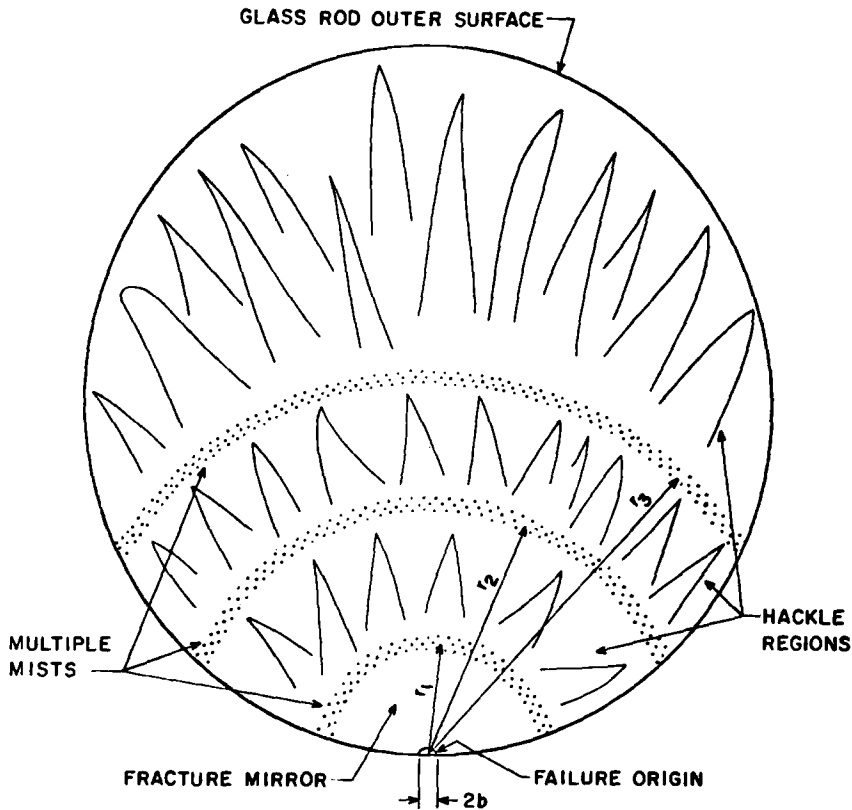


FIG. 2.—Schematic drawing of the appearance of multiple mist regions on the fracture surfaces of a glass rod. Primary, r_1 ; secondary, r_2 ; and tertiary, r_3 mists are depicted along with the mirror and hackle regions.

that ensued. It is apparent that the mist regions are essentially precursors of the instabilities that develop fully in the creation of the hackle region.

Although only a few of the specimens actually exhibited complete multiple-mist development, a number of the flexural specimens, particularly the higher-strength ones, had partial or poorly developed secondary and tertiary mist regions. These partially developed multiple mists were visible only at the high-stress regions of the flexural specimens and failed to mature toward the neutral axis, analogous to the incomplete mirrors discussed by Shetty et al. [6]. The high-stress region situation, discussed in relation to the appearance of partial mists in flexural specimens, is actually a fundamental factor in the appearance of multiple-mist regions on fracture surfaces. All of the specimens that exhibited multiple-mist regions had relatively high strengths for glass, approximately 100 MN/m^2 and above.

Virtually all of the studies of fracture mirrors in glass have confirmed that the strength and the mirror-primary mist boundary can be described by the empirical relationship

$$\sigma_f r_i^{1/2} = "A_i" \quad (1)$$

where σ_f is the fracture stress, r_i is the radius of the mirror-primary boundary from the fracture origin, and " A_i " is the boundary constant. A_1 is generally referred to as the fracture mirror constant for the mirror-primary mist region boundary. Figures 3, 4, and 5 illustrate the applicability of Eq 1 to the

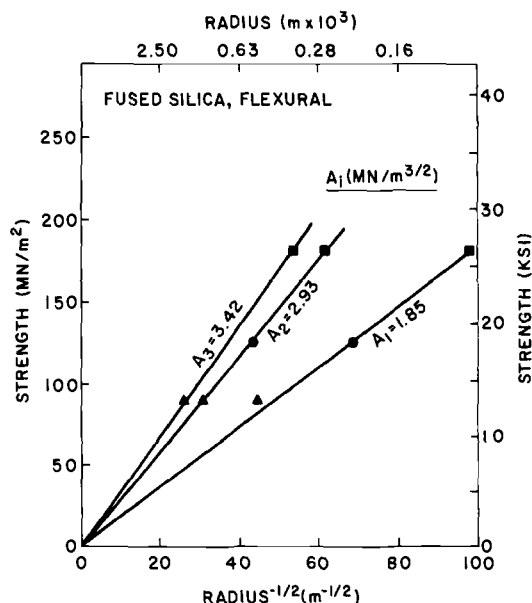


FIG. 3—The classic mirror radius plot for fused silica in the flexural test. The primary mist radii are on the A_1 line, the secondary mist on the A_2 , and the tertiary mist on the A_3 line.

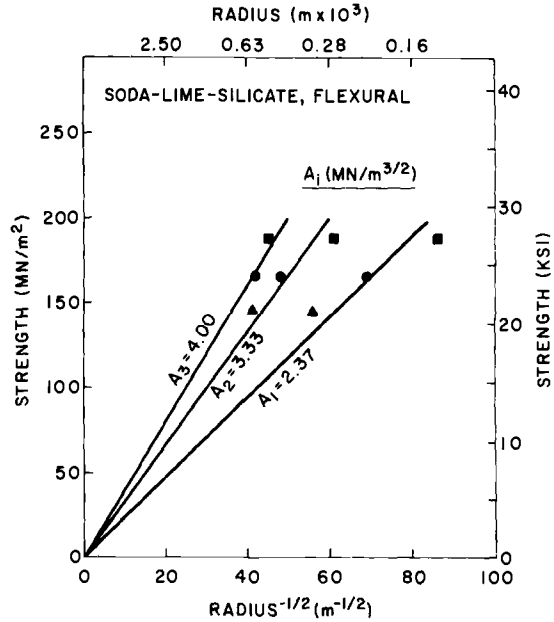


FIG. 4—The soda-lime-silicate glass mist radii plot for flexural testing.

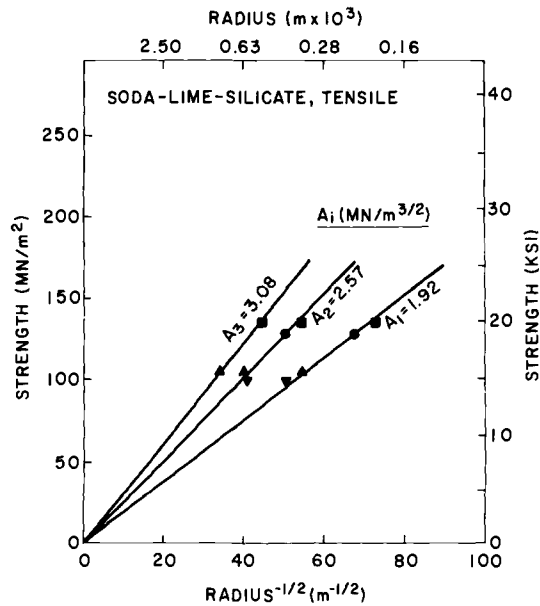


FIG. 5—The mist radii plot for the tensile fibers of the soda-lime-silicate glass.

multiple-mist region radii for these glasses. Even though the number of observations is too small for an extensive statistical analysis, the results suggest that Eq 1 is a reasonable first approximation to the data. It is also noteworthy that the tensile results (Fig. 5) appear to be more consistent than the flexural results in Figs. 3 and 4. The A_1 values for the primary mist regions, or the fracture mirror constants listed in Table 1, are in general agreement with the extensive compilations of Mecholsky and Freiman [4] and Abdel-Latif et al [5], even though the number of these observations is limited.

Several trends are apparent from these multiple-mist region boundaries, the most obvious of which is that each successive mist constant appears to be nearer to its predecessor than the previous one. Specifying A_2 as the secondary and A_3 as the tertiary mist region constant, the A_2/A_1 ratio varies from 1.3 to 1.6, but the A_3/A_2 ratio is nearly constant at 1.2 for all the glasses under the conditions studied here. Although the total data available for these multiple-mist regions are quite limited, the 1.2 ratio of A_3/A_2 is very nearly the same in the three distinct cases, which strongly supports the concept of a constant ratio between the multiple-mist radii.

There have been various attempts by the proponents of the stress-intensity factor criterion for primary mist formation to specify empirically the A_1 , or the fracture mirror constant, in terms of a specific ratio of the fracture toughness K_{Ic} . A logical extension of this concept is that each of the " A_i " values might also occur at specific K_{Ic} ratios. The limited number of observations in this study does not lend support to the extension of that concept to secondary and tertiary mist constants, for in each instance the fused silica, although having a higher K_{Ic} , possesses much lower " A_i " values, as noted in Table 1.

Observations of the consecutive mist regions, as well as the development of additional hackle following each of the secondary and tertiary mists, confirms that the mist is the precursor instability that develops before the hackle. If the reasonable assumption is made that the mist formation criterion is fundamentally the same as that for the primary, secondary, and tertiary mists, then sev-

TABLE 1—Multiple-mist region boundary constants.

		SLS ^a	FS ^b
Flexure	$A_1 \text{ MN/m}^{3/2}$	2.37	1.85
	$A_2 \text{ MN/m}^{3/2}$	3.33	2.93
	$A_3 \text{ MN/m}^{3/2}$	4.00	3.42
Tension	$A_1 \text{ MN/m}^{3/2}$	1.92	...
	$A_2 \text{ MN/m}^{3/2}$	2.57	...
	$A_3 \text{ MN/m}^{3/2}$	3.08	...

^a Soda-lime-silicate ($K_{Ic} = 0.76 \text{ MN/m}^{3/2}$).

^b Fused silica ($K_{Ic} = 0.79 \text{ MN/m}^{3/2}$).

eral conclusions regarding the proposed criteria for the mirror boundary and for all subsequent mist regions can be reached. The fact that the same form of empirical equation, Eq 1, appears to describe the formation boundaries of the secondary and tertiary mist regions lends credence to these arguments. One must conclude that the onset of each of the mist regions (primary, secondary, tertiary, and so on) can be described successfully by the empirical equation. The flexural results are moderately supportive of this, and the tensile strengths more strongly confirm it.

The development of multiple-mist regions does not appear to be very supportive of either the crack velocity criterion or the static stress-intensity criterion for fracture mirror-primary mist boundary formation. In the tensile case (Fig. 5), if each new mist region develops at the terminal crack velocity or even some critical crack velocity characteristic of the onset of this hackle precursor instability, then the region demands a series of velocity increases and decreases during the crack growth. The sequence would be that of rapid acceleration across the mirror region to the critical (terminal?) velocity at the mirror-primary mist boundary, and then a velocity decrease upon formation of the mist region and hackle. This would be followed by another acceleration in the hackle region until the critical velocity was again reached to yield the development of the secondary mist region. The sequence of deceleration and acceleration for each subsequent mist region formation would have to be repeated. Although these types of velocity fluctuations are not impossible, they have never been observed for very rapidly moving cracks in glass. For example, Kerkhof [15] reports a steadily increasing velocity to the terminal value without any indication of a decrease when mist formation occurs.

The application of a static stress-intensity criterion is similarly questionable, for the stress intensity, K_I is related to the product of the applied stress and the square root of a flaw length parameter

$$K_I = \sigma_{app} c^{1/2} Y \quad (2)$$

where Y is a geometric factor, σ_{app} is the applied stress, and c is the flaw length dimension. In a typical tensile strength measurement, such as that depicted in the results of Fig. 5, the applied stress across the crack plane is constant (or increasing as the remaining ligament supports the total load), and the primary flaw is increasing constantly. With each successive mist region formation, on a macroscale the K_I must be larger than it was for the previous mist region. On a microscale the K_I condition is considerably more complex, as has been demonstrated by Kobayashi et al [16] for macrocrack branching, where K_I decreases. However, it is the need for the succession of repetitions of the K_I criterion at multiple mists that leads one to question its applicability.

Recognition of the fracture mirror and multiple-mist formation problem as an extremely complex dynamic crack problem is not in itself a very difficult task, once the alternatives are examined. Solution of the problem in closed

form is a formidable task indeed. However, Broek [17] has outlined an approach to the dynamic problem of crack bifurcation which can be qualitatively applied, in modified form, to explain the formation of multiple mists on glass fracture surfaces. Broek suggests, on the basis of an energy release rate criterion, that once the kinetic energy available is sufficient for two cracks, then bifurcation is possible; furthermore, additional bifurcation of one of the original branches may occur once the energy criterion is met again. By analogy, once the energy conditions for primary mist formation have been met, they can occur again in a suitable situation.

Figure 6 illustrates a possible sequence of events leading to multiple-mist region formation. This standard graph of strain energy release rate versus crack size, or radius, contains a series of R lines for energy demands for mist formation and a single strain energy release rate line, G . The critical condition is reached at $G = R$, beyond which G exceeds the R value, the difference being available to satisfy kinetic energy demands. However, once a Δc equal to r_1 is reached, there is sufficient availability of kinetic energy to reach the R_1 line, characteristic of the primary mist demands. The primary mist demands a greater R level as a consequence of the surface roughness, as well as possible kinetic needs; thus R_1 lies above the original R . The r_1 is characteristic of the mirror-primary mist boundary radius. In this diagram (Fig. 6) the primary mist and hackle are both denoted by the same horizontal R_1 level, whereas, in fact, the hackle development may increase the R_1 level so that an upward slope may be more appropriate. Once the primary mist and hackle have formed at r_1 , the difference between G and the R_1 line governs the potential shift to R_2 and the development of a secondary mist at r_2 . The sequence repeats for the

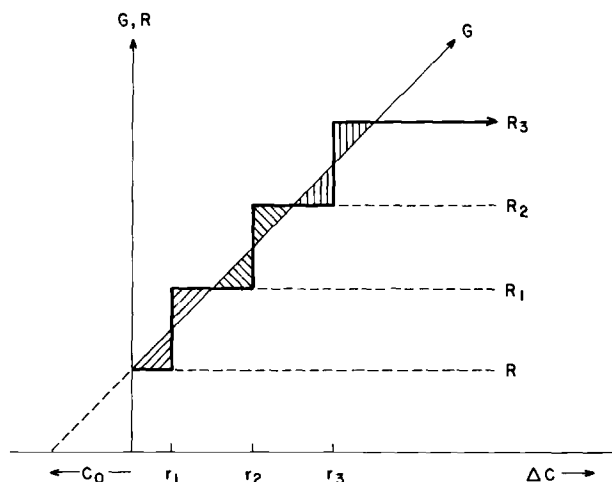


FIG. 6—The schematic plot illustrating a possible sequence for multiple mist development on the fracture surface.

tertiary mist at R_3 and r_3 , and so on. In theory, at least, any number of mist regions could develop in this manner. However, practically, specimens are of finite dimensions, and the strain energy release rate, G , is limited by the strength of the glass, $G\alpha\sigma_t^2$. This explains why multiple mists occur only in high-strength specimens, as these are the only specimens with sufficiently large G values to reach the secondary mist- R_2 , and tertiary mist- R_3 levels.

Conclusions

Observations of multiple-mist regions on soda-lime-silicate and fused silica glass fracture surfaces have yielded results comparable in some respects to the initial mirror-mist boundary. Secondary and tertiary mists appear to ascribe to the same empirical equation, but with larger constants. These results suggest that these constants may be in specific ratios; however, additional studies are needed for verification of this point. It is also apparent that the existence of multiple mists creates some concerns with velocity and stress-intensity criteria for mist region development. The authors suggest that an energy criterion that incorporates dynamic considerations is more appropriate, and a qualitative diagram for such a criterion is offered.

Acknowledgments

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Localized Deformation and Fracture of Magnesium Oxide

REFERENCE: Adler, W. F. and James, T. W., "Localized Deformation and Fracture of Magnesium Oxide," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 271-290.

ABSTRACT: Particulate erosion of brittle materials is a complex process of fracture nucleation, growth, and interaction which leads to removal of material from the exposed surface. The temporal and spatial development of the localized pressure loading imparted to a material surface by a water-drop or soft-body collision is not clearly defined. For the impact conditions investigated, the duration of the applied pressure pulse is generally less than 100 ns. Correspondingly, the exact form of the transient stress states generated within the impacted body is not easily determined. However, insights into the nature of the damage produced by these relatively short stress cycles are being obtained from microscope examinations of the residual deformation and fracture patterns in transparent materials. The response of single crystals of magnesium oxide to water-drop collisions is described. It was found that for the high strain rates imposed slip and fracture occur in the same systems and planes as are associated with quasi-static deformations. However, water-drop impacts produce slip only on the four {110} 45-deg slip systems, whereas rounded solid particles produce dominant slip on two {110} 90-deg slip systems, as well as on the {110} 45-deg slip systems. The fractures associated with each condition are also distinctly different. It was also found that fracture initiation could be suppressed with the introduction of numerous active slip sources on the impact face of the crystal.

KEY WORDS: impact, water-drop collisions, fractography, stress waves, deformation, fracture, crystalline materials, magnesium oxide, lithium fluoride, materials, materials science

Water-drop impacts on materials impose a rapid, localized loading which is just beginning to be understood. The general features of the impact event depend on the magnitude of the target-water drop interaction, which is primarily a function of the impact velocity. For solid target materials there is a velocity-drop size threshold for single drop collisions, below which no damage occurs, and a less well-defined threshold for multiple water-drop collisions. Since the mechanics of water-drop impacts on material surfaces has been de-

¹ Associate manager, Materials Group, and member of the technical staff, respectively, Effects Technology, Inc., Santa Barbara, Calif. 93111.

scribed by Adler[1],² a very cursory treatment of water-drop impingement will be given here.

Fractographic analyses can be used in conjunction with an understanding of the transient stress conditions that are generated in the target material to construct the temporal development of the primary modes of failure. This information can then be used to select alternative materials or to suggest ways in which the particle-impact resistance of a specific material can be improved. Optically transparent ceramics and polymers afford the opportunity to view the extent and nature of the damage due to a single water-drop collision as well as that due to multiple water-drop interactions [2-4]. Because of the rapidly changing, inhomogeneous stress distribution in the target material, interpretation of the fracture surfaces is quite complicated. Fractographic analyses of this kind are almost nonexistent in the particulate erosion field. The present investigation is a preliminary effort to examine the general character of the fractures produced by water-drop impacts on crystalline solids [single crystals of magnesium oxide (MgO) and lithium fluoride (LiF)] at moderate impact velocities. Moderate impact velocity is taken to mean a level above the fracture threshold but not one that produces large deformations in the impacted surface.

Experimental Procedures

Materials

Single crystals of MgO were purchased from Norton Chemical Corp., Niagara Falls, Ontario, Canada, in the form of cleaved, undoped high-purity single crystals. The specimens were prepared by cleaving and by mechanical and chemical polishing. The specimens were first cleaved to size (~15 by 15 by 5 mm) on {100} planes. The sized pieces were then mechanically polished on one of the 15 by 15-mm faces to remove the cleavage steps and artifacts. Chemical polishing was done with a hot (~100°C) 10 *N* phosphoric acid (H₃PO₄) solution. The action of this polish is described in Sangwal and Sutaria [5]. The polishing was continued until no evidence of mechanical polishing-induced dislocations was apparent when the surface was etched. The prepared surfaces were then flat and oriented to within 2 to 3 deg of a {100} plane. The impact face was labeled the (001) plane.

A dislocation etchant consisting of 0.5 *N* sulfuric acid (H₂SO₄) solution at 50°C was used for revealing ingrown dislocations, low-angle tilt boundaries, and slip bands associated with impact. The etching times varied from 0.5 to 1.0 min. The action of this etchant is also described in Sangwal and Sutaria [5]. In addition to being a dislocation etchant, this solution preferentially dissolves material at fractures even if they are so tightly sealed that they are invisible to a light microscope before etching. This characteristic of the etchant was used to reveal the very fine fracture structure around the impact site.

² The italic numbers in brackets refer to the list of references appended to this paper.

TABLE 1—*Properties of single crystals.*

Properties	MgO	LiF
Crystal structure	rock salt	rock salt
Density, kg/m ³	3580	2600
Knoop hardness, ^a kg/mm ²		
(001) <100>	400	87 to 96
(001) <110>	780 to 800	93 to 103
(110) <001>	420	87 to 98
(110) <111>	930	97 to 120
(110) <110>	810	93 to 116
Slip system	{110} <110>	{110} <110>
Cleavage plane	{100}	{100}
Water solubility, mg/100 g H ₂ O	0.62	270

^aTaken from Ref 6.

The relevant properties of MgO and LiF are listed in Table 1. Lithium fluoride is included for comparison, since it has also been used in other particle impact studies. Its water-drop impact response was found to be quite similar to that for MgO: the difference is essentially a matter of degree.

Single-crystal LiF in the form of {100} cleaved and annealed blocks approximately 12 by 12 by 8 mm was purchased from Harshaw Chemical Co., Solon, Ohio. Spectrographic analysis performed by Harshaw indicated only trace impurities of calcium, magnesium, and silicon.

The as-received crystals contained cleavage steps and deformation damage on the surfaces. The following procedure was used to prepare the surfaces for impact.

1. The cleaved surface was polished flat mechanically.
2. The flat surface was then polished chemically with fluoboric acid at room temperature until the damage layer due to mechanical polishing was removed.
3. Since Step 2 left a microscopically rough surface, the specimen was given a dislocation etch [7], which left the surface generally smooth, with the ingrown dislocation content revealed. The ingrown dislocation density was approximately $3 \times 10^5/\text{cm}^2$, not counting those present in the low-angle tilt boundaries.

Water-Drop Impact Experiments

A laboratory-scale, water-drop impact facility employing a 30-mm powder gun was developed for studying water-drop impacts on brittle ceramic materials. An extensive developmental effort was required to establish suitable stopping and retrieval conditions for brittle crystalline specimens fired down the range that would not introduce any damage other than that due to the im-

part of a single water drop. A closed system with rail guides for the specimen mounted on a sabot was selected to meet the imposed recovery requirements.

In the experimental procedure used water drops are formed at the tip of a hypodermic needle mounted above the test section and fall in the path of the moving specimen. As a single drop falls from the tip of the hypodermic needle, it interrupts a pair of fiber optics links situated at right angles to each other. If both beams are disrupted, this action initiates the firing sequence. The system is calibrated so that the sabot reaches the drop when it has fallen to a point within the limits of the exposed surface of the specimen. After impacting the water drop, the sabot enters the recovery tube. A graded distribution of material in the recovery tube controls the deceleration of the sabot and protects the face of the specimen from extraneous damage.

The tests are conducted in a helium atmosphere at an ambient pressure of 13.33 Pa (0.1 torr) in order to minimize distortion of the drop caused by the small quantity of helium gas that precedes the sabot down the gun barrel and engages the drop before its collision with the specimen's surface. In order to operate at pressures well below the vapor pressure of water, a solution of 80 volume percent distilled water and 20 volume percent ethylene glycol ($C_2H_6O_2$) is used. From a pragmatic viewpoint, this solution behaves in essentially the same way as pure water during the collision process.

The stated conditions within the water-drop facility allow spherical water drops to impact the specimen with direct photographic measurement of the drop just before impact. Deviations from a spherical shape are negligible.

Experimental Results and Discussion

The water-drop impact damage in (001) MgO comprises a characteristic slip-band pattern and a superimposed annular fracture pattern. The slip-band pattern will be discussed first.

Slip-Band Structure

The slip-band intersections with the (001) impact face shown in the upper half of Fig. 1 are almost entirely in the [100] and [010] directions. There is a central region seen in Fig. 1 with a very low density of slip, referred to as the central undamaged zone. There are a few exceptions associated with some of the larger impact fractures. These exceptions appear in the [110] and $[1\bar{1}0]$ directions. Due to their scarcity and short range, the slip bands associated with the fractures are thought to be a result rather than a cause of the fractures.

The subsurface slip-band pattern was investigated by sectioning the impact on a plane that passes through the impact axis. The sectioning was accomplished by cleaving through the impact on an (010) plane. The surface was given a light dislocation etch and the slip-band patterns were revealed. The lower halves of Fig. 1a and b show the etched cross sections and their relation to the slip-band structure on the impact face.

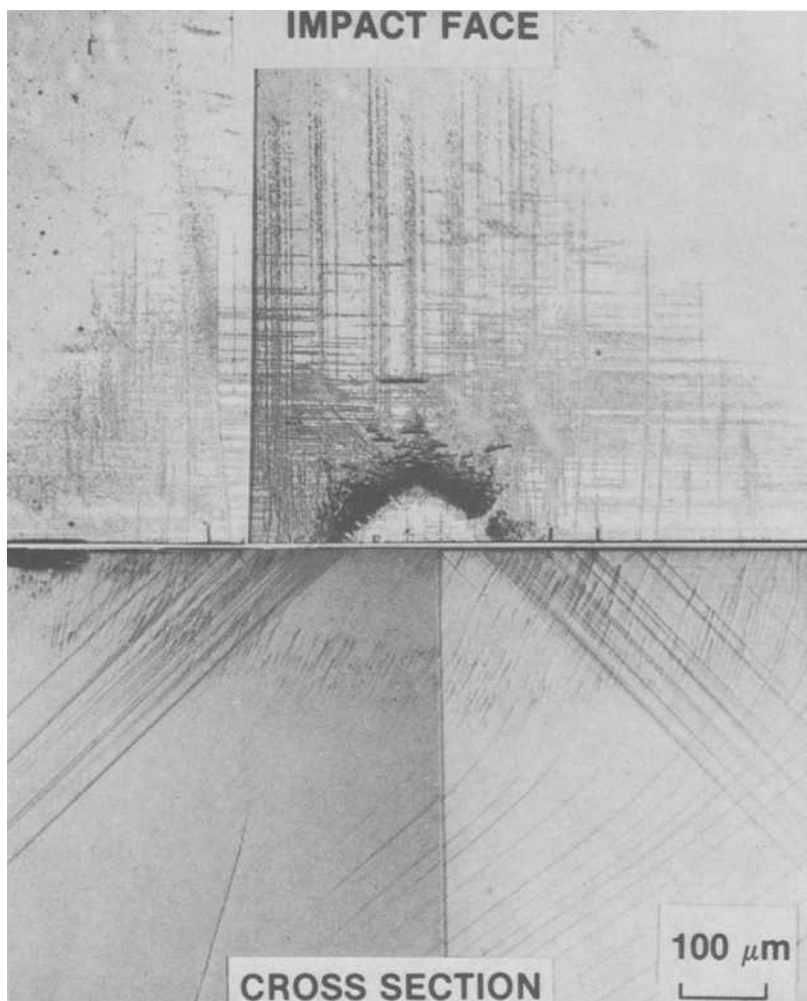


FIG. 1a—Dislocation-etched impact face and cross section of an MgO specimen: 1.68-mm water-drop impact at 563 ms^{-1} .

From cross sections for impacts up to 700 ms^{-1} , it could be seen that there are essentially two types of slip bands present. One type, apparent in the cross section in Fig. 1a, originates at the impact surface and proceeds at a 45-deg angle into the crystal away from the center of the impact. These bands are labeled A in Fig. 2. In addition to the Type A slip bands, there is another set of slip bands present for the higher velocity impacts (above 600 ms^{-1}) as shown in Fig. 1b. These slip bands are both at 45-deg angles and parallel to the impact face in the region bounded by the Type A slip bands and the central undamaged zone. These slip bands are labeled Type B in Fig. 2.

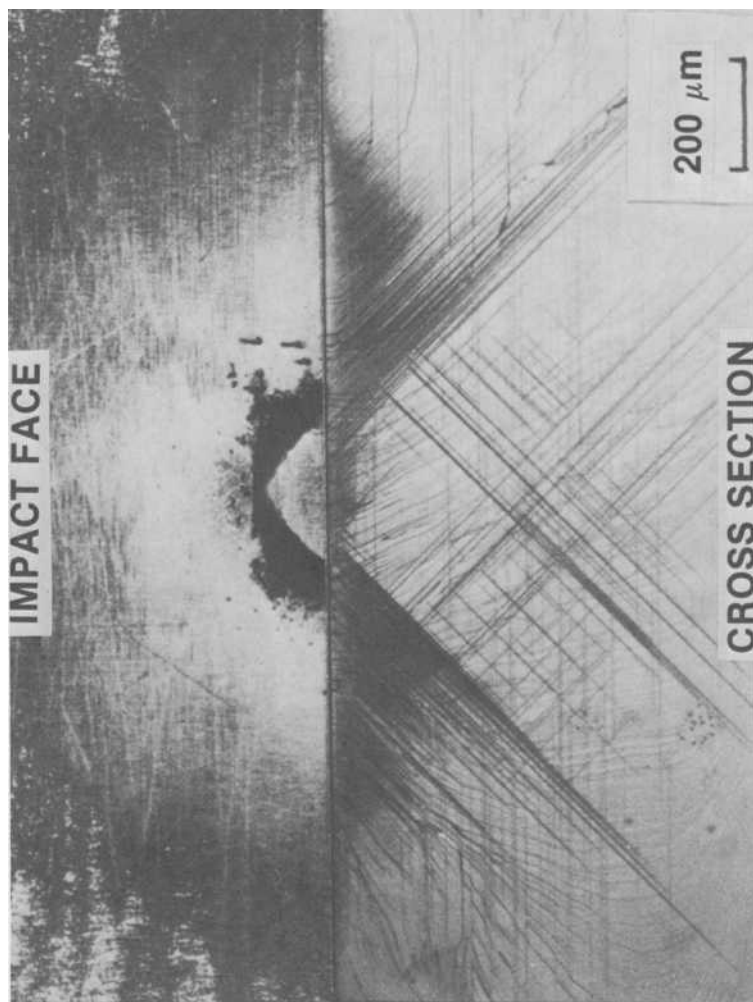


FIG. 1b—Dislocation-etched impact face and cross section of an MgO specimen: 1.82-mm water-drop impact at 672 m s^{-1} .

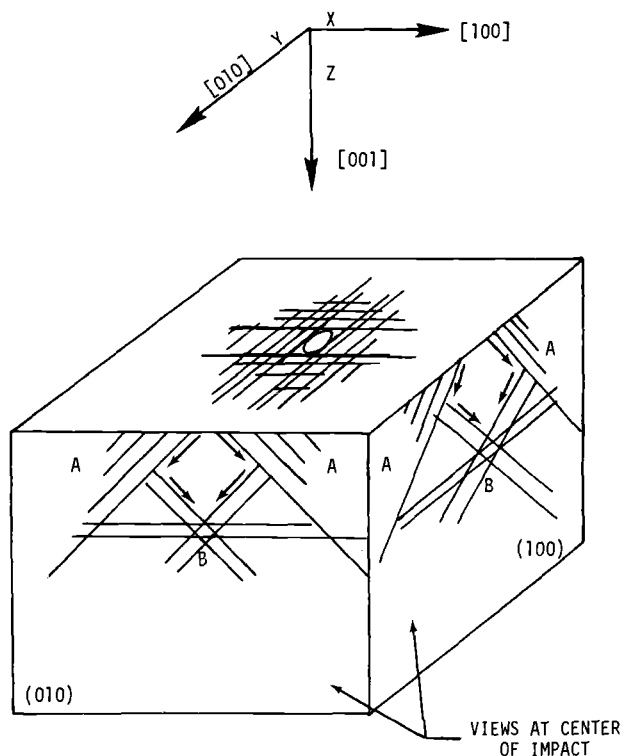


FIG. 2—Sketch of the slip bands present and the coordinate system used for single water-drop impacts on MgO.

The resultant deformations due to the water-drop impact can be constructed from these observations and consideration of the slip systems in MgO. Since MgO has cubic symmetry, there will be four planes of symmetry for water drops impacting in the $[001]$ direction on the (001) face. Figure 2 is a simplified sketch of the slip-band structure constructed from the symmetry information and the slip-band observations. Magnesium oxide is known to slip on $\{110\}$ planes in the $\langle 110 \rangle$ directions characteristic of ionic crystals with rock-salt structure.

It became apparent that the slip due to water-drop collisions takes place predominantly on the four $\{110\}$ planes that make an angle of 45 deg to the (001) surface. These slip bands will be labeled $\{110\}$ 45 deg. Specifically, these four planes are (101) , $(\bar{1}01)$, (011) , and $(0\bar{1}1)$. This can be easily seen on the (010) face and (001) impact face in Fig. 2. The Type A slip bands are on the (101) and $(\bar{1}01)$ planes. The Type B slip bands at an angle of 45 deg to the surface are also on the (101) and $(\bar{1}01)$ planes. The Type B slip bands in the (100) direction are also $\{110\}$ slip planes; specifically, they are the intersection of the (010) cross-section face with the (011) and $(0\bar{1}1)$ planes, which appear at 45-

deg angles to the surface, generally below the impact site on the (100) cross-section face. Because of the cubic symmetry of the impact, a similar explanation applies to the slip bands present on the (100) cross section. The two remaining slip planes of the $\{110\} \langle 110 \rangle$ slip system are the (110) and $(\bar{1}10)$ planes. Slip bands on these planes would appear in the [110] and $[\bar{1}10]$ direction on the impact face and in the [001] direction on the cross-section faces. These slip bands are referred to as $\{110\}$ 90 deg. The slip on these planes for water-drop impacts on (001) MgO was found to be insignificant over the range of conditions investigated.

Now that the dominant active slip planes have been established, the slip directions will be identified consistent with the known slip systems $\{110\} \langle 110 \rangle$ and the generally compressive nature of the impact stresses.

Slip on the (101) Type A slip system must be such that the material interior (that is, the positive x side) to the slip band moves in either the $(\bar{1}01)$ or the $(10\bar{1})$ direction to be consistent with the $\{110\} \langle 100 \rangle$ slip systems. The fact that the impact pressures are primarily compressive at the center of the impact makes $(\bar{1}01)$ the obvious choice. A somewhat similar argument applies to the Type B slip bands present in the impacts at higher velocities. For these bands the slip is such that the material on the positive side of the band moves in a (101) direction. Both of these deformations are indicated in Fig. 2 by small arrows. By symmetry, the mirror-image bands slip as indicated in Fig. 2.

The Type B slip bands that appear in the [100] direction on the (010) cross section are as indicated: the same Type B slip bands that appear at 45-deg to the surface on the (100) cross section. By symmetry, the slip bands on the (010) cross section have the same slip as those on the (100) cross section. Therefore, the slip on the bands appearing in the [100] direction on the (010) cross section is in either the $[011]$ or the $[0\bar{1}1]$ direction. The cumulative effect of slip in the indicated directions on these six slip planes is that the region of the crystal generally below the impact, bounded by the six slip planes (A and B), and the impact face moves in the [001] direction without being deformed by slip itself.

The described deformation response of (001) MgO for water-drop impacts is distinctly different from quasi-static indentations and solid-particle impacts on (001) MgO and LiF. All investigators report having seen slip on all six $\{110\} \langle 110 \rangle$ slip systems [8–13]. The presence of all systems produces a characteristic “rosette” pattern (examples can be seen in Refs 9, 10, and 13). In addition to the four $\{110\}$ 45-deg slip systems identified for water-drop impacts, two $\{110\} \langle 110 \rangle$ slip planes that intersect the (001) surface at 90 deg (designated $\{110\}$ 90 deg) are also active. Slip on the $\{110\}$ 90-deg planes is in the $[\bar{1}10]$ direction in the impact plane. This slip plane network indicates not only displacement of the material under the impact site in the [001] direction, as for water-drop impacts, but also displacement of material parallel to the impact plane.

The description of the slip deformations observed for water-drop impacts has not considered the temporal development of slip or possible strain-rate

effects. Although it is not possible to observe the step-by-step development of slip during an impact, there are several clues in the slip pattern left by the impact, which, when combined with an understanding of the temporal nature of the loading function, do provide some understanding of the evolution of the resulting deformation.

For impacts generating Type B slip bands, there is an apparent blockage of the two orthogonal sets of Type B slip bands by both interaction with each other and the Type A slip bands. This blockage indicates that, although slip did not occur at the same time, it did occur later than the slip on the interior Type A bands. In addition to the evidence of slip blockage, the fact that the lower velocity impacts (below 600 ms^{-1}) only begin to produce Type B slip bands indicates that slip on the Type B slip bands occurs later than slip on Type A bands and most likely is a result of the Type A slip deformations.

A reasonable explanation of this sequence of events is that the Type B slip bands were activated by $[100]$ and $[010]$ tensile stresses in the region of the crystal immediately below the impact site, which was being driven in the $[001]$ direction. The $[100]$ tensile stresses in the (010) plane would result from the $[\bar{1}00]$ and $[100]$ components of $[\bar{1}01]$ and $[101]$ slip, respectively, on the Type A $\{110\}$ 45-deg slip systems that originated at the impact surface. Similar stresses would arise in the $[010]$ direction in the (100) plane.

The stresses required for nucleation (activation) and expansion (motivation) of slip bands in MgO have been extensively studied by Stokes et al [14] and in LiF by Gilman and Johnston [15, 16]. Stokes and co-workers found the activation stress to be approximately twice the motivation stress in chemically polished single-crystal MgO. The facts that higher stress is required for activation of dislocations than for motivation and that dislocations move at a velocity that is a function of the applied stress partially account for the slip band development during impact [16]. The Type A slip bands are numerous, narrow, and independent, as can be seen from Fig. 1.

This slip-band pattern is very different from quasi-static deformations. Quasi-static slip-band behavior in MgO has been studied by Argon and Orowan [17] and by Stokes [14] and that in LiF by Johnston and Gilman [16]. For single crystals the slip bands (composed of dislocation half loops) in quasi-static deformations widen and expand through various mechanisms described in these references, which results in plastic shear of the crystal. The end result is a few very wide and extensive slip bands. Only a few slip bands develop due to the shortage of active sources in chemically polished specimens and the larger stress required to activate rather than to motivate slip bands.

In the water-drop impact situation, there are many narrow slip bands also composed of dislocation half loops, which indicate that the stresses are large enough for slip-band activation, but the temporal development of strain is more rapid than can be accommodated by slip-band motivation. A quantitative feeling for the order of magnitude of the strain rates occurring in a 550-ms^{-1} water-drop impact can be gained by realizing that the total time that the

peak impact pressures are acting on the surface is estimated to be of the order of 50 ns.

Although Klein and Edington [18] have identified slip at high strain rates on {100}, {111}, and {112} planes in the $\langle 110 \rangle$ directions, the only slip systems identified for the MgO water-drop impact conditions are the {110} $\langle 110 \rangle$ systems. Even though slip is only observed on {110} $\langle 110 \rangle$ systems for impact on a {100} plane, it is possible that other slip systems may be active for other impact conditions. Slip on other systems may occur for impacts on planes other than (001). Impacts orientated so that the stresses on the {110} $\langle 110 \rangle$ systems are less preferential may also cause slip on other systems. Hulse et al [19] have used a similar approach to activate slip on {100} $\langle 110 \rangle$ systems at elevated temperatures for MgO crystals in compression.

Narayan [11] and Narayan and Washburn [12] have reported that low-velocity aluminum oxide (Al_2O_3) microsphere impacts on (100) MgO produce slip in the {110} $\langle 110 \rangle$ slip systems and that in addition the dislocations consist of dislocation dipoles (two closely spaced dislocations of opposite sign). In contrast, low strain-rate indentations made by a sharp pin show single dislocations. Whether or not the dislocations present in a water-drop impact damage site are single or dipole pairs and what consequence that would have on slip-band development will have to be investigated further.

Water-drop impact damage on (001) LiF, which is considerably softer than MgO, was also investigated. As shown in Table 1, LiF exists in the same rock-salt cubic crystal structure as MgO and has the same {110} $\langle 110 \rangle$ primary slip systems.

An impact velocity of 209 ms^{-1} is below the threshold for introduction of organized slip or fracture in LiF. The slip patterns at velocities above this threshold are shown in Fig. 3. The magnification is the same in each case, so the increased area indicates the influence of increasing the velocity for the same drop diameter. The initial development of the slip pattern is thus displayed; this is assumed to be characteristic of MgO except that a higher threshold velocity would be required for organized slip in MgO. The impact in Fig. 4 shows that fracture response of LiF. The impact at 385 ms^{-1} in LiF is very similar to that shown for MgO at 560 ms^{-1} in Fig. 1a.

Fracture Initiation Mechanisms

In addition to the characteristic slip-band pattern for a single-drop impact on (001) MgO, there is a characteristic fracture annulus for impact velocities in the range from 450 to 700 ms^{-1} . When etched and viewed on the impact face, the fracture types display the following features (refer to Figs. 1 and 5):

1. Due to the fourfold symmetry around the [001] impact axis, the impact damage is similar in eight octants.
2. The fractures exist in an annulus whose inner boundary is approximately square. The diagonals of this square are the [100] and [010] directions.

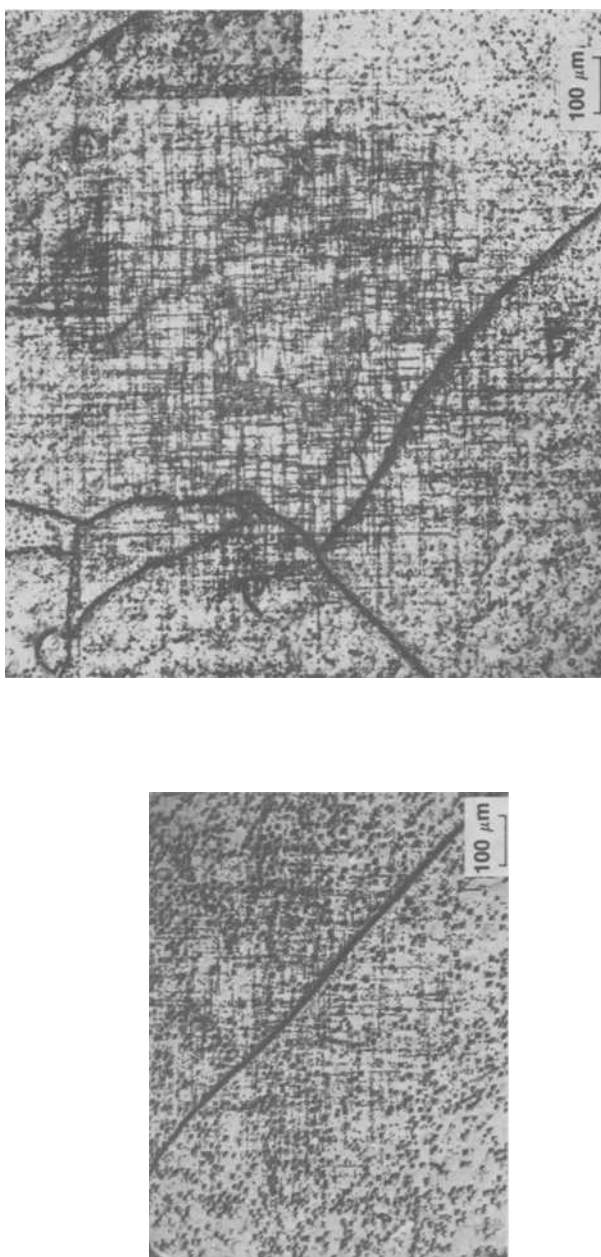


FIG. 3.—Slip patterns on (001) LiF. The impact surface has been etched: (left) 2.25-mm water-drop impact at 220 ms^{-1} ; (right) 2.25-mm water-drop impact at 288 ms^{-1} .

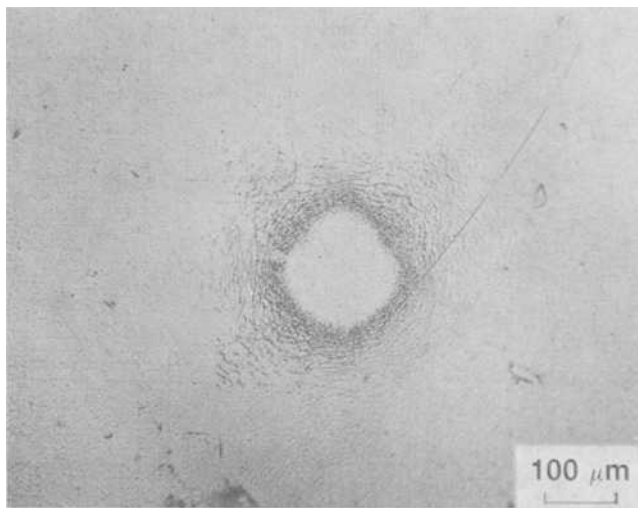


FIG. 4—Fractures on (001) LiF due to a 1.63-mm water-drop impact at 385 ms^{-1} . The impact surface was etched and viewed in transmitted illumination.

3. The fractures found at the inner radii tend to be of very short range and high density.

4. In the $\langle 100 \rangle$ radial directions, the larger fractures tend to intersect the surface tangent to the impact annulus.

5. The fractures lying near a $\langle 110 \rangle$ radial direction are of two types: (1) those whose intersection with the (001) impact face is tangent to the impact, and (2) those that are apparent distortions in a radial direction of the Type 2 fractures. Intersections of these radially distorted fractures from two quadrants form the W-shaped fractures seen in Fig. 5.

6. The density of the fractures falls off rapidly outside the fine fractures described in 4. The fractures present farthest from the center are Type 4 fractures and are generally in the $\langle 100 \rangle$ radial sectors.

In cross section the Type 3 fractures are found to be very shallow ($< 3 \mu\text{m}$), and the larger Type 4 fractures are found to be $10 \mu\text{m}$ or less in depth. The Type 4 fractures typically consist of two straight sections, as seen in Fig. 6. These crack types will be described in greater detail, along with the manner in which they may have been formed.

The fracture annulus appears to be the cumulative effect of dislocation and the phenomena of dislocation slip-band interaction, which are active in the water-drop impact situation. Fracture initiation mechanisms have been suggested by several investigators [14, 17, 20–26]. All of these mechanisms require intersecting slip bands for their operation. In Fig. 6(*top*) both orthogonal and nonorthogonal intersecting $\{110\}$ 45-deg slip bands are seen. Although intersecting slip bands were not observed after etching, they could be seen when

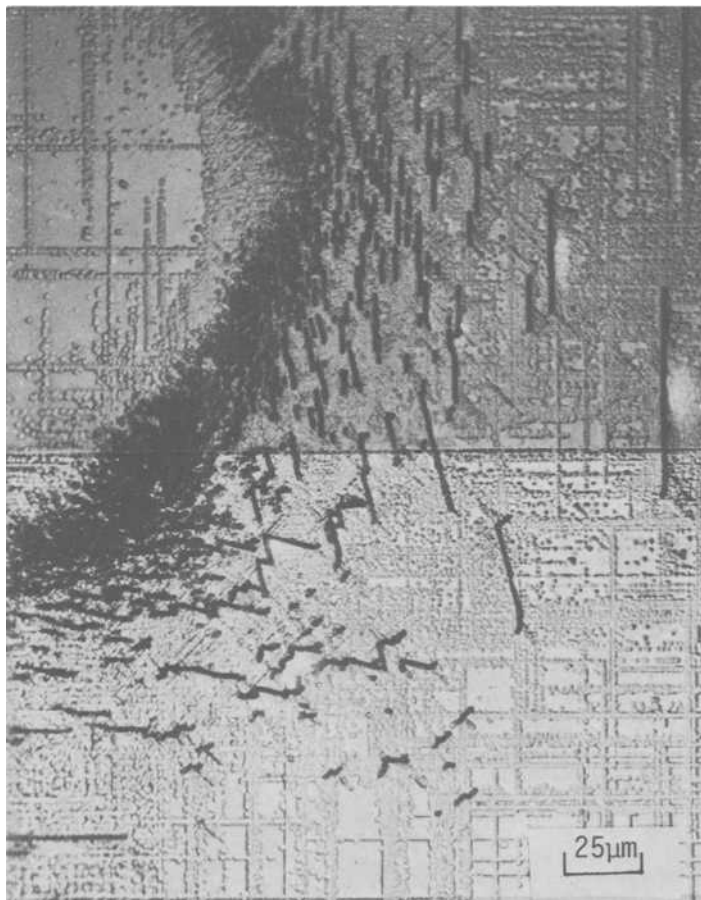


FIG. 5—One quadrant of the fracture annulus for a 1.5-mm drop impact on MgO at 556 ms^{-1} .

viewed with transmitted polarized light. A possible sequence of events for slip-band development, which is consistent with previous observations and includes dislocation slip-band interactions in the region of the fracture annulus, is described.

Consider a 2-mm water drop colliding with (001) MgO at 500 ms^{-1} . The calculated longitudinal and shear wave velocities along the $\langle 100 \rangle$ directions in MgO are 8.96 and $6.56 \text{ mm}/\mu\text{s}$. The rate of expansion of the contact zone between the drop and the face of the MgO target will be approximated by a compressible drop impact on a rigid plane. Initially the periphery of the contact zone moves faster than the wave speeds in MgO or water; however, it only takes 5.8 ns for the shear wave in MgO to separate from the contact boundary. This occurs at a radial distance of $76 \mu\text{m}$ from the point of impact. The drop continues to load the surface for at least an additional 40 ns, and the boundary

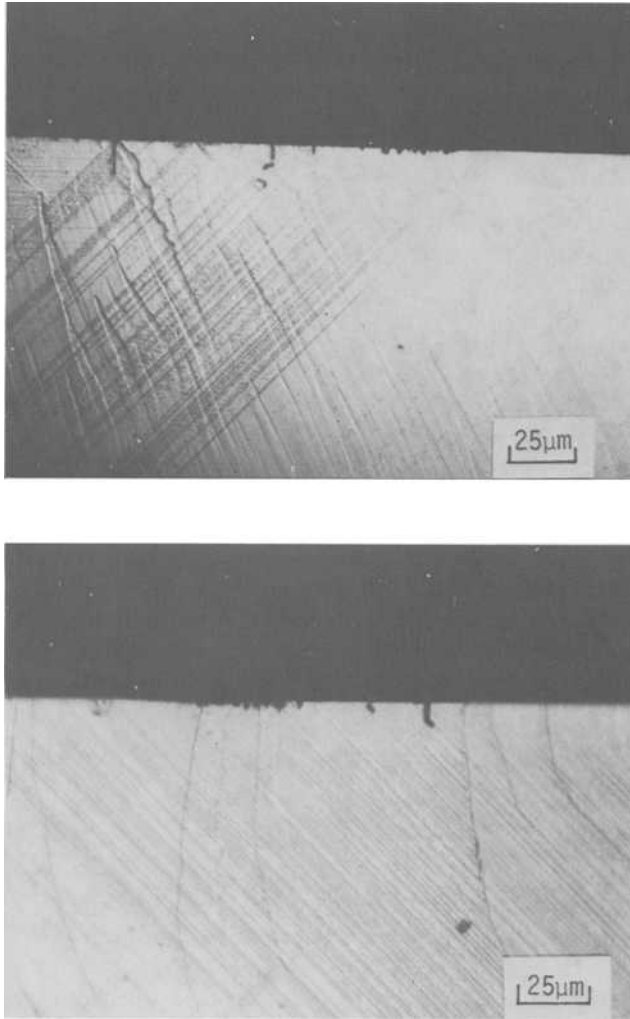


FIG. 6—The etched cross sections showing slip bands and fractures in water-drop-impacted MgO: (top) 1.68-mm drop-impact at 563 ms^{-1} ; (bottom) 1.82-mm drop-impact at 672 ms^{-1} .

of the contact zone reaches a radial distance in excess of $200 \mu\text{m}$. At this point pressure release occurs, and the applied pressure precipitously declines. During this time the contact zone is being deformed in the $[001]$ direction.

The impact region in MgO will experience both the effects of the stress waves propagating in the material and the direct deformation of the contact zone. A possible mechanism for activation of intersecting active slip bands in the vicinity of the impact face is formulated based on the water-drop loading sequence. When the shear wave separates from the contact zone, it is capable

of activating Type A slip bands (Fig. 2). The initiation of the Type A slip bands at the boundary of the central undamaged zone can be clearly seen in Fig. 1. Next, due to the direct deformation in the $[001]$ direction of the material in the contact zone, the surface exterior to the contact zone is in a tensile state. While these tensile stresses exist in the vicinity of the impact face, the orthogonal $\{110\}$ 45-deg slip bands seen in Fig. 6 are activated, intersect with the active Type A bands already present, and cause the observed fractures. As the contact zone expands and engulfs the fractures, the attendant compressive state halts further fracture development at that location. The short period of time that the tensile stresses exist would account for the extremely shallow ($3\text{-}\mu\text{m}$) fractures at the inner radii and somewhat deeper ($\sim 10\text{-}\mu\text{m}$) fractures at the outer range of the fracture annulus. As described here, the $(\bar{1}01)$ slip bands activated in tension have time only to propagate to shallow depths and interact with other bands.

The fracture initiation mechanism proposed by Stokes and co-workers [14, 20, 21] occurs at intersections where two expanding slip bands have completely cut through one another. This situation exists at the surface during the short time it is in a tensile state. A fracture is nucleated by a pileup of edge dislocations in the slip bands, activated in tension against the barrier provided by the orthogonal slip bands (see Fig. 7). Once a crack is nucleated, it tends to propagate along the $\{110\}$ plane at the edge of the barrier slip band. At some point the fracture is large enough to initiate cleavage on the $\{100\}$ plane normal to the applied tension. This mechanism generates a characteristic $(\bar{1}01)$ $[010]$ slit source. This characteristic shape can be seen in cross section in Fig. 6.

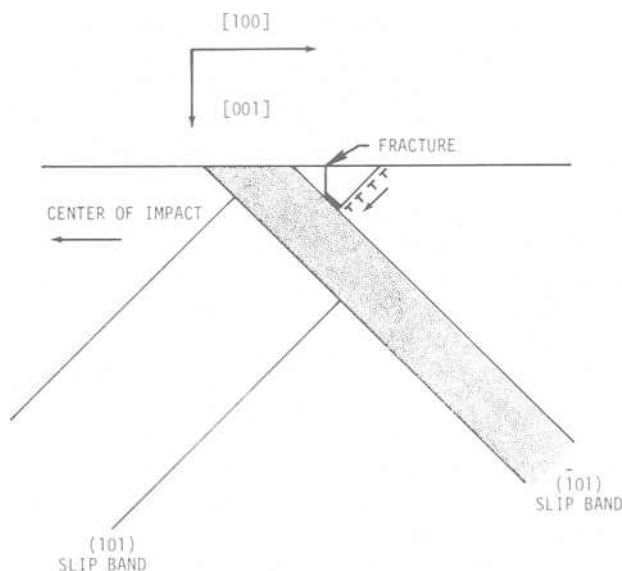


FIG. 7—Slip-band interaction for fracture initiation (proposed by Stokes et al [14, 21, 22]).

Stokes et al [14] have noticed that by introducing many active slip systems this fracture initiation mechanism can be suppressed for quasi-static deformations. Their conclusion is that one or both of the following mechanisms is responsible: (1) if the spacing between slip bands is very small, the slits, although they may form, will not be able to grow to a size critical enough to cause catastrophic fracture on a {100} plane, and (2) the numerous slip sources allow deformation to proceed homogeneously in the crystal and eliminate large local strains. If either or both of these mechanisms are active, introducing numerous active slip systems in the surface layer to be impacted should suppress fracture formation in single-crystal MgO.

Fracture suppression by the introduction of numerous slip sources was investigated by preparing (001) MgO specimens with sufficiently numerous surface dislocation sources that slip could be motivated at a lower stress than that required to activate slip. The objective of this experiment was to see if impact resistance could be improved. The specimen was prepared by mechanical polishing with an Al₂O₃ submicron-size polishing compound on nylon. The surface contained such a high density of dislocations that etching could not distinguish individual dislocations or slip bands. In addition to the dislocations, the surface contained numerous scratches.

After water-drop collisions at 512 ms⁻¹ and 574 ms⁻¹, the impact sites were located by transmitted polarized light (etching revealed nothing) and sectioned. The etched cross section and impact face are shown in Fig. 8. The impact shows numerous slip bands but no detectable fractures.

These results indicate that introducing dislocation sources in the impact surface does improve the impact-fracture resistance. It is possible that the surface was in a partially compressive state due to the polishing, which also may have helped to eliminate the fractures. One surprising result is that the polishing scratches did not act as flaws to promote fracture. Stokes et al [27] have developed a method for introducing dislocation sources in the surface without leaving scratches or a compressive layer. This method could be used to check these results.

A mechanism for fracture nucleation as a result of interacting nonorthogonal {011} <110> slip systems has been proposed by Keh et al [23]. Close examination of Fig. 1 reveals numerous fractures, which were apparently nucleated at the intersection of two nonorthogonal {110} 45-deg slip systems. During the time the surface layer is in tension, the two intersecting Type A slip systems with dislocation motions are the proper geometry to interact as in Keh's model. A diagram of Keh's model is shown in Fig. 9. The fractures predicted by this model would lie in a radial direction in relation to the impact. Close examination of Fig. 1 reveals that there are short <110> radial fracture segments (approximately the width of the intersecting slip bands) in the fractures present at the intersections of the nonorthogonal {011} 45-deg slip bands. The extension of these radial fractures in a tangential direction is consistent with the radial tensile forces; that is, a flaw was initiated in the <110> radial direc-

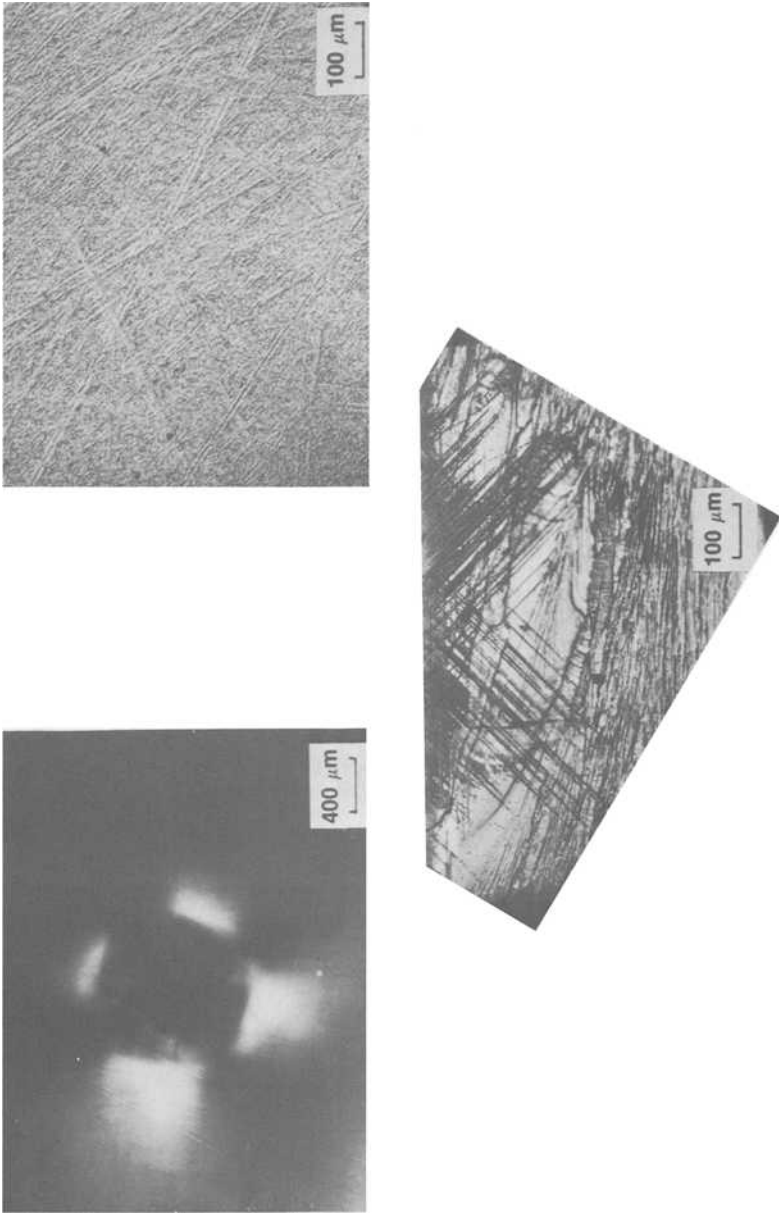


FIG. 8—The nature of water-drop-impact damage on mechanically polished MgO. (Top left) the site of 1.63-mm water-drop collision at 574 ms^{-1} , viewed in transmitted polarized light; (top right) the dislocation-etched impact site shown in Fig. 6 (top) (note the absence of a fracture annulus); (bottom) a dislocation-etched cross section for a water-drop collision at 512 ms^{-1} .

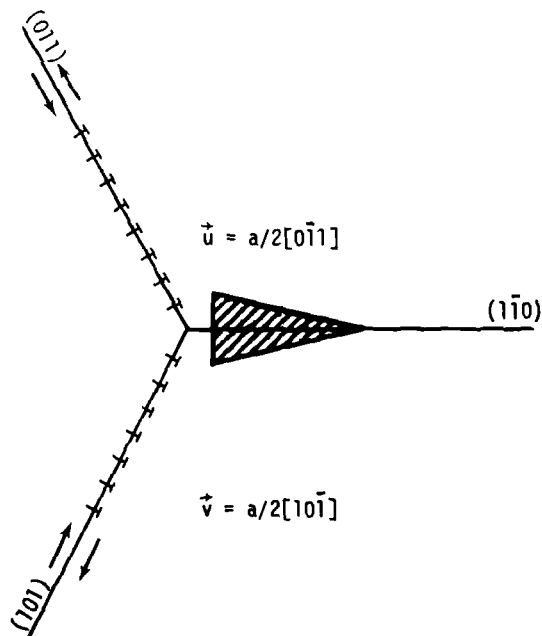


FIG. 9—A crack formation on the (110) plane in MgO (from Keh et al [23]).

tion by the Keh mechanism and then propagated in the tangential direction by the radial surface tensile stresses.

Cottrell [22] proposed that a fracture on the (001) plane may be initiated by the intersection of edge dislocations moving on (101) and (10 $\bar{1}$) planes in metals. The Cottrell mechanism could account for the (001) fracture at the surface but would not account for the (101) [010] slits observed in Fig. 6. The fracture-extension mechanism proposed by Clarke et al [24], in which a microcrack grows to critical size by generating dislocations at its front, is also not likely to be active in this case because of the change in direction in the fracture from one that is parallel, or inclined at a 45-deg angle, to the surface to one that is perpendicular to the surface.

Argon and Orowan [17] have a view of the fracture initiation process for slip-band interactions in MgO different from that of Stokes et al [14, 20, 21], Keh et al [23], Clarke et al [24], Cottrell [22], and Washburn et al [25]. Argon and Orowan maintain that fractures are caused at slip-band intersections because of high macroscopic stresses resulting from the geometrical incompatibilities of plastic deformation across the intersecting slip bands rather than from the coalescence of individual dislocations. It is conceivable that, due to the high strain rates associated with water-drop collisions, even though the slip bands are very narrow, stresses large enough to cause fracture could be attained because of the inability of additional plastic deformation to relieve

the stresses fast enough. This mechanism may operate during the surface tensile state at the intersection of both the orthogonal and the nonorthogonal $\{011\}$ 45-deg slip planes. Presumably the stresses due to slip-band intersection would initiate a fracture at the intersection; then the surface-layer tensile stresses would cause the fracture to propagate to the surface. This two-stage fracture mechanism would explain the observed sharp turn in the fractures (see Fig. 6). It was apparent in some photomicrographs that interaction occurred between a low-angle tilt boundary that passed through the slip-band pattern visible on the impact face and the slip bands, which either caused or enhanced the fractures present. Slip across a low-angle boundary was suggested as a mechanism of crack formation by Orowan [28] and was observed by Gilman [29]. For both LiF and MgO, Washburn et al [25] have observed that dislocation pileups against subgrain boundaries (that is, low-angle boundaries) are the exception rather than the rule. They have postulated that subboundaries are effective barriers only when certain geometric relations exist between the dislocation Burgers' vector and the boundary. Washburn's work was done at low strain rates.

Conclusions

At the initiation of the investigation of water-drop impact damage in single crystals of MgO and LiF, it was thought that, because of the high strain rates imposed, slip and fracture might occur in systems and planes other than those associated with quasi-static deformations. However, slip and fracture occurred in the same systems and planes that are active in quasi-static deformations for these materials for the impact conditions considered.

Identification of the slip bands generated by water-drop collisions using section and etch procedures revealed a characteristic of the water-drop impact which is significantly different from that for solid particle impacts. The deformation due to water-drop impacts is such that material is displaced in directions away from the impact plane and not in directions in the impact plane. Rounded solid particle impacts, in contrast, cause displacements in directions in the impact plane as well as away from it.

For impacts on (001) MgO, it was demonstrated that introduction of numerous active slip sources into the impact face is an effective means of reducing the impact damage and completely eliminating fracture for the impact velocities tested, which approached 600 ms^{-1} . The numerous active sources allow deformation to take place by slip (rather than fracture) at rates greater than can be accommodated by dislocation multiplication methods in surfaces with very few active sources.

Acknowledgment

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Fatigue Fracture Surface Micromorphology in Poly(vinyl chloride)

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ABSTRACT: The fatigue fracture surface micromorphology of poly(vinyl chloride) (PVC) was studied as a function of molecular weight (M), plasticizer content, and residual orientation, with particular attention given to how these variables affect the formation of discontinuous growth bands (DGBs). Discontinuous growth bands, which form on a number of polymeric solids under cyclic loading, represent successive increments of crack extension through a craze zone. As expected, DGB width showed little change in size over the molecular weight range or specimen orientations examined and increased in size with increasing plasticizer content. On the other hand, the cyclic stability of these bands increased markedly with an increase in molecular weight, but to a lesser extent with additions of a plasticizer. The internal structure of the bands became more ragged with increasing M, whereas the addition of a plasticizer reestablished the crisp and well-defined micromorphology associated with DGBs in low-M polymers.

KEY WORDS: plastic, poly(vinyl chloride), fatigue crack propagation, electron fractography, discontinuous growth bands, fatigue striation, materials, materials science, fractography

In recent years, increasing attention has been given to an evaluation of fatigue crack propagation (FCP) behavior in engineering plastics as a function of the crack-tip stress intensity factor [*1-3*].² Such information is useful from an engineering viewpoint in that it allows the designer the opportunity to predict the fatigue life and overall cyclic response of numerous polymeric solids. To fill an important gap in our knowledge of fatigue, studies have been undertaken to explore the effect of material variables such as molecular weight [*4-7*], molecular weight distribution [*8*], external [*4*] and internal plasticization [*7*], and processing history [*9*] on FCP behavior in both amorphous and semicrystalline homopolymers and impact-toughened copolymers; parallel

¹ Research assistant, New Jersey Zinc professor of metallurgy, and professor of chemistry, respectively, Materials Research Center, Lehigh University, Bethlehem, Pa. 18015.

² The italic numbers in brackets refer to the list of references appended to this paper.

studies have been conducted as well to examine the role of external test variables (for example, test frequency [10–12] and temperature [13–15] on the fatigue process.

In concert with these studies, efforts have been made to identify fatigue fracture micromechanisms and to relate them to known deformation processes such as crazing and shear [16–19]. In this regard, interpretation of fracture surface morphological features with the aid of optical and electron microscopic techniques has proved to be especially useful. Perhaps the most common morphological feature found in the fatigue fracture of numerous polymeric solids and metal alloys is the fatigue striation. Such markings, which represent the successive positions of the crack front at the end of each loading cycle, are oriented normal to the crack growth direction. Furthermore, the distance between these lines represents the increment of crack growth during a particular load excursion and hence can be compared with the macroscopic crack growth rate when striation formation is the sole operational fatigue mechanism. For this reason, the spacing between adjacent fatigue striations increases with increasing magnitude of the stress intensity factor range, ΔK .

A second type of fracture line has been identified in many glassy [17] and some semicrystalline polymeric [20, 21] laboratory specimens. Here, too, the fracture bands lie perpendicular to the direction of crack extension and increase in width with ΔK , but, unlike fatigue striations, these bands form discontinuously. It has been shown that a craze at the crack tip advances continuously over many hundreds to thousands of loading cycles before the crack suddenly strikes through the craze. Numerous studies have shown that the size of these discontinuous growth bands (DGBs) corresponds to the length of the plastic zone directly ahead of the crack tip [17, 19]. This conclusion follows from a comparison of fracture band widths at different stress intensity levels with computed values of the crack-tip plastic zone dimension computed using the Dugdale formulation [22]

$$\text{band width} \approx \text{plastic zone width} \approx \frac{\pi}{8} \frac{K_{\max}^2}{\sigma_{ys}^2} \quad (1)$$

where $K_{\max}$ = maximum stress intensity factor, and σ_{ys} = yield strength. From Eq 1, the computed value of σ_{ys} should (and does) [17] correspond to the stress for crazing.

Though the generality of the DGB formation process has been established for several polymeric solids, almost no efforts have been made to examine this fracture process as a function of material variables. It is known that DGBs are formed in poly(methyl methacrylate) (PMMA) only at low values of molecular weight [7]. A related study of molecular weight distribution effects on DGB formation in PMMA will be reported shortly [23]. The objective of this investigation is to examine the micromechanisms of fatigue crack propagation advance in poly(vinyl chloride) (PVC) as a function of molecular weight, plas-

ticizer content, and specimen orientation. Poly(vinyl chloride) was chosen for this study because clear-cut DGBs develop readily under cyclic loading, and considerable FCP data have been generated for comparison. In addition, some preliminary fractographic observations have been reported for this material [4].

Experimental Procedure

The specimens in this study were obtained from two sources. Cadillac Plastics supplied extruded PVC of unspecified composition, which was found to possess a molecular weight (M) of 108 000 (by gel permeation chromatography). In addition, two series of milled and compression-molded PVC were supplied by the B. F. Goodrich Co.; the Series 1 and Series 2 specimens together encompassed a weight-average molecular weight range from 0.61×10^5 to 2.25×10^5 (by gel permeation chromatography). Specimens of Series 1 were prepared with varying amounts of the plasticizing agent dioctyl phthalate (DOP) [4], whereas Series 2 specimens were received only in the unmodified state. The specific designations of these specimens, their plasticizer contents, and their molecular weights are given along with their thickness in Table 1. Since the values of weight-average to number-average molecular weights, M_w/M_n , fell between 2.07 and 2.17, with no trend as a function of M , the breadth of molecular weight distribution is relatively narrow and essentially independent of M . The fatigue crack propagation data for these materials have been reported previously [4, 6] and are shown in Fig. 1 for those specimens that do not contain any plasticizing agent. (Skibo et al reported that

TABLE 1—*Materials.*

Series (source)	Specimen Designation	Thickness, mm	$M_w \times 10^{-5}$
1 (B. F. Goodrich)	A	6.3	0.605
	A(13) ^a	6.3	0.605
	B	6.3	0.965
	B(13)	6.3	0.965
	C	6.3	1.41
	C(6)	6.3	1.41
	D	6.3	2.25
	D(6)	6.3	2.25
	D(13)	6.3	2.25
2 (B. F. Goodrich)	131-1	6.3	0.67
	132-1	6.3	0.95
	133-1	6.3	1.06
	134-1	6.3	1.69
	135-1	6.3	2.08
Cadillac Plastics	CP	3.2	1.08

^a Numbers in parentheses represent the DOP content in parts per hundred of resin.

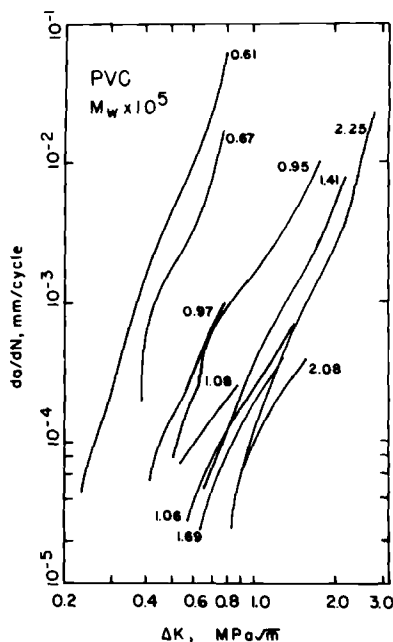


FIG. 1—Effect of molecular weight on the fatigue crack propagation of PVC; $R = 0.1$, 10 Hz.

DOP additions to PVC had little influence on FCP rates [4].) The fatigue specimens were of the compact tension geometry, with a height-to-weight ratio (H/W) of 0.6, in accordance with the ASTM Test for Constant-Load-Amplitude Fatigue Crack Growth Rates Above 10^{-8} m/Cycle (E 647-78T). The fatigue tests were conducted using sinusoidal loading and a frequency of 10 Hz. Precracking was carried out at 100 Hz and was terminated when the crack had grown from the starting chevron region to a distance of approximately one half the specimen thickness. The stress ratio, $\sigma_{\max}/\sigma_{\min}$, was 0.1, and the stress-intensity factor, ΔK , was determined from the relationship

$$\Delta K = Y \Delta \sigma \sqrt{a} \quad (2)$$

where a is the crack length, $\Delta \sigma$ is the change in applied stress, $\sigma_{\max} - \sigma_{\min}$, and Y is a geometrical factor based on the ratio of crack length, a , to the overall specimen length, W . For this specimen configuration

$$Y = 29.6 - 185.5 (a/W) + 655.7 (a/W)^2 - 1017 (a/W)^3 + 638.9 (a/W)^4$$

One specimen was examined for each test condition with the anticipated crack plane being oriented normal, parallel, and at ± 45 deg to the original processing direction.

All fracture surfaces were analyzed with the aid of an optical metallograph and an ETEC scanning electron microscope (SEM), using an accelerating voltage of 20 kV. The fracture surfaces for study in the SEM were prepared by

coating them with carbon and then with gold, or by coating them with a gold-palladium alloy alone.

Results and Discussion

Macromorphology of PVC

Molecular Weight and Plasticizer Effects—Unmodified specimens from the Series 1 and 2 material supply were used to study the effect of molecular weight on the fatigue fracture morphology. All the specimens having molecular weights in the range between $M_w = 0.95 \times 10^5$ and $M_w = 2.25 \times 10^5$ failed by discontinuous crack growth. In sharp contrast, the specimens with weight-average molecular weights of 0.61×10^5 and 0.67×10^5 showed no evidence of DGB formation and presumably failed by some continuous cracking process. Furthermore, in all but one case, DGBs were found to extend over the entire fatigue fracture region of these specimens. The exception was Specimen 132-1 from Series 2 ($M_w = 0.95 \times 10^5$), which revealed DGBs only at low values of ΔK ; fatigue striations were observed at high ΔK levels.

Measurements of band sizes were made from all specimens and compared with the corresponding ΔK levels. These results are shown in the form of least-square fit lines, as shown in Fig. 2. (The symbols shown in these plots are for

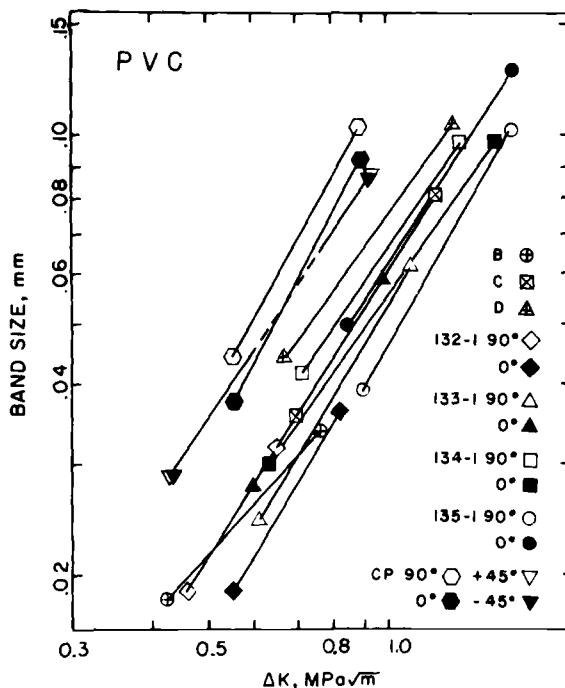


FIG. 2.—Discontinuous growth band size as a function of ΔK and test orientation for PVC. No DGBs were observed below $M_w = 0.95 \times 10^5$.

TABLE 2—*Calculated yield stresses for PVC material, CP and Series 1 and 2, using the Dugdale model.^a*

Series	DOP, percent	σ_{ys}^b , MPa
1	0	70 to 80 [4]
1	6	56
1	13	40
2	0	75 to 85
CP	?	53 to 61 [17]

^a In all cases $R(\sigma_{min}/\sigma_{max}) = 0.1$.^b An average.

the purpose of identification of each material, with each line being derived from roughly ten separate data points.) Note that the data from Specimens B to D and 132-1 to 135-1 all lie within a relatively narrow scatter band and reveal no clear effect of molecular weight on the size of the DGBs. On the other hand, the band spacings associated with the Cadillac Plastic (CP) specimens were consistently larger than measurements from other specimens, regardless of test direction.

The logarithmic slopes of the band size versus ΔK plots for these specimens are similar (1.7 ± 0.2) and in reasonably good agreement with the second power relationship of K_{max} to the plastic zone size, as proposed in the Dugdale model [22]. Consequently, Eq 1 was used to calculate an inferred yield strength for these materials at different ΔK values over the test range (see Table 2). The inferred yield strengths for Specimens B, C, and D from Series 1 varied over a small range from 70 to 80 MPa, whereas the yield strength values from Specimens 132-1 to 135-1 from Series 2 ranged from 75 to 85 MPa. These values are somewhat higher than those reported by Mills [24], who found an upper yield strength of 65 MPa in rigid PVC when tested at a strain rate of 10^{-4} s^{-1} . Since the strain rate associated with fatigue testing at 10 Hz is surely several decades higher than that employed by Mills, one would expect the inferred yield strengths, derived from these fatigue experiments, to be higher. In fact, using a yield strength-strain rate dependency in PVC of approximately 6 MPa per decade change in strain rate, as determined from results generated by several investigators [25-28], the 10 to 15 MPa difference between yield strength values reported by Mills and inferred in this study from fractographic measurements would correspond to a twofold to threefold difference in the strain rates used in these two studies. This difference appears reasonable based on some preliminary computations of the strain rate at the crack tip. Overall, the inferred yield strengths for these specimens varied little with molecular weight—a finding that is consistent with the lack of dependence of yield strength on molecular weight for this range of M_w values [29, 30].

As expected, the addition of 6 and 13 percent DOP plasticizing agent to Se-

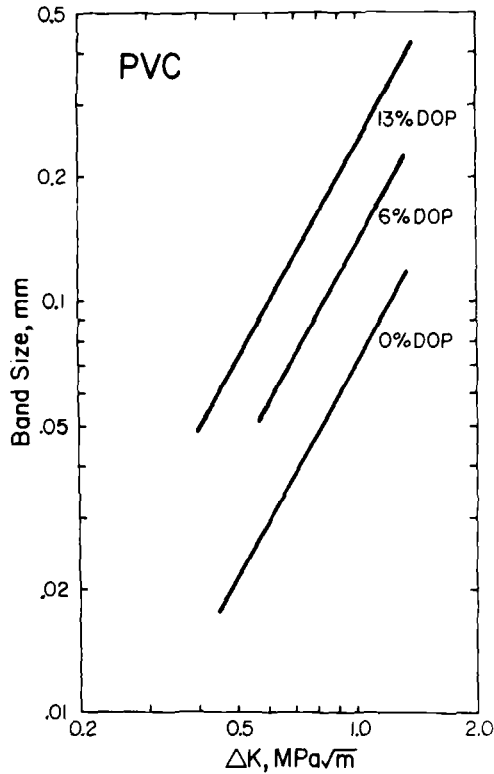


FIG. 3—Effect of DOP content on DGB sizes on the fatigue fracture surface of PVC.

ries 1 specimens reduced the inferred yield strength by 25 and 47 percent, respectively (Table 2), which reflects the increased DGB widths associated with the addition of DOP (Fig. 3). Finally, the larger DGB spacings in the CP material, mentioned earlier, translate to a lower inferred yield strength—an observation that suggests a significant degree of plasticization in these specimens.

Recalling that a DGB is developed over many loading cycles, the cyclic stability or life of each band, N^* , may be determined by dividing the band width by the corresponding fatigue crack growth rate so that

$$N^* = \frac{\text{band width}}{da/dN} \quad (3)$$

From Fig. 4 it can be seen that N^* depends strongly on the stress-intensity factor range; in all cases, the number of loading cycles necessary to strike through the craze decreases as ΔK increases. This is attributed to the greater amount of cyclic damage introduced within the craze zone at the higher ΔK levels. Of particular significance is the fact that the overall craze stability is

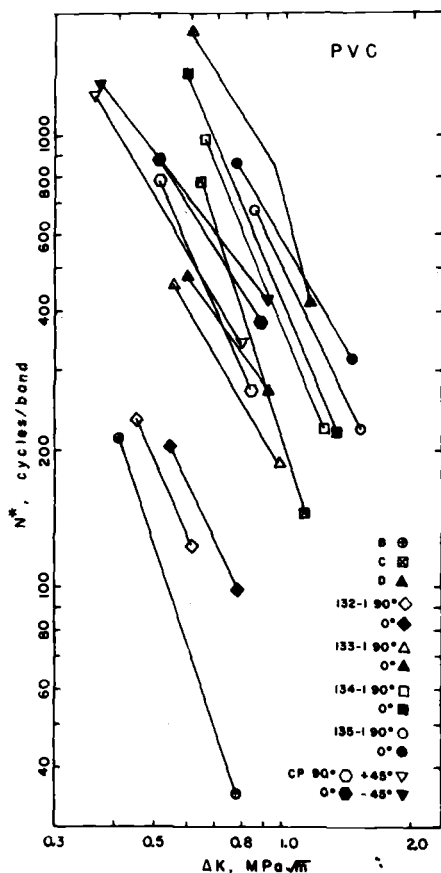


FIG. 4—Effect of molecular weight on cyclic stability, N^* , of crazes in PVC as a function of ΔK and the test orientation.

seen to improve with increasing molecular weight and is quite consistent with the markedly greater degree of fatigue crack propagation resistance observed with high- M specimens (Fig. 1). The largest improvement in craze stability is found by increasing M in the low- M regimen, with craze stability tending to reach some limiting value at high- M levels. This saturation in craze stability at high M is to be expected because improvement in FCP response likewise reaches a limiting growth rate plateau at high M [4, 6] (see Fig. 1). That is, since DGB widths do not change significantly with M , and the growth rate approaches an asymptotic value at high molecular weights, N^* tends toward a limiting level for a given ΔK (see Eq 3 and Fig. 5).

Finally, the cyclic stability of DGBs in the plasticized material is greater than that of the unplasticized materials. This arises from the fact that the FCP resistance of PVC with different values of M is little affected by the plasticizer content [4]. Consequently, N^* should increase with increasing plasticizer con-

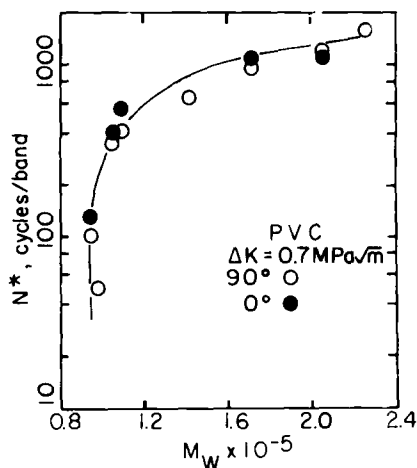


FIG. 5—Cyclic stability, N^* , as a function of molecular weight, M_w .

tent, since the band size increases (Fig. 4) while da/dN remains essentially unchanged. For this same reason, the cyclic life of the DGB in the CP material supply is greater than the DGB cyclic life in Specimen 133-1 even though the molecular weight is the same in these two materials (Fig. 4). A similar trend in cyclic stability of the DOP-plasticized specimens is shown in Fig. 6. N^* increases with increasing DOP content for the three molecular weights studied. There is, however, a change in relative ranking between Specimens D(6) and D(13), which is attributed to the lower FCP rates exhibited by Specimen D(6) (recall Eq 3).

Effect of Residual Orientation—Specimens of Series 2 at each molecular weight were tested with the load axis both parallel (0 deg) and perpendicular (90 deg) to the milling direction. The fracture mechanisms at each M in the two orientations were found to be fundamentally the same. That is, Specimen 131-1 revealed no DGBs in either test orientation, whereas Specimens 132-1 to 135-1 revealed DGBs in both test orientations. Referring again to Fig. 2, it appears that although there are small differences in DGB width with orientation at each molecular weight, there is no well-defined trend. However, a consistent relationship is revealed between craze stability, N^* , and the test orientation (Fig. 4). Discontinuous growth bands produced in 0 deg specimens tend to be slightly more stable than those found on the 90 deg specimens. Consequently, for this series of milled and compression-molded PVC specimens, the test orientation does not have a pronounced effect on the inferred yield strength, but does affect the craze stability. When the test is conducted so that the loading axis is parallel to the original milling direction, the crazes have a small but consistently greater stability at all molecular weights. This is consistent with the existence of a mill-induced directionality of chain entanglements, as discussed elsewhere [6].

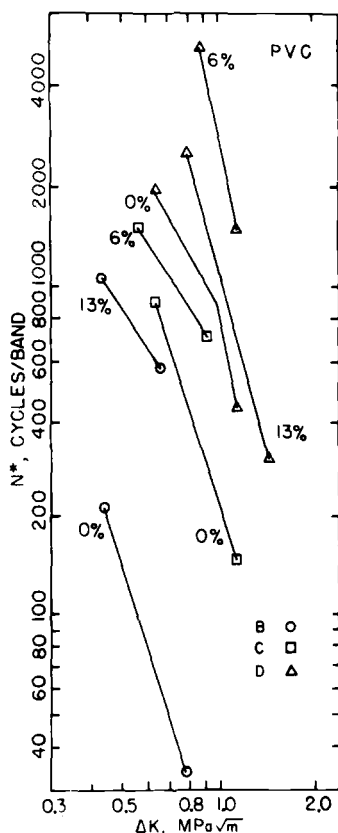


FIG. 6—Effect of DOP content on cyclic stability, N^* , of crazes in PVC as a function of ΔK .

The effect of residual orientation on DGB formation in the extruded CP PVC (tested with the load axis at 0, 90 and ± 45 deg to the extrusion direction) is somewhat more complicated. The 90 deg specimen shows DGBs across the entire width of the fracture surface and at all stress-intensity levels up to fast fracture. The other three orientations have a rough, "mist" region in the interior of the fracture surface in the intermediate ΔK range (~ 0.47 to $0.75 \text{ MPa} \sqrt{\text{m}}$). This mist region interrupts DGB formation either mildly (0 deg) or strongly (± 45 deg). A similar mist region has been noted on the fracture surfaces of polycarbonate and polysulfone [17] and is believed to be the result of the crack growing through multiple crazes, rather than a single craze, during fatigue crack growth. The reason for this transient behavior in some CP specimens is not clear at this time. Band size measurements for these specimens are shown in Fig. 2. (For the ± 45 -deg specimens, measurements were made only at those ΔK values where the DGB extended across the entire fracture surface width, that is, at low and high ΔK .) The results show that the 90-deg orientation specimen possessed the largest band size at a given ΔK , which implies that

the yield strength is lowest in this testing direction. It is interesting to note again that the craze stability is greater for the 0-deg orientation specimen than for the 90-deg specimen. At this time, we can provide no explanation for the difference in craze stability associated with the ± 45 -deg specimens.

Micromorphology

Molecular Weight Effect—When DGBs are examined in the SEM at increased magnification, some general trends in micromorphology become apparent as a function of ΔK , molecular weight, and plasticizer content. Each discontinuous growth band reveals an array of microvoids that decrease in size in the direction of crack growth. These microvoids reflect the passage of the fatigue crack through the midplane-region of the porous craze zone. As discussed earlier, a DGB is formed over many loading cycles, so that the craze accumulates an additional increment of damage with each loading cycle. A gradual breakdown of molecular entanglements results, along with the nucleation and growth of microvoids. It is believed that the size of the voids depends on the magnitude of the crack-tip opening. Since this displacement decreases with increasing distance from the crack tip, the resulting voids should vary in size in similar fashion. Figure 7 illustrates this by showing the last half of one DGB (small voids) and the beginning of the next DGB (large voids). The darker band, which separates these two contiguous bands, is referred to as the stretch zone and represents the region of crack blunting associated with crack arrest as the crack suddenly encounters initially uncrazed polymer material.

At lower molecular weights ($M_w = \sim 0.95$ to 1.10×10^5), it appears that with increasing ΔK , the stretch zone width increases in relation to the overall band size (Fig. 8a and b). Since the crack opening displacement is directly related to K_{max}^2 ($COD = K_{max}^2 / E\sigma_{ys}$), one would expect crack-tip blunting to be greater at higher stress-intensity levels.

At higher molecular weights ($M_w = \sim 1.4$ to 2.25×10^5) the DGBs become less well defined. As is seen in Fig. 9 for Specimen 134-1 ($M_w = 1.69 \times 10^5$), the void gradient no longer reveals a smooth transition from large to small voids. Also, although the stretch zone is still apparent at high ΔK for this molecular weight, it is not a distinct feature of the band. That is, the large voids seem to be "overlapping" into the stretch zone. Finally, an examination of the highest M specimen of Series 2 ($M_w = 2.08 \times 10^5$) reveals that the stretch zone is completely obscured by large voids and that no clear void gradient can be distinguished (Fig. 10a).

As discussed earlier, an increase in molecular weight serves to enhance craze stability (Fig. 4). We believe that this enhanced craze stability is due to more effective entanglements of the longer molecular chains at high M [31, 32], thus, craze breakdown is delayed, and void coalescence occurs more slowly. In addition, the enhanced craze stability allows the void growth to proceed further

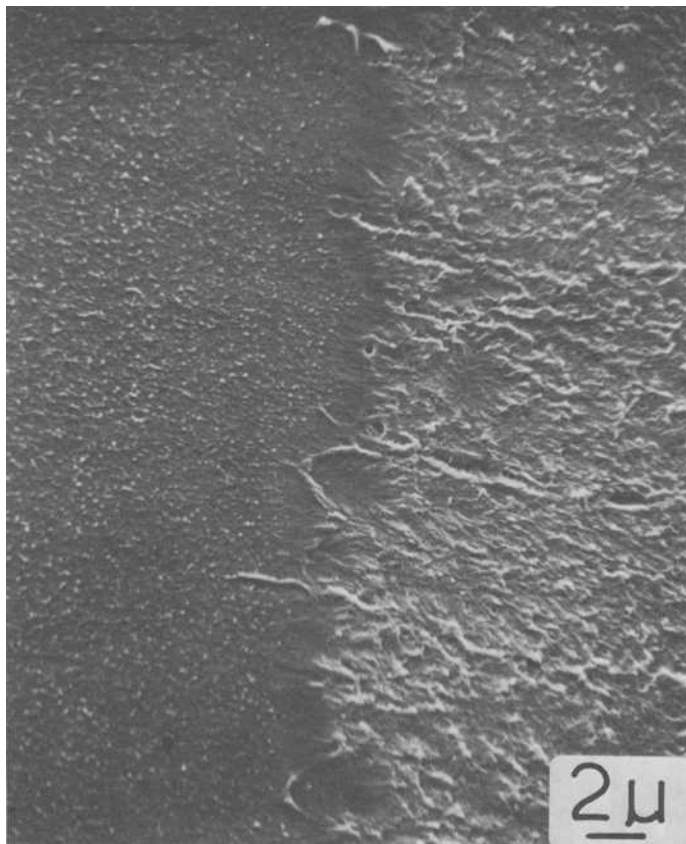


FIG. 7—An SEM fractograph showing the void gradient and stretch zone associated with adjacent DGBs. The arrow indicates the direction of crack growth; $M_w = 0.96 \times 10^5$, 13 percent DOP, $\Delta K = 0.5 \text{ MPa } \sqrt{m}$.

prior to instability, which results in the formation of a more mottled fracture surface.

As noted in the previous section, the addition of a plasticizer to the PVC matrix has the effect of decreasing the yield strength, thereby increasing the size of the DGBs. It is most striking that the addition of plasticizer makes the DGBs more crisp and well defined. Figure 10*a* and *b* shows the difference in DGBs at high M in the unplasticized and plasticized (13 percent DOP) states. Notice that there is considerably less tufting on the fracture surface of the plasticized specimen than on the unmodified PVC specimen. Furthermore, when the plasticizing agent is added, the gradient from large to small voids within the bands becomes smooth again.

It is interesting to note that the CP material supply revealed a more clearly defined set of bands than specimens of the same M_w ($\sim 1.10 \times 10^5$) from Series



FIG. 8—Discontinuous growth band morphology of low- M PVC as a function of the stress intensity level. (a) $\Delta K = 0.42 \text{ MPa} \sqrt{\text{m}}$. (b) $\Delta K = 0.77 \text{ MPa} \sqrt{\text{m}}$. $M_w = 0.97 \times 10^5$.

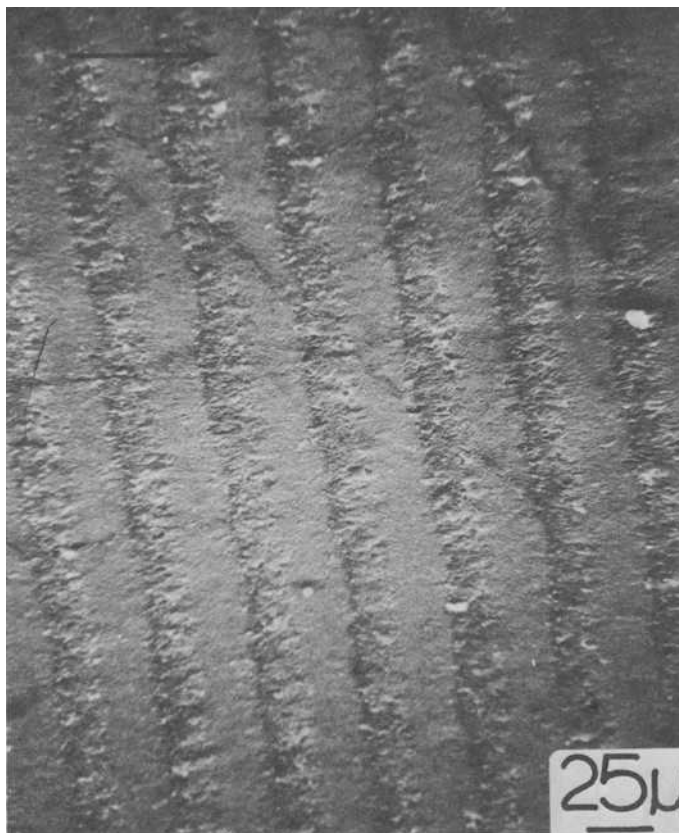


FIG. 9—Discontinuous growth band morphology of high- M PVC revealing a more ragged structure than that at low M . $M_w = 1.69 \times 10^5$, $\Delta K = 0.66 \text{ MPa } \sqrt{\text{m}}$.

1 or 2 (see Fig. 11a and b). From the previous discussion, this would suggest that the CP material contained a plasticizing component. This conclusion is consistent with the previously observed fact that the inferred σ_{ys} in the CP material was low. In addition, it was observed in a previous study [6] that the FCP slope for the CP material was considerably lower than the FCP slopes of the other molecular weight specimens (Fig. 1) and that its glass transition temperature was approximately 13 deg lower than that for the unmodified PVC materials.

Additional Morphological Features—It is interesting to note that although Series 1 and Series 2 materials were provided by the same supplier and were presumably processed in the same manner (milled and compression molded), they exhibited some differences in fracture surface morphology. This suggests that the processing conditions for these two materials were, in fact, different.

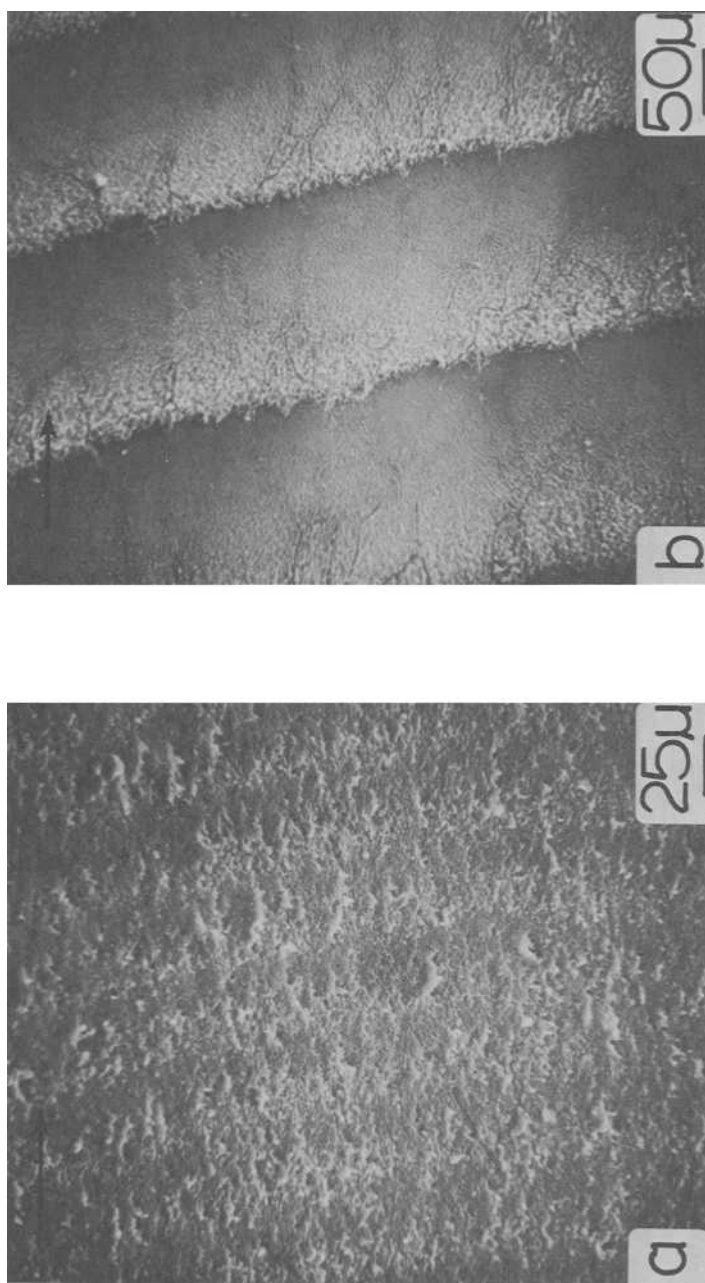


FIG. 10—The effect of a plasticizer on the size and structure of DGB morphology at high M . (a) $M_w = 2.1 \times 10^5$, 0 percent DOP, $\Delta K = 0.89$ MPa $\sqrt{m}$. (b) $M_w = 2.25 \times 10^5$, 13 percent DOP, $\Delta K = 0.81$ MPa $\sqrt{m}$.

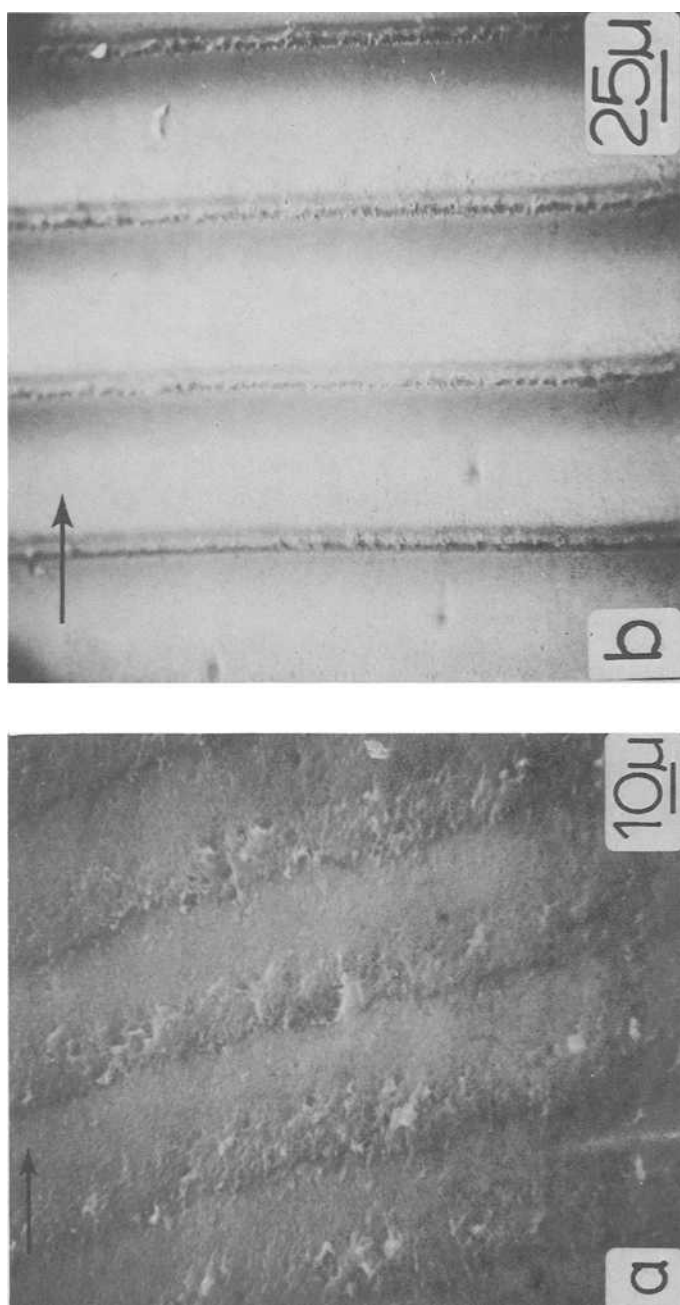


FIG. 11—Comparison of DGB morphology of an unmodified PVC fracture specimen and a CP PVC fracture specimen. (a) $M_w = 1.06 \times 10^5$, $\Delta K = 0.74 \text{ MPa } \sqrt{\text{m}}$. (b) $M_w = 1.07 \times 10^5$, $\Delta K = 0.68 \text{ MPa } \sqrt{\text{m}}$.

Series 1 specimens exhibited particle boundary failure [4], especially at high stress intensities and high molecular weight. The interfacial breakdown between the original resin particles can be clearly seen in Fig. 12 and indicates that at high stress-intensity levels the interfacial polymer material is less resistant than the particles themselves to fatigue crack growth. (Note that PVC particles straddle adjacent DGBs.) Apparently, the milling conditions in the Series 1 material supply resulted in a lower degree of fusion and homogenization than was the case with the Series 2 mill charge. In contrast, interparticle failure was not observed on the Series 2 specimens.

A reexamination of the fracture surfaces from the lowest *M* materials (Specimens A and 131-1) revealed bands running perpendicular to the direction of crack growth. Measurement of these fracture surface markings in Specimens A and 131, such as those shown in Fig. 13a, indicate that their spacing for the



FIG. 12—A photomicrograph of a PVC fracture surface showing boundary failure around the original resin particles. Note the prominent stretch zone. $M_w = 1.41 \times 10^5$, 6 percent DOP, $\Delta K = 1.00 \text{ MPa } \sqrt{m}$.

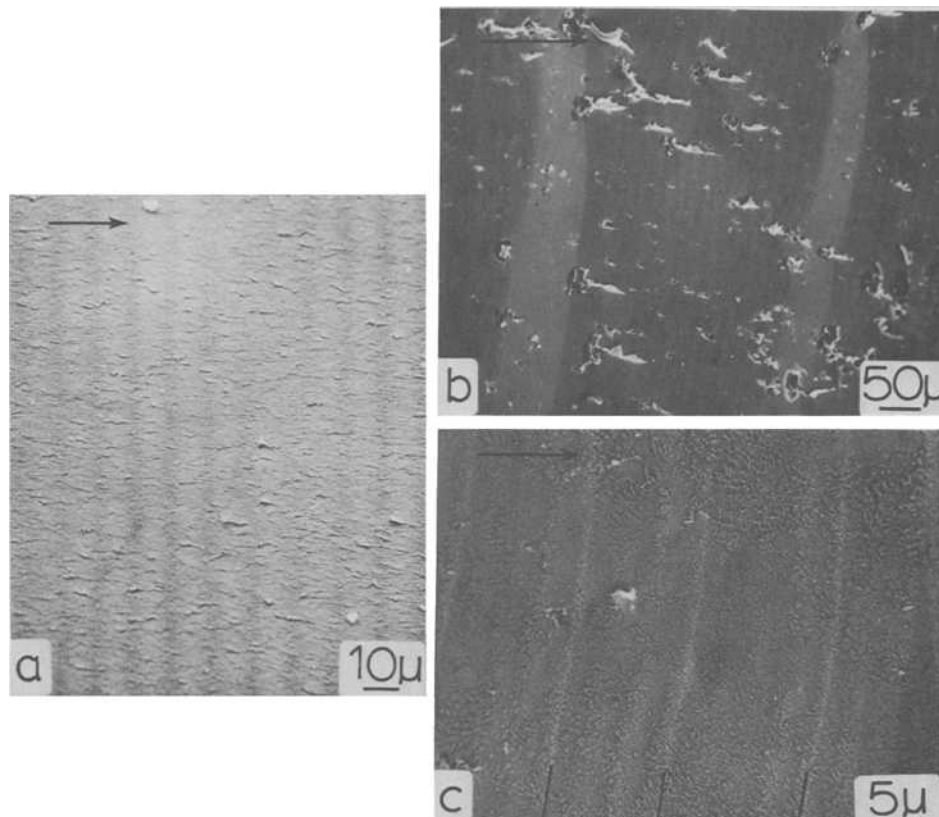


FIG. 13—Fracture surface of PVC at very low M . (a) Constant-sized bands, found at low ΔK levels ($\Delta K = 0.4 \text{ MPa } \sqrt{\text{m}}$). (b) Striation-like lineage (within crack arrest marks) corresponding to macroscopic growth rate, near fast fracture. $\Delta K = 0.64 \text{ MPa } \sqrt{\text{m}}$. (c) High magnification of the bands in (b), which reveals the finer internal lineage. The marks indicate the boundaries of each "striation." $M_w = 0.61 \text{ to } 0.67 \times 10^5$.

most part is invariant with ΔK . Note that this differs from discontinuous growth bands, which increase in size as a function of ΔK^2 . Therefore, it is not clear whether these lines represent specific crack front positions during the test. Furthermore, the micromorphology of these bands does not include the microvoid gradient found to be present on markings known to reflect discontinuous crack extension. Therefore, the determination of N^* for each of these bands would generate information of questionable significance, and for this reason it was not included in Figs. 4 and 5. These bands are reminiscent of the constant-sized bands found on polystyrene fracture surfaces over a limited ΔK range [16]. Finally, at stress-intensity levels near fast fracture, one finds yet another set of fracture markings in Specimen A (Fig. 13b). The broad light bands represent arrest lines associated with periodic interruption in the test

for the purpose of crack length measurements. The number of narrower lines sandwiched between these arrest bands corresponds to the number of loading cycles between successive crack length readings and implies that these markings are striations. And yet these "striations" are themselves composed of an integral number of narrower lines similar to those found at low ΔK levels (Fig. 13c). To our knowledge, a striation substructure of this type does not correspond to any specific cyclic loading event such as a part of the loading cycle. Rather, these additional fracture lines are believed to be caused by plastic flow-induced perturbations of an already formed fatigue fracture surface. Similar ripple-type markings have been shown to exist in metals [33].

Specimen 132-1 ($M_w = 0.95 \times 10^5$) was found to exhibit a change in the fatigue crack growth mechanism midway through the fatigue test. This transition is associated with the discontinuity in crack growth rate at a ΔK of 0.8 to 1.0 MPa $\sqrt{\text{m}}$ (Fig. 14). Furthermore, the slope of the da/dN versus ΔK plot after the transition decreased from 3.25 to 2.75. This transition occurred in both the 0 and 90-deg test orientations at approximately the same stress-intensity level. At low ΔK levels, the fracture surface was yellowish but then changed sharply at the transition to a multicolor rainbow pattern. This occurrence is similar to the transition noted in polystyrene, where the multicolor appear-

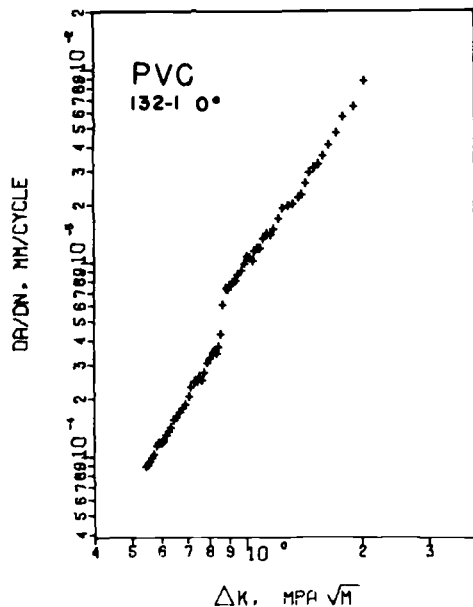


FIG. 14—Fatigue crack propagation Specimen 132-1, showing the discontinuity in crack growth rate at $\Delta K \approx 0.8 \text{ MPa } \sqrt{\text{m}}$ associated with the fracture mechanism transition. $M_w = 0.95 \times 10^5$, 0 deg test orientation.

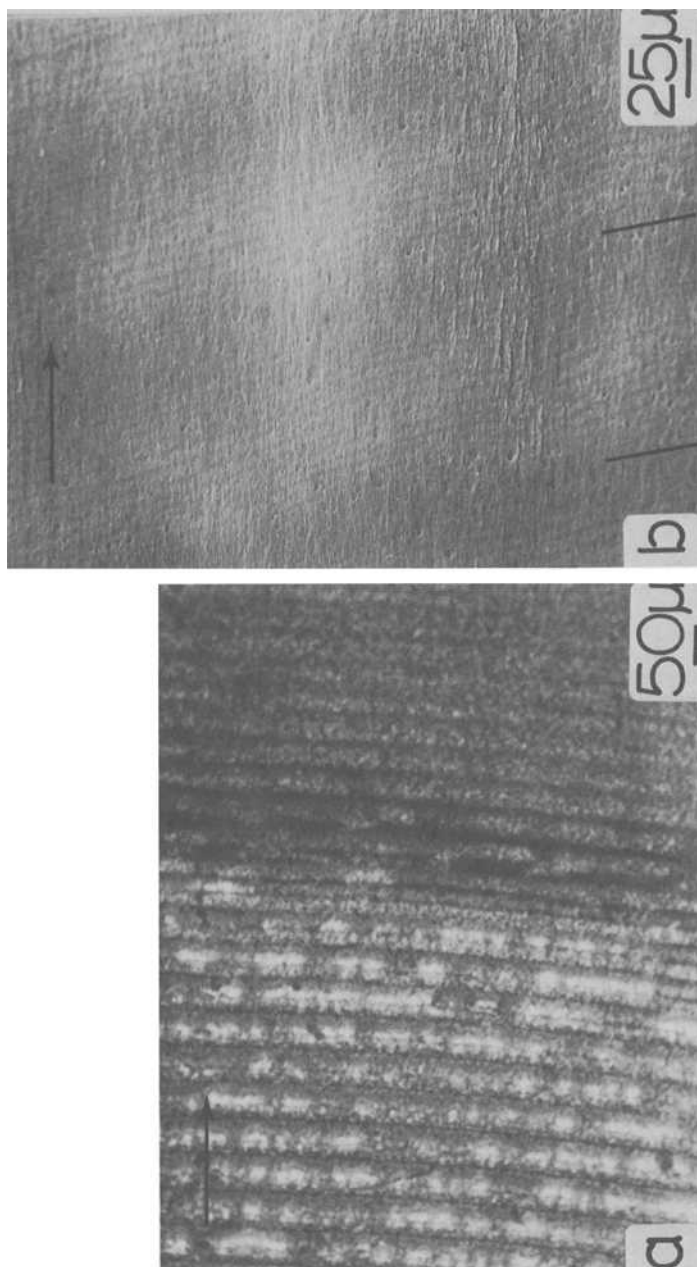


FIG. 15—(a) Light photomicrograph of the fracture transition of Specimen 132-1. The light patchy areas indicate the roughness of DGBs, whereas the lower contrast region indicates a smoother texture, $\Delta K = 0.8 \text{ MPa } \sqrt{\text{m}}$. (b) A periodic array of finer fracture lines which comprise each larger band. The marks indicate the boundaries of large bands. $\Delta K = 1.76 \text{ MPa } \sqrt{\text{m}}$, $M_w = 0.95 \times 10^5$.

ance was found to be due to the early stages of crack growth through craze bundles [16].

Prior to the transition, cracking occurred by discontinuous growth, as observed in the higher molecular weight specimens. When viewed under the light microscope, these bands revealed a rough texture due to the void gradient and stretch zone that make up each band. Beyond the transition, the bands appeared as undulations on the fracture surface and were composed of small, regular-sized voids, which thereby gave them a smoother texture (Fig. 15a). With increasing ΔK , these new bands increased in size and were found to be close to, but smaller than, predicted DGB widths. At still higher ΔK levels, these bands were seen to contain a periodic array of finer fracture lines which changed in spacing across the dimension corresponding to the larger band widths (Fig. 15b). Just short of fast fracture, the distance between these fine lines was found to correspond to the macroscopic growth rate; at the same time, the larger undulations were no longer identifiable. Similar fatigue behavior has been noted by Mackay et al [34] for the case of fatigue in polycarbonate. Further studies are needed to provide explanations for the transition in fatigue crack growth mechanisms in these two materials. To be sure, the fatigue micromorphology in amorphous plastics is surprisingly complex.

Conclusions

Over the molecular weight range studied, discontinuous crack growth is found to be the major fatigue cracking mechanism in PVC. The inferred yield strength is not strongly dependent on M or test orientation but does vary markedly with plasticizer content. Craze stability, that is, the number of loading cycles required to strike through a craze, is found to be strongly dependent on molecular weight. Plasticizer additions also contribute to improvement in discontinuous growth band stability, though to a lesser degree.

Whereas DGB size is not dependent on M , the micromorphology of the discontinuous growth band varies strongly with molecular weight. With increasing M , the DGBs become less clean; both the void gradient and the stretch zone lose their distinctiveness, and the boundaries between the bands become difficult to identify. Finally, the crisp internal structure of the DGBs found at low M is restored to that observed in the high- M specimens through the addition of a plasticizing agent.

Acknowledgments

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Fracture of Tungsten Wire in Metal Matrix Composites

REFERENCE: Kim, C., Phillips, W. L., and Weimer, R. J., "Fracture of Tungsten Wire in Metal Matrix Composites," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 314-333.

ABSTRACT: Mechanical properties were measured for the tungsten-fiber-reinforced Type 6061 aluminum and Type 1010 steel matrix composite systems. The tensile strength of the latter system was appreciably lower than the theoretical rule-of-mixture value. Auger spectroscopy and electron microprobe analysis showed significant iron contamination in the tungsten fibers. In addition, the tensile fracture mode at room temperature was shown to change from ductile to completely brittle upon annealing at 800 to 950°C, that is, just slightly above the fabrication temperature. Quantitative microanalysis showed that penetration of the iron diffusion front into the tungsten fiber coincided exactly with an annular fracture feature observed by scanning electron microscopy of tensile fracture surfaces. The strength degradation in steel/tungsten composites was due to fiber embrittlement caused partly by microstructural polygonization during the recovery process, but primarily by grain boundary diffusion of elemental iron from the steel matrix.

KEY WORDS: aluminum, annealing, diffusion, fractography, materials, materials science, metal matrix composite, steel, tensile strength, tungsten fiber

Fiber-reinforced metal matrix composites have the potential to improve dramatically the performance of advanced propulsion and power generation systems because the high-temperature strength and creep resistance is far superior to that of conventional monolithic materials. Equally important from a structural design viewpoint is the fact that significant increases in strength and stiffness are obtained from composite materials at substantial weight reductions over those obtained from their monolithic counterparts. Present and future applications of composite materials include airframe components, turbine blades, advanced land and sea vehicles, and space satellites. This increased use demands an improved understanding of the initiation of structural damage and the subsequent failure of these materials in a variety of design applications.

¹ Materials engineer, physical scientist, and supervisory materials engineer, respectively, Naval Research Laboratory, Washington, D.C. 20375.

Although many techniques have been developed to fabricate metal matrix composites, most systems of engineering significance employ either liquid metal infiltration of a fiber array or solid-state diffusion bonding of fiber arrays to metal foils. In both cases, exposure of the materials to necessarily high temperatures alters the properties of the constituents and often produces undesirable reactions between fiber and matrix, thereby making the final composite less than ideal.

Specifically, such high-temperature exposure recrystallizes and embrittles the tungsten fibers, thereby reducing their mechanical properties. The recrystallization temperature may be greatly increased by the presence of trace amounts of alumina or thoria [1]², whereas other impurities such as nickel [2-4] and iron [3] appreciably reduce the recrystallization temperature. One study of the effects of various contaminants on the room temperature tensile properties of tungsten fibers infiltrated with a liquid copper alloy concluded that the reduced ductility and tensile strength were due to a notch effect [5]. The room temperature mechanical properties of highly strained tungsten fibers change gradually with annealing temperatures less than one third the melting point due to rearrangement of the submicrostructure, that is, polygonization during the recovery process [6, 7]. However, it remains unclear just how the mechanical properties of tungsten fibers change when diffusion bonded to common structural metals such as commercial aluminum and steel alloys.

The purpose of the present study is to determine thermal effects on tungsten reinforcing elements in terms of both isothermal fabrication anneals and interfacial reaction products. The mechanical properties were measured on tungsten-reinforced aluminum and steel composites, as well as on tungsten fibers, both isothermally annealed and extracted from failed test specimens. The failure surfaces were examined metallographically and fractographically using scanning electron microscopy. Additional characterization included Auger spectroscopy, electron microprobe analysis, and energy-dispersive X-ray analysis. These microscopic results were used to explain various aspects of material property degradations.

Experiment

Three commercially available metal matrix composite materials were evaluated in this study. One composite system consisted of Type 6061 aluminum alloy unidirectionally reinforced with 45 volume percent of NS-55 tungsten fibers with diameters of 110 μm (Manufacturer A) and 125 μm (Manufacturer B). The other system investigated was Type 1010 steel unidirectionally reinforced with 45 volume percent of 110- μm diameter NS-55 tungsten fibers. These three composite materials are designated Al/45W(A), Al/45W(B), and St/45W(A).

²The italic numbers in brackets refer to the list of references appended to this paper.

The composites were fabricated in a vacuum retort by a solid-state diffusion bonding technique in which sandwiches built up by alternating layers of metal matrix foils and aligned fiber arrays were subjected to high temperatures and pressures to achieve consolidation. The fabrication parameters were 69 MPa (10 ksi) pressure for 30 min at temperatures of 482°C for Al/45W(A,B) and 760°C for St/45W(A). After air cooling, the composite plates were cut into appropriate mechanical test specimens. Microscopic examination of the fibers and composite materials both before and after testing took many forms, and each approach is described in the following section.

Results

Uniaxial Strength of Composite Materials

Mechanical test specimens were cut from composite plates and diamond ground to final dimensions so that surfaces were as flaw-free as possible. Figure 1 illustrates the specimen configurations used for longitudinal tension and compression tests. The tensile specimens were 10 cm long, 1 cm wide at the shoulders, 0.5 cm wide in the gage section, and 0.25 cm thick. The gage length was 2.5 cm and was blended into the shoulders with a 1.25-cm radius of curvature. The compression specimens were 1.7 cm long and 0.42 cm in diameter. Because the strength of composites usually depends critically upon maintaining uniformly axial loads, the tensile coupons were fitted with special gripping tabs fabricated from Type 6061-T6 aluminum and bonded with Hysol E9309-2 Aerospace Adhesive. A rather different problem that must be dealt with in compression testing is the tendency of the specimen ends to "broom" before maximum load can be developed in the composite. Brooming restraint rings were fashioned from hardened Type 4340 steel and were individually bored to provide a good friction fit to each specimen.

Room temperature tension and compression tests were conducted on all three composite materials using a closed-loop servo-hydraulic testing machine operated under displacement control at strain rates of the order of 10^{-4} s⁻¹. The results are presented in Table 1, which also includes rule-of-mixture (ROM) calculations of composite strength. The observed tensile strengths of the aluminum/tungsten composites generally agreed quite well with ROM values, whereas steel/tungsten composite tensile strength was considerably less than the expected ROM value; this suggests that perhaps the fibers were embrittled and notch sensitive. This would account for the loss in tensile strength for this system. The low transverse strength is, unfortunately, characteristic of unidirectionally reinforced composites, and microstructural sources of this effect will be examined in a later section.

Tensile Properties of Tungsten Fibers

A large range of consolidation temperatures is possible to effect diffusion bonding during the composite fabrication process, and the temperature actu-

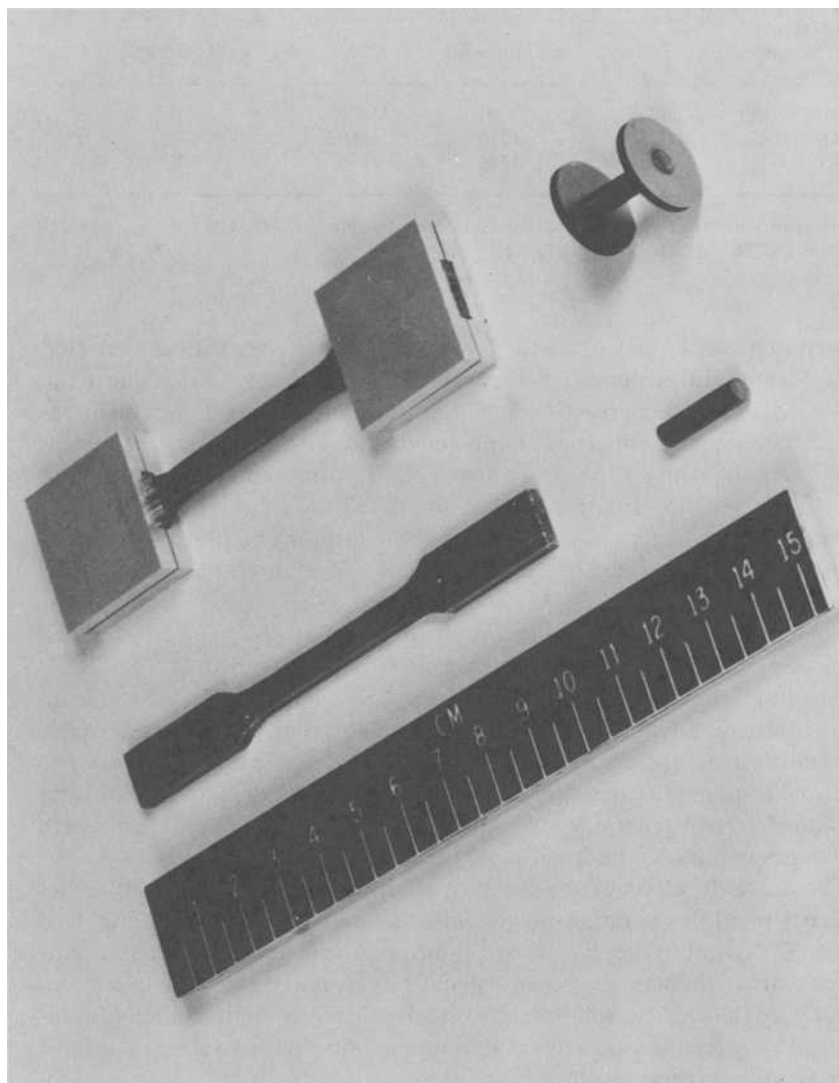


FIG. 1.—Test specimen configurations for uniaxial strength tests.

TABLE 1—*Uniaxial strength of composite materials.*

Material System	Longitudinal Strength, MPa			Transverse Strength, MPa
	Tension	Compression	ROM ^a	Tension
Al/45W(A)	1248	890	1427	131
Al/45W(B)	1280	1216	1289	155
St/45W(A)	627	1346	1496	232

^a Rule-of-mixture (ROM) calculations used matrix strengths of 240 MPa for Type 6061-T4 aluminum and 365 MPa for Type 1010 steel.

ally used depends largely upon the fabricator's experience with the selected matrix. Meaningful evaluation of mechanical property degradation due to the fiber-matrix interfacial reaction and the thermomechanical treatment required a base line on properties of unreacted materials. This base line was established by exposing 125- μm diameter NS-55 tungsten fibers to 30-min, isothermal, annealing treatments that simulated the fabrication processes. The tensile test specimens utilized the paper-tab gripping method described in the ASTM Test for Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials (D 3379-75), and these specimens were tested at room temperature using a fixed strain rate of $1.8 \times 10^{-3} \text{ s}^{-1}$. The effect of annealing temperature on the ultimate tensile strength is shown in Fig. 2.

Annealing temperatures up to approximately 700°C produced slight increases in strength. Above 700°C, the strength decreased, which suggests some sort of microstructural change in the fiber. It is reasonable to expect this trend in thermal response to carry over into the composite. Interfacial reactions and diffusion effects, in general, would tend only to alter the temperature at which the changes commence and, perhaps, the severity of those changes.

To evaluate the effect of the composite fabrication processes on the fiber properties in all three composite systems, the matrix material was dissolved away in 37 percent hydrochloric acid solution. The ultimate tensile strengths of these extracted fibers are shown also in Fig. 2. Limited exposure to concentrated hydrochloric acid solution had no adverse effect on fiber strength; this was verified by testing virgin fibers after immersions for up to 200 h. The most striking feature of these results is the wide range of strength fluctuations exhibited by fibers extracted from the steel/tungsten composites. Broad scatter in strength values is characteristic of brittle fibers that are sensitive to surface damage or irregularities. On the other hand, fibers extracted from the aluminum/tungsten systems had well-defined strength values and retained their ductility. In the case of steel/tungsten composites, the fiber strength degradation was probably aggravated by severe interfacial reactions and diffusion of

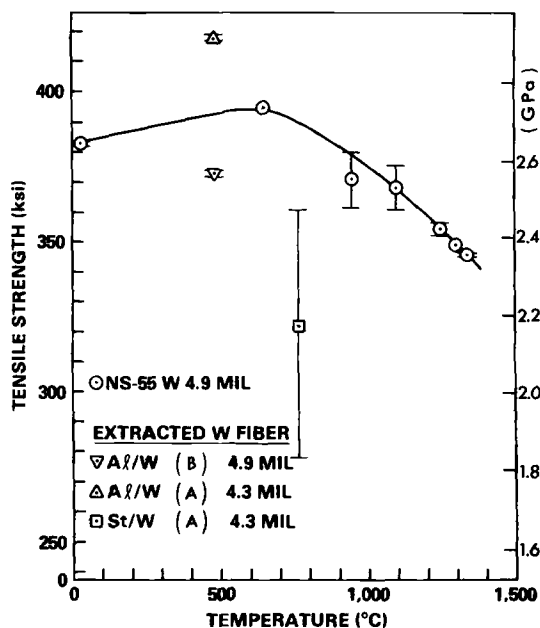


FIG. 2—Influence of isothermal annealing treatments and composite fabrication processes on the room-temperature tensile strength of NS-55 tungsten fibers.

contaminants, and a quantitative microanalysis of the constituent materials was performed.

Fractographic and Metallographic Observations

Scanning electron microscope (SEM) fractography was used extensively in this study to assess qualitatively the ductility of constituent materials in the composites as well as the quality of the interfacial bonds. A tensile fracture surface in an Al/45W(A) composite is shown in Fig. 3. The fibers were generally well aligned and arranged in a hexagonal array. The fracture surface revealed clearly the interlaminar failure of the aluminum matrix. In optical metallography, features of this type are usually obliterated. Inadequate bonding between foils of matrix material might be caused by the thin surface oxide layers that did not dissolve into the matrix during the diffusion bonding process. Also prevalent were separations at the fiber-matrix interfaces, where there were no perceptible reaction layers. This conclusion was based on the observation that the exposed wire surfaces retained the original wire drawing marks, which were replicated on the matrix cavities. The separations of fiber-matrix and matrix-matrix interfaces were due to transverse tensile stresses caused by necking of fibers and matrix. Radial cracks propagating along the grain boundaries of the tungsten fibers were caused by the circumferential stresses

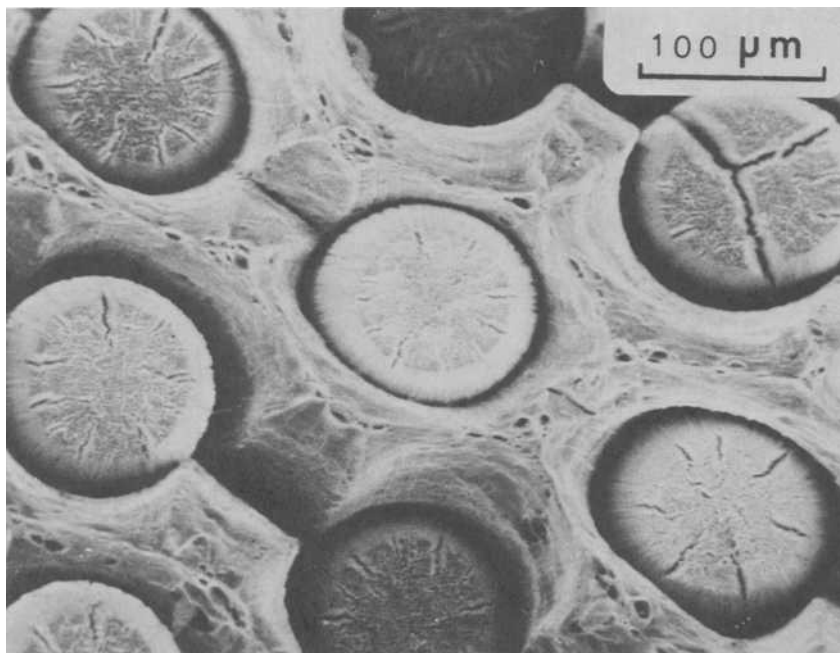


FIG. 3—An SEM fractograph of tensile failure in an aluminum/tungsten composite, top view.

developed from the macroscopic reduction of the cross-sectional area at necking. This reduction in the area of the tungsten wire was about 50 percent. Higher magnifications showed fibrous ductile fractures, which will be discussed later.

A typical fractograph of tensile failure in the St/45W(A) is shown in Fig. 4a. Where the tungsten fibers were diffusion bonded adequately to the steel matrix, the ductile matrix became deformed around the fiber. The two sides indicated by arrows are unbonded regions where there was insufficient pressure during consolidation of the composite. The macroscopic pressure direction in the diffusion-bonding process was tangential to the fiber circumferences at those points, as shown in the optical metallograph presented in Fig. 4b. Some of the tungsten fibers filled the voids during the specimen-polishing procedure. The entire tungsten fiber failed in a brittle transgranular fracture mode. However, there were numerous secondary fractures that propagated along the elongated grain boundaries present in the periphery of the fiber. These random secondary intergranular cracks were thought to be heterogeneously nucleated. Each fiber had at its periphery an annular feature that was typically 15 to 20 μm thick, and the river pattern in the core region indicated that the cracks originated somewhere inside that peripheral ring. In addition to showing this more clearly, Fig. 5 also demonstrates the result of inadvertent omis-

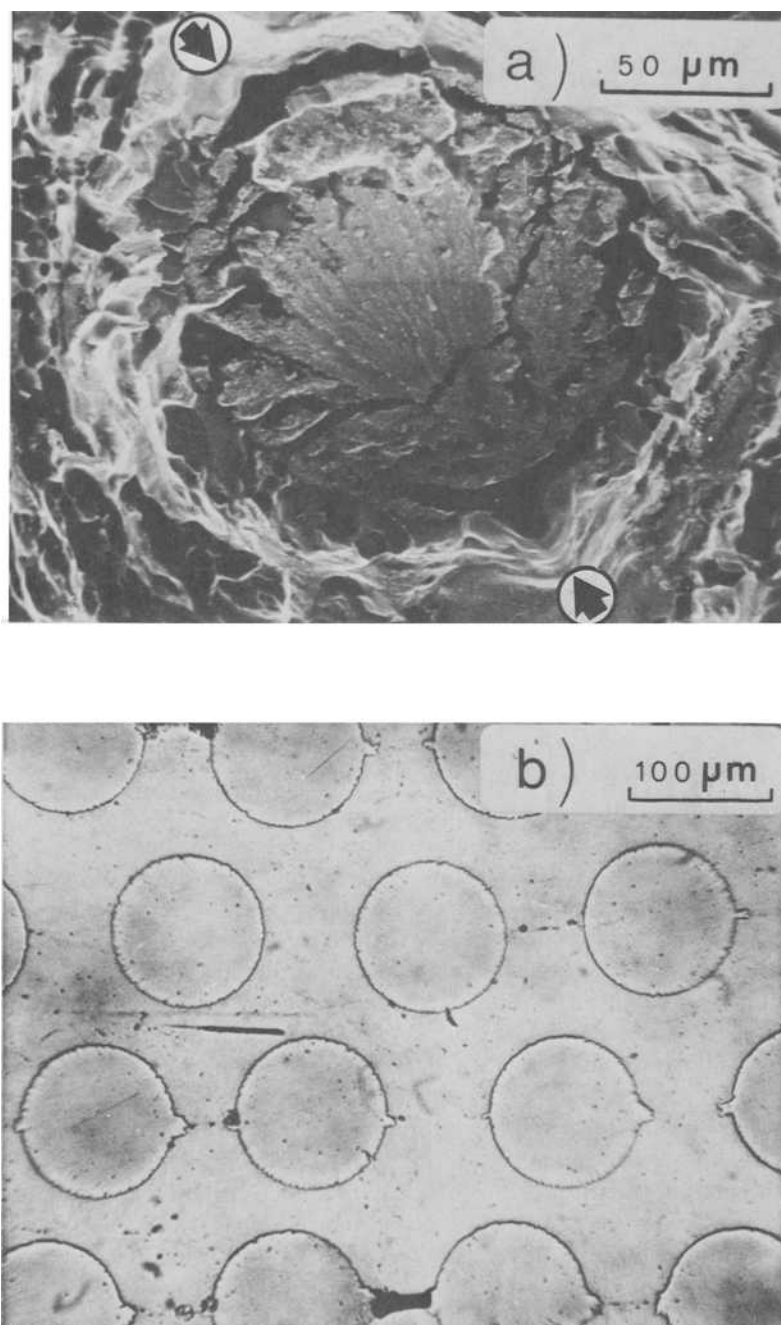


FIG. 4—Steel/tungsten composite: (a) SEM fractograph of tensile failure, top view; (b) optical metallograph.

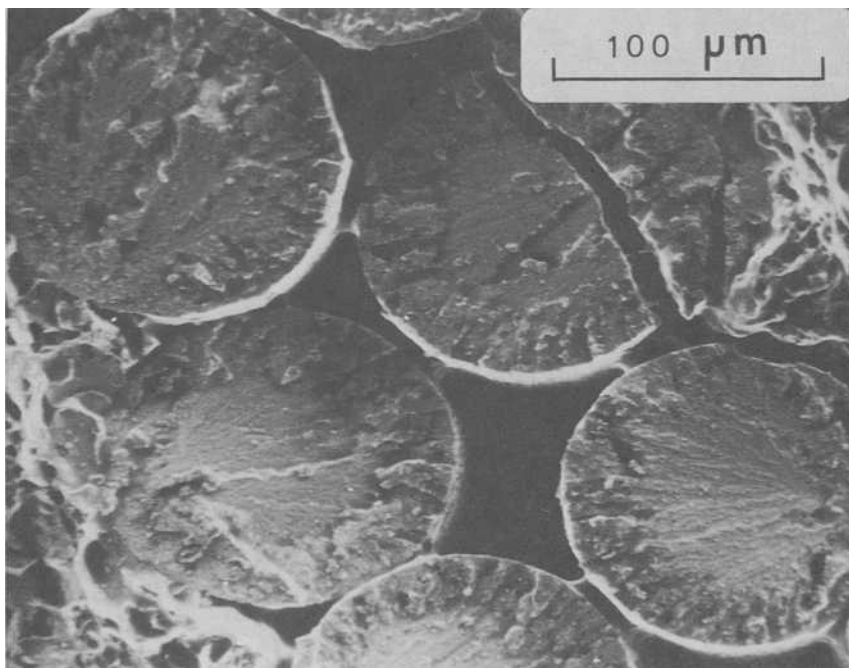


FIG. 5—An SEM fractograph of tensile failure in a steel/tungsten composite, top view.

sion of a matrix foil from between two fiber layers. Fiber fracture in that annular region at the fiber periphery proceeded quite distinctively from fracture in the core region. The major (that is, transverse) fractures in the fibers originated systematically at the circumference of the smooth interior core in regions of good fiber-matrix bonding. This observation was consistent with the expectation that matrix deformation in the tensile loading direction produced high fiber stresses by transmitting strains through a well-bonded interface. The fiber alignment and spacing were generally good, and no fiber pullout was observed. Also interesting was the presence of many longitudinal cracks in the fiber, as illustrated in Fig. 6, which shows a pair of mating fracture surfaces (one is an inverted image). The transverse fracture proceeded toward the upper left of the fiber, and the intergranular longitudinal cracks proceeded radially inward. Because the traces of the longitudinal cracks matched so well on the mating fracture surfaces, it was concluded that they nucleated independently and prior to the transverse fracture.

A striking change in the fracture mode came about when these composite materials failed under transverse tension loading. The failure was predominantly by fiber splitting, as is shown in Fig. 7. More than 80 percent of the fibers were split in the St/45W(A) composite. In the case of Al/45W(A), about 70 percent of the fibers split, and the remainder failed by separating from the

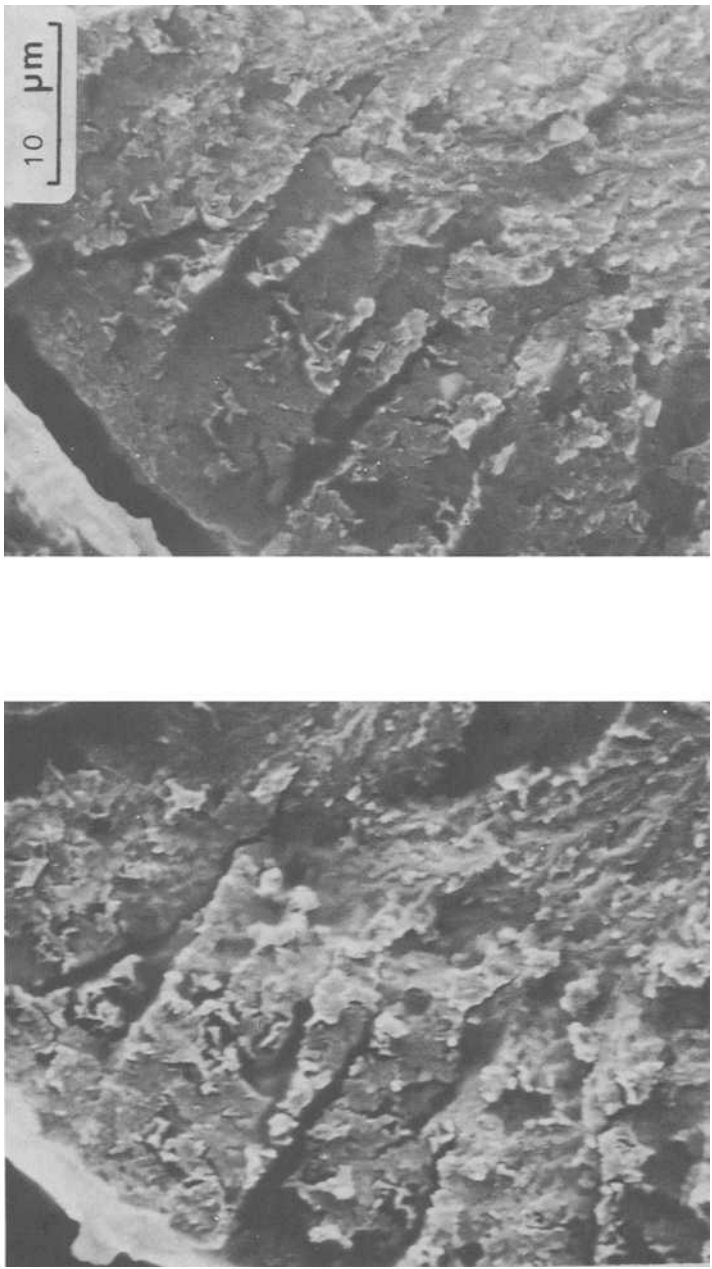


FIG. 6—An SEM fractograph of tensile failure of tungsten fiber in a steel/tungsten composite using an inverted image to compare mating fracture surfaces.

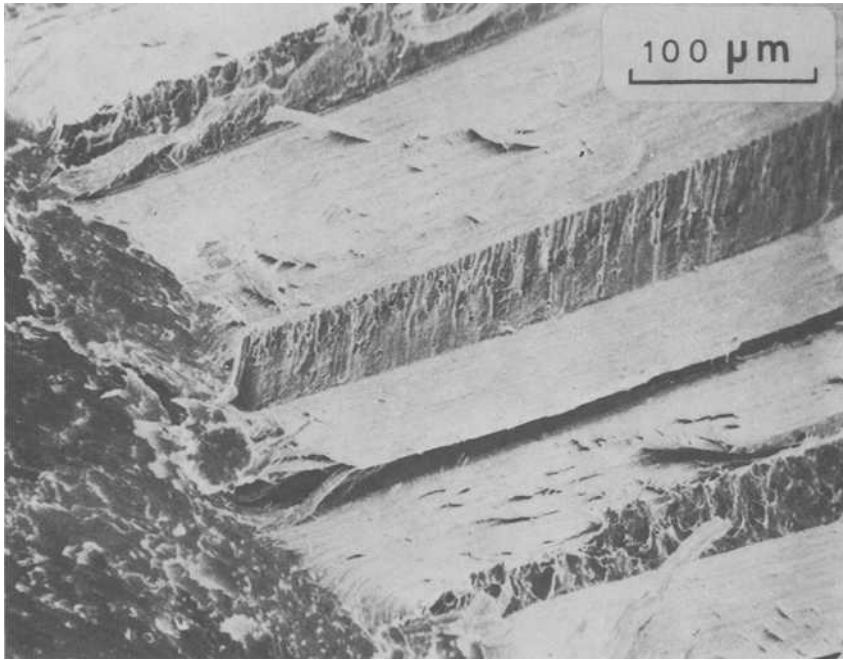


FIG. 7—An SEM fractograph of transverse tensile failure of a steel/tungsten composite.

fiber-matrix interface. The fiber failures in Al/45W(B) were equally divided between splits and interfacial separations.

The fracture morphology for NS-55 tungsten tensile specimens heat treated below the “brittle” transition temperature, presented in Fig. 8, shows a highly ductile fibrous type of fracture. Considerable necking had occurred. Figure 9 shows a specimen annealed above that transition temperature, and the fracture was a flat, transgranular cleavage, and quite brittle (that is, no necking had occurred). Optical metallographs of these annealed tungsten fibers are presented in Fig. 10; these show clearly the microstructural changes that commence in the temperature range of 800 to 950°C.

Auger Spectroscopy

The preponderance of split fibers observed in transverse tensile fracture of both steel/tungsten and aluminum/tungsten composites suggested that the tungsten fibers were intrinsically weak in the transverse direction, possibly because of chemical contaminants. Auger spectra from the split tungsten-fiber surface of the steel/tungsten system gave three principal iron peaks and a combined form of aluminum peak in the vicinity of 1400 eV; the results are presented in Fig. 11. In the aluminum/tungsten system there were no iron peaks, but there were overlapping peaks from metallic aluminum and alumi-

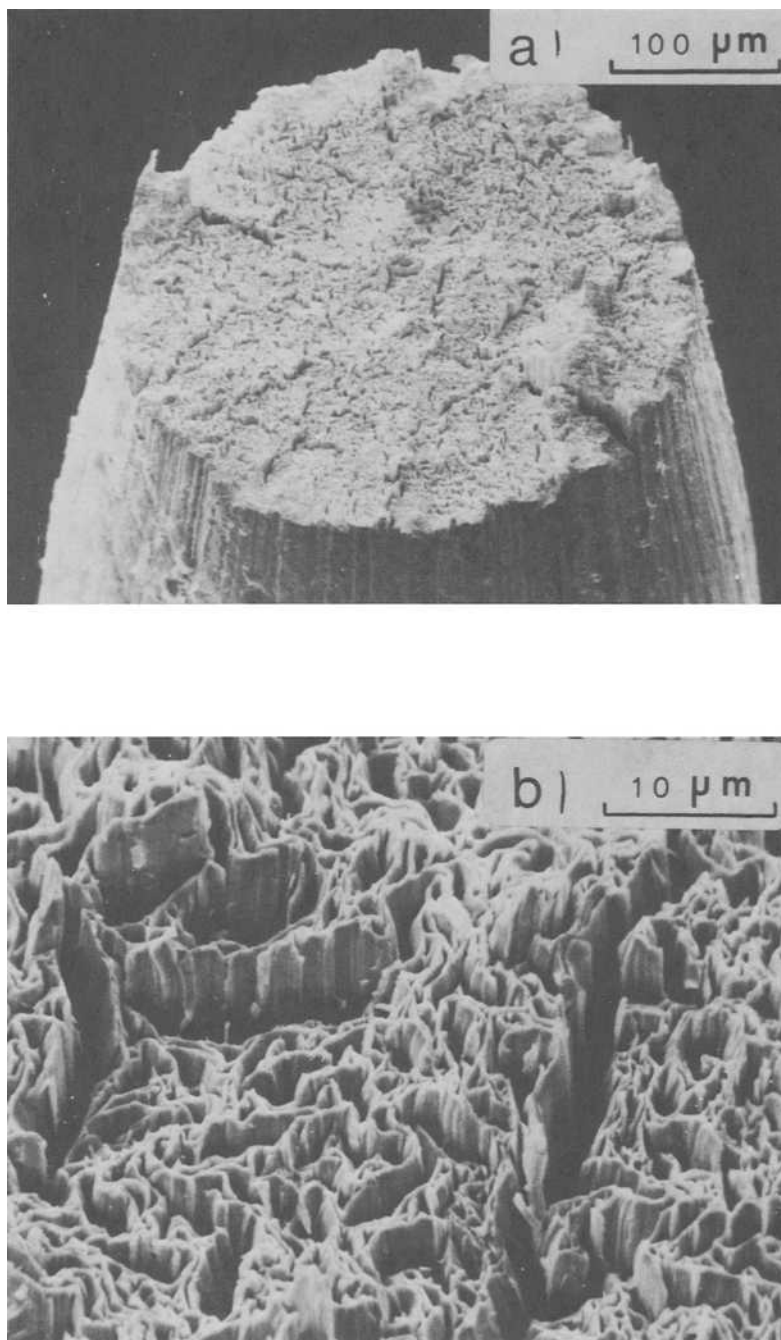


FIG. 8—Scanning electron microscope fractographs of tensile failure in NS-55 tungsten fiber after annealing for 33 min at 801°C.

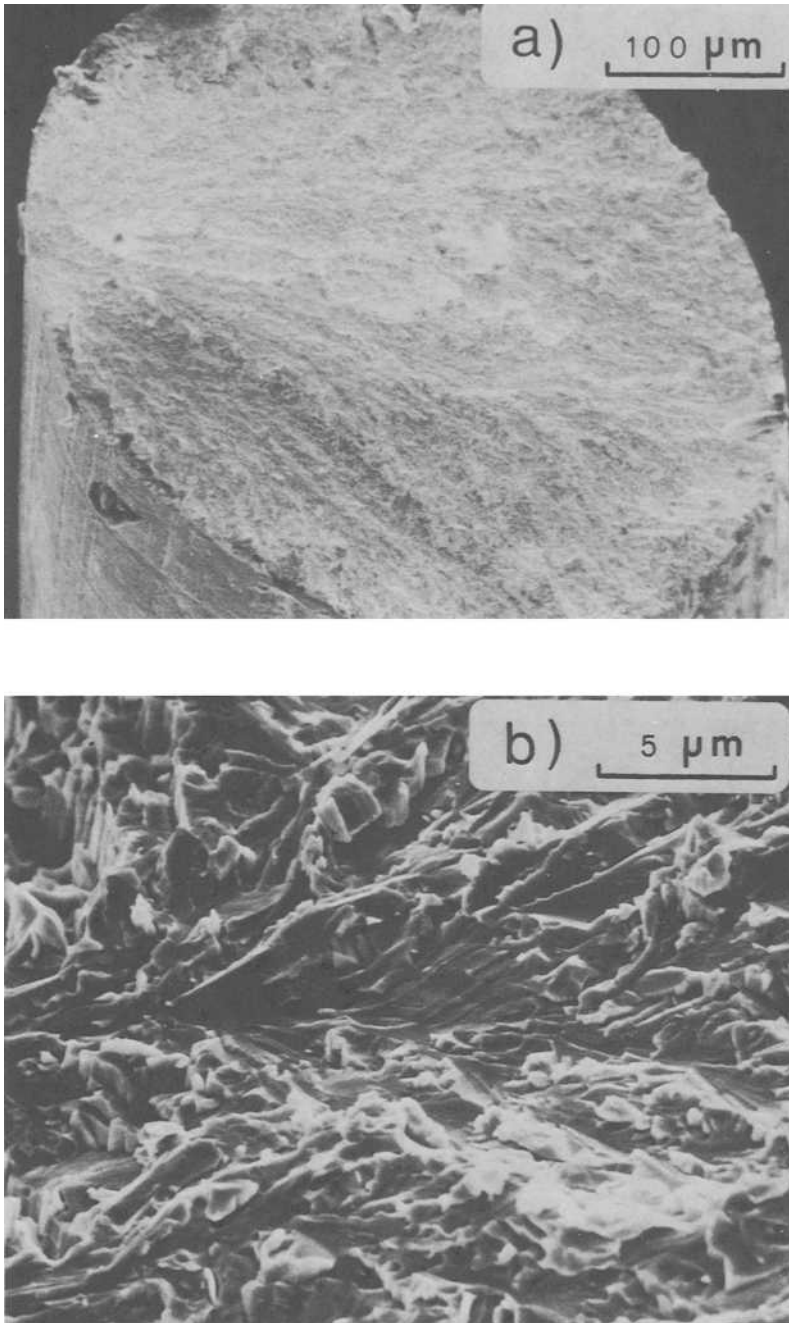


FIG. 9—Scanning electron microscope fractographs of tensile failure in NS-55 tungsten fiber annealed for 31 min at 951°C.

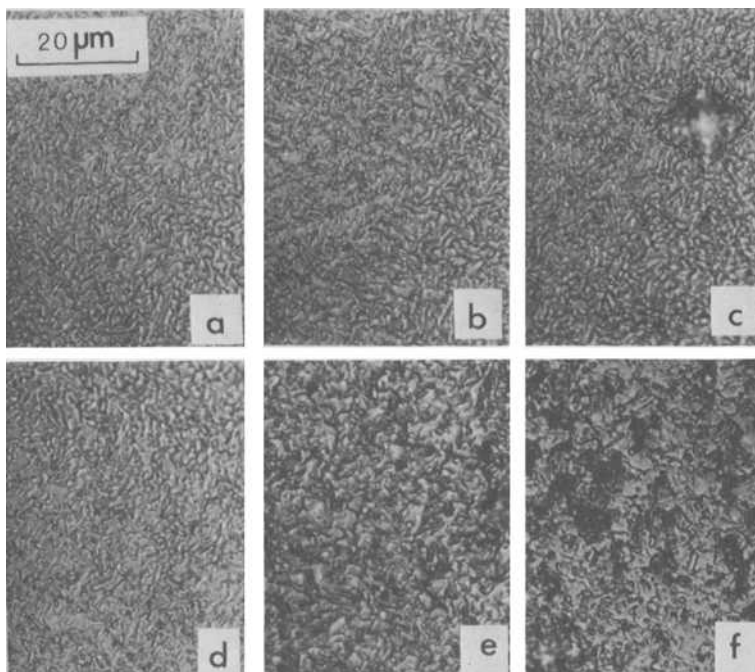


FIG. 10—Microstructures of NS-55 tungsten fibers on planes perpendicular to fiber axes after annealing for 30 min at the following temperatures: (a) as-received, (b) 492°C, (c) 646°C, (d) 801°C, (e) 951°C, and (f) 1104°C.

num oxide. The strong carbon peak was caused by extraneous hydrocarbons, which were broken up by the electron beam and deposited on the specimen surface.

The peak-to-peak amplitude of the Auger electron spectrum of iron was measured by monitoring the iron signal at constant sensitivity and changing only the position of the beam on the specimen. Since the peak-to-peak amplitude of a given spectrum is directly related to the surface concentration of that element, a relative concentration profile could be estimated, as shown in Fig. 12a. Note that the iron diffusion front penetrated about 20 μm into the fiber, corresponding to the annular feature in the fractographs.

Electron Microprobe Analysis

Auger spectroscopy and electron microprobe analysis differ significantly in the volume of material being analyzed. The former utilizes a beam size of about 5 μm and an electron escape depth of about 10 $\text{\AA}$ and is ideally suited for analysis of surface constituents, whereas the latter has an effective beam diameter of about 2 μm and a penetration depth of at least 1 to 2 μm and is suited to analysis of bulk constituents. Because the electron microprobe ana-

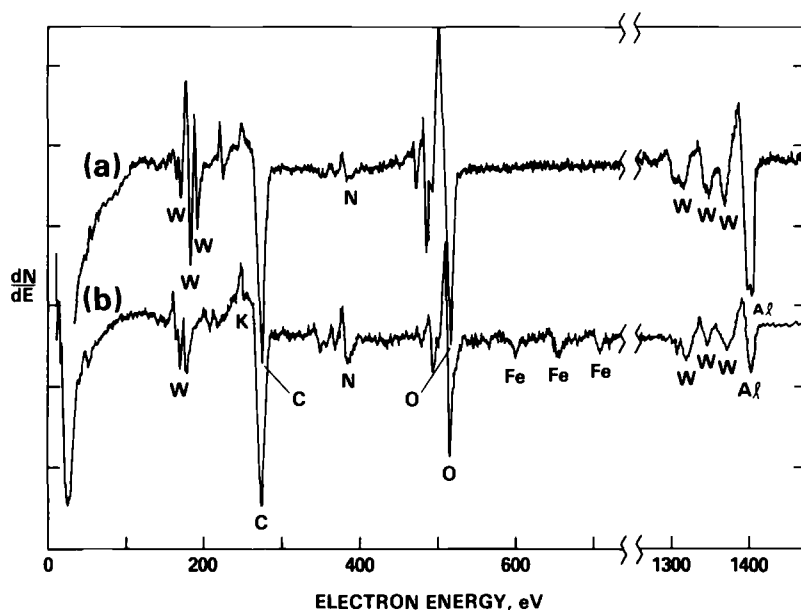


FIG. 11—Auger electron energy spectra of the fiber-matrix interface region of transverse fractures in (a) aluminum/tungsten and (b) steel/tungsten composites.

lyzes a volume of material several orders of magnitude greater than does Auger spectroscopy, it was undertaken both as a check on the latter and to see if volume contaminants were present. Typical results are presented in Fig. 12*b*. The iron diffusion front was again about 20 μm into the fiber, although the concentration gradient was significantly different, according to this analysis, and will be discussed later.

Discussion

Thermomechanical Effects

Ambient temperature tensile tests of highly strained tungsten fibers showed that annealing at temperatures in the 800 to 950°C range and higher caused these normally ductile fibers to fracture in a completely brittle manner that corresponded to the distinct microstructural changes shown in Fig. 10. Figure 10*a* through *d* showed essentially similar structures. The fibrous microstructure of curved grains was attributed to the nonuniform mode of deformation of the grains associated with [011] textured body-centered cubic metal in drawing operations [8]. With the tensile axis parallel to [011], two of the four $\langle 111 \rangle$ slip directions were oriented favorably to accommodate extension parallel to [011] and contraction parallel to [100]. For axially symmetric flow, the other two slip directions operated to decrease the [100] diameter while increasing the [011] diameter [7,9]. Thus, the ribbon-shaped grains observed in

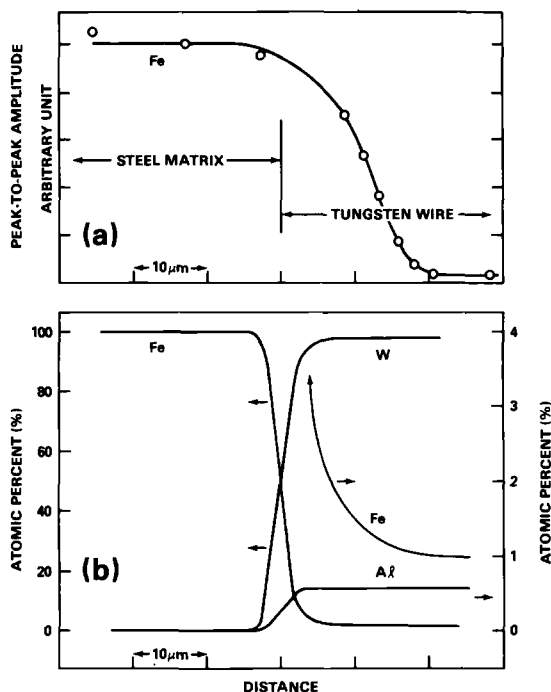


FIG. 12—Concentration profiles near the fiber-matrix interface in steel/tungsten composites: (a) auger spectroscopy at 5 keV Excitation energy; (b) electron microprobe analysis.

this photomicrograph and in the associated fractograph (Fig. 8) exemplified the fibrous nature of this system in its extremely ductile failure mode. The curly ribbon shape was attributed to the axially symmetric deformation in the drawing processes, since sheet drawing operations do not display a similar feature. Such twisted grains are divided by subgrain boundaries that are typically 1 μm wide and 2 or 3 μm long, as may be seen from comparison of transmission electron diffraction patterns of different areas that cover a few boundaries [7]. Annealing such strained fibers at 950°C changed the structure to that shown in Fig. 10e.

Careful examination of this structure revealed that some of the subgrain boundaries were migrating, and, subsequently, neighboring subgrains were merged. It was extremely difficult to differentiate the grain boundaries from the boundaries of the substructure; thus, the observed grain width included both. In a given elongated fibrous grain, the structure of adjacent cells had closer crystallographic orientation than did the cells belonging to other grains, and subgrain coarsening apparently was operative within each grain.

The average width of fibrous grains was reported to increase even at 700°C, ranging from 0.1 to 5 μm [9]. This temperature marks the point at which the ultimate strength began to decrease from its maximum value in the present

study. However, optical metallographs of longitudinal cross sections of tungsten fibers did not show significant increases in width as the annealing temperature was increased up to 1000°C. This seeming contradiction may be resolved by observing that additional longitudinal subgrain boundaries emerge parallel to the fiber boundaries due to the polygonization process. Moreover, such polygonization increases the brittleness by allowing greater effective stress concentration at the subgrain boundaries. For example, the fracture shown in Fig. 9 propagated mostly by cleaving the grains, because the low fracture stress in the polygonized configuration precluded activation of any favorable arrangements of active slip systems. Polygonization can be considered the preliminary stage of recrystallization, and the embrittlement of tungsten that accompanies recrystallization at these temperatures can be explained on the basis of theories of brittle fracture [10, 11].

Others [9] have reported that no dislocation networks were observed in specimens annealed at the relatively low temperature of 800°C, because the fine subgrains permitted only small dislocation pileups. However, annealing at 900°C increased the dislocation mobility as well as the subgrain size to allow larger dislocation pileups to develop. The consequent decrease in effective surface energy would promote brittleness [6]. It was also known that the yield strength of tungsten depended upon the subgrain diameter in a manner well described by the Petch equation [12], a fact that supports subgrain coarsening in view of the declining yield strength in the fabrication temperature range reported here.

Fiber Contamination Effects

The fracture of annealed tungsten fibers has been discussed so far without any consideration of possible effects due to contamination of the fibers by other chemical species during composite fabrication. Because iron was already known to embrittle tungsten [13, 14] the issue was particularly important for steel/tungsten composites. The brittle fracture of tungsten fibers in the steel/tungsten composite materials studied here was due principally to such iron contamination because the fabrication temperatures were generally below those shown to be responsible for the transition to completely brittle behavior in the fiber material.

The iron concentration profiles determined by both Auger spectroscopy and electron microprobe analysis showed that the iron diffusion front penetrated the tungsten fiber to a depth of about 20 μm . However, there was a distinct, and significant, difference in the concentration profiles that could be accounted for in terms of the ratio of the grain and subgrain boundary area to the bulk tungsten volume that was analyzed by each method. Because the Auger analysis was on the surface of an intergranular fracture and the depth of penetration was so shallow, that ratio is approximately 40 times greater than that resulting from electron microprobe analysis. This ratio was approxi-

mately the factor by which the iron concentration measured by Auger spectroscopy exceeded that measured by electron microprobe analysis in the region of the profile marked by a significant concentration gradient. In fact, this difference demonstrated conclusively that the iron contamination proceeded by grain boundary diffusion rather than by bulk diffusion.

The fractographs of tungsten fibers in the steel/tungsten system presented a consistent fracture topography showing an annular fracture feature of 15 to 20- μm thickness that corresponded exactly to the excessive iron contamination. In the optical metallography studies, the etchant [3 percent sodium hydroxide (NaOH), 10 percent potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$) and 87 percent water (H_2O)] did not reveal any diffusion reaction layer, and the diffusion distance obtained experimentally in this study was substantially greater than might be expected on the basis of existing data [15, 16]. It was concluded that a short-circuit diffusion path along the dislocation and grain boundaries was responsible for the rapid rate of contamination. Throughout the interior of wire, there was a slight iron contamination of approximately 1 atomic percent that would contribute to the brittle fracture behavior of the tungsten fiber core region in terms of increased notch sensitivity, particularly with the contaminated periphery of the fiber providing the notches in abundance [17]. The solid solubility of iron in tungsten is approximately 1 weight percent. Because the bulk diffusion of iron in tungsten was considerably less than the grain boundary diffusion, most of the iron resided in the grain and subgrain boundaries where, together with the tungsten dopants such as aluminum oxide, it served to reduce the cohesive strength of the fibrous grains in the drawn tungsten.

In some systems, a brittle intermetallic phase formed at the reaction layers in the composite, thereby producing a similar notch effect. For example, a composite of Type 304 stainless steel reinforced with fibers of W-2ThO₂ alloy formed a reaction layer at elevated temperatures that was isomorphous to the iron-tungsten compound (Fe_3W_2), and the fiber strength was significantly reduced by an embrittlement effect [18]. However, the harmful element (iron, in this case) could be used effectively to stabilize the fibrous structure of the drawn tungsten by adding certain alloying elements, such as chromium and zirconium, to the matrix [19]. Multicomponent iron-based alloys formed by powder metallurgical techniques did not produce even solid-solution reaction layers when used as the matrix for thoriated tungsten fibers [19].

Conclusions

Several important observations have been made concerning thermomechanical and chemical behavior of tungsten fibers which, in turn, suggest directions that effective development efforts might take to utilize such materials in metal matrix composites. Specifically, it was found that isothermal annealing of tungsten fibers in the temperature range of 800 to 950°C or higher for

relatively short times induced a microstructural transition to a brittle condition. The microstructural changes were due to strain-induced grain boundary migration and polygonization.

In addition, comparisons of experimental strength data to theoretical rule-of-mixture values showed the aluminum/tungsten composites to be in excellent agreement. Subsequent microscopic examinations indicated no perceptible fiber-matrix reaction layer, but they did indicate that the bonding and strengthening was due principally to mechanical interlocking of the constituents. The steel/tungsten composites, on the other hand, possessed good compressive strengths but only half the theoretically expected tensile strengths. This was indicative of notch sensitivity and was eventually found to be due to fiber embrittlement and strength degradation caused by excessive contamination with elemental iron and, to a lesser degree, by substructure coarsening.

Finally, microanalytical studies of interface chemistry disclosed that the iron contamination of the fibers in steel/tungsten composites was associated with grain boundary rather than bulk diffusion processes. The excessive concentration of elemental iron in the boundaries caused intergranular delamination and splitting of the fibers in planes parallel to the tensile axis during longitudinal strength tests. These secondary fractures grew radially into the fibers from the outer surface and substantially weakened them before primary failure of the fibers occurred by means of transgranular cleavage fracture in a plane normal to the tensile axis.

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Techniques

Quantitative Fractography of a Fatigued Ti-28V Alloy

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ABSTRACT: A quantitative fractographic analysis was undertaken in order to relate important microstructural and fractographic features to fatigue fracture in a Ti-28V alloy. Special emphasis is placed on facet characteristics and subsurface cracking in the single-facet and multifacet regions.

The stereological treatment of this problem develops methodologies and relationships based primarily on vertical sections cut through the fracture surface and parallel to the crack propagation direction. The evaluation of fracture profiles (in terms of true length, roughness, and degree of orientation) permits calculation of fracture surface characteristics (such as true surface area, roughness, and type of surface).

True mean facet areas, $\bar{A}_{\text{facets}}$, are calculated from combined measurements on scanning electron microscope photographs and two parallel, vertical sections along the edge and center of the fracture surface. The values of $\bar{A}_{\text{facets}}$ for the single-facet and multifacet regions are 22.2 ± 4.9 and $12.7 \pm 4.0 \text{ nm}^2$, respectively, with indicated 95 percent confidence intervals. True mean subsurface crack lengths, $\bar{L}_{\text{cracks}}$, in the edge section are 0.05 ± 0.07 and $0.16 \pm 0.07 \text{ mm}$ for the single-facet and multifacet regions, respectively, whereas in the center section the corresponding values are 0.13 ± 0.08 and $0.34 \pm 0.13 \text{ mm}$. Based on an upper-lower bound approach, the fracture surface is found to conform most closely to a partially oriented surface with strong planar elements. The results obtained here are consistent with the fatigue crack growth model of Chakraborty.

KEY WORDS: fractography, quantitative fractography, fatigue crack growth, subsurface cracks, true facet area, fracture roughness, degree of orientation, lineal roughness, materials, materials science

Quantitative fractography refers to the quantitative evaluation of the fracture surface and features within it in terms of true fracture surface areas, area fractions, sizes, spacings, and other quantities of interest. Central to this undertaking is the ability to determine the true fracture surface area. Methods exist whereby this can be done; but, if possible, we would like to avoid indirect or destructive experimental procedures that are prohibitively laborious or time-

¹Professor of metallurgy and senior research scientist, respectively, Fracture and Fatigue Research Laboratory, Georgia Institute of Technology, Atlanta, Ga. 30332.

consuming. Unfortunately, the standard equations of stereology [*1*]² are not directly applicable in this case because we are dealing with nonplanar surfaces.

A practical objective for such a quantitative treatment would be to base as much of the experimental and analytical procedures as possible on the direct scanning electron microscope (SEM) photographs of the fracture surface. These procedures would invoke the basic principles of stereology and projected images [2]. However, with the usual fairly flat fracture surface, the critical requirement of randomness in the orientation of surface elements is not completely satisfied with one angle of viewing. The problem of randomness can be solved, in principle, by increasing the number of viewing angles. However, methods have been worked out to deal with "partially oriented" surfaces [3], and the typical fracture surface appears to be another case of such classifications.

A less direct approach to the quantification of features in the fracture surface is to use two-dimensional sections cut through the fracture surface. Vertical sections appear to possess several advantages. They are easily prepared, and measurements on the planar sections are straightforward. Moreover, a considerable portion of the fracture surface and the underlying alloy is available for statistical sampling and measurements. Most important, the linear trace provides an essential quantitative link between accessible two-dimensional measurements and the desired three-dimensional quantities. And, finally, with little additional preparation, a direct visual comparison can be made between the fracture profile and the fracture surface.

Naturally, there has been considerable activity in the area of quantitative fractography. Recent reviews and publications by Chermant et al [4-6] and El-Soudani [7,8] have laid much of the groundwork for quantitative treatments. Some attempts have been made, especially by Chermant and Coster [4], to systematize the available relationships for quantifying fracture surfaces. This is an extremely difficult task because so many types of relationships are required. For example, to list a few, we need precise relationships that are valid

- (a) in the fracture surface only,
- (b) between the fracture surface and its projection plane, or
- (c) between the fracture surface and a parallel plane of polish.

If vertical sections are employed, then we need to know

- (d) the equations relating the fracture surface and its profile, and
- (e) those equations that connect the fracture profile to its projection line.

In addition we require

- (f) the basic relationships of projections, as well as

²The italic numbers in brackets refer to the list of references appended to this paper.

(g) the appropriate stereological equations between planar sections and projections.

A general solution to this difficult problem does not appear to exist. Thus, indirect and rather laborious experimental techniques have been employed in this investigation. Some useful relationships and procedures are developed for the quantitative evaluation of microstructural and fractographic parameters important in Chakrabortty's fatigue crack growth model [9, 10]. Specifically, these include true fracture surface areas, true facet sizes, and subsurface crack characteristics, as well as degree of orientation and roughness parameters.

Experimental Procedures and Results

A Ti-28V alloy fatigue specimen was selected for fractographic analysis. Two vertical and parallel longitudinal sections were cut, one near the specimen edge and, the other close to the center of the specimen. Figure 1 depicts the positions of the vertical sections and fracture profiles, as well as the locations of the single-facet and multifacet fracture regions [9, 10]. Typical oblique SEM views of the fracture surface and vertical sections are shown according to their location on the fracture surface. Note the large facets in the single-facet regions in comparison with those in the multifacet region.

The fracture surface was first electroplated with a layer of copper; then the specimen was mounted and prepared metallographically to show the fracture trace and underlying microstructure near the edge of the specimen. Figure 2 shows examples of the fracture trace from the single-facet and multifacet regions. A sequence of photomicrographs was taken along the fracture trace and then assembled into one continuous strip for analysis. The photostrip was marked off into 40 equal lengths, or compartments, for local sampling. This procedure was repeated for a vertical section near the center of the specimen.

Trace Lengths

The trace lengths were measured in two ways. The first method used a plastic sheet marked with a grid of parallel test lines, which gave P_L as a function of angle within each compartment at 12 different positions, each 15° apart. The trace length was then calculated according to [11]

$$L_t = (\pi/2) \overline{P_L(\theta)} A_T \quad (1)$$

where $\overline{P_L(\theta)}$ is the number of intersections of the trace with the test grid, per unit length of grid, averaged over all 12 angular positions. A_T is an arbitrary test area that encloses the fracture trace within the compartment.

The trace lengths were also measured using the semiautomatic Zeiss MOP-3 operator-interactive image analysis system [12]. Here, the values of L_t are obtained directly and rapidly simply by tracing the fracture profiles with the electronic pencil.

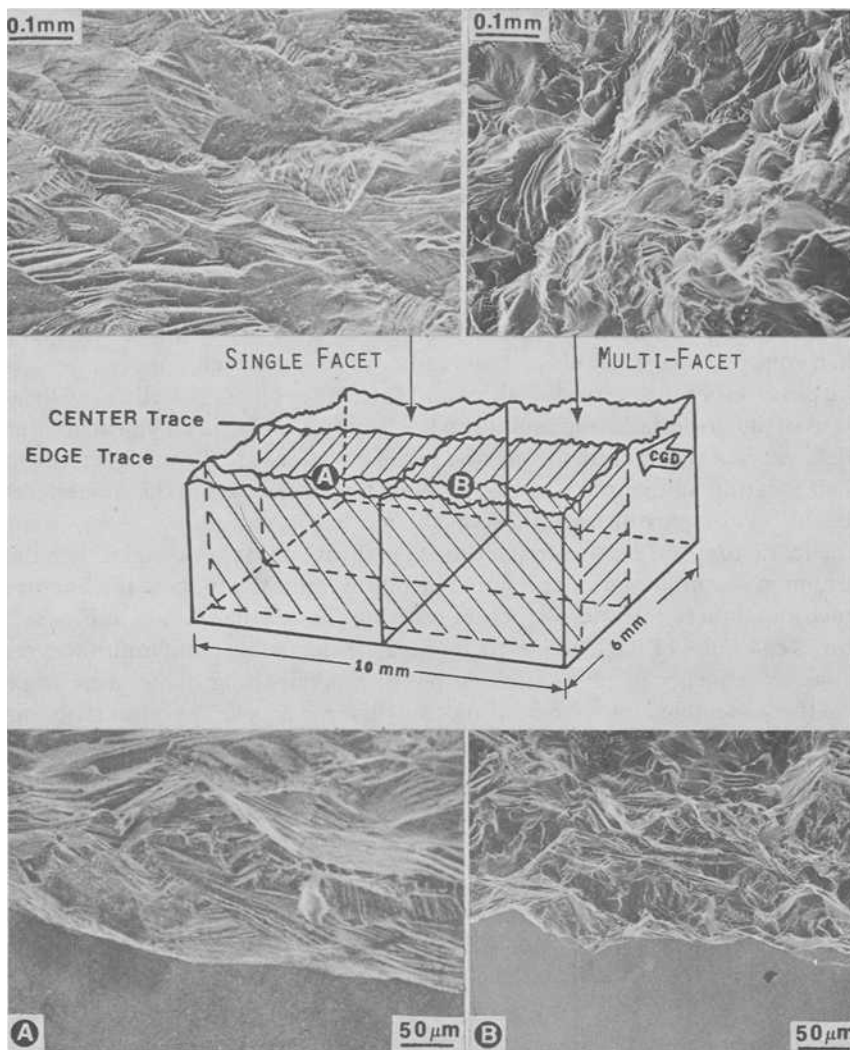


FIG. 1—Overview of specimen geometry showing the fracture surface, edge and center traces, and single-facet and multifacet regions, including typical views of fracture surfaces and the edge trace by oblique SEM.

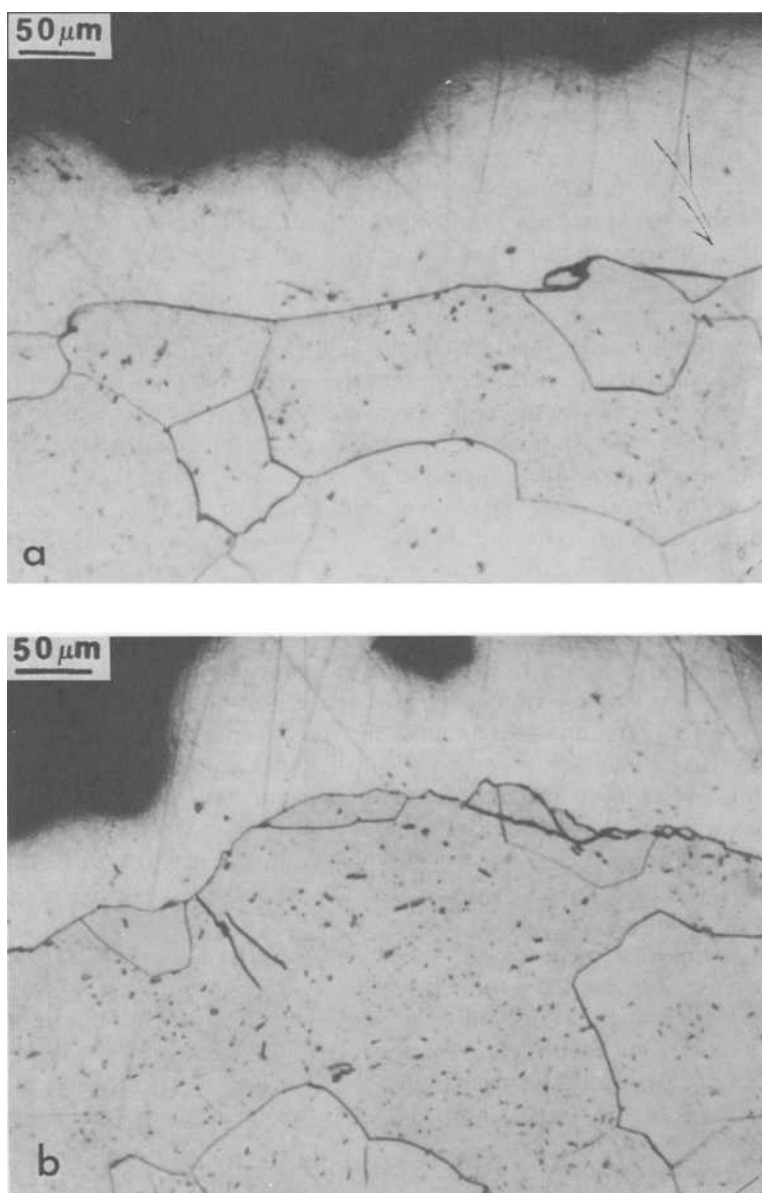


FIG. 2—Examples of fracture profile from the edge vertical section: (a) single-facet region, (b) multifacet region. Note the copper coating above the fracture traces, and subsurface cracks in (b).

The primary quantity calculated from the measurements of L_i is the ratio L_i/L' , which Gurland [13] called the lineal roughness parameter, R_L . (L_i) is the true length of the fracture trace in compartment i , whereas L' is the projected width [equivalent here to the width of the compartment measured along the mean crack propagation direction (CPD)]. Thus, the "rougher" the fracture trace, the larger the value of R_L .

The results of these measurements are presented in Table 1 and Fig. 3 for the edge and center sections as well as for the single-facet and multifacet regions. The average values of R_L for the two traces are 1.06 in the single-facet regions and 1.15 in the multifacet regions, which amount to about an 8 percent difference. It is noteworthy that the averages of R_L agree as closely as they do (± 0.001) between the edge and center traces, because manual methods were employed for the edge trace measurements, and the semiautomatic MOP-3 was used for the center trace. Based only on these trace length measurements, it would seem that edge effects are absent, at least for this alloy, fatigued under these conditions, and for this type of specimen geometry. However, as we shall see, other parameters indicate real differences in the trace characteristics from section to section and from region to region. In any event, R_L is a valuable parameter in its own right and plays an important role in calculations of fracture surface areas and facet areas.

Rose of the Number of Intersections [3, 14]

The other experimental quantity obtained from the fracture trace is the rose of the number of intersections, called the "rose" for short. It is merely a polar plot of P_L as a function of the angle θ , measured from some selected orientation direction (here, the mean crack growth direction). For a completely "random" line (one in which the small linear segments assume all possible directions with equal probability), the rose plot would appear as a circle centered on the origin of coordinates. For a partially oriented line, the rose plot would deviate from circularity to an extent depending on the degree of orientation of the line.

Angular measurements of $P_L(\theta)$ were made as described earlier. However, since the average rose was desired for the four regions (single-facet and multifacet regions along the edge and center traces), all P_L values obtained at the same angle within each region were collected and averaged. The results in terms of $P_L(\theta)$ are plotted as solid lines in Fig. 4. Also given are the results of a least-squares analysis (dashed lines) based on the theoretically exact equation

$$P_L(\theta) = a + b \sin \theta \quad (2)$$

This expression applies to a partially oriented line in a plane and embodies the assumption that the small linear segments comprising the line are either completely random or fully oriented (parallel to the CPD). The constant a represents the radius of the $P_L(\theta)$ circle centered at the origin (which describes the

TABLE 1—Parameters for fracture profiles in fatigued Ti-28V alloy.

	Single-Facet Region, 3.75 mm long	Multifacet Region, 6.25 mm long
Mean lineal roughness parameter, $\bar{R}_L$		
edge trace ^a	1.060	1.147
center trace ^b	1.061	1.148
True profile length, $\sum_{i=1}^{40} (L_i)$		
edge trace	3.975	7.169
center trace	3.995	7.204
Degree of orientation, Ω_{12}		
experimental data		
edge trace	0.676	0.389
center trace	0.644	0.562
least-squares data		
edge trace	0.691	0.470
center trace	0.754	0.610

^a Manual measurements.^b Zeiss MOP-3 measurements.

completely random linear segments), whereas the $b \sin \theta_i$ term gives two $P_L(\theta)$ circles tangent to the origin (which characterize the fully oriented system of linear segments). The vectorial summation of the circular figures results in the complete roses, half of which are shown in Fig. 4.

Comparisons of the experimental and least-squares roses reveal a slight tendency to peak toward the 90° direction. This probably means that complete orientational randomness is not achieved in the subset of "random" linear

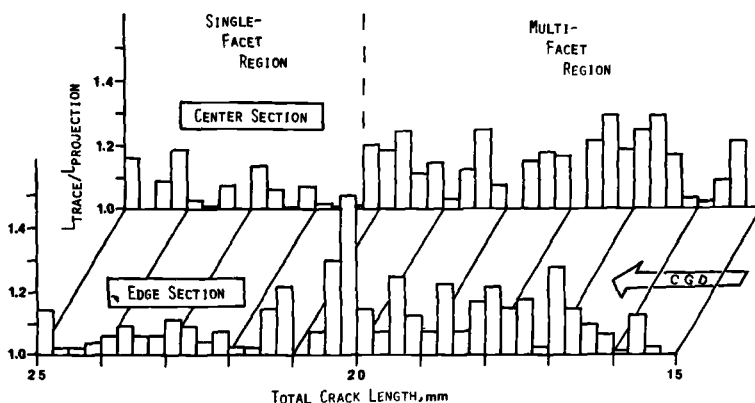


FIG. 3—Lineal roughness parameter, R_L , for fracture surface traces along the edge and center sections.

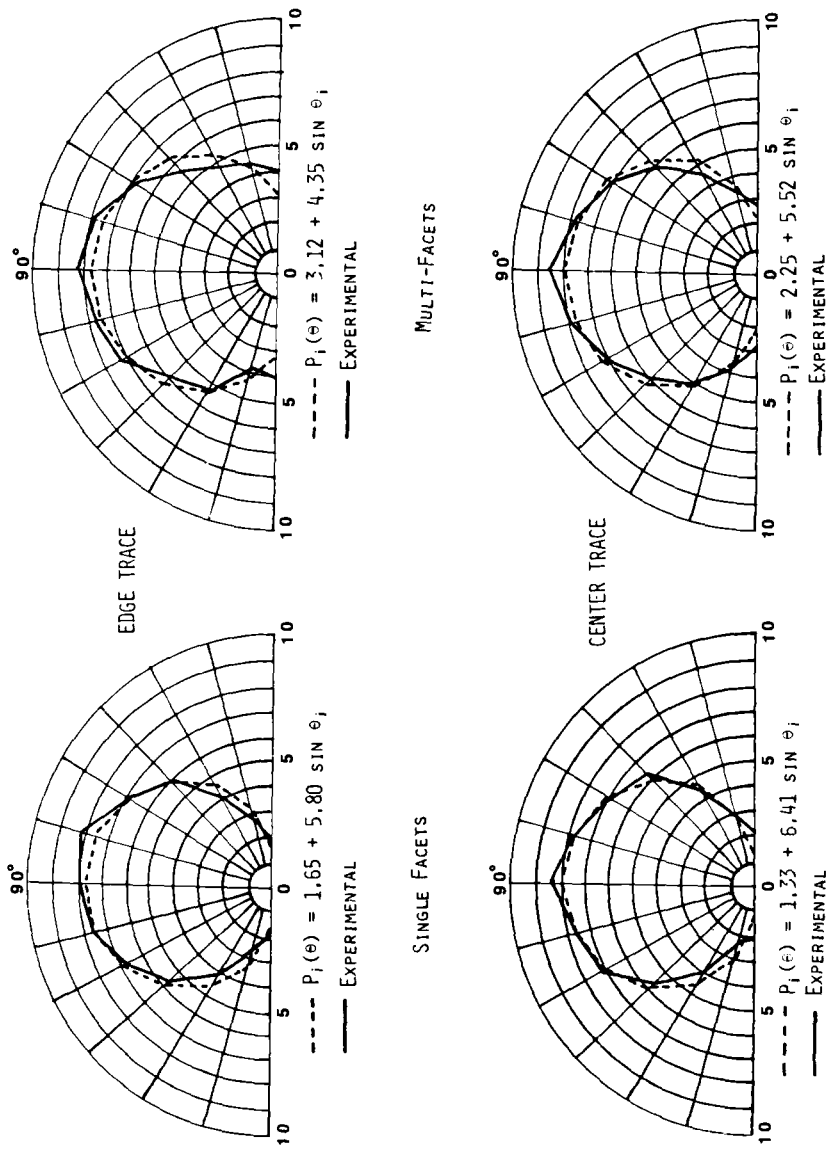


FIG. 4—Experimental and least-squares polar plots of the rose, $P_i(\theta)$, for edge and center traces.

segments; rather, in this fairly flat fracture trace there is an undue proportion of linear segments lying parallel or close to the reference line.

Degree of Orientation [3, 15]

A quantitative index for evaluating the degree of orientation of a line in a plane is Ω_{12} , which is defined by

$$\Omega_{12} = \frac{(P_L)_\perp - (P_L)_\parallel}{(P_L)_\perp + 0.571 (P_L)_\parallel} \quad (3)$$

Here we need only the P_L values perpendicular and parallel to the orientation direction. For completely oriented lines, $\Omega_{12} = 1$, whereas for completely random lines, $\Omega_{12} = 0$. The intermediate values of Ω_{12} indicate quantitatively the proportion of linear segments oriented parallel to the orientation direction.

The results of the Ω_{12} calculations are given in Table 1 for both the experimental data and the least-squares curves. It is clear that the degree of orientation is higher (by 25 to 28 percent) in the single-facet region than in the multifacet region. Moreover, the degree of orientation is generally higher (by 13 to 17 percent) in the center trace than in the edge trace. Also noteworthy is the fact that the spread between single-facet and multifacet data is large along the edge trace, whereas the comparable data along the center trace show a relatively small spread.

Thus, although the lineal roughness parameter, R_L , indicates negligible differences in average trace lengths between the edge and center traces, the degree of orientation, Ω_{12} , shows definite differences between these traces. Obviously one parameter is insufficient to represent completely the fracture trace characteristics. In fact, both the rose and the degree of orientation, as well as R_L , provide the essential information needed for determining the type and extent of fracture surfaces and their areas.

Fracture Surface Area

Ultimately, of course, we would like to relate measurements made on vertical SEM pictures of the fracture surface to true spatial quantities in the fracture surface. As a first step toward developing these equations, we use vertical longitudinal sections lying parallel to each other and the CPD.

We start the analysis with a definition of fracture surface roughness, S_A , expressed as

$$S_A = S_t/A_t^* \quad (4)$$

where S_t is the true fracture surface area (in three-dimensional space), and A_t^* is the projected (test) area (in a two-dimensional plane), as seen in the SEM picture. Obviously, for a constant projection area there can be a wide range of true fracture surface areas with $S_t \geq A_t^*$. As the true fracture surface area in-

creases (gets "rougher"), the parameter S_A will also increase. If reentrances or self-overlap occur, the general concept, the total projection, may be required [16].

We look next for a direct relationship between the fracture surface area and the trace length in a vertical section through the fracture surface. For our model, we take a test cube of volume, $V_T (= L_T^3)$, enclosing a portion of the fracture surface whose projected area, $A_T' (= L_T^2)$, is related to the test volume through $V_T = A_T' L_T$. The trace length in one side of the cube, L_A , is equal to L_t/L_T^2 .

Accordingly, we can write

$$\frac{S_t}{A_T'} = \frac{(S_v)_{\text{fracture}}}{(A_v)_{\text{projected}}} = \frac{(4/\pi) (L_A)_{\text{trace}}}{A_T'/V_T} = \frac{4}{\pi} \frac{L_t/L_T^2}{1/L_T} = \frac{4}{\pi} \frac{L_t}{L_T} \quad (5)$$

where $S_v = (4/\pi)L_A$ is one of the basic equations of stereology, whose validity for any type of surface depends only on adequate sampling and measurements. Since L_T in this case is also equal to L' , the edge length of the projected area, we have

$$S_A = \frac{4}{\pi} \frac{L_t}{L'} = \frac{4}{\pi} R_L \quad (6)$$

where R_L is the lineal roughness parameter defined earlier, and L_t is obtained from Eq 1. Also, the true surface area may be obtained from

$$S_t = \frac{4}{\pi} R_L A_T' \quad (7)$$

where A_T' is the arbitrary projection area with dimensions consistent with our choice of L' .

In order to ensure adequate randomness in the sampling of the trace, intersection counts of $P_L(\theta)$ were obtained as described previously at 12 angular positions, and then averaged for each compartment. Moreover, to provide some three-dimensional coverage, the results from the two vertical sections were also averaged. The values of $\bar{S}_A$ and S_t obtained in this way for the single-facet and multifacet regions are entered in Table 2. Future experiments are planned that will include additional sections at other angles and in other directions in order to improve randomness in the spatial sampling. By proper utilization of any symmetry elements, however, the number of sections required can be held to a minimum.

Upper-Lower Bounds

The results reported in Table 1 demonstrate conclusively that there is a significant degree of preferred orientation in this fracture surface. Accordingly, we can utilize the procedures worked out for "systems of partially oriented surfaces," described in the literature [17]. These classifications include par-

TABLE 2—Parameters for fracture surface in fatigued Ti-28V alloy.

	Single-Facet Region, 3.75 mm long	Multifacet Region, 6.25 mm long
Mean surface roughness parameter, $\bar{S}_A$	1.32	1.45
True fracture surface area, S_t	29.7 mm ²	54.4 mm ²
Facet sizes ^a		
mean projected facet area, $\bar{A}_{\text{facets}}$ (SEM)	16.42 ± 3.64	8.68 ± 2.78
mean true facet area, $\bar{A}_{\text{facets}}$	22.17 ± 4.94	12.68 ± 4.04

^a With 95 percent confidence intervals.

tially oriented systems of surfaces with linear or planar orientations, which may apply to our fracture surface. The reference or orientation plane is taken parallel to the average macroscopic crack plane.

One method for determining whether the fracture surface belongs to any particular category of surfaces is to use upper and lower bounds, based on assumed types of surfaces. Four types of surfaces are evaluated: minimum, random, planar, and linear. The pertinent equations for these four cases are as follows:

Case I—Minimum Surface Area. These are ruled surfaces, that is, surfaces that lie along the trace but are perpendicular to the vertical section. The trace can have any degree of complexity.

$$(S_v)_\perp = L_A = (\pi/2) \overline{P_L(\theta)} \quad (8)$$

where $(S_v)_\perp$ refers to the area of fracture surfaces perpendicular to the vertical sectioning plane per unit volume, L_A is the trace length per unit area in the vertical section, and $\overline{P_L(\theta)}$ is the number of intersections of the trace with the grid, per unit grid length, averaged over angular positions of the test grid. Note that we do not refer here to the case of a perfectly flat surface.

Case II—Random Fracture Surface. Here we hypothesize a surface with elements oriented randomly in all directions

$$S_v = \frac{4}{\pi} L_A = 2 \overline{P_L(\theta)} \quad (9)$$

which applies to *any* type of surface, as well as a completely random one, as long as the sampling is sufficient.

Case III—Partially Oriented Surface with Planar Orientation. This type of surface consists of both random and partially oriented surface elements. The latter are relatively flat and equiaxed (planar), and they lie mostly parallel to the mean fracture plane. The surface area per unit volume for just the planar surface elements is given by

$$(S_v)_{\text{pl}} = (P_L)_\perp + (P_L)_\parallel \quad (10)$$

where the subscripts $\perp$ and $\parallel$ refer to directions taken with respect to the mean CPD in the vertical section. Thus P_L is measured in only two directions.

Case IV—Partially Oriented Surface with Linear Orientation. This type of surface consists of both random and partially oriented surface elements. The latter are not equiaxed, but show some degree of elongation in the mean CPD. The surface area per unit volume for just the linear surface elements is

$$(S_V)_{\text{lin}} = \frac{\pi}{2} (P_L)_{\perp} + 0.429 (P_L)_{\parallel} \quad (11)$$

where $\perp$ and $\parallel$ refer to directions, as described earlier.

The calculations for the first two cases are straightforward and based on well-known equations of stereology. Cases III and IV apply to partially oriented surfaces with assumed planar or linear orientations. The equations given refer only to the oriented portions of the surface and are determined experimentally by $(P_L)_{\perp}$ and $(P_L)_{\parallel}$ measured directly or taken from the rose plots.

The results obtained for these four cases, based on the experimental data, are given in Fig. 5 in terms of S_A . The width of each band is determined by the edge-trace and center-trace values. Our "best" values of S_A , 1.32 for the single-facet and 1.45 for the multifacet regions, are well within the upper and lower bounds established by the linear and minimum bands, respectively. Moreover, these "best" values also lie close to their planar bands, and thus it appears reasonable to assume that the fracture surface investigated here belongs more to the planar category of partially oriented surfaces than to the other categories.

Facet Areas

One of the more important microstructural features in this investigation is the facets observed in the fracture surface. Since they can be identified in the SEM photostrip, the mean projected area, $\overline{A'}_{\text{facets}}$, may be determined by

$$\overline{A'}_{\text{facets}} = A'_A / N'_A \quad (12)$$

where $A'_A = P'_P$, and N'_A is the number of facets per unit area in the SEM photographs (the projection plane). A'_A can be obtained either manually, with a square point count grid marked on a plastic sheet or with the semiautomatic Zeiss MOP-3.

The values of $\overline{A'}_{\text{facets}}$ were obtained at selected locations and then corrected to true mean facet areas by a relationship developed for this case. Basically, we postulate that the projection ratio, S_A , for the entire fracture surface should equal the corresponding projection ratio for a selected feature in the fracture surface [namely, $(A'_A)_{\text{facets}}$]. Accordingly, we write

$$(S_A)_{\text{fracture}} = (A'_A)_{\text{facets}} \quad (13)$$

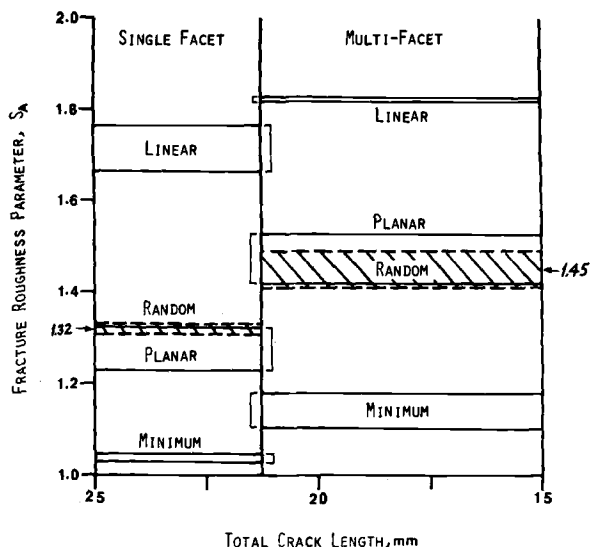


FIG. 5—Upper and lower bounds of fracture roughness parameter, S_A , in the single-facet and multifacet regions. The best values for S_A are 1.32 in the single-facet region and 1.45 in the multifacet region, indicated by arrows.

where $(A'_A)_{\text{facets}}$ is defined by

$$(A'_A)_{\text{facets}} = \bar{A}_{\text{facets}} / \bar{A}'_{\text{facets}} \quad (14)$$

which is simply a ratio of the mean true facet area to the mean projected area. From this follows

$$\bar{A}_{\text{facets}} = (S_A)_{\text{fracture}} \bar{A}'_{\text{facets}} \quad (15)$$

or finally

$$\bar{A}_{\text{facets}} = \frac{4}{\pi} R_L \bar{A}'_{\text{facets}} \quad (16)$$

which utilizes Eq 6. Mean values of true $\bar{A}_{\text{facets}}$ and projected $\bar{A}'_{\text{facets}}$ facet areas are given in Table 2.

The mean true facet areas measured at 14 positions along the SEM photostrip are plotted in Fig. 6 as a bar graph. Average values for the single-facet and multifacet regions are indicated; these values reveal that the mean facet size in the single-facet region is almost twice that in the multifacet region. The choice and delineation of facet areas in the SEM photostrip is a highly subjective operation, and therefore better experimental methods must be devised to ensure better reproducibility. It should be noted that some results obtained previously for the fracture surface also apply to the facet areas; namely, the fracture surface roughness, S_A , the degree of orientation, and the identification with planar orientation.

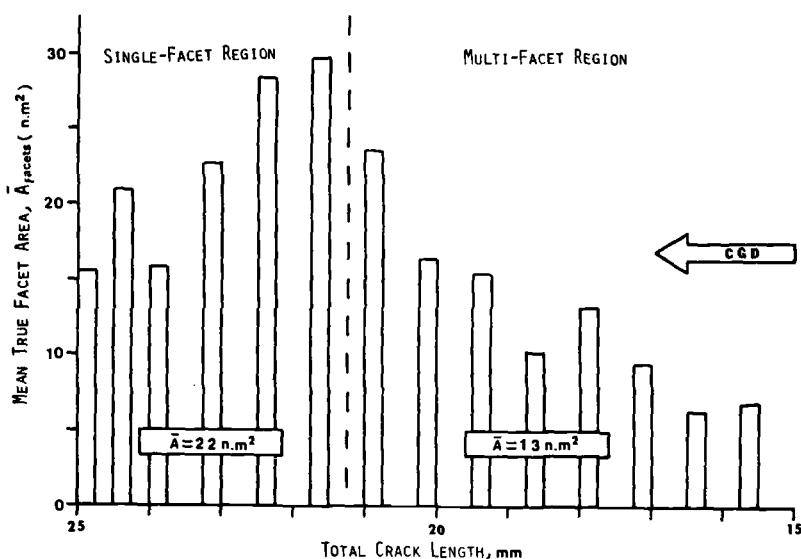


FIG. 6—Variation of the mean true facet areas, $\bar{A}_{facets}$, in single-facet and multifacet regions.

Subsurface Cracks

Because subsurface cracks were noticeably more prevalent in the multifacet region (see the example in Fig. 2), methods were investigated for their quantitative characterization. The crack traces are exposed in the vertical sections in much the same way as the fracture surface traces; therefore, procedures generated for the fracture surface are generally applicable to crack surfaces. A lineal crack roughness parameter for crack traces, similar to the lineal roughness parameter, R_L , for fracture surface traces, was deemed useful, as were the true total crack length and a crack surface roughness parameter similar to S_A for the fracture surface.

Crack lengths were determined in two ways: manually, with a grid of parallel lines on a plastic sheet, much as has been described for measurements of fracture surface trace lengths, and semiautomatically, simply by tracing the crack profile with the MOP-3 electronic pencil. The results of the calculations are summarized in Table 3. Note that $(R_L)_{cracks} \geq 0$ is possible, whereas $(R_L)_{trace} \geq 1$.

Several interesting observations can be made about the data in Table 3. The multifacet region has a mean crack roughness parameter about three times greater than that in the single-facet region. Also, the total crack length in the multifacet region is four to five times greater than that in the single-facet region. Comparisons between the two traces show that the mean value of the $(R_L)_{cracks}$ parameter for the center trace is about 2.5 times greater than that for the edge trace. Thus, subsurface cracking appears to be a very inhomogeneous process indeed, and one that is linked to the facet sizes and local mechanisms of fracture. If interest dictates, a more detailed study of crack branching, orientation, curvature, and so on is possible [11].

TABLE 3—Parameters for subsurface cracks in fatigued Ti-28V alloy.

	Single-Facet Region, 3.75 mm long	Multifacet Region, 6.25 mm long
Mean lineal crack roughness parameter, $(R_L)_{\text{cracks}}$		
edge trace	$0.206 \pm \begin{smallmatrix} 0.28 \\ 0.21 \end{smallmatrix}$	0.654 ± 0.26
center trace	0.526 ± 0.30	1.364 ± 0.50
Total subsurface crack trace length, $\sum_{i=1}^{40} (L_i)_i$		
edge trace	0.773 mm	4.085 mm
center trace	1.973 mm	8.528 mm
Mean crack surface roughness parameter, $(S_A)_{\text{cracks}}$		
edge trace	$0.262 \pm \begin{smallmatrix} 0.36 \\ 0.26 \end{smallmatrix}$	0.833 ± 0.33
center trace	0.670 ± 0.38	1.737 ± 0.64

The results of the local measurements of $(R_L)_{\text{cracks}}$ are presented graphically in Fig. 7. Although subsurface cracks are seen to occur irregularly over much of the specimen, the bulk of the cracking and its greatest intensity occur in the multifacet region.

Discussion

Useful procedures and relationships have been developed for this investigation in order to assess the true fracture surface area, true facet sizes, and the subsurface crack characteristics. The new equations for quantitative fractography are based mainly on the stereological analysis of the fracture trace in vertical sections. To summarize, the principal equations used here are as follows:

For the trace the equations are

- (a) lineal roughness parameter, $R_L = L_i/L'$,
- (b) true profile length, $L_i = (\pi/2)\overline{P_L(\theta)}A_T$, and
- (c) degree of orientation,

$$\Omega_{12} = \frac{(P_L)_\perp - (P_L)_\parallel}{(P_L)_\perp + 0.571 (P_L)_\parallel}$$

For the fracture surface they are

- (d) surface roughness parameter, $S_A = S_i/A'_i$,
- (e) true fracture surface area, $S_i = (4/\pi)R_L A'_T$, and
- (f) mean true facet area, $\overline{A}_{\text{facets}} = (4/\pi)R_L \overline{A}'_{\text{facets}}$.

The primes indicate projected quantities, that is, measurements made parallel to the mean CPD in the vertical sections, or parallel to the mean fracture plane in the SEM photomicrographs.

The most important experimental quantity is $P_L(\theta)$, the directional dependence of P_L . From these measurements we obtain $\overline{P}_L(\theta)$, as well as $(P_L)_\perp$ and $(P_L)_\parallel$. Thus, the two-dimensional quantities R_L , L_i , and Ω_{12} can be calculated directly and without difficulty. However, the accuracy of the results obtained for the fracture surface depends to a great extent on the adequacy of the sampling of sections through the fracture surface. The main problem, of course, is the lack of complete randomness in the orientation of fracture surface elements. Suitable weighting functions must be devised, or else sampling by sections must include more positions, directions, and angles with respect to the fracture surface.

One means of assessing the fracture surface area is the analysis based on partially oriented surface elements, provided we know whether we are dealing with the linear, planar, or planar-linear category. Thus, the upper-lower bound approach gives us essential information on the *type* of surface that is under scrutiny, and then permits quantitative calculations based on that type. Here the evidence points overwhelmingly to the partially oriented system of surfaces with planar orientation, and this is entirely consistent with experimental observations of faceting in the fracture surface.

The comparison of results for the single-facet and multifacet regions is of interest with regard to the predictions of Chakraborty's fatigue crack growth model [9, 10]. As a consequence, we should find that, compared with the single-facet fracture surface, the multifacet fracture surface:

- (a) has a lower degree of orientation,
- (b) has higher roughness parameters,
- (c) has greater true fracture surface area,
- (d) has a smaller mean facet size, and
- (e) has more subsurface cracking.

These expectations have been confirmed quantitatively by the present study.

The parameters obtained in this quantitative fractographic study vary from center trace to edge trace and from single-facet to multifacet regions. This reveals the extreme heterogeneity of the fracture process and emphasizes the need for representative sampling if meaningful results are to be achieved.

Conclusions

The experimental procedures utilized in this investigation are quite laborious and not well-suited to routine quantitative evaluation of fracture surfaces. However, the authors hope that experience gained here will shorten the time and labor required in subsequent work without appreciably diminishing the accuracy of the results.

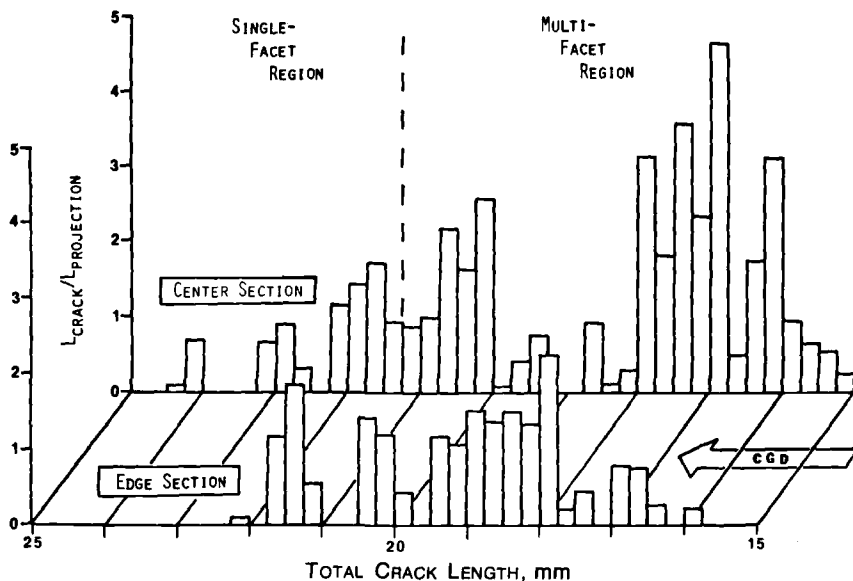


FIG. 7—Linear crack roughness parameter, $(R_L)_{cracks}$, for subsurface crack traces along the edge and center sections.

With further developments in the quantitative analysis of fracture surfaces, further advances in the formulation of meaningful, quantitative theories for predicting fatigue crack growth rates can be anticipated.

Acknowledgments

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Fourier Transform Techniques— Fracture and Fatigue

REFERENCE: Passoja, D. E. and Psioda, J. A., "Fourier Transform Techniques—Fracture and Fatigue," *Fractography and Materials Science*, ASTM STP 733, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 355–386.

ABSTRACT: Real-time analysis of the fatigue crack growth process has been achieved by means of phase-sensitive signal detection methods used in conjunction with spectrum analysis. During crack growth, modulations of the displacement signal occur as a result of the crack's interaction with the microstructure. It was found that the character of the modulation signals was not random but periodic. Utilization of quantitative fractographic and Fourier transform techniques facilitated the formulation of the relationships existing between the microstructure and the signals measured during fatigue crack growth testing. There is a direct correspondence between the Fourier spectrum of the crack profile and the Fourier spectrum of the displacement modulations; the space-time correspondence between the test and the fracture surface was established by the determination of the microscopic fatigue crack velocity. The spectra of the fracture surface and the displacement modulations arise from secondary cracking along second-phase particles in the Type 7075-T6 alloy that was studied.

KEY WORDS: fatigue, fractography, spectrum analysis, signal processing, Fourier transform methods, aluminum alloys, microconstituents, materials, materials science

The formation of a fracture surface in a metal during fatigue or impact testing takes place by means of a series of complex deformation processes that occur within the crack-tip plastic zone. The microstructure of the metal, which constitutes the medium in which the fracture process occurs, is composed of a variety of heterogeneous subunits that deform in concert during the formation of the fracture surface. Within any range of spatial dimensions, it is usually possible to identify sets of microstructural features that are salient to the fracture process. It is often extremely difficult to isolate these features and to account for the mechanism by which the full spectrum of microstructural subunits cooperatively interacts with the crack tip to form the fracture surface.

In general, any quantitative evaluation of a microstructure or a fracture surface has required that a selective identification and measurement be made

¹Senior research scientist and project scientist, respectively, Union Carbide Corp., Central Scientific Laboratory, Tarrytown Technical Center, Tarrytown, N.Y. 10591.

[1-7].² Once a microstructural feature (or features) of interest is determined, an average microstructural dimension is computed and correlated with a particular property such as fracture toughness. In some cases this method works well, but a great deal of microstructural information is lost or ignored in the determination of "averages." Our experience with mechanical testing of real microstructures indicates that it is difficult to describe an average microstructure, for microstructures tend to be statistically heterogeneous, and their properties tend to reflect this fact.

The fracturing of a microstructure occurs by means of a set of deformations that include a range of microscopic and macroscopic dimensions. The statistical nature of the deformation process makes it difficult to isolate any one dimension from others. It is highly likely that no one set of deformations that fully describes the actual fracture can be isolated, but somehow the deformations occur cooperatively and fracture is realized. The authors suggest instead that *all* of the microstructure is important and that the spectrum of microstructural subunits must be properly scaled and sequenced in order to account fully for the formation of a fracture surface.

Fourier transform techniques offer a number of advantages for attacking this problem. One particularly attractive feature is that the frequency representation of spatial or temporal coordinates can be described as a spectrum. The spectral representation eliminates the need to rely only on "averages." Arbitrary assignment of importance to any one microstructural subunit is no longer necessary, and hence a more collective description of the fracture process is possible. In previous work it has been possible to show that the fracture profile of HY-180 weldments is not random, but has an amplitude frequency relationship that is characteristic of a surface having fractal dimensions [8,9]. The genesis of this characteristic is not known, but the study demonstrates that an alternative description of the fracture geometry can be facilitated by Fourier transform methods.

Theoretical

The objective of the following work is to establish direct relationships between the microstructure and the mechanical properties during fatigue testing without separating the cause-and-effect relationships that might exist between them. A theoretical basis and subsequent analytical method have been established that relate the frequency content of the *x* and *y* displacement signals measured on the specimen to the spatial frequencies present on the profile of the fracture surface that formed during testing. Since the information concerning fatigue crack propagation in real microstructures is somewhat complex, it was necessary to make extensive use of Fourier transform techniques in order to accomplish a thorough analysis of the problem. The general theme and a cursory development of the theoretical background is presented here.

²The italic numbers in brackets refer to the list of references appended to this paper.

Extensive use of communications theory concepts and statistical signal processing methods was made to guide the development of our methods.

General Considerations

Since fatigue crack growth testing normally takes place under sinusoidal load control conditions, the time-dependent force applied to the specimen is

$$F_i(t) = A \cos (\omega_c t) \quad (1)$$

where A = the load amplitude, and ω_c = the test frequency. The resulting displacement of the specimen is related to the compliance and can be written formally as

$$F = -Kx \quad (2)$$

If the specimen's compliance changes as a result of crack propagation, then the force on the specimen becomes

$$F_0(t) = \int_0^t K(t - \tau)x(\tau)\tau \quad (3)$$

under the assumption of linear superposition [10]. Experimentally, the authors have observed that as crack growth proceeds, the frequency of the displacement signal does not change, but its amplitude does. Experience dictates that Eq 3 probably has the form

$$F_0(t) = K(t)X_0 \cos (\omega_c t) \quad (4)$$

When written in this manner, Eq 4 bears a striking similarity to the equation that describes the amplitude-modulated carrier wave used in the transmission of AM radio signals. In the case of radio transmission, the AM signal can be represented as [11]

$$S(t) = P(t) \cos (\omega_c + \phi) \quad (5)$$

Here the information in the signal is contained as a time-dependent modulation term, $P(t)$; the phase shift, ϕ , is included in the equation for completeness. We may thus state our first two assumptions:

Assumption 1: Crack propagation during fatigue crack growth results in a modulation of the specimen's sinusoidal displacements resulting from the small compliance changes that occur during crack propagation.

Assumption 2: In analogy with AM communications transmission, the base-band signal, which contains the signals of interest, lies in the vicinity of the sinusoidal test frequency, ω_c , that is, $0 < \omega < 2 \omega_c$.

When viewed in this manner, the microstructural information of interest (Eq 4) is contained in the time-dependent compliance term, $K(t)$.

Our objective in this work is to demodulate this signal and to interpret the information content of this signal with respect to the fracture process. Since the crack travels through different portions of the microstructure during the fatigue test, information is collected from the microstructure over the time interval of the test, ΔT . In order to relate a distance (microscopic) on the fracture surface to a time interval (macroscopic), the crack growth velocity must be known.

Assumption 3: A microscopic distance on the fracture surface can be related to a time interval of the test in some consistent manner.

Assumption 3 thus provides a means of relating the spatial and temporal coordinates to each other and states that, in the most general terms, the crack growth velocity probably obeys an equation similar to Langevin's [12]

$$\frac{dv}{dt} = -\beta v + \zeta(t) \quad (6)$$

where β is a constant, v is the microscopic crack velocity, and $\zeta(t)$ is a fluctuating component, which may or may not be a random fluctuating force. As a first approximation, it will be assumed that the fluctuating component, $\zeta(t)$, is small and that the microscopic growth velocity can be approximated by

$$v = \lambda_{ss}\omega_c \quad (7)$$

where λ_{ss} = the average microscopic distance moved per cycle (the average fatigue striation spacing), and ω_c = the frequency of the test.

Fourier Transform Methods

The use of Fourier transform techniques and the representation of signals in the frequency domain were used in the development and understanding of the signal processing strategy and in analyzing the test specimen's signals during fatigue testing.

The representation of signals in the frequency domain greatly simplifies the separation of "signal" from Gaussian noise, since periodic signals usually appear as delta functions of discrete frequencies, whereas random noise usually appears as a spectrum of constant amplitude over a range of frequencies that terminates at some cutoff frequency, ω_u . The general use of Fourier analysis in communications theory, and in other fields as well, is probably related to its ability to represent complex signals in the frequency domain in a more manageable form.

Assuming that Eq 3, representing the time-dependent force measured on the specimen during testing, can be written as a Fourier transform, we have

$$F_0(\omega) = \int_{T_1}^{T_2} F_0(t) \exp(-i\omega t) dt$$

and

$$(8)$$

$$F_0(\omega) = X(\omega)K(\omega)$$

where $X(\omega)$ = the Fourier spectrum of the displacement, and $K(\omega)$ = the Fourier spectrum of the compliance.

Equation 8 is a convolution integral and is a product of two functions in the frequency representation. In the time domain, demodulation of the compliance information is equivalent to deconvolution of the compliance from the displacement term in the frequency representation. Figure 1 shows the frequency representation of a double sideband (DSB) signal convolved with a carrier frequency, ω_c . The carrier is shown to be a spectrally pure sinusoid, and the information or signal is shown as two flat-band spectra cut off at the upper cutoff frequency, $\omega_c + \omega_u$, and at the lower cutoff frequency, $\omega_c - \omega_u$, on either side of the carrier.

If the anticipated form of the signal, $K(t)$, is considered, one can quickly appreciate that its form is unknown, and some method is necessary to relate the measured signal to the microstructure. Evidently, random Gaussian noise could be an acceptable result [13]. In this case, Fourier transform analysis techniques would prove to be advantageous in separating periodic signals from noise or in analyzing the noise content of the signal [14].

Figure 2 shows the relationship between the power spectrum and the autocorrelation function. The autocorrelation function is defined as

$$R_{11}(t) = \int_0^\omega S(\omega) \exp(i\omega t) d\omega \quad (9)$$

where $S(\omega)$ = the power spectrum and is merely the inverse transform of the Fourier power spectrum [14].

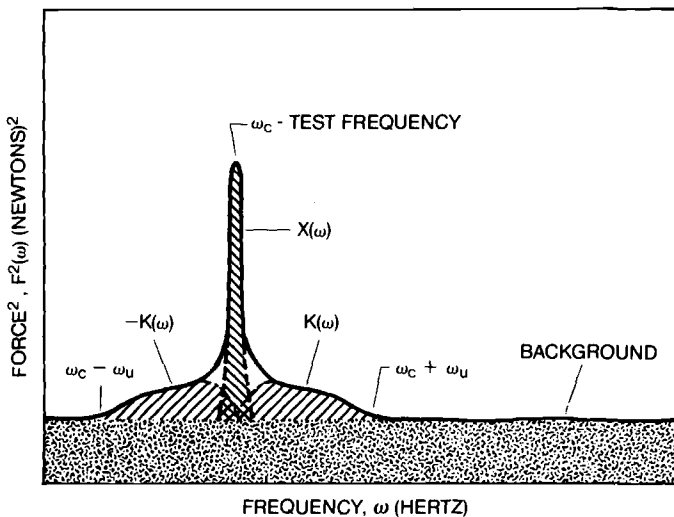


FIG. 1—Theoretical representation of the double sideband compliance spectrum of bandwidth $\omega_c - \omega_u$ convolved with the displacement carrier spectrum. The carrier spectrum is assumed to be a spectrally pure sinusoid of frequency ω_c . This spectrum is analogous to a double sideband AM radio frequency spectrum. The baseband of the compliance spectrum is different from that of the test frequency.

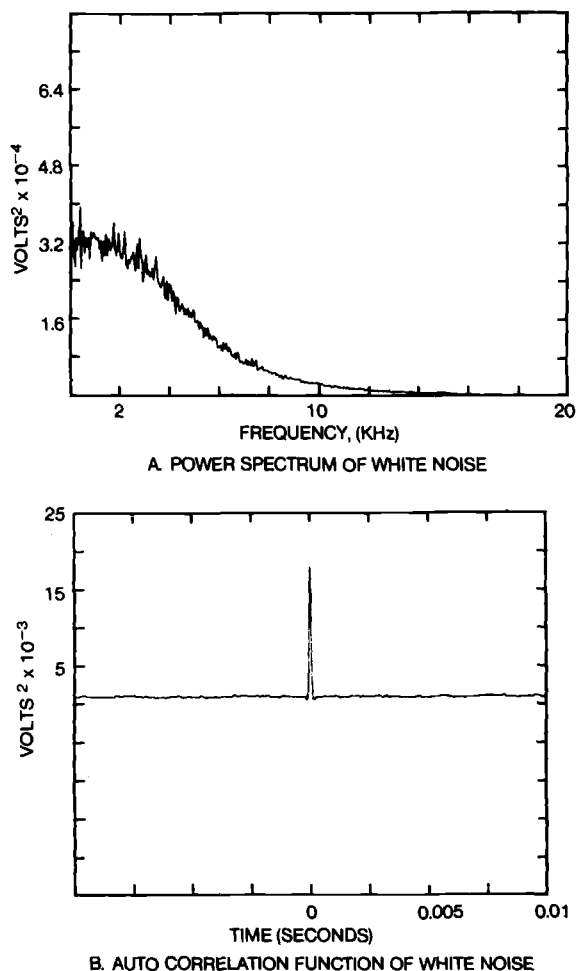


FIG. 2—The Fourier transform pair: (a) the power spectrum of white noise; (b) the autocorrelation function of white noise—a delta function in the vicinity of $t = 0$ showing no long-range periodicity.

The example shown in Fig. 2 is random Gaussian noise with a bandwidth of 5 kHz. The power spectrum shown indicates that the spectrum is flat-band to approximately 4 to 5 kHz and falls off to zero above $\omega_c + \omega_u$. The corresponding autocorrelation function shows that no long-range periodicities exist and that the autocorrelation function is essentially a δ function in the vicinity of $t = 0$. Any periodicities present in the signal would yield a periodic autocorrelation function for $t > 0$.

In order to form the relationship between temporal and spatial coordinates [time $\leftrightarrow$ distance and frequency (Hz) $\leftrightarrow$ frequency (cm^{-1})] Assumption 3 must be used. The Fourier transform of the fracture described in the coordinates of the specimen is

$$Y(K) = \int_{L_1}^{L_2} Y(x) \exp(-iKx) dx \quad (10)$$

where the integration interval corresponds to some distance on the specimen (L_2 to L_1) over which the fatigue crack has traveled in the time interval $\Delta T = T_2 - T_1$.

Material

Standard Charpy-size specimens were used throughout this investigation to study crack growth, using Fourier spectrum analysis. The Type 7075 aluminum-zinc-magnesium-copper alloy was a rolled 5.1-cm (2 in.) thick plate of commercial grade quality. The chemical composition of this plate of high-strength aluminum is given in Table 1. The analysis is typical of commercial quality Type 7075 aluminum. The alloy was solution heat treated and artificially aged. The temper designation applied to this condition is T6. The yield strength of the material evaluated in the plate longitudinal direction was 490 MN/m² (71 ksi). The elongation-to-fracture in a 5.1-cm (2-in.) tensile gage section was 10 percent. Although it was not measured in this investigation, the fracture toughness of material in the longitudinal-short transverse (L-S) orientation [15] is expected to be no more than 38.5 MN/m^{3/2} (35 ksi $\sqrt{\text{in.}}$) since the material is of commercial quality.

The rolled grain structure is shown in the composite photomicrograph in Fig. 3. The directionality of the structure should be noted. A schematic drawing of the L-S oriented Charpy-size fatigue test specimen is sketched in Fig. 3 in relation to the base-plate orientation. The length of the test specimen was parallel to the longitudinal (L) or rolling direction. The fatigue crack was designed to propagate across the short transverse or S direction. Due to mechanical working, the second-phase particles, commonly referred to as microconstituents, are elongated in the rolling direction. The shape and size of these second-phase particles are shown in the unetched composite photomicrograph shown in Fig. 4.

The S and T directions define the fatigue fracture plane after the specimen is finally fractured. While growing in the S direction, the fatigue crack front must interact with grain boundaries and microconstituents (Figs. 3 and 4) that are contained within the crack-tip plastic zone. The spacing between the grain

TABLE 1—Chemical analysis of Type 7075 aluminum alloy investigated, compositions in weight percent.

Cr	0.20
Cu	1.49
Fe	0.26
Mg	2.22
P	<0.26
Si	0.07
Mn	0.09
Ti	0.05
Zn	5.7
Al	Balance

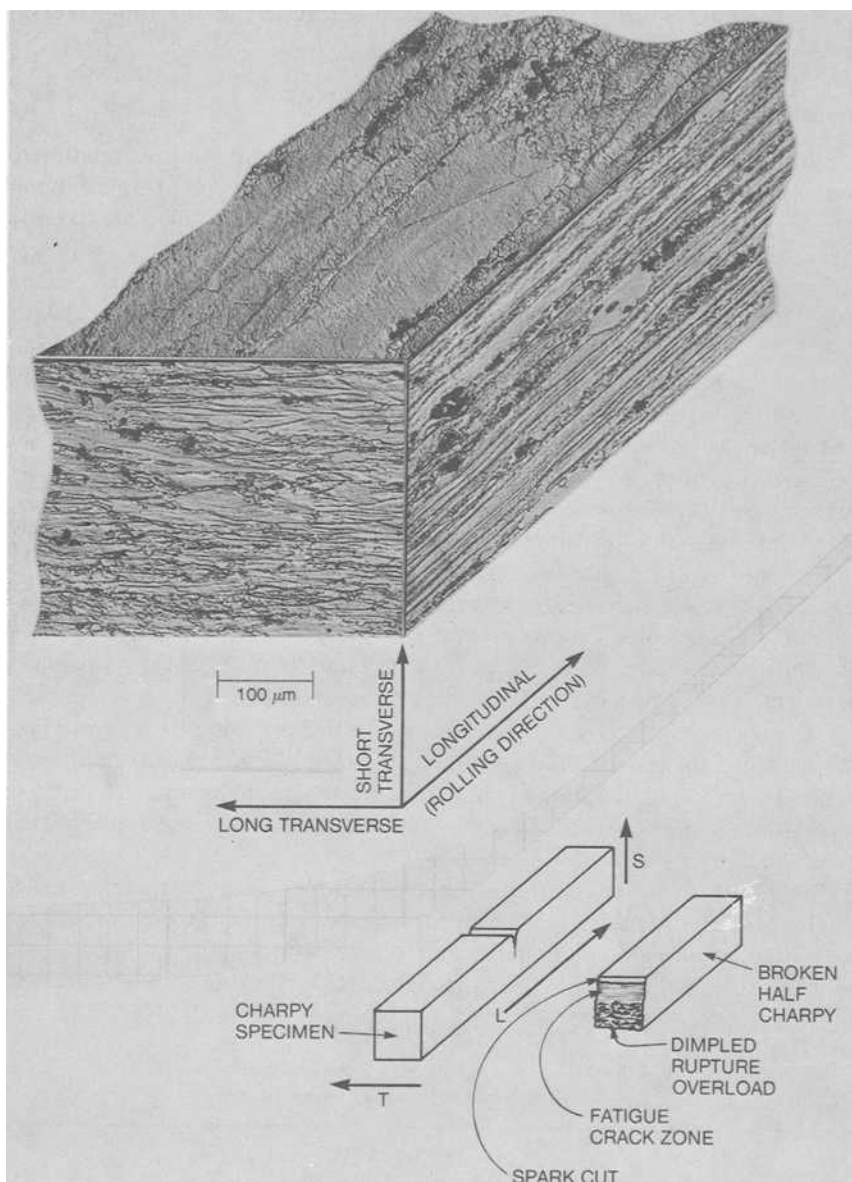


FIG. 3—Composite etched optical photomicrograph of the rolled Type 7075-T6 aluminum grain structure.

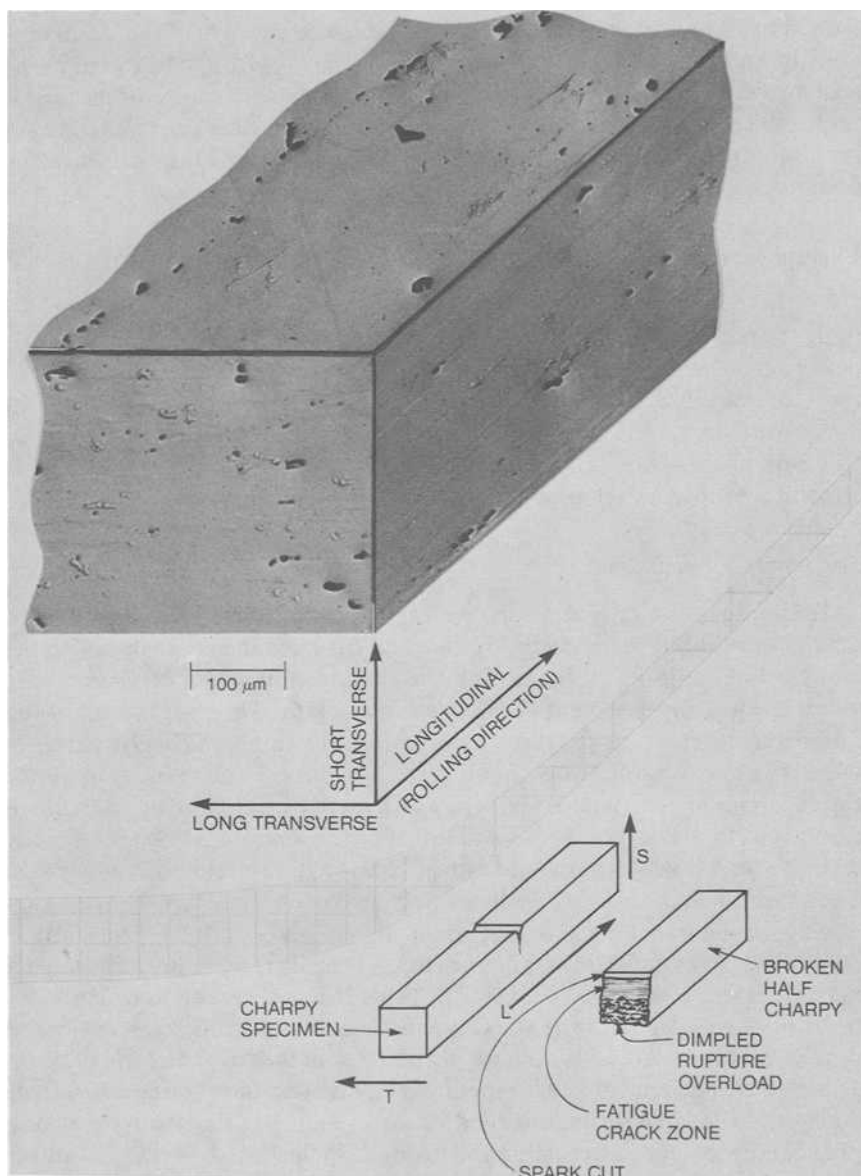


FIG. 4—Composite unetched optical photomicrograph of the rolled Type 7075-T6 aluminum alloy showing microconstituents elongated in the rolling direction.

boundaries, which are traversed by the crack along the S direction, ranges from 0 to 30 μm . In the same orientation, the separation between the microconstituents that the fatigue crack encounters ranges from 0 to 250 μm . The spacing between the microconstituents and grain boundaries is of particular importance to the crack growth behavior in this material. The role of microstructure on the mechanism of cracking will be discussed in the following sections.

Experimental Methods

The following work is divided into several sections, which describe the fatigue testing conditions and the metallographic, fractographic, and signal-detection techniques. We will show that the use of a sequence of complementary techniques results in a more thorough description of the effect of microstructure on the fatigue mechanism in this alloy, and, in particular, we will show how Fourier transform techniques can be used to elucidate the basic structure-properties relationships that are inherent in the problem.

Fatigue Test Conditions

Tension-tension fatigue tests were carried out in three-point bending on Charpy-size specimens. The test fixture was designed in accordance with the British Welding Society for testing the fracture toughness of Charpy-size crack-opening displacement (COD) specimens [16]. The progression of the fatigue crack front or (COD) was monitored with an MTS extensometer which had linear response over a range of ± 0.5 mm. The impulse response of the gage-mount-specimen assembly was found to be flat-band up to frequencies >500 Hz. All fatigue tests were run at a frequency of 37 Hz using a sine wave under load control on an Instron servo-hydraulic closed-loop system. The extensometer, or COD feedback and stroke feedback signals, were analyzed with the signal detection technique described further on. Four fatigue conditions were studied in detail. The stress-intensity ranges, stress ratios, and maximum stress intensities are listed in Table 2. These were chosen in order to investigate the effects of the stress intensity level, K_{mean} , and stress intensity range, ΔK , on the fatigue crack growth behavior of the alloy. The ΔK values in Table 2 have been calculated for the stress intensity at the tip of a sharp fatigue crack in a Charpy specimen loaded in three-point bending, using the compliance correction and stress-intensity formula in the British Welding Institute recommended standard [16]. The crack length, a , at which most measurements were taken is given in Table 2 as a fraction of the specimen width, W .

Fracture Profile Measurements

After specimen failure, the fracture topography was examined using metallographic sectioning of the crack profile. The cross sections were mounted and

TABLE 2—Charpy-size specimen fatigue test conditions.

Specimen No.	ΔP , N	P_{mean} , N	P_{max} , N	a/W , MN/m ^{3/2}	ΔK , MN/m ^{3/2}	K_{max} , MN/m ^{3/2}	Stress Ratio, R
20	1783.7	1779.3	2735.7	0.54	21.6	33.1	0.35
21	778.4	1779.3	2224.1	0.50	8.3	23.5	0.65
24	1490.2	1112.1	1935.0	0.55	18.7	24.2	0.23
25	911.9	1121.0	1543.5	0.53	10.7	18.0	0.41

polished in two orientations, using standard metallographic techniques: one to reveal the crack path in relation to the growth direction, and the other to expose the advancing crack front. Vacuum impregnation and alumina bead infiltration were used in order to preserve edge retention. The profiles were examined at $\times 100$ and $\times 500$ magnification in both the unetched and etched conditions. The fatigue crack growth direction S lies in the crack profile plane defined by the L and S directions. The plane of the advancing crack front is defined by the L and T directions.

A photomontage was made, covering an interval of 51.2 cm at $\times 500$ on the crack growth direction profiles only. Graph (tracing) paper was mounted over the photomontage and a base line was drawn on the graph paper. The x and y coordinates of the profiles were digitized at 0.1-cm intervals by measuring the coordinates of the edge of the fracture profile under a $\times 10$ binocular microscope and recording them on data sheets. The data were then punched on paper tape for computer processing. Data collected from the fracture profiles were used to compute both the Fourier transform and its inverse by means of a computer program based upon the fast Fourier transform [17]. The program was taken from the IBM PL1 library, rewritten in FORTRAN IV, and adapted to a time-sharing system. The computed spectra were plotted on an x - y plotter. The frequencies of each spectrum were then related to the grain boundary and microconstituent spacings within the microstructure that served to modulate the crack advance in the fatigue crack growth process.

Fractography

Stereo-pair fractographs were examined at magnifications ranging from $\times 100$ to 5000 to establish differences in fracture surface topography that were related to the changes in loading conditions. At $\times 5000$, the fatigue striations were resolved and measured. At $\times 100$ to 500, the frequency of grain boundary and microconstituent cracking was easily seen by stereographic observations.

Signal Detection Methods

There are two methods that are generally used to demodulate AM signals [18]—envelope detection and synchronous detection using phase-locked detection techniques. We chose to use a synchronous detection method for demodulating the compliance signal for the following reasons:

1. By detecting the signal that was in phase with the load feedback signal, detection would take place in relation to a reference; *coherency* for the entire mechanical testing system could thus be established. That is, displacement signals were detected synchronously, with load feedback being used as a reference signal.
2. A considerable improvement in the signal to noise ratio could be realized and, if necessary, weak signals could be detected.

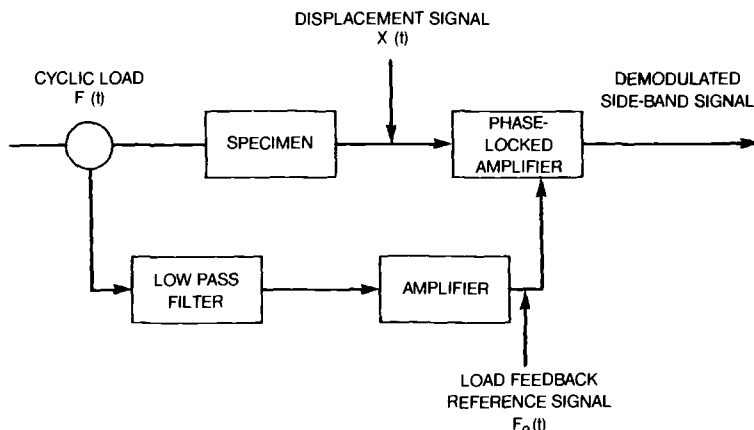


FIG. 5—Block diagram of phase-locked detection demodulation methods used.

3. Any frequency drift that might occur during testing would be eliminated from the signal measurement.

Figure 5 shows a block diagram used for the signal detection method. The servo-hydraulic testing system was operated under load control, and the load feedback signal was used for the signal reference. The reference signal was low passed through a filter and amplified to eliminate higher frequency noise and to provide a strong enough reference signal to eliminate any drift and phase jitter that might arise. Figure 6³ shows a frequency representation of the shifts that occur in the sideband information using this signal-detection method. The lock-in amplifiers synchronously rectify the signals that pass through them, resulting in an upward translation of the carrier from $\omega_c \rightarrow 2\omega_c$ and a downward translation of the upper sideband from $\omega_c + \omega_u$ to $0 + \omega_u$. A low-pass filter in the lock-in amplifier is used to eliminate the high-frequency components and to pass the d-c components present in the signal.

Two Ithaco 393 lock-in amplifiers were used to demodulate synchronously the strain and stroke signals measured for the specimen during fatigue crack growth testing. A clip-on extensometer (MTS No. 632.05 C-60) and an LVDT supplied with the Instron servo-hydraulic test system were used to generate the signals. This is shown schematically in Fig. 7. The demodulated signals were fed into a Nicolet 660 A dual-channel spectrum analyzer; the real time, power spectra, autocorrelation function and cross-correlation functions for the signals were determined in this manner.

Results and Discussion

Fatigue crack propagation was examined in a single heat of Type 7075-T6 aluminum in one orientation in relation to the rolling direction. This restric-

³ Fisher, E., Ithaco Corp., Guilford, Conn., personal communication, "The Evolution of the Modern Lock-In Amplifier," Dec. 1978.

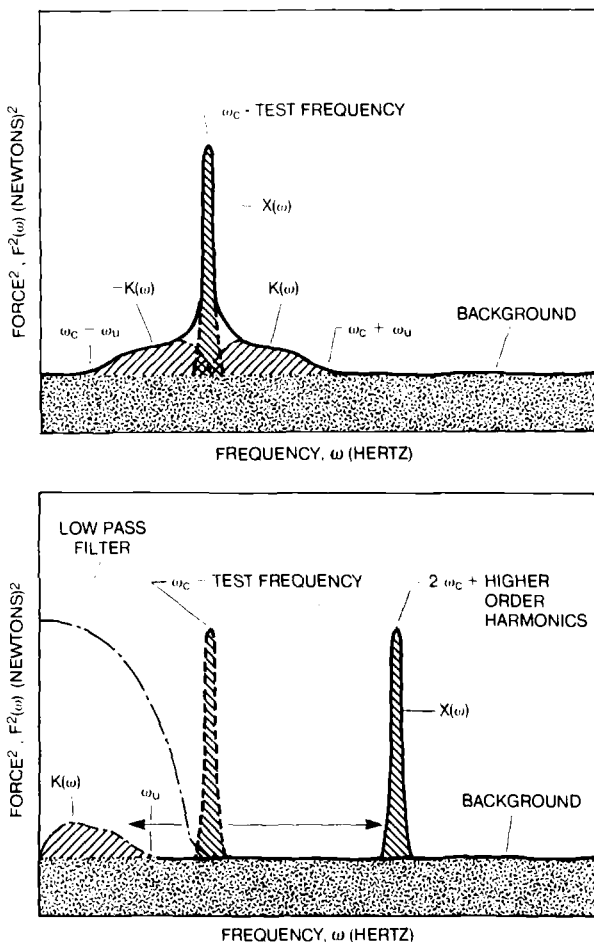


FIG. 6—Frequency representation of signal shifts resulting from demodulation methods used in this work: (top) representation of spectrum showing carrier and sideband information before demodulation; (bottom) spectrum after demodulation using phase-locked techniques. The carrier is heterodyned upward to $2\omega_c$, the sideband information is translated downward between 0 and ω_u , and a low-pass filter is used to eliminate the unwanted high-frequency terms [19].

tion was imposed so as to concentrate on the main purpose of establishing the spectrum analysis testing technique. Two mean stress levels and two stress-intensity ranges were employed. With this testing program, scanning electron microscope (SEM) fractographic and metallographic crack profile analyses were performed in conjunction with dynamic testing in order to establish direct correspondence between the techniques. The macroscopic fatigue crack growth per cycle, da/dN , was not measured since the intention was to develop the signal detection technique. For ease of specimen preparation, standard Charpy-size specimens were used.

The results of the complementary approach are described in the three sec-

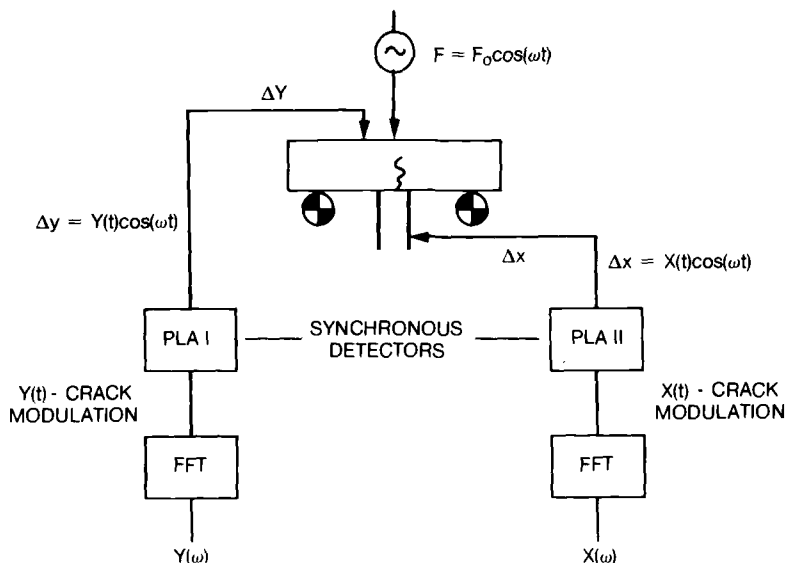


FIG. 7—Schematic diagram of the signal detection setup used in this program for measuring crack-tip deformation interference.

tions that follow. The behavior of a particular specimen (Specimen 24) is used to exemplify the methodology. Since the utility of the method is based upon establishing a direct relationship or relationships that might exist between the fracture surface and a measured property, an entire sequence of techniques was required to develop a foundation for the overall relationship. The sequence of techniques described in detail in the following sections is as follows:

- (a) SEM fractographic observations,
- (b) dynamic signal detection, and
- (c) fracture profile analysis.

The detailed descriptions presented in these sections provide an example of the new analytical method illustrated for a single testing condition. Comparisons between different testing conditions follow in the section on differences related to ΔK and $K_{\max}$ levels.

Fractography

Figure 8 is a plot showing the stress-intensity levels that were present during the fatigue test at the crack lengths where measurements were taken. The fractographic work was restricted to fractured areas where spectrum analysis measurements had been taken during cyclic testing.

In Fig. 9, the fatigue striation spacing is shown at a crack length of 5.5 mm, a ΔK level of $18.7 \text{ MN/m}^{3/2}$ ($17.0 \text{ ksi} \sqrt{\text{in.}}$), and a $K_{\max}$ of $24.2 \text{ MN/m}^{3/2}$ ($22.0 \text{ ksi} \sqrt{\text{in.}}$). If the assumption is made that the crack advanced one striation spacing

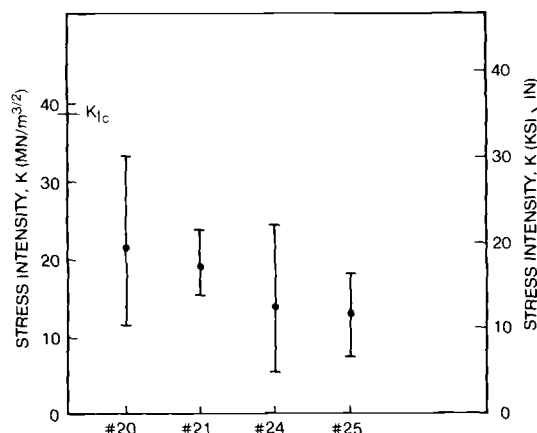


FIG. 8—Stress intensity range used in fatigue testing of each of the specimens listed in the graph. Values correspond to K levels at the crack lengths listed in Table 2.

per cycle, then the average microscopic growth rate, $d\lambda/dN$, measured from 15 fractographs was $0.57 \pm 0.05 \mu\text{m}/\text{cycle}$. Since the specimen was tested at a frequency of 37 Hz, the resultant average microscopic crack velocity is $21.2 \pm 1.8 \mu\text{m}/\text{s}$.

The fractographic evidence for the effect of microstructure on crack propagation in Specimen 24 was revealed by studying stereo-pair fractographs at magnifications ranging from $\times 200$ to 500. Figure 10a is an SEM fractograph that illustrates the fracture surface roughness resulting when the crack-tip plastic zone encountered a microconstituent or a grain boundary. The photomicrograph shows fragmented particles, tear lines, and the general topography change that resulted from localized redirection of the crack through its

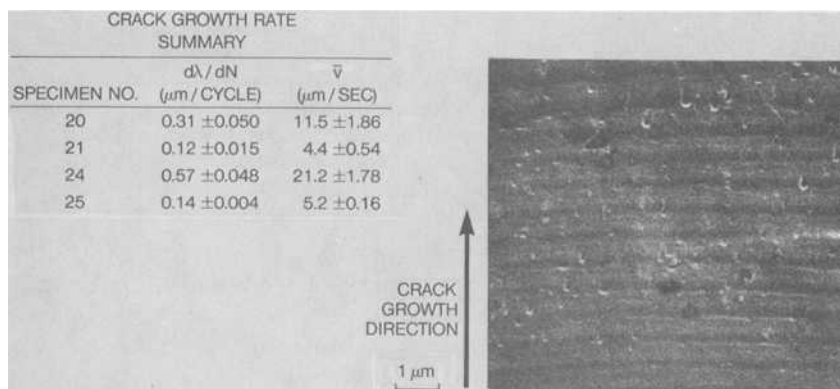


FIG. 9—An SEM fractograph of fatigue striations in fractured Specimen 24 corresponding to a crack length of 5.5 mm, measured from several regions, $d\lambda/dN = 0.57 \pm 0.05 \mu\text{m}$.

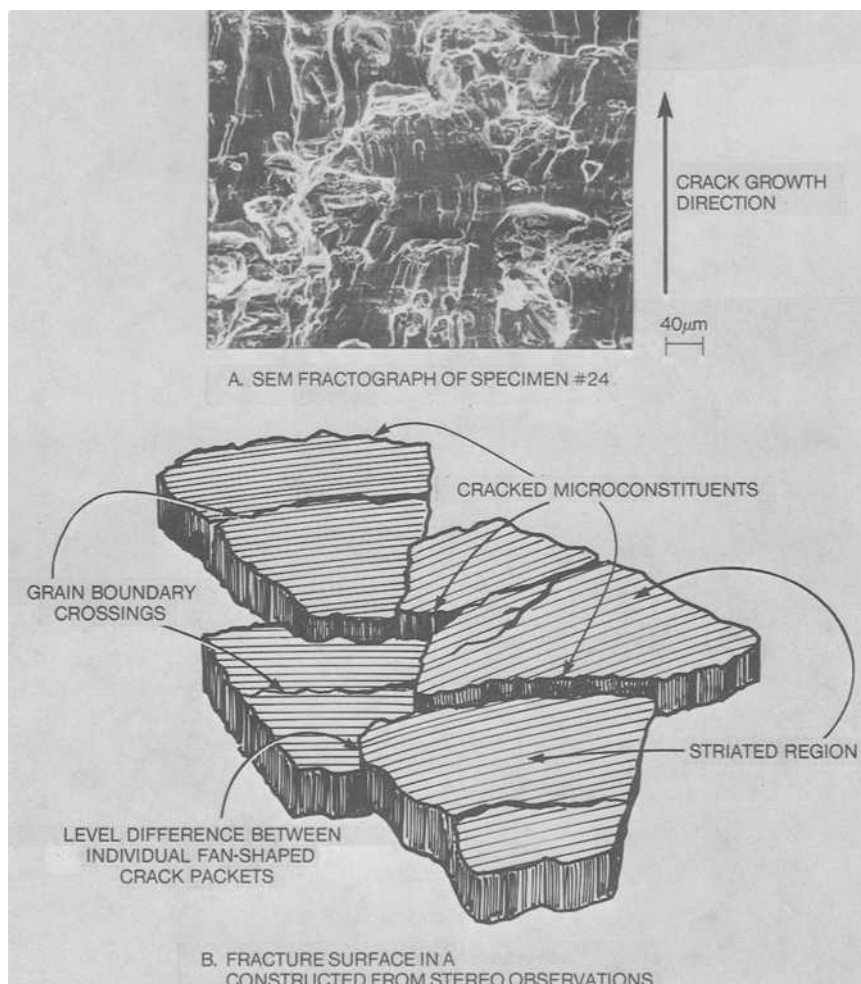


FIG. 10—An SEM fractograph and schematic drawing of the fracture surface to illustrate the topography and crack direction changes associated with crack-tip interactions with microconstituents and grain boundaries.

interaction with the microstructure. Particles with dimensions in excess of some critical size and boundaries that were sufficiently weakened by Mg_2Si or some other phase were evidently activated by the approaching crack front. The frequency of occurrence and extent of transverse cracking is strongly dependent upon the ΔK and $K_{\max}$ conditions of loading. Figure 10b is a schematic drawing of the fracture surface in Fig. 10a. This figure shows boundary and microconstituent crossing events that have caused a series of tear lines and a localized change in the crack growth direction. Generally, the overall crack front appears to have been broken up into many individual fan-shaped

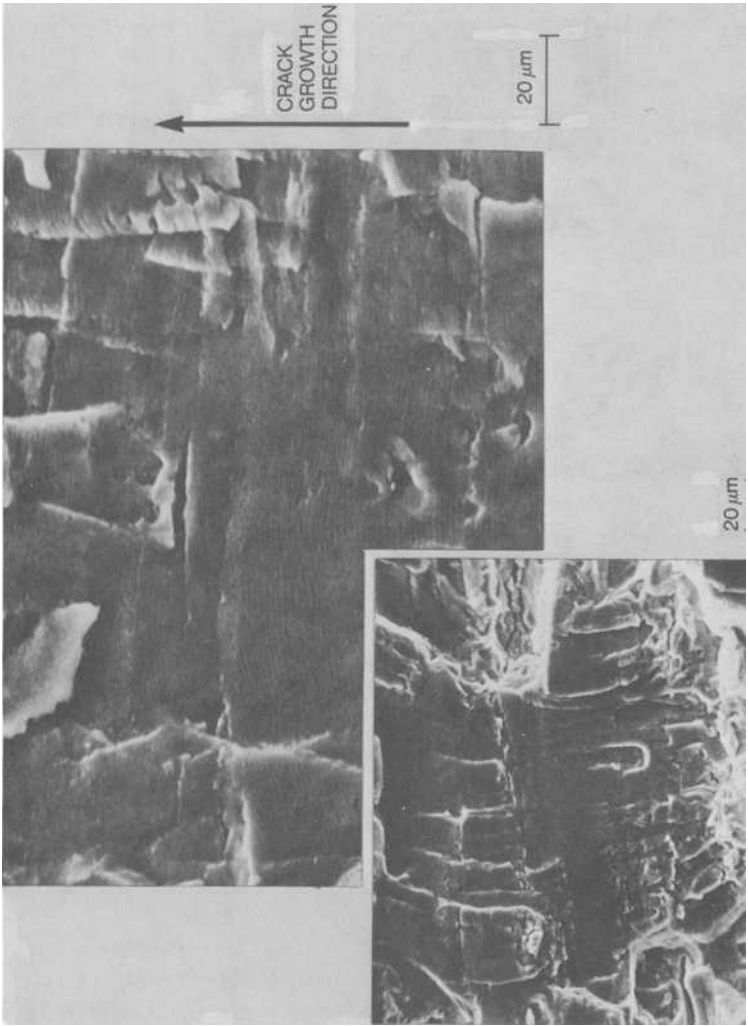


FIG. 11—An SEM fractograph from Specimen 24 at higher magnification, showing more detail in the secondary cracks at microconstituents and grain boundaries.

packets where crack growth occurred by a fatigue striation mechanism. Figure 11 shows the same fracture surface area at slightly higher magnification. The spacing between regions where secondary cracking occurred lies between 10 and 100 μm . The smooth regions between these microstructural disturbances are striated in a manner similar to the fracture surface in Fig. 9.

Quantitative metallography was implemented to confirm whether the spacing between secondary cracks observed in stereo-pair fractographs was caused by particles. The grain boundary and microconstituent nearest neighbor spacings were measured in the direction of crack propagation (S). Random lines were placed parallel to the crack growth direction on the L-S plane similar to those in Figs. 3 and 4. Over 700 grain spacings were measured on five different photographs. The intercept spacing between second-phase particles was measured in a similar manner. Separations between more than 400 particles were measured. The distributions of spacings between grain boundaries and microconstituent particles are plotted in Figs. 12 and 13, respectively. The grain boundary spacings ranged between 1 and 30 μm . The most probable boundary separation was between 6 and 8 μm . Along the S direction, particles were spaced between 0 and 260 μm apart. The most probable spacing between microconstituents shown in the distribution in Fig. 13 was about 40 μm . Three measurements between secondary cracks in Fig. 11 were 29, 44.5, and 78 μm . These spacings fall well within the distributions plotted in Figs. 12 and 13.

In order to relate a characteristic dimension on the specimen such as the spacings between the secondary cracks to a time interval of the test, the microscopic crack velocity must be used in the following manner. It was shown earlier that, by knowing the average fatigue striation spacing and the test frequency, the microscopic crack growth velocity was discovered to be 21.2 $\mu\text{m/s}$. If the crack had encountered a series of microstructural features having

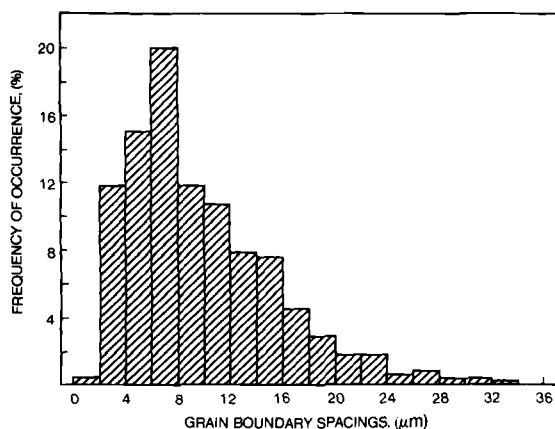


FIG. 12—Distribution of spacings in Type 7075-T6 aluminum between grain boundaries in the S direction measured in the L-S or crack-profile plane.

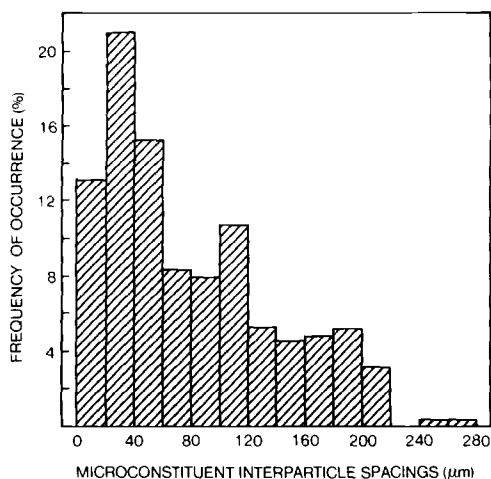


FIG. 13—Distribution of spacings in Type 7075-T6 aluminum between microconstituents in the L-S plane measured along the S or crack-profile direction.

spacings of 29, 44.5, and 78 μm , the time intervals between the encounters would be 1.36, 2.09, and 3.67 s, respectively. If the features were uniformly distributed in the microstructure, then, during fatigue crack growth, periodicities of 0.73, 0.48, and 0.27 Hz would be observed. The space-time relationships will be utilized further in the section on dynamic signal detection in order to interpret the temporal periodicities observed during testing.

Reconstruction of the fatigue crack growth mechanism from fracture surfaces requires that observations be made of many different areas at several magnifications. Although fracture interpretations are often quite adequate, the data comes after the fact of fracture. The sequence of events that occur at the crack tip determine the fracture topography. Detection of the sequence of microstructural interactions as they occur can be provided by real-time analysis of fatigue crack growth coupled with quantitative fractographic analysis. An understanding of fatigue crack growth rate and mechanism need not be reached by fractographic observations alone.

Dynamic Signal Detection

According to the theoretical development outlined here, it has been hypothesized that crack propagation during fatigue cycling creates modulations of the specimen's sinusoidal displacements resulting from small compliance changes. These modulations occur particularly when the microconstituents interrupt the more steady-state striated crack growth.

The demodulated stroke feedback and COD feedback signals were analyzed and recorded with the Nicolet 660 system. Basically three functions were measured and plotted for each of the test conditions illustrated in Fig. 8. The real-time signals are shown in Fig. 14 for Specimen 24. These signals were re-

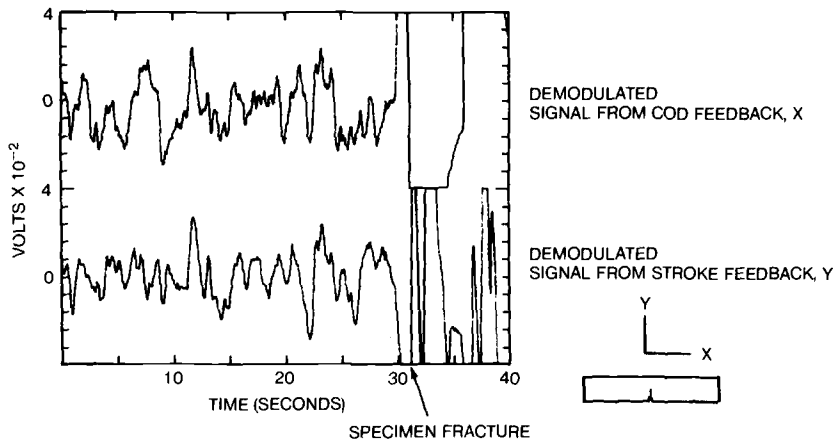


FIG. 14—Real-time signals from a Nicolet 660 spectrum analyzer after demodulation of the side-band content of the COD feedback and stroke feedback signals.

recorded in regions from which fracture surfaces were analyzed by SEM observations. The upper trace in Fig. 14 is the demodulated COD feedback signal. The lower trace is the simultaneously detected and demodulated stroke feedback signal. Simultaneous detection of the displacement modulations by two independent, orthogonal detector systems proved to be very useful since similarities and differences could be observed in the signals. A thorough analytical description of the signals measured by the detectors is not available at present, however. It should be noted that the COD feedback signal varies with time in an irregular sawtooth fashion. The sudden change in Fig. 14 after 30 s is due to specimen overload and fracture. This section of the output is to be ignored as far as fatigue crack growth is concerned.

The problem of pattern or signature recognition is significantly reduced by use of Fourier analysis techniques. The spectrum of the demodulated real-time COD feedback signal is shown in the upper plot in Fig. 15. The envelope of frequencies from zero to slightly more than 2 Hz, shown in Fig. 15, is related to the spacings between the microconstituents and grain boundaries from approximately 10 to 110 μm which were active secondary crack nucleation sites. The primary peaks in Fig. 15 are listed in Table 3 along with corresponding spacings in micrometres calculated based upon the fatigue crack velocity, v , of 21.2 $\mu\text{m/s}$, determined from striation spacing data. Division of the crack velocity by each of the frequencies detected from the demodulated displacement signal results in the interparticle spacings recorded in Table 3. These frequencies and spacings correspond very closely to those expected based upon the fractographic evidence discussed earlier. It would appear that the spatial frequencies observed are a set of interparticle spacings, described by the distribution shown in Fig. 13. The particular set of spacings sampled during the test acted as primary nucleation sites for secondary cracking and thus gave rise to the small compliance changes that acted to modulate the displacement signal.

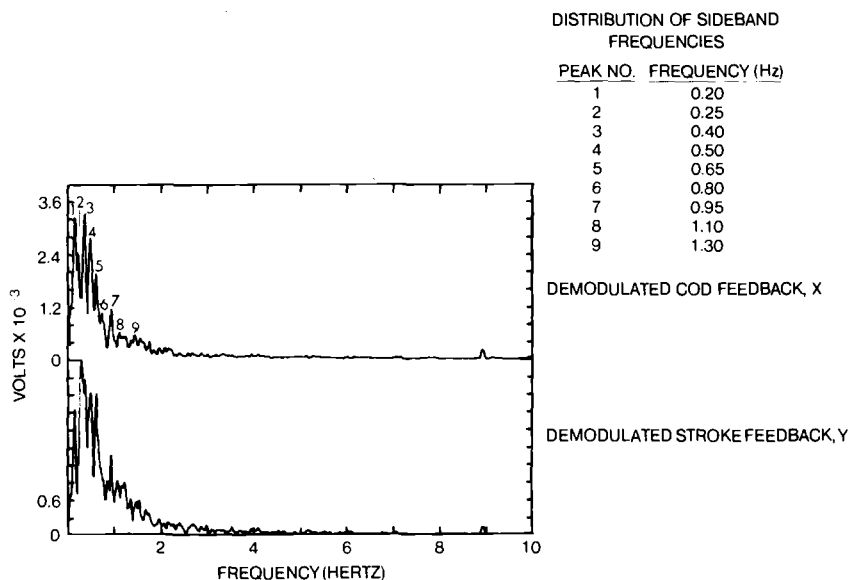


FIG. 15—Envelope of sideband frequencies from demodulated COD feedback (upper curve) and stroke feedback (lower curve) signals. Individual frequencies are related to the spacings between microconstituents (Table 3).

It should be noted that the crack front interacts with the microstructure in a volume of material and not in a single plane. As such, the frequencies indicated on the spectrum analysis do not necessarily correspond exactly to the same set measured from a localized spot in a fractograph or from a planar spacing, as determined from Figs. 12 and 13. The problem is further complicated by the fact that the crack front does not move uniformly across the spec-

TABLE 3—Summary of frequencies from demodulated displacement signal recorded for Specimen 24.

Peak No.	Frequency, Hz	Expected Microstructural Spacing, λ , μm , Computed Based upon $\bar{v} = 21.24 \mu\text{m/s}$
1	0.20	106.2
2	0.25	85.0
3	0.40	53.1
4	0.50	42.5
5	0.65	32.7
6	0.80	26.6
7	0.95	22.4
8	1.10	19.3
9	1.30	16.3

imen width, but rather moves in discrete packets, as evidenced by stereo-pair fractographic evidence.

The autocorrelation function for the demodulated signal measured during the test is shown in Fig. 16. The autocorrelation function eliminates phase information and distinguishes similarities between delayed portions of the same signal to itself [19]. If the modulation variation due to the microstructure is periodic but is randomly delayed in time (randomly phased), the autocorrelation function will distinguish the periodic part of the signal. The positions marked with arrows on Fig. 16 are approximately 2 s apart. Based upon the average microscopic fatigue crack velocity, the corresponding distances between the microstructural units are 42.4, 48.8, and 84.8 μm . These distances are directly related to the most probable spacings between microconstituents measured in the S direction, as shown in Fig. 13.

Fracture Profile Analysis

To extend further the understanding of the Fourier analysis approach, an additional technique was employed. Figure 17 is a series of composite optical photomicrographs that reveal the profile of each of the fatigue cracks for the four conditions considered in this investigation. The Fourier spectrum of the digitized crack profile is shown in Fig. 18. Table 4 summarizes the spatial frequencies associated with the peaks of the spectrum. The spacings that correspond to these frequencies are shown along with the temporal frequencies converted by using the microscopic crack growth velocity. These frequencies should be compared with those measured during testing.

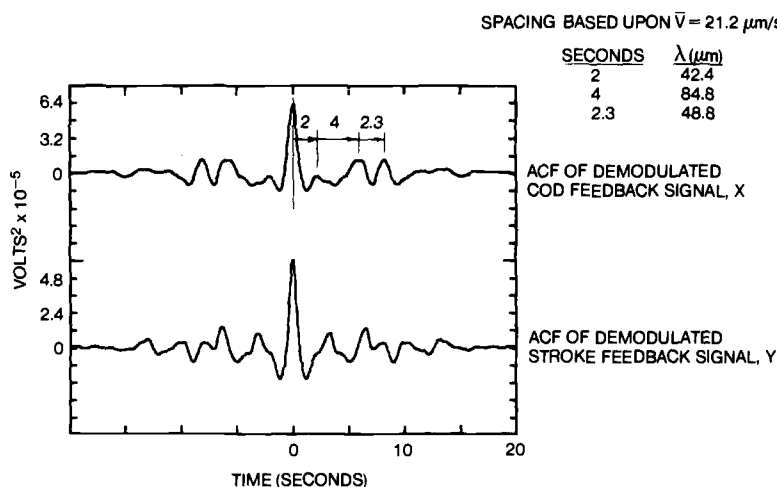


FIG. 16—Autocorrelation functions from the demodulated COD feedback and stroke feedback signals from fatigue testing Specimen 24. Notice most periodic parts of the signal correspond to microstructural spacings based upon crack velocity, $\bar{V}$.

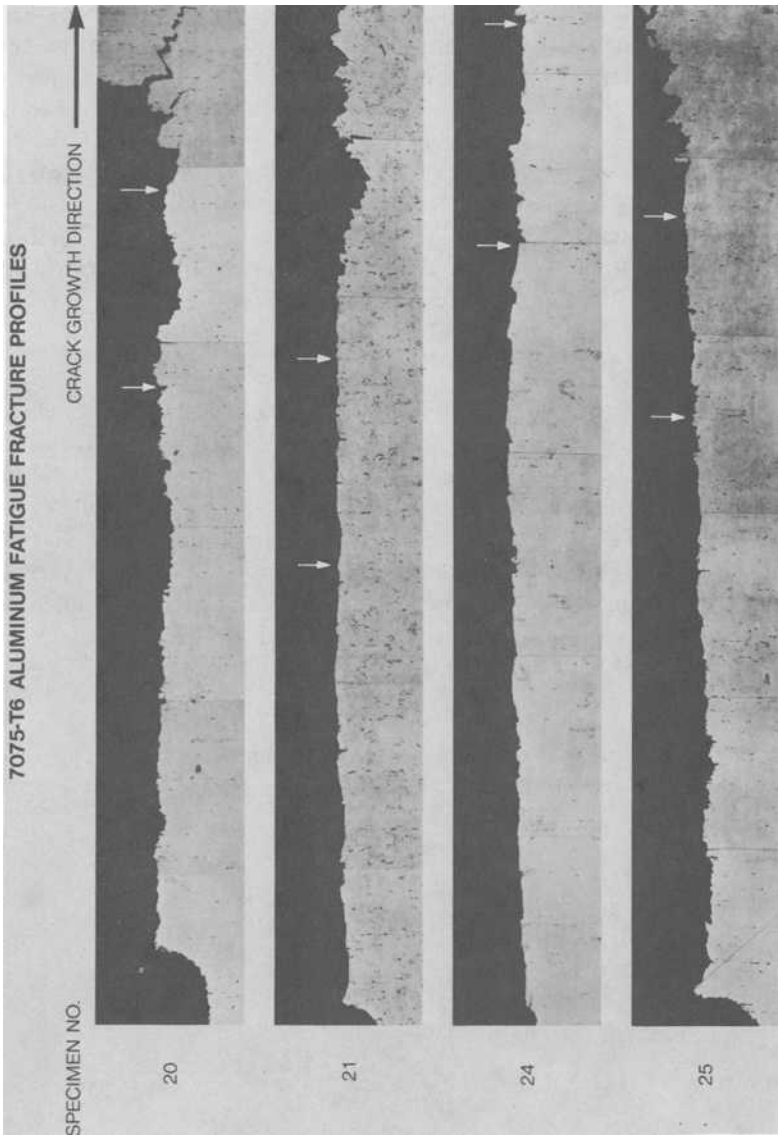


FIG. 17—Composite optical photomicrographs of crack profiles showing the relative topography of the fatigue cracks for the four conditions listed in Table 2. The digitized regions are indicated by arrows.

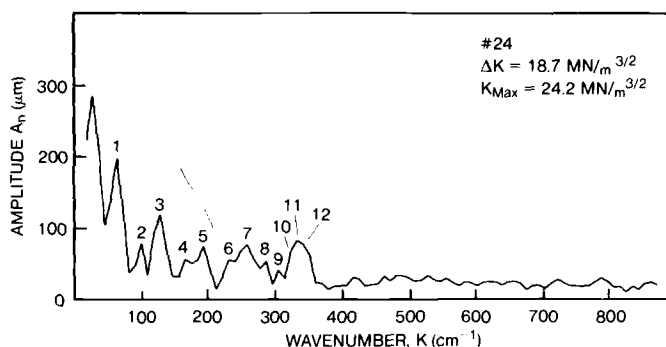


FIG. 18—Fourier spectrum of the digitized crack profile of Specimen 24 shown in Fig. 17. The numbered peaks, wave numbers, and corresponding microstructural spacings are given in Table 4.

Differences Related to ΔK and K_{max} Levels

To examine the versatility of the spectrum analysis technique, fatigue in the same plate of material was compared for the test conditions illustrated in Fig. 8. As indicated earlier, from stereo-pair fractographic analysis, the striation spacings were measured for each test condition. Considering the Paris [20] relationship for crack growth rate, $da/dN = C\Delta K^m$, where c is a constant and m is the exponent required to curve fit the data, the growth rate is expected to change with the variation in ΔK . At similar ΔK levels, one might not have expected $d\lambda/dN$ to decrease from 0.57 to 0.31 $\mu\text{m}/\text{cycle}$ when the mean load was shifted upward, causing the K_{max} value to increase from 24.2 to 33.1 $\text{MN}/\text{m}^{3/2}$ (22.0 to 30.1 $\text{ksi}\sqrt{\text{in.}}$.) When operating at a higher K_{max} value, a large fraction of the total energy is expended in the secondary cracking perpendicular crack

TABLE 4—Summary of peaks present in fracture profile spectrum Specimen 24.

Peak No.	$k(\text{cm}^{-1})$	$\lambda, \mu\text{m}$	ω , Computed ^a Frequency, Hz
1	68.5	146	0.15
2	107.5	93	0.22
3	136	73	0.29
4	178	56	0.38
5	205.3	48.7	0.44
6	244	41	0.52
7	273.9	36.5	0.58
8	303	33	0.64
9	322	31	0.68
10	344.8	29	0.73
11	362	27.6	0.77
12	371	26.9	0.79

^aThe computations were made by using $\bar{\nu} = \omega/k$.

growth direction (S). When a different localized overload is activated, less driving force is available for the striation growth mechanism. By referring to Table 3, it can be seen that, based upon a test frequency of 37 Hz, the local crack velocity, $\bar{v}$, in Specimen 20 was $11.5 \mu\text{m/s}$, whereas the crack velocity in Specimen 24 was nearly twice that rate at $21.2 \mu\text{m/s}$.

Compared at equivalent magnifications, the fractograph from Specimen 20 (shown in Fig. 19, *left*) indicates that more secondary cracking occurred in Specimen 20 than in Specimen 24 (shown in Fig. 19, *right*). The distance over which the fatigue crack moved by striating without longitudinal splitting is significantly less in Specimen 20, which is loaded to a higher K_{max} condition. In Specimen 24 the fatigue crack moved more coherently with the cyclic load over greater distances, which resulted in more rapid crack growth.

A comparison of the fracture profiles in Figs. 20 and 21 reveals differences that were evident when the average K_{mean} was increased with ΔK remaining fixed. The transverse cracking increased concomitantly with the increase in K_{mean} . This yielded a rougher fracture profile resulting from the more frequent linkup of the transverse cracks over shorter distances. The striated regions were broken up into smaller packets because of the increased influence of the microstructure. This phenomenon has a beautiful optical analogue [21] that is known as coherency length, the distance over which a light wave packet travels before it loses coherency with the light source. In the case of fatigue, a particular striation packet moves coherently with the cyclic load until it becomes so strongly influenced by the microstructure that the individual striation packet is broken up. Since this occurs over different regions of the fracture surface, apparently the "striation coherency length" is dependent on the statistical distribution of the microstructural features.

Since the crack in Specimen 20 takes nearly twice as long to traverse a given distance than that in Specimen 24, it is not at all surprising to find that there is a slight shift in the envelope of sideband frequencies for the real-time signals to lower values, as detected by spectrum analysis. The spectra for Specimens 20 and 24 are plotted in Fig. 22. When the individual frequency components are divided into the $\bar{v}$ value of $11.5 \mu\text{m/s}$ for Specimen 20, the critical interparticle and grain boundary spacings that cause crack-tip modulations are reduced in relation to Specimen 24. At a higher K_{max} value, Specimen 20 had a crack front that grew in a larger number of individual packets than that in Specimen 24. The effective distance between secondary cracks was reduced accordingly.

Figure 23 shows fracture profile spectra for Specimens 20 and 24; these should be compared with the two spectra measured for the real-time signals shown in Fig. 22. Evidently, there are similarities between the two spectra, but there are distinct differences too. The fracture surface observations of the specimens revealed that localized overload occurred during crack growth, and this resulted in gross roughening of the fracture surface. Since localized overload occurs randomly from point to point on the fracture surface during fa-

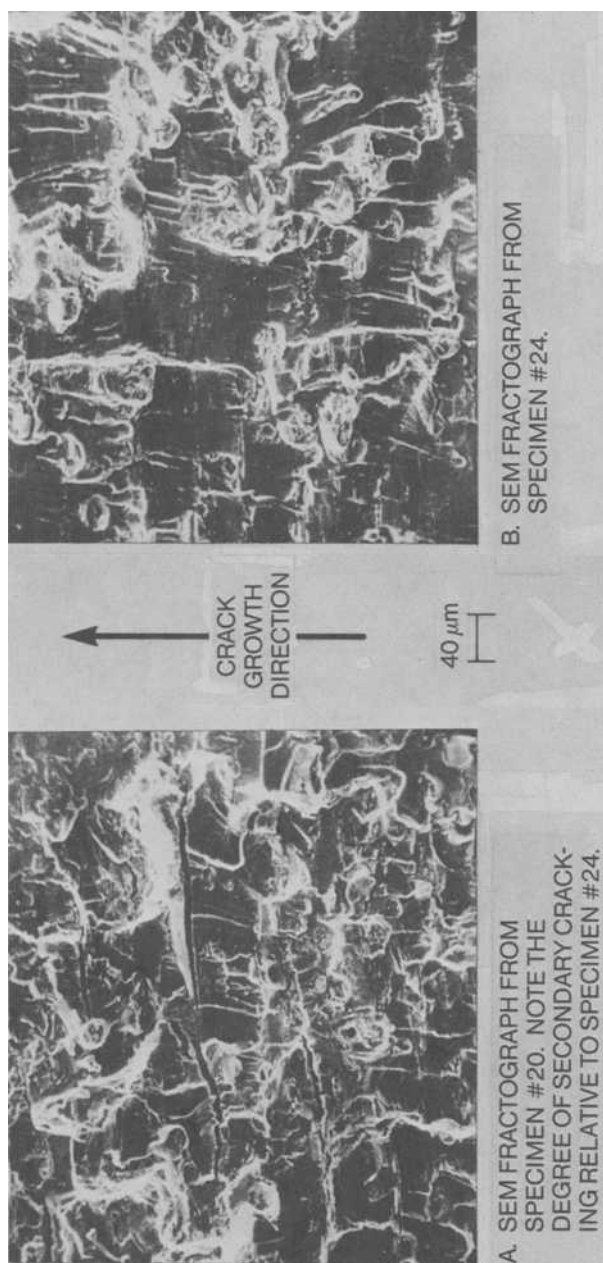


FIG. 19—SEM fractographs from comparable regions of the fatigue fractures in Specimens 20 and 24. The loading conditions are illustrated in Fig. 8. (Left) An SEM fractograph from Specimen 20. Note the degree of secondary cracking in relation to Specimen 24. (Right) An SEM fractograph from Specimen 24.

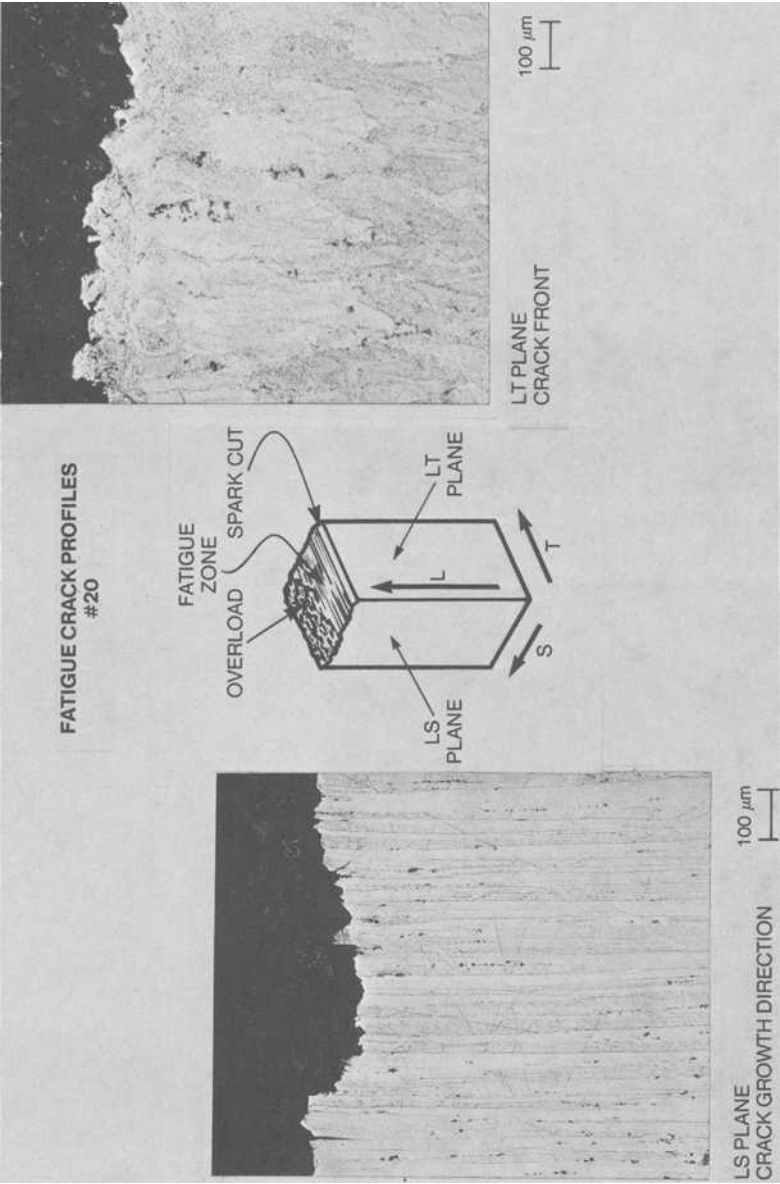


FIG. 20—Etched optical photomicrographs of the fatigue crack profile of Specimen 20. Observations are made parallel to the crack growth direction and as viewed in relation to the advancing crack front.

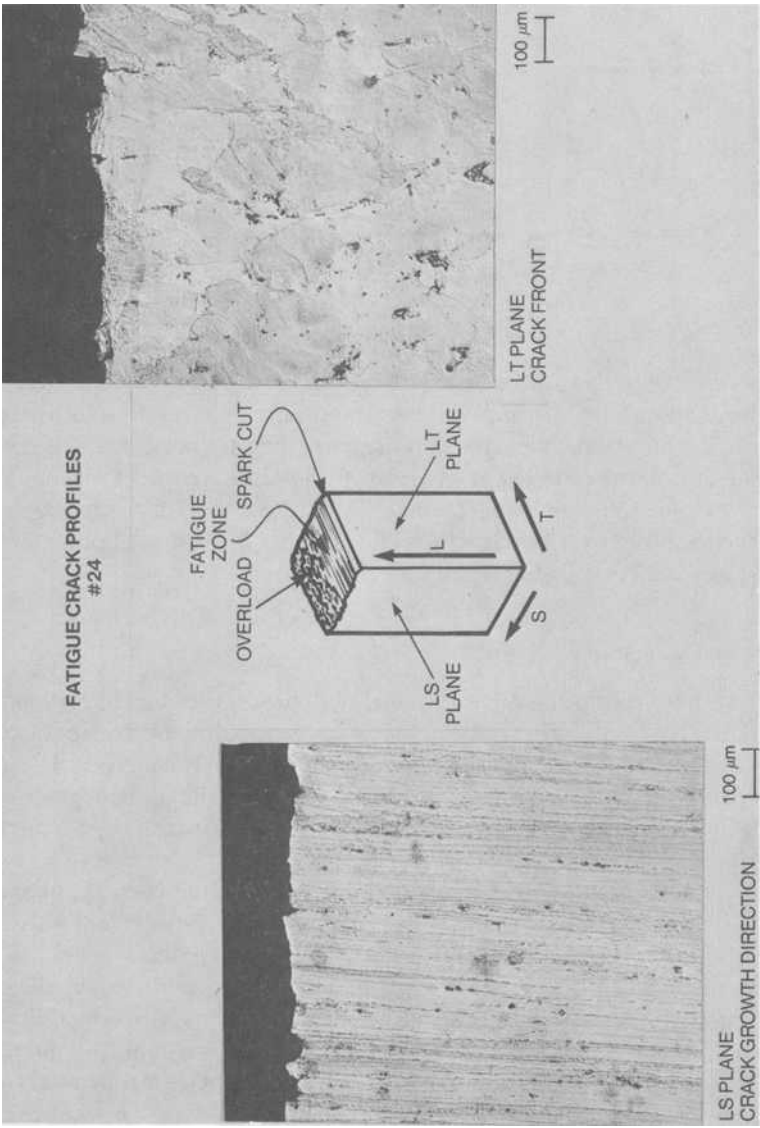


FIG. 21—Etched optical photomicrographs of the fatigue crack profile of Specimen 24. Comparison is made equivalent to Specimen 20 in Fig. 20. Notice that the crack profile of Specimen 24 is flatter than that of Specimen 20.

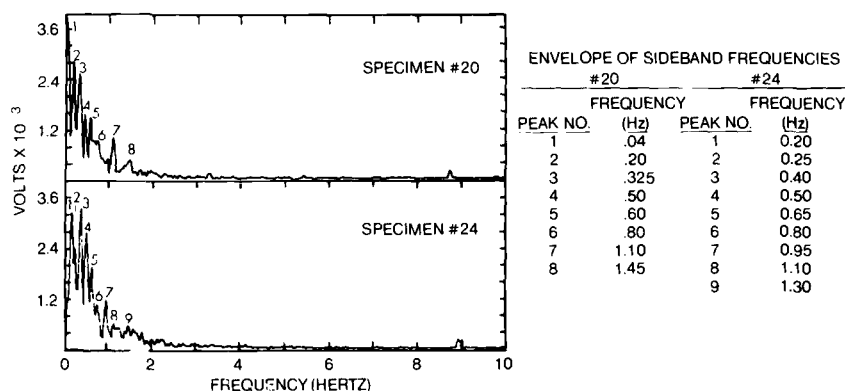


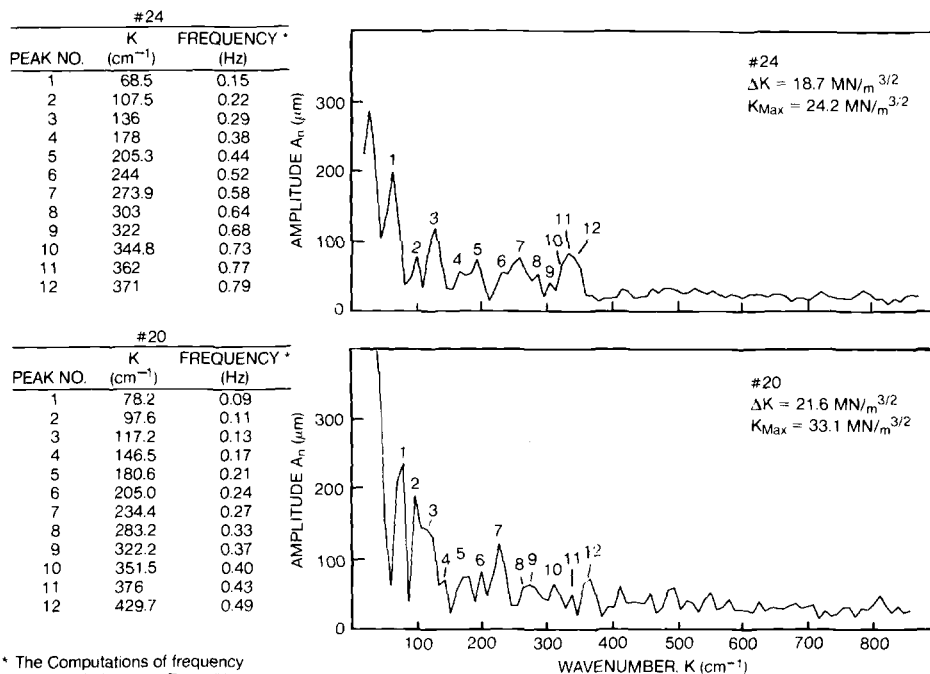
FIG. 22—RMS frequency spectra of demodulated COD feedback signals from fatigue tests of Specimens 20 and 24.

tigue, it would not be detectable by means of our phase-sensitive signal detection method. Spontaneous compliance fluctuations due to random localized crack growth would appear as system noise since they are not generated synchronously during the test. The fracture profile spectra are therefore similar insofar as their features originated from the fatigue test; their differences originate in the localized overload fracture that resulted from $K_{\max}$ approaching K_{Ic} on a localized basis in Specimen 20.

Summary and Conclusions

Real-time analysis of the fatigue crack growth process studied in conjunction with quantitative fractographic analysis has resulted in the development of a new mechanical testing method for studying fatigue. The method, based upon Fourier transform techniques, is able to delineate the influence of microstructural inhomogenieties present at the crack tip during fatigue crack growth.

The new analytical method developed in this program has been applied to the study of fatigue crack growth in a commercial grade Type 7075-T6 high-strength aluminum alloy. Demodulation of the small, dynamic compliance signal present on the cyclic displacement signals was accomplished by using lock-in amplifiers; analysis of the signal was accomplished by using a dual-channel spectrum analyzer. Fatigue crack growth occurs by the commonly observed striation mechanism until a second-phase particle or a weak grain boundary is encountered; at a sufficiently high stress intensity, a secondary crack is nucleated, which serves to interrupt the striated crack advance and to reduce the crack growth rate. The frequency spectrum of the demodulated signals is directly related to the frequency spectrum obtained from the fracture profile.



* The Computations of frequency were made by using $\bar{\nu} = \omega/K$

FIG. 23—Fourier spectra of fatigue fracture profiles of Specimens 24 and 20. The crack profiles are shown in Fig. 17.

Acknowledgments

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Application of Scanning Electron Microscopy for Correlating Fracture Topography and Microstructure

REFERENCE: Nenonen, P., Törrönen, K., Kemppainen, M., and Kotilainen, H., "Application of Scanning Electron Microscopy for Correlating Fracture Topography and Microstructure," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 387-393.

ABSTRACT: A new method for correlating fracture topography and microstructure has been developed. The details of the method, which utilizes scanning electron microscopy, are described. The method is applied in a microstructural analysis of cleavage crack propagation in a bainitic pressure-vessel steel.

KEY WORDS: fractography, microstructure, scanning electron microscopy, fracture mechanisms, materials, materials science

To gain a better understanding of the structure-property relationships and micromechanisms of fracture, a simultaneous observation of the fracture surface and the corresponding fracture profile in the underlying microstructure is of vital importance. The use of scanning electron microscopy (SEM) in fracture surface studies has already gained widespread acceptance. The possibilities of SEM in microstructural studies, however, are less well known.

The application in fracture profile studies is especially useful, as SEM is capable of extending microstructural resolution far beyond that of an optical microscope. Three techniques for preparing specimens suitable for simultaneous viewing have been introduced so far. The first technique is one in which the fracture surface of a specimen is first protected by a suitable material, such as electrolytic nickel [1,2]³ or epoxy [3,4]. The specimen is subsequently sectioned, polished, and etched on a plane perpendicular to the fracture path. This technique suffers from two deficiencies: the removal of the mounting material may damage the fracture surface, and edge rounding may

¹ Head and research engineer, respectively, Metallographic Section, Laboratory of Metallurgy and Mineral Technology, Technical Research Centre of Finland, SF-02150 Espoo 15, Finland.

² Head, Reactor Materials Research Section, and head, Materials Testing Section, respectively, Metals Laboratory, Technical Research Centre of Finland, SF-02150 Espoo 15, Finland.

³ The italic numbers in brackets refer to the list of references appended to this paper.

occur [5]. The second technique utilizes direct electropolishing of the fracture surface to reveal the underlying microstructure. During electropolishing, those areas of the fracture surface that are to be examined in relation to the microstructure are protected by stopoff lacquer [5]. Both methods involve the simultaneous SEM observation of the fracture surface and the corresponding perpendicular microstructural section from a 45-deg angle to both planes. Quantitative dimensional and angular measurements of the fracture profile are, however, difficult to perform using these methods.

An improvement on the previous methods was achieved by the precision-sectioning method [6, 7], which involves photography of the fracture surface before sectioning. This technique was used, however, at relatively low magnifications.

To avoid the deficiencies of the previous techniques, a new specimen preparation and viewing technique for SEM, which gives detailed and highly informative results, was developed. The details of this technique, as well as an application concerning the cleavage fracture of a bainitic steel, are described in the following section.

Method

The method consists of the following steps:

1. A desired fracture surface area (such as 0.2 by 1 mm, which corresponds roughly to 20 photomicrographs at a magnification of $\times 1000$) is viewed and photographed using SEM. The exact location of the area is measured using the goniometer of the specimen stage.
2. The fracture surface is plated electrolytically with nickel [8].
3. A surface perpendicular to the overall fracture surface is mechanically ground and polished to a plane crossing the area that was photographed earlier. A micrometer is used for measuring the amount of grinding.
4. The polished plane is etched conventionally to reveal the microstructure.
5. The specimen is viewed in the scanning electron microscope, and the fracture surface profile that corresponds to a location observed in Step 1 is photographed.
6. The corresponding locations in each series of SEM photomicrographs are identified. If desired, the grinding, polishing, and etching sequences can be repeated, and a new series of photomicrographs can be obtained.

A great advantage of the method arises from the nickel plating of the fracture surface after it has been photographed, but before it is sectioned. The fracture surface is seen untouched on both sides of the profile, and edge rounding is prevented during further specimen preparation. Thus, the fine details of the fracture profile can be photographed for analysis. A disadvantage is the relatively lengthy and tedious specimen preparation and viewing process required. Examples of the results are seen in Figs. 1 and 2.

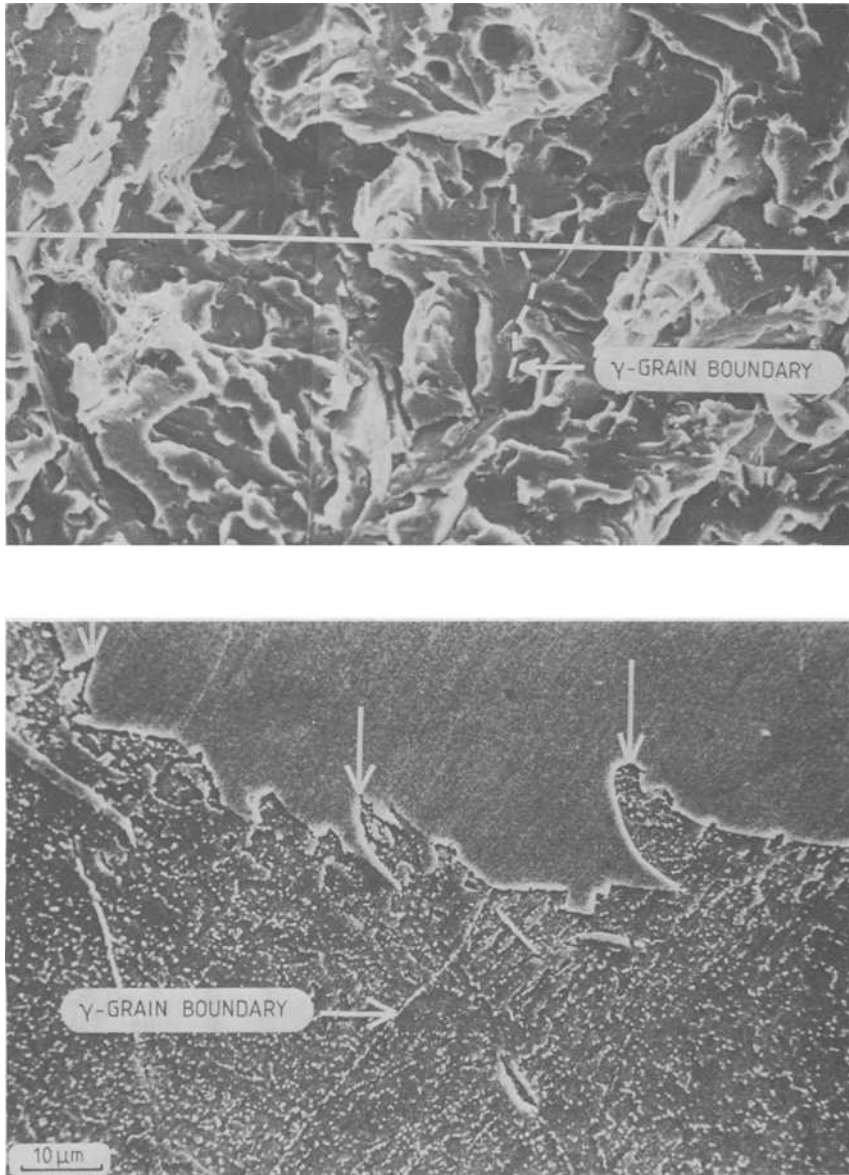


FIG. 1—Scanning electron photomicrographs of the fracture surface and the corresponding fracture profile of a bainitic chromium-molybdenum-vanadium steel. Some equivalent points are marked with arrows. Note the weak effect of a prior austenite grain boundary on the fracture path. Charpy V-notch specimen, tested at 77 K.

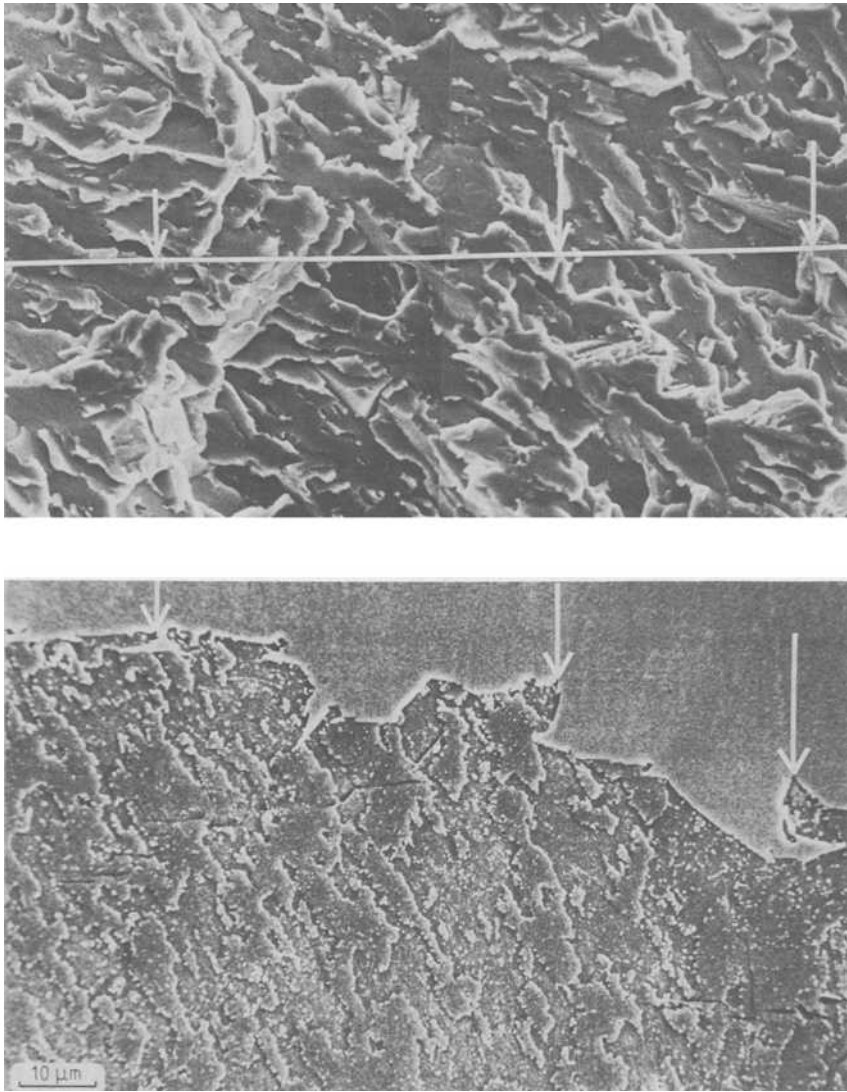


FIG. 2—Scanning electron photomicrographs of the fracture surface and the matching fracture profile. Tension test specimen of a bainitic chromium-molybdenum-vanadium steel tested at 77 K. Note the elongated shape of the packets seen on the cross section as well as the cleavage facets. See the text for further explanation.

Application

The previously described method was utilized in studying the micromechanisms of brittle fracture in a medium-strength chromium-molybdenum-vanadium alloyed pressure-vessel steel. A careful transmission and SEM study of the tempered bainitic microstructure revealed four different grain-size units to be considered in regard to the fracture path [9]. Prior austenite grains were found to be divided into constituents called packets. These packets are the smallest units surrounded by high-angle boundaries. Parallel, or nearly parallel, groups of packets form so-called bundles. The shape of the packets depends on the austenite-ferrite transformation temperature. At higher transformation temperatures cross sections of the packets are irregular, but the packets are definitely elongated. At lower transformation temperatures, the shape becomes platelike. Within a single bundle the long or growth direction of the packets is always approximately parallel to one $\langle 111 \rangle_a$ direction. The orientation relationship between neighboring packets in the same bundle is usually twin orientation; only two ferrite orientations are seen within a single bundle. The packets are further subdivided into parallel laths. The lath boundaries inside a packet are always low-angle boundaries. Further details of the microstructural studies are reported elsewhere [9].

The packets were revealed in SEM photomicrographs because of differential attack by the etchant used (3 percent nital). The most severe etching of the ferritic matrix occurs in the $\langle 111 \rangle$ directions and the least severe in the $\langle 100 \rangle$ directions. The effect of the etching was checked with the etch pit method [1]. Combining this selective etching behavior with the knowledge of the relationship between the elongated packet morphology and the crystallography, an analysis of the fracture path could be made. An example is shown in Figs. 2 and 3.

Figure 2 (*bottom*) shows the packet microstructure revealed by etching. The propagating crack is seen to change direction at every crossing of a packet boundary. Several nonpropagating microcracks are also seen adjacent to the main crack path. These microcracks generally go through one packet. The cracks never propagate along packet boundaries but cross the packets at various angles. Since the $\{100\}$ -type planes, which are the expected cleavage planes in ferritic materials [10–12], are never parallel to the long direction of the packets, it is suggested that the cleavage facets seen on the fracture surface are equivalent to cross sections of the packets cut along one of the three possible $\{100\}$ -type planes. Because of their low-angle nature, the lath boundaries inside a packet do not change the direction of the fracture path. The elongated shape of the cleavage facets and the corresponding perpendicular cross sections of the packets clearly show that the packets are roughly platelike.

The three possible fracture facets belonging to one of the two packet orientations are shown in Fig. 3 (*top*), which is an enlargement of Fig. 2 (*top*). The traces of the corresponding fracture facets in the fracture profile are indicated in Fig. 3 (*bottom*).

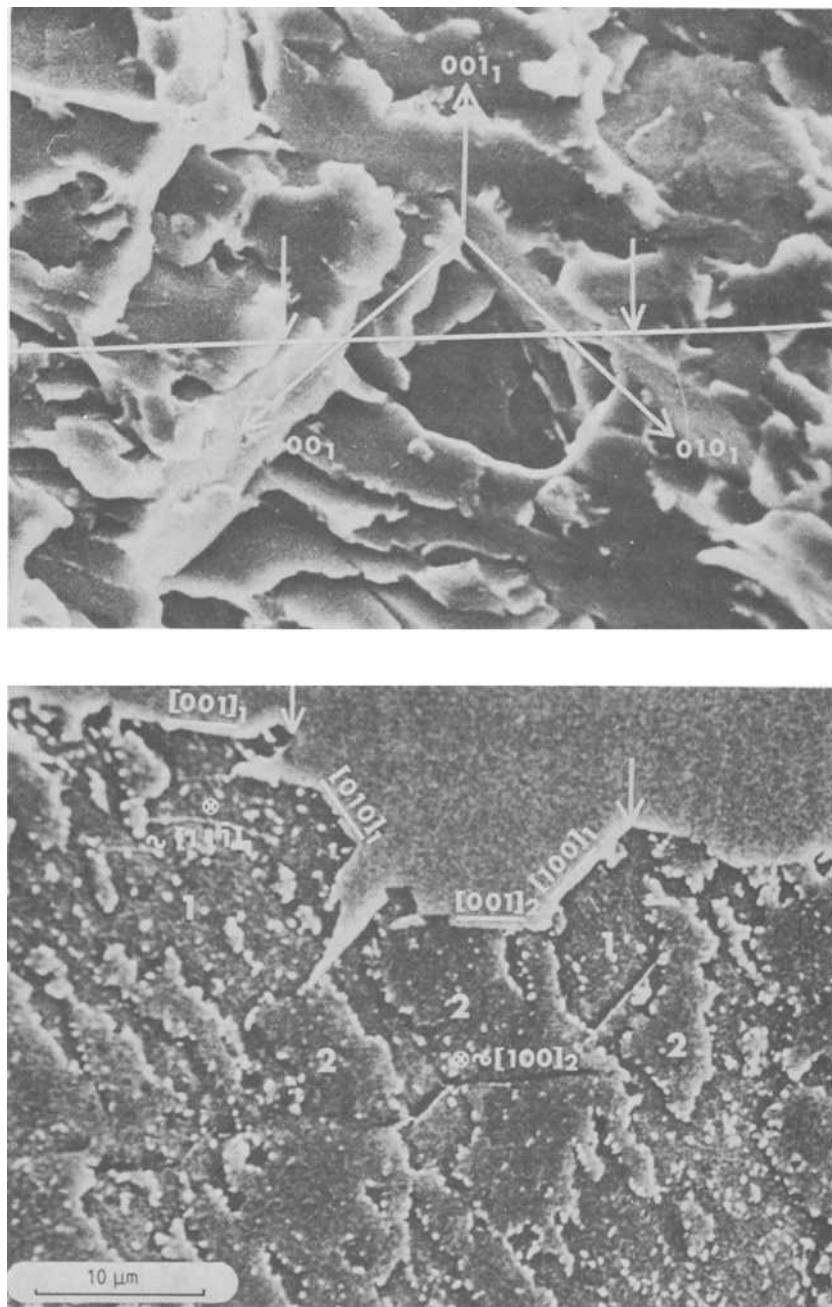


FIG. 3—Photomicrograph of the three fracture planes belonging to one of the two packet orientations (top) and the corresponding traces of the fracture planes in the fracture profile (bottom). This is an enlargement of a portion of Fig. 2.

A comparison of the average sizes of the packets and cleavage facets indicates also that the packet boundaries limit the crack propagation. The average width of the packets is $3.0\text{ }\mu\text{m}$ (as measured from SEM photomicrographs perpendicular to the long direction of the packets), and the average size of the cleavage facets is $3.6\text{ }\mu\text{m}$ (as measured from the fracture profiles on SEM photomicrographs). The angle between the long direction of the packets $\langle 111 \rangle$ and the cleavage plane $\{100\}$ is 35.3 deg . Thus the average length of the trace of the cleavage facet in the fracture profile must be between 3.0 and $3.96\text{ }\mu\text{m}$, which is the case.

Summary and Conclusions

A method for correlating the fracture topography and the microstructure utilizing SEM has been presented. The advantage of the method arises from the fact that the fracture surface can be viewed untouched and, for direct comparison, a matching fracture profile can be generated. The applicability of the method is shown in an analysis of the fracture path in a tempered bainitic steel. This analysis shows that the fracture facet of the bainitic structure equals the bainitic packet size.

Acknowledgments

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M. R. Krishnadev,¹ L. R. Cutler,² G. J. Sojka,² P. Gauvin,³
and G. Hamel³

Characterization of the Fracture Behavior of Fine-Grained High-Strength Low-Alloy (HSLA) Steels and Iron-Base Alloys Under Low-Temperature and Mechanical Environments

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ABSTRACT: This paper discusses the use of different fractographic techniques to characterize the fracture morphology of high-strength low-alloy (HSLA) steels with a variety of fine-grained microstructures and iron-base alloys strengthened by intermetallic precipitates in order to understand the various fracture mechanisms controlling their fracture resistance under low-temperature and mechanical environments. The fractographic techniques that have been used are conventional techniques, scanning electron fractography, sectioning techniques, and a newly developed chemical polishing method in conjunction with the Nomarski interphase contrast technique. The mechanical tests that have been used are tensile, notched-tensile, instrumented impact testing with pre-cracked Charpy V-notch specimens, low-cycle fatigue, and high-cycle fatigue. A comprehensive characterization of the fracture morphology of the HSLA steels developed for arctic applications has resulted. Important features of the fracture (such as "splitting," striations, ductile tearing, quasi-cleavage, microvoid coalescence, unit fracture path, persistent slip bands) have been identified and related to the mechanical behavior of the materials.

KEY WORDS: fractographic techniques, fractography, characterization, high-strength low-alloy (HSLA) steels, fracture mechanisms, materials, materials science

During the last 5 years the authors have been actively involved in characterizing the mechanical properties and the fracture behavior of high-strength low-alloy (HSLA) steels that have been developed for pipeline and other

¹ Associate professor, Department of Metallurgy, Laval University, Quebec, Canada G1K 7P4.

² Graduate students, Department of Metallurgy, Laval University, Quebec, Canada G1K 7P4.

³ Students, Department of Metallurgy, Laval University, Quebec, Canada G1K 7P4.

structural applications in the arctic. In particular, we were investigating the effect of different environments (chemical and mechanical) on the fracture characteristics of these steels in order to (1) gain a better understanding of the operative fracture mechanisms and (2) develop comprehensive fracture photomicrographs under controlled laboratory conditions to serve as a norm for identifying the fracture mode and failure mechanisms occurring under actual service conditions. Such characterization studies would also be useful in establishing the performance limits of these newly developed steels and in pointing out the way to develop steels with improved fracture resistance.

Because of the variety of mechanical tests used (tensile, notched-tensile, precracked Charpy, low-cycle fatigue), some of which involve specimens of complicated geometry, and also because of the variety of microstructures involved, we had to develop new surface preparation and sectioning techniques. These techniques will be described in detail.

In addition to the fracture-characterization studies of commercial HSLA steels for use in the arctic, we have a program to develop steels with improved strength (500 to 700 MPa) and toughness (transition temperature from -40 to -100°C), in which we depend heavily on fractographic techniques to evaluate the effect of composition and heat treatment. In these alloy development studies, we have found the scanning electron microscopy (SEM) of obliquely sectioned specimens to be extremely useful in relating the fracture morphology to the matrix microstructure. This will also be briefly discussed.

Experimental Techniques

Fractographic Techniques

The fractographic techniques that have been used are (1) scanning electron microscopy, (2) the oblique sectioning technique, and (3) a newly developed chemical polishing method in conjunction with the Nomarski interphase contrast technique.

For the oblique sectioning technique, which permits simultaneous observation of the fracture surface, the microstructure, and their interface, the following procedure is used:

1. The fracture surface is coated with several coats of microstop lacquer until the deep cavities are almost filled. The specimen is heated at 60°C for 20 min between coats.
2. The specimen is then sectioned on an Isomet low-speed saw with the fracture and sectioned plane forming a 135-deg angle.
3. The specimen is mounted in Lucite and polished in the normal manner. Texmet paper greatly improves the edge retention during the polishing stage.
4. The specimen is removed from the Lucite. The microstop lacquer is dissolved, and pieces of Lucite that remain trapped in the specimen surface can be removed easily.

The advantages of this technique are that even very rugged fractures can be examined without contaminating the fracture surface, and excellent edge retention can be achieved also.

For the chemical polishing technique, the following procedure is used:

1. The specimen is ground to a 600-grade finish. This can be achieved by either polishing by hand (this is preferable when the specimen has a complex geometry) or using a polishing wheel.
2. The specimen is then swabbed vigorously with cotton dipped in a chemical bath consisting of 80 parts of hydrogen peroxide, 5 parts of hydrofluoric acid, and 15 parts of distilled water.
3. Immediately after the polish, the specimen is rinsed in hydrogen peroxide, then in water, and finally with alcohol and dried.
4. The specimen is then etched with an etchant (3 percent nital) to reveal the microstructure and subjected to mechanical testing in the usual manner (in our case, plate-bending high-cycle fatigue tests).
5. After testing, the specimens can be examined directly without any further surface preparation. The use of the Nomarski interphase contrast during examination greatly enhances the fine surface features (such as slip bands, microcracks, and others).

Mechanical Tests

Mechanical tests that have been used are (1) tensile, (2) notched-tensile, (3) instrumented impact testing of precracked Charpy V-notch specimens, (4) low-cycle fatigue, and (5) high-cycle fatigue tests.

For tensile testing, 6.3-mm diameter specimens with a 25-mm gage length have been used. For notched-tensile testing, the specimens have a 60° notch. For impact testing, full-size (10 by 10 by 55 mm) Charpy V-notch specimens

TABLE 1—Composition of the commercial HSLA steels.^a

Steel Alloy	C	Mn	Cr	Ni	Mo	Cu	Cb
I-Cb-Mo acicular ferrite steel	0.035	1.95	...	...	0.25	...	0.09
II-Cb-Ni-Mo acicular ferrite steel	0.059	1.93	0.024	...	0.428	...	0.065
III-Cu-Ni-Cb polygonal ferrite steel	0.04	0.60	0.73	0.97	0.18	1.18	0.04
IV-High-Cb polygonal ferrite steel	0.03	1.8	...	...	...	...	0.14

^a The balance is iron.

TABLE 2—*Composition of the experimental HSLA steels and iron-columbium alloy.^a*

Steel Alloy	C	Mn	Si	P	Cr	Ni	Ti	Cb	Cu	B
Weathering steel	0.07	1.18	0.72	0.084	0.58	0.59	0.18	0.05	1.23	...
Cu—B	0.039	1.52	0.25	...	...	...	0.035	...	1.79	0.0009
Cu—Ni—Cb	0.020	1.94	0.26	...	...	1.56	...	0.115	1.94	...
Fe—Cb	...	...	...	...	...	...	...	1.0	...	...

^aThe balance is iron.

with a gage length of 13.8 mm have been used. For high-cycle fatigue tests, 355-mm long constant-stress tapered specimens have been used and tested under reverse bending.

Materials

The composition of the commercial HSLA steels used in the study is shown in Table 1, and Table 2 shows the composition of the experimental steels and iron-niobium alloys under development.

Different physical metallurgy principles, as well as different technologies, are used in the production of these HSLA steels, which have been developed mainly for arctic pipeline applications. In the acicular ferrite steels, molybdenum is added to retard the $\gamma \rightarrow \alpha$ transformation so that highly substructured acicular ferrite rather than polygonal ferrite is formed. These steels also require controlled rolling and controlled cooling. In contrast, the other two commercial steels require a somewhat less rigid rolling schedule and are air cooled from the finish-rolling temperature. In the copper-nickel-columbium steel (Steel III), copper is used for precipitation strengthening. In Steel IV, high columbium levels are used to obtain large carbonitride particles which refine the grain size. Figure 1a shows the optical microstructure of the columbium-molybdenum acicular ferrite steel, and Fig. 1b shows the optical microstructure of the high-columbium HSLA steel.

Results and Discussion

In tensile testing as well as impact testing, we have carried out an in-depth study of the mode of formation and the morphology of "splits" (that is, separations that open up parallel to the rolling plane in tensile and impact testing). The study was prompted by the fact that most of the newly developed HSLA steels show splits, and a better understanding of the split formation mechanism, as well as the effect of splits on the fracture resistance, is needed [1, 2].⁴

⁴The italic numbers in brackets refer to the list of references appended to this paper.

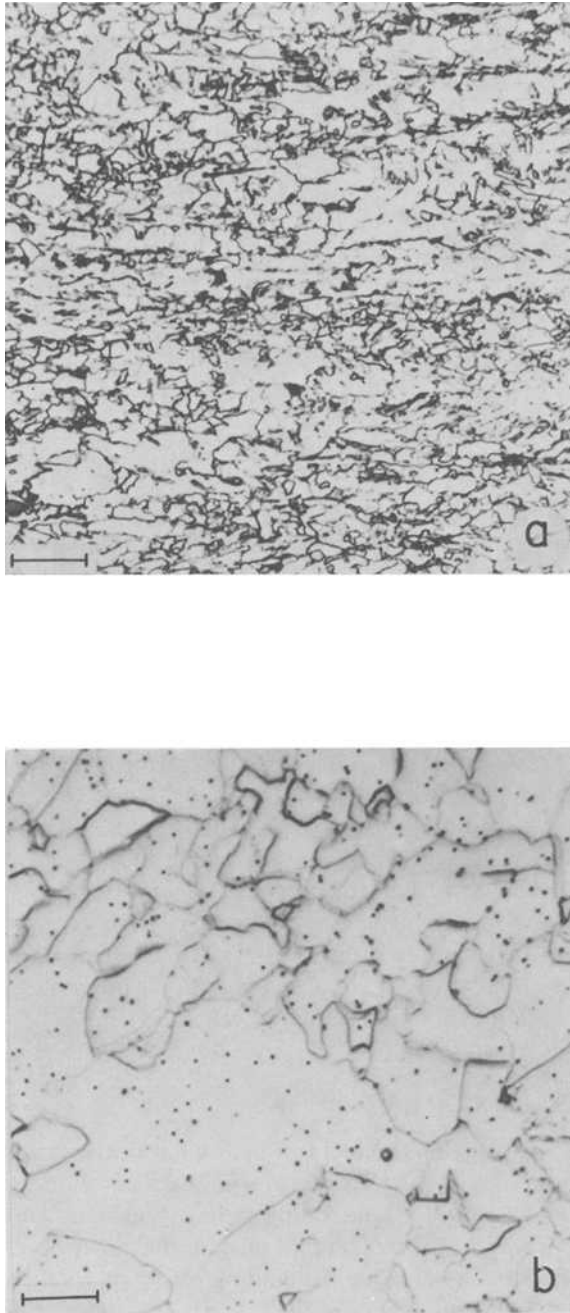


FIG. 1—Optical microstructures of commercial HSLA steels: (a) columbium-molybdenum acicular ferrite steel (scale mark indicates $20\text{ }\mu\text{m}$); (b) high-columbium HSLA steel (scale mark indicates $10\text{ }\mu\text{m}$).

Tensile Tests (Unnotched and Notched)

Figure 2 illustrates the effect of low temperature and notches on the tensile fracture of the columbium-molybdenum acicular ferrite steel (Steel I). In the unnotched specimen (Fig. 2a) broken at -115°C , extensive splitting, as well as ductile dimples, is evident (Fig. 2b). The presence of many ductile dimples even at -115°C attests to the excellent ductility of this acicular ferrite steel; the elongation at fracture at this temperature was over 30 percent. It may also be noted that split walls show considerable tearing with traces of quasi-cleavage areas and a network of elongated dimples (see arrows in Fig. 2b), which indicates that splits need not be composed entirely of the cleavage type of fracture,

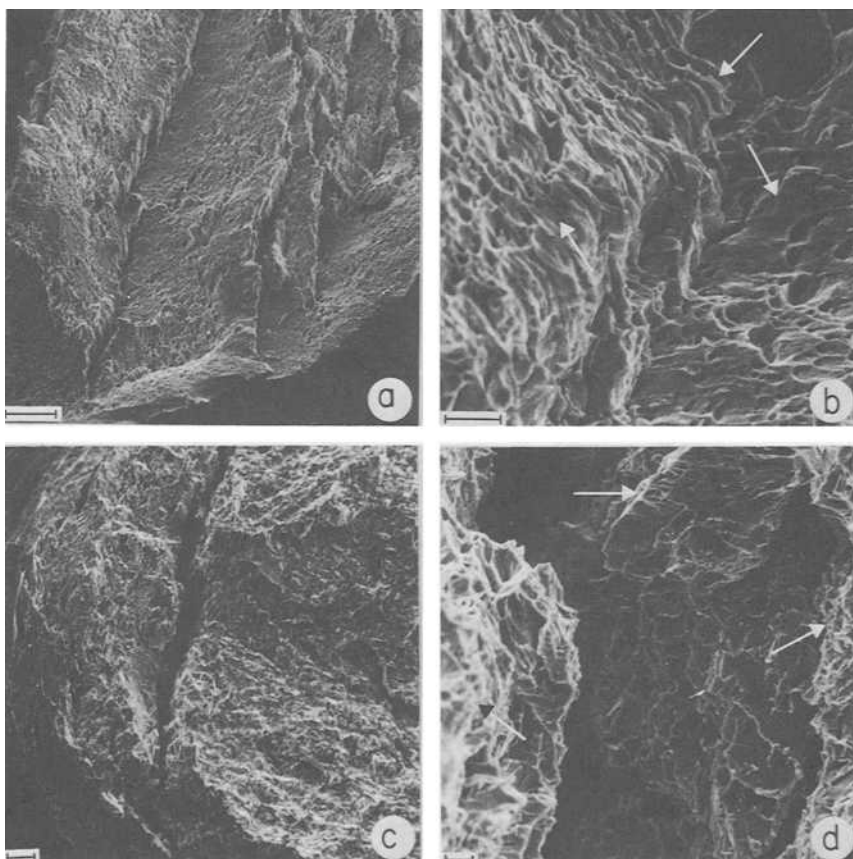


FIG. 2—The effect of temperature and notch on fracture morphology, in particular the “splitting” of columbium-molybdenum acicular ferrite steel tested at -115°C . (a) SEM fractograph of a tensile specimen (scale mark indicates $100\text{ }\mu\text{m}$); (b) SEM fractograph of a tensile specimen (scale mark indicates $10\text{ }\mu\text{m}$); (c) SEM fractograph of a notch tensile specimen (scale mark indicates $100\text{ }\mu\text{m}$); (d) SEM fractograph of a notch tensile specimen (scale mark indicates $100\text{ }\mu\text{m}$).

as is generally reported, but rather may be composed of mixed types (cleavage and ductile).

In the presence of notch, splitting is considerably reduced (see Fig. 2c), and there is an increase in the proportion of the transgranular cleavage fracture mode (see Fig. 2d). However, ductile dimples are also apparent (see the arrows in Fig. 2d), which attests to the inherent ductility of the acicular ferrite matrix. This ductility is also borne out by the fact that the notch sensitivity ratio (NSR) values (that is, the ratio of the strength of the notched specimen to the unnotched specimen) were quite impressive (>1.4).

By comparing the split-wall morphology in Fig. 2b with that in Fig. 2d, it is evident that notch has increased the propensity for transgranular cleavage. Thus, split morphology depends upon the chemical (that is, temperature) and mechanical environments.

Slow-Bend and Impact Tests

We have also examined the split morphology of specimens subjected to slow-bend and impact tests with a view to studying the effect of strain rates on its formation. In both cases, splitting occurred (see Fig. 3a and b) at all temperatures except at very low temperatures ($<-120^{\circ}\text{C}$), when the specimens were completely brittle. Thus the occurrence of splitting appears to be independent of the strain rate. It may also be noted that the splits are separated by areas profuse with ductile dimples, which indicates that splitting is not a continuous process but an intermittent one. It is also worth noting that split walls show a different morphology (tunnel-type secondary splits; see arrows in Fig. 3b) than that observed in tensile testing.

To examine the mode of split formation, precracked Charpy V-notch specimens of columbium-nickel-molybdenum acicular ferrite steel (Steel II) were broken in the impact tester at different temperatures. These were then sectioned, polished, and etched. Figure 4 shows the relevant scanning electron photomicrographs, with Fig. 4a and b tracing the beginning of the splits. It is evident from these photographs that splits form as sharp microcracks that link up with the continued application of the load by a ductile deformation of the ligaments between the splits. For example, in Fig. 4b sharp microcracks that are present in the initiation area of a split may be observed (as at Point A). Microcracks linking up and expanding into splits may be observed (as at Points B and C). Figure 4c indicates that splits formed at low temperatures show transgranular cleavage with evidence of tearing.

It has been suggested that voids occur well in advance of the primary fracture and nucleate splits, which propagate at least concurrently with primary fracture or just ahead of it [3]. However, our fractographic work indicates that splits are formed by the nucleation of several microcracks and their subsequent link-up with continued deformation, this link-up taking place by a basically ductile deformation mechanism.

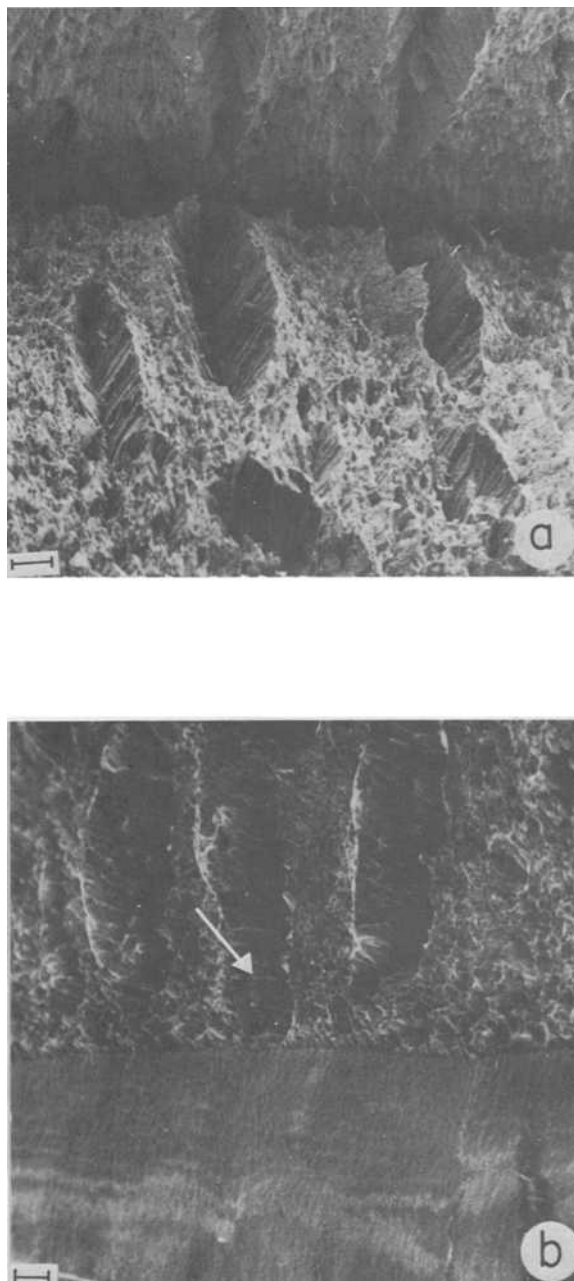


FIG. 3—The effect of the strain rate on split morphology of columbium-molybdenum acicular ferrite steel tested at room temperature: (a) SEM fractograph of a slow-bend specimen (scale mark indicates 200 μm); (b) SEM fractographs of a precracked Charpy V-notch specimen (scale mark indicates 200 μm).

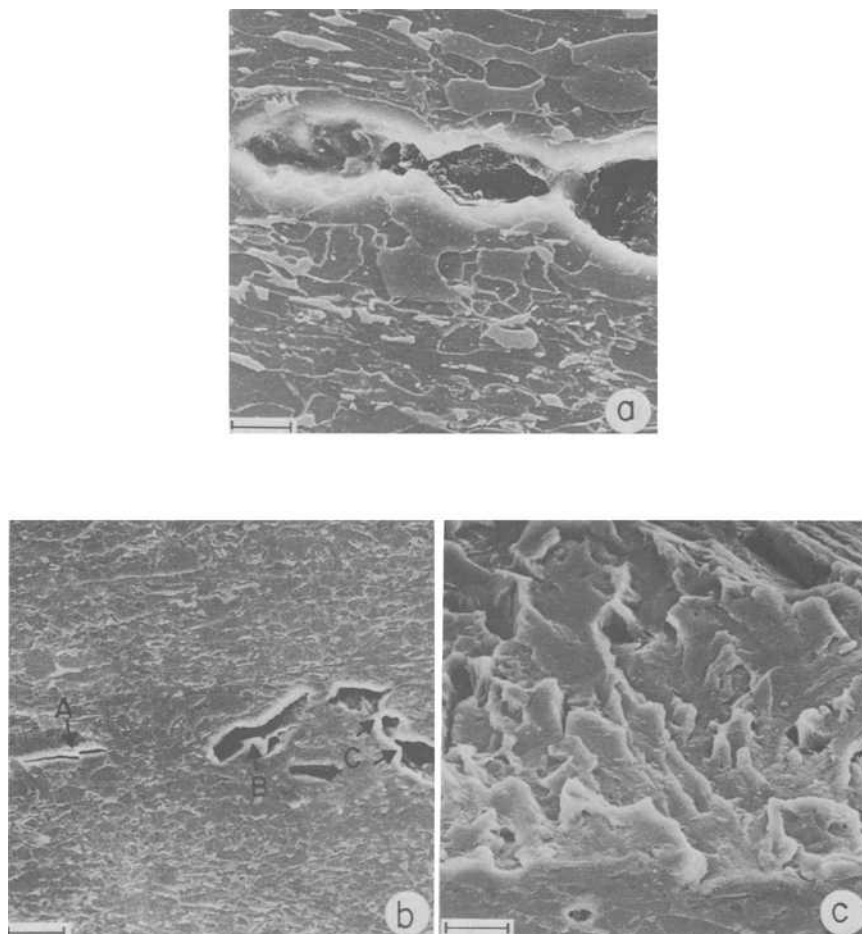


FIG. 4—Scanning electron microscope fractographs of oblique-sectioned Charpy specimens of columbium-nickel-molybdenum acicular ferrite steel tested at different temperatures, showing the mode and nature of the split formation: (a) at room temperature (scale mark indicates 5 μm); (b) at -60°C (scale mark indicates 20 μm); (c) at -100°C (scale mark indicates 10 μm).

Fracture Toughness

Apparent fracture toughness, K_{I_d} , values have been calculated for all four steels, and the variation in K_{I_d} may be related qualitatively (as indicated in Fig. 5) to the changes in the fracture mode: high fracture toughness values corresponding to a predominantly ductile mode of fracture (Fig. 5a), moderate fracture toughness values corresponding to a mixed type (ductile plus cleavage) of fracture (Fig. 5b), and very low fracture toughness values corresponding to a predominantly cleavage type of fracture (Fig. 5c).

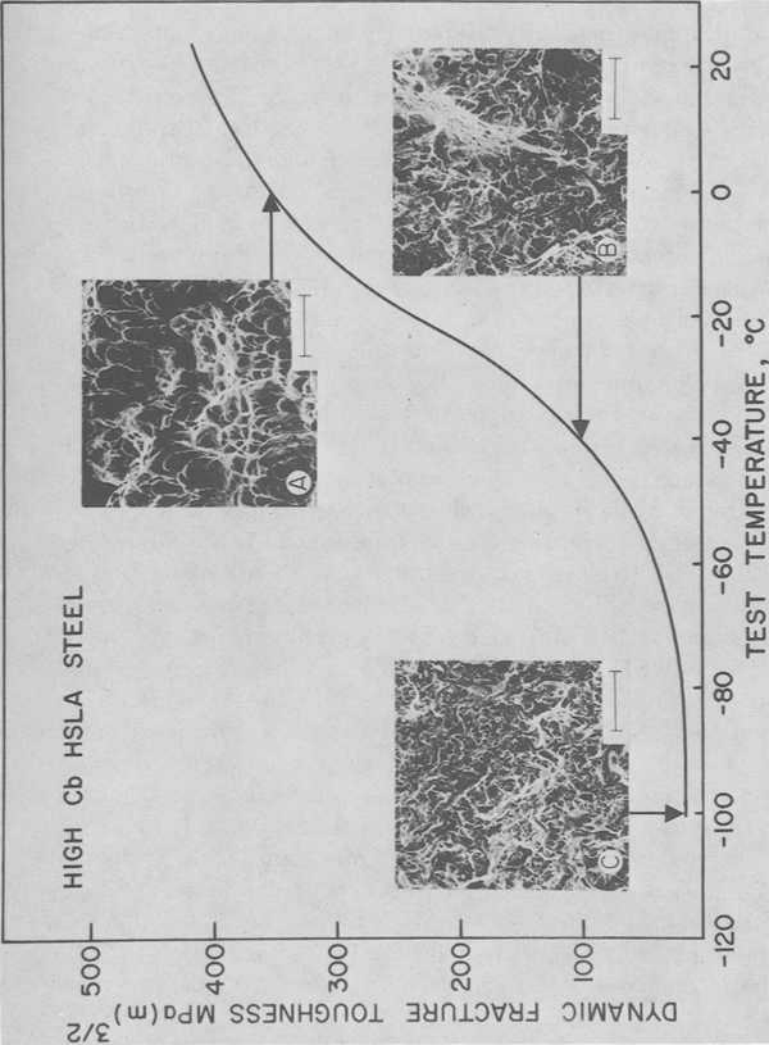


FIG. 5.—The relationship between fracture morphology and apparent dynamic fracture toughness (high-columbium HSLA steel): (a) the scale mark indicates 100 μm ; (b) the scale mark indicates 100 μm ; (c) the scale mark indicates 100 μm .

Low-Cycle Fatigue

In the literature there is hardly any information on the low-cycle fatigue fracture morphology of these commercial HSLA steels. We have investigated the effect of strain level and temperature on the fracture morphology by extensive scanning electron microscopy. Our fractographic work indicates that all four steels exhibit similar fracture characteristics—a mixed mode of fracture involving different fracture mechanisms, namely, (1) fatigue striation formation, (2) transgranular cleavage, (3) microvoid coalescence, (4) decohesion of the particle–matrix interface, (5) shearing of inclusions, and (6) tearing. Similar mixed-type fracture modes have been observed in high-cycle fatigue specimens [4]. Thus, fatigue initiation and propagation do not take place by a single mechanism but by a combination of several mechanisms, and the fracture topography is quite complex compared with that observed in pure metals. These points will be emphasized with some typical fractographs of the columbium-molybdenum acicular ferrite steel (Figs. 6 and 7).

Figure 6a shows the general appearance of the low-cycle fatigue fracture at low strain levels (0.002 strain amplitude). Figure 6b shows the initiation area. It is evident that numerous cracks originate at the surface. Some of these may have been nucleated at inclusions (as at Point A). Pores or voids running adjacent to the crack (see the arrows in Fig. 6b) may also be noted. Figure 6c shows that fracture proceeds by transgranular cleavage as well as by striation formation. Extensive secondary cracking may also be noted. The striations are highly localized (see the arrows in Fig. 6c), and some of these have the characteristic “tire track” morphology. These could well be classic tire tracks, as the steel has columbium (carbon, nitrogen) particles precipitated in the matrix: tire tracks are formed when small second-phase particles are detached and buried in a film of metal dust, which moves as the crack propagates [5]. Figure 6d indicates that fracture propagates in different plateaus. Figure 6e shows the central area of the specimen. It exhibits basically quasi-cleavage features with extensive secondary cracking. There are no well-defined striations. A cracked inclusion and the void associated with it may be seen (at Point A in Fig. 6e). Very fine pores or voids occur at the beginning or along some of the secondary cracks (as at Point B), which indicates that these cracks may be caused by the formation and subsequent linking up of the pores.

Figure 7 shows the effect of increasing strain level (strain amplitude = 0.0181) on the fracture morphology of the acicular ferrite steel. Well-defined striations are present to a greater extent in the initiation area (see Fig. 7a). Areas showing quasi-cleavage features, as well as voids associated with inclusions and second-phase particles, may also be seen in the center of the specimen (see Fig. 7b). Ductile dimples separated by regions of transgranular cleavage and ductile tearing (see Fig. 7c) are evident in the final fracture region.

To relate the fracture characteristics to the underlying microstructure, some of the specimens fractured in low-cycle fatigue have been sectioned and exam-

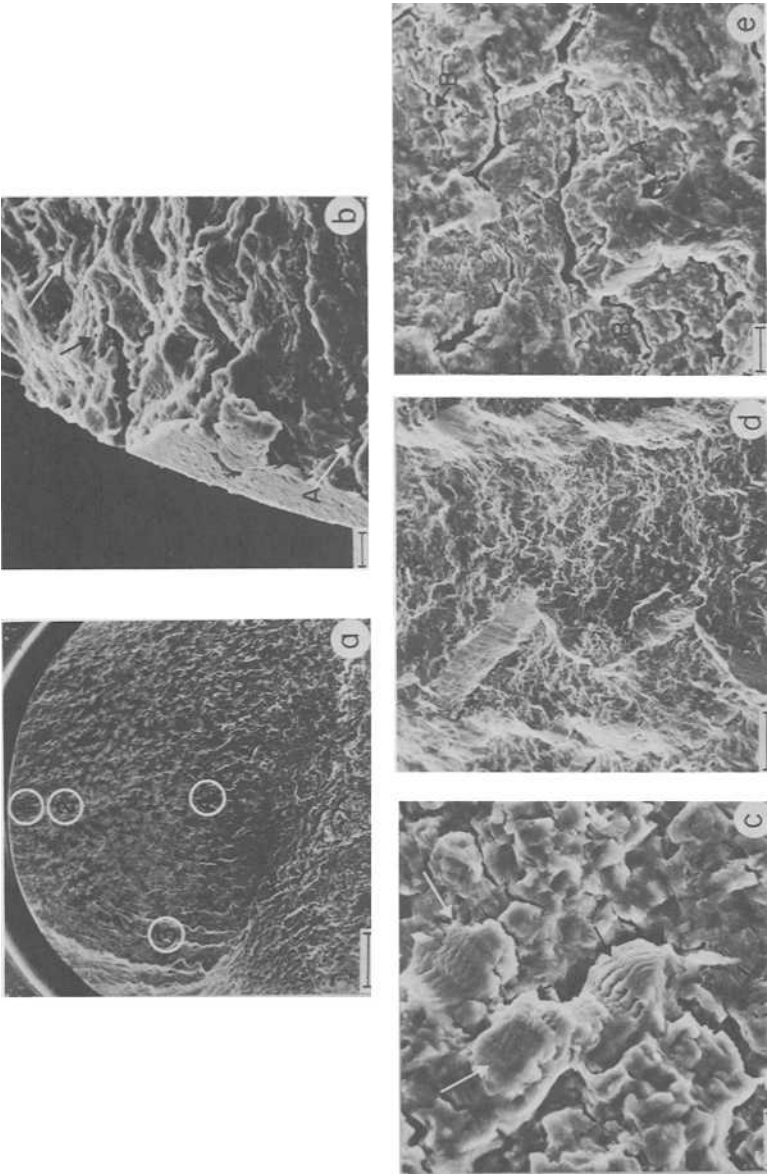


FIG. 6—Scanning electron microscope fractographs of columbium-molybdenum acicular ferrite steel subjected to a low-cycle fatigue (strain amplitude = 0.002), illustrating the complexity of the fracture mechanisms involved: (a) overall fracture topography (scale mark indicates 1000 μm); (b) details from the initiation region (the area indicated as 1) (scale mark indicates 10 μm); (c) Region 1 (scale mark indicates 10 μm); (d) Region 2 (scale mark indicates 10 μm); (e) Region 3 (scale mark indicates 100 μm); (f) Region 4 (scale mark indicates 25 μm).

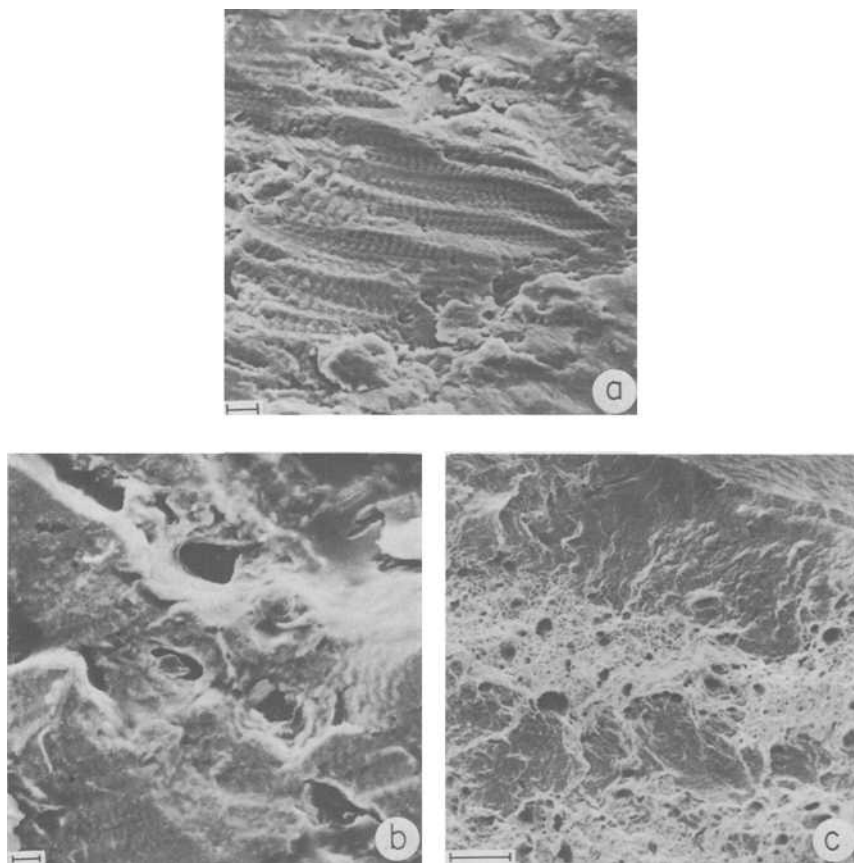


FIG. 7—Scanning electron microscope fractographs of columbium-molybdenum acicular ferrite steel low-cycle fatigued at a higher strain amplitude of 0.0181: (a) initiation area (scale mark indicates 10 μm); (b) center of specimen (scale mark indicates 10 μm); (c) final fracture zone (scale mark indicates 100 μm).

ined (see Fig. 8). In Fig. 8, the crack morphology of the columbium-molybdenum acicular ferrite steel is compared with that of a high-columbium HSLA steel, both steels having been tested at -60°C with a strain amplitude of 0.004. The acicular ferrite steel shows numerous surface cracks as well as the ability to deform plastically, whereas the high-columbium steel shows a continuous outer rim with little evidence of plastic deformation and cracking. These differing fracture characteristics may account for the differing fatigue resistance of the steels; for this condition of testing, the life of the acicular ferrite steel was twice that of the high-columbium steel (15 000 versus 7 000 cycles).

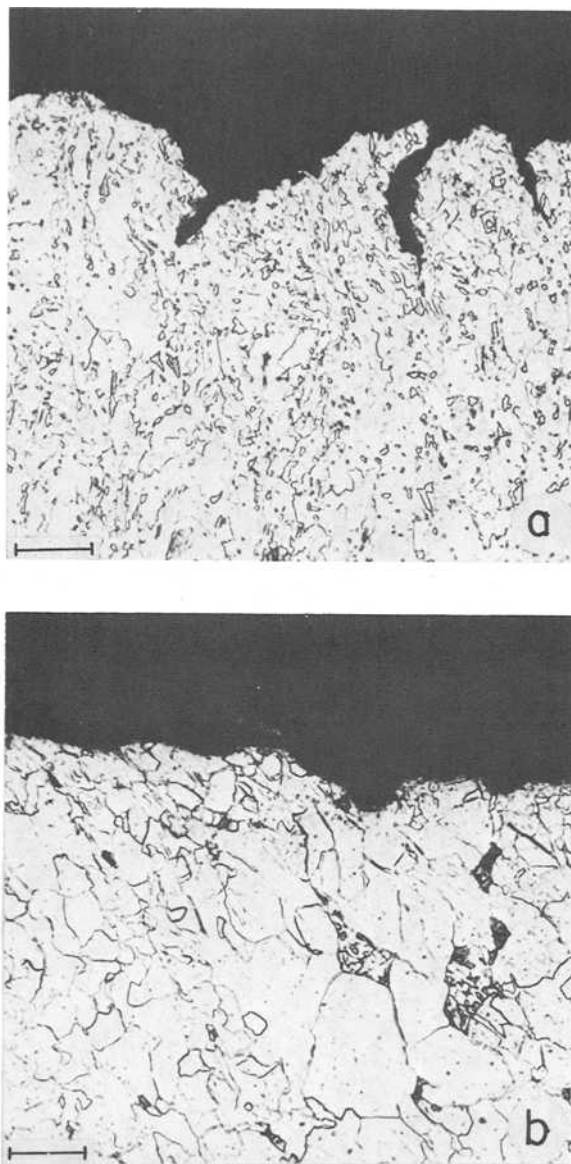


FIG. 8—The effect of the microstructure on the crack morphology of HSLA steels tested at -60°C with a 0.004 strain amplitude: (a) columbium-molybdenum acicular ferrite steel (scale mark indicates $20\text{ }\mu\text{m}$); (b) high-columbium HSLA steel (scale mark indicates $20\text{ }\mu\text{m}$).

High-Cycle Fatigue

In the literature there are hardly any studies of the fatigue initiation mechanisms of these complex commercial HSLA steels. To overcome this drawback, we have evaluated the effect of the stress level and temperature on the nature of slip-band formation and its effect on the fatigue behavior. We will report some preliminary results to illustrate the usefulness of the chemical polishing technique that we have developed to study the fracture mechanisms in HSLA steels. Figure 9 shows some representative photomicrographs. It compares the slip morphology of polygonal ferrite steel (copper-nickel-columbium steel) (Fig 9a and b) with that of acicular ferrite steel (Fig. 9c). Figure 9a shows the main fatigue crack, which is transgranular and associated with slip bands (as

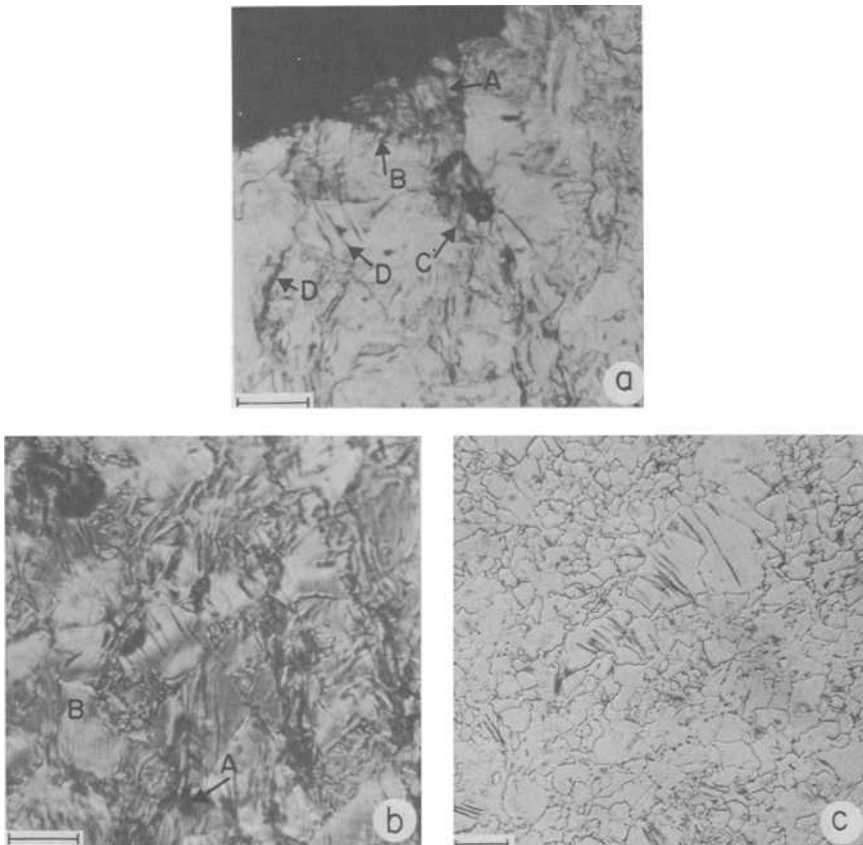


FIG. 9—The effect of the microstructure on the slip-band formation in commercial HSLA steels (chemical polishing method in conjunction with Nomarski interphase contrast): (a) copper-nickel-columbium ferritic steel (scale mark indicates 20 μm); (b) copper-nickel-columbium ferritic steel (scale mark indicates 20 μm); (c) columbium-nickel-molybdenum acicular ferrite steel (scale mark indicates 13 μm).

at Point *A*) and microcracks (as at Point *B*). Slip bands radiating from an inclusion (at Point *C*) and cracks initiated at persistent slip bands may also be seen (as at Point *D*). Figure 9*b* shows the area away from the main crack. The diffuse nature of the slip distribution is evident. Persistent slip bands (PSB) and microcracks initiated along them may also be seen (as at Point *A*). Very fine slip making is evident in some grains (as at Point *B*).

In contrast to the general slip distribution observed in the copper-nickel-columbium ferritic steel, the columbium-nickel-molybdenum acicular ferrite steel shows highly localized slip bands, which are confined mainly to large grains (see Fig. 9*c*). Thus, slip morphology appears to depend on the nature of the matrix, although fatigue cracks initiate at persistent slip bands, conforming to the classical theory of fatigue-crack initiation.

Alloy Development Work

Copper-Columbium-Titanium-Phosphorus Weathering Steel—In general, if phosphorus levels higher than 0.04 percent are used, embrittlement results, and it is very difficult to get low transition temperatures. However, we have found that by resorting to a controlled-rolling and cooling practice, a bainitic matrix can be achieved that is relatively more immune to embrittlement than a ferritic matrix. This is evident from Fig. 10*a* and *b*, which show the fracture morphologies of ferritic and bainitic specimens aged for 24 h at 550°C. The ferritic specimen shows intergranular fracture, whereas the bainitic specimen shows ductile dimples.

Copper-Boron HSLA Steel—There is a lot of controversy regarding the effect of boron on the toughness of structural steels. However, we have found that by controlling the amount of boron and the heat treatment, strength levels of over 690 MPa, with transition temperatures lower than -40°C, can be realized. Again, this observation can be interpreted in terms of the effect of boron on “the effective grain size” [6]. This is illustrated more clearly in Fig. 10*c* and *d*. In comparing the figures, it is evident that martensite packets in the quenched and tempered specimen deflect the fracture more effectively (see the arrows in Fig. 10*d*) than the ferrite grains (see the arrows in Fig. 10*c*) in the air-cooled specimen.

Iron-Niobium Alloy—Another project that we have undertaken is the development of iron-base alloys strengthened by intermetallic precipitates for low-temperature applications. The advantage of strengthening by intermetallic precipitates over strengthening by hard metal carbides is that the intermetallic precipitates have ductility, which results in a propagating crack in their path being arrested due to stress relaxation. However, development work has been hampered by the fact that these intermetallic precipitates [for example, iron-niobium intermetallic compound (Fe_2Nb), and iron-tantalum intermetallic compound (Fe_2Ta)] tend to segregate to grain boundaries and subboundaries, which reduces the toughness. We have found that by controlled-rolling tech-

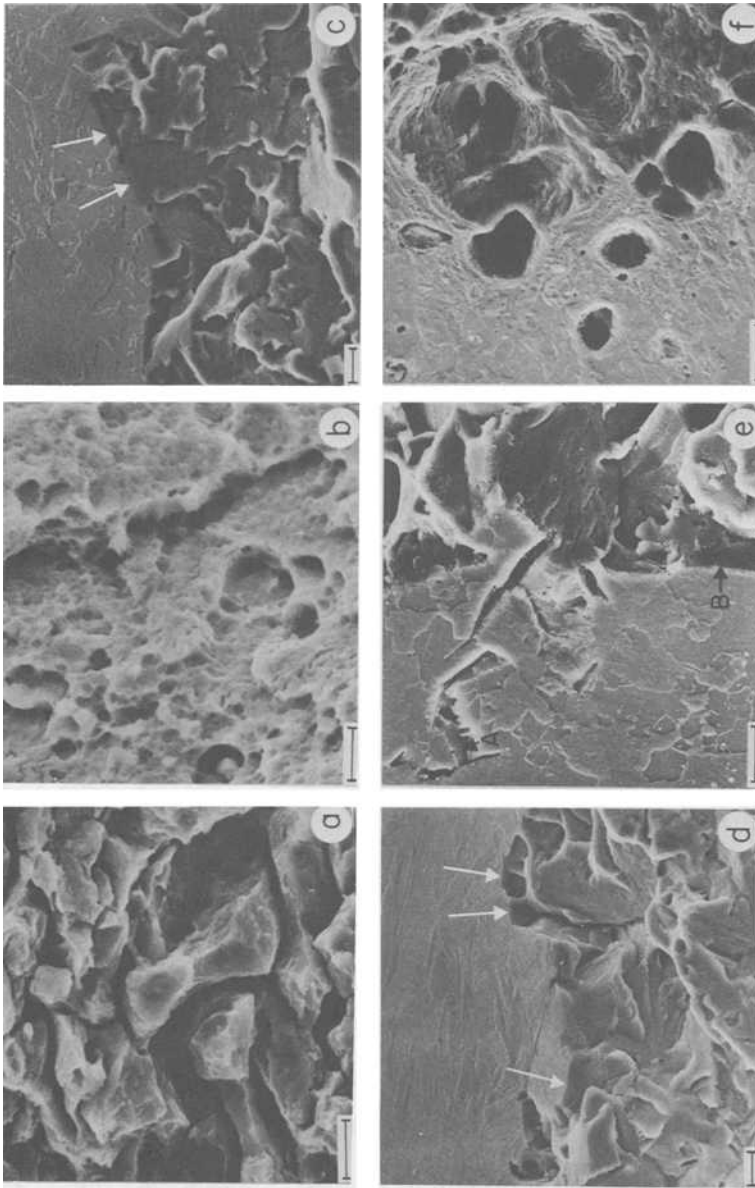


FIG. 10—Scanning electron microscope fractographs of broken Charpy V-notch specimens to illustrate the use of fractographic techniques in alloy development work: (a) and (b) are copper-columbium-titanium-phosphorous weathering steel fractured at 0°C : (a) ferrite (scale mark indicates $5\mu\text{m}$); (b) bainite (scale mark indicates $5\mu\text{m}$). (c) and (d) are copper-boron HSLA steel fractured at -80°C : (c) normalized at 950°C (scale mark indicates $10\mu\text{m}$); (d) re-austenitized at 950°C , water-quenched, and tempered for 1 h at 600°C (scale mark indicates $10\mu\text{m}$). (e) and (f) are iron-niobium alloy fractured at -196°C : (e) air-cooled from 1380°C (scale mark indicates $10\mu\text{m}$); (f) controlled rolled (scale mark indicates $10\mu\text{m}$).

niques, which are normally used for HSLA steels, the ductility and toughness of iron-niobium alloys can be improved tremendously. For example, even at -196°C the controlled-rolled alloy is tough, whereas the alloy that is air cooled from the soaking temperature of 1380°C has poor toughness even at moderately low temperatures (for example, 0°C). Again, scanning electron fractography, in conjunction with the oblique sectioning technique, indicates the reasons for this (see Fig. 10e and f).

Figure 10e and f shows the fracture characteristics of the iron-niobium alloy as a function of processing and heat treatment. Sharp cleavage cracks along the grain boundaries can be seen in Fig. 10e (as at Point A). These are probably due to the segregation of the Fe_2Nb precipitates. A long fracture path can also be seen at Point B. In contrast to this, the controlled-rolled alloy (Fig. 10f) shows extremely fine-grained structure and ductile dimples even at -196°C , which, attests to its inherent toughness.

Conclusions

Different fractographic techniques have been used effectively to characterize the fracture behavior of commercial HSLA steels and experimental alloys. The following important conclusions can be drawn from the characterization study:

1. Splits occur in specimens fractured in a variety of mechanical tests (such as tensile, slow-bend, impact, and fatigue tests). The chemical composition, temperature, strain rate, and loading mode affect the split morphology. The occurrence of splits, however, is independent of the microstructure.
2. On the Charpy V-notch specimens, the unit fracture path or effective grain size controls the toughness at low temperatures.
3. The transition in the apparent dynamic fracture toughness, K_{Id} , with test temperature is related to changes in the fracture mode.
4. Fatigue fractures of the commercial HSLA steels exhibited complex fracture characteristics. A mixed mode of fracture occurred, indicating interaction between various individual mechanisms (transgranular cleavage, microvoid coalescence, tearing, fatigue striation formation, intergranular cleavage).
5. Fatigue cracks initiate at persistent slip bands and eventually link up to form the major crack. The slip band morphologies, however, vary with the microstructure.

Acknowledgments

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Application of Load Pulsing to the Fractographic Study of Stress-Corrosion Cracking of Austenitic Stainless Steels

REFERENCE: Hahn, M. T. and Pugh, E. N., "Application of Load Pulsing to the Fractographic Study of Stress-Corrosion Cracking of Austenitic Stainless Steels," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 413-427.

ABSTRACT: A study has been made of stress-corrosion fracture surfaces produced in a Type 310S and a Type 304 steel tested in boiling aqueous magnesium chloride (154°C). During cracking, small load pulses were periodically superimposed onto an otherwise constant load, and, in the case of transgranular cracking, these produced markings on the fracture surfaces that delineated the instantaneous position of the crack front. A one-to-one correspondence was observed between these crack-front markings and the number of load pulses for time intervals between pulses exceeding 15 s. For smaller intervals, there were fewer markings than pulses. Moreover, the distance between the markings did not decrease to zero as the intervals decreased below 15 s but attained an approximately constant value. These observations are considered to indicate that transgranular cracking is a discontinuous process and that crack advance involves brittle mechanical fracture. No crack-front markings were detected on intergranular stress-corrosion surfaces, presumably because the crack tip is normally blunt in this case. The authors suggest that intergranular stress-corrosion cracking in this system occurs by continuous anodic dissolution at the crack tip, probably by the film-rupture model.

KEY WORDS: austenitic stainless steels, stress-corrosion cracking, fractography, materials, materials science

The interpretation of transgranular stress-corrosion fracture surfaces has been hindered by uncertainty concerning the position of the crack front in relation to the fractographic features. In the present work, small load pulses were superimposed onto an otherwise constant load during crack propagation, which caused limited plastic blunting at the crack tip and thus permitted the position of the crack front to be identified on the fracture surface by scan-

¹ Research assistant, Department of Metallurgy and Mining Engineering, University of Illinois, Urbana, Ill. 61801; presently research assistant at Lehigh University, Bethlehem, Pa. 18015.

² Professor, Department of Metallurgy and Mining Engineering, University of Illinois, Urbana, Ill. 61801; presently a metallurgist at the National Bureau of Standards, Washington, D.C. 20234.

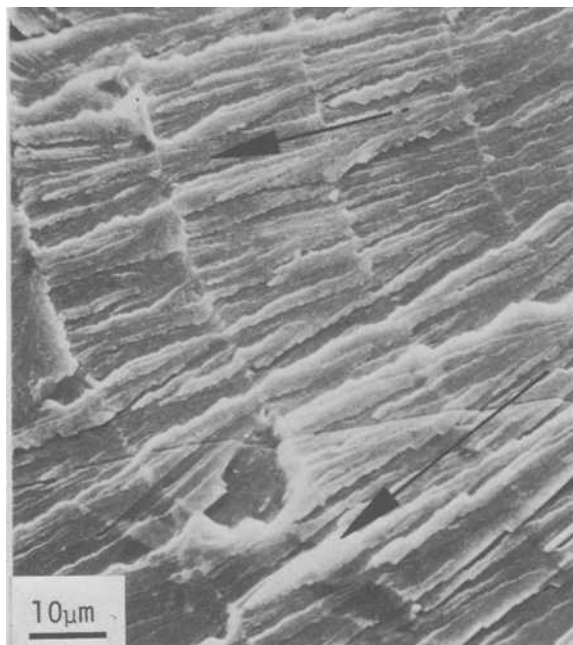


FIG. 1—Scanning electron photomicrograph of the transgranular stress-corrosion fracture surface for an admiralty metal bend specimen tested in aqueous ammonia, illustrating four crack-front markings introduced by periodic increases of the deflection ($\Delta t = 100$ s). After Beavers [1, 2].

ning electron microscope (SEM) examination. This approach had been used earlier in our laboratory by Beavers [1,2]³ in a study of transgranular stress-corrosion cracking (SCC) of admiralty metal in aqueous ammonia. Those studies were carried out on single-crystal bend specimens, and the position of the crack front was marked periodically by incrementally increasing the deflection. Figure 1 is a typical fractograph of a fracture surface for which the interval between the incremental deflections was 100 s. The fractography is typical of that for the transgranular SCC of a number of different alloy systems [3,4] and consists of a series of flat, parallel facets separated by serrated steps; the facets are of (110) orientation for admiralty metal, and the steps consist of two alternating {111} surfaces [1,2,5]. Four well-defined crack-front markings are visible in Fig. 1, and one can see that they are approximately perpendicular to the serrated steps. Assuming that each deflection produces a marking, Fig. 1 indicates that the crack velocity is $\sim 2 \times 10^{-5}$ cm/s, which is in reasonable agreement with values obtained by conventional methods [2,6].

It is thought that transgranular fracture surfaces of the type shown in Fig. 1 are produced by discontinuous cleavage and that they result from the embrittlement of the material ahead of the crack front by reaction with the environ-

³The italic numbers in brackets refer to the list of references appended to this paper.

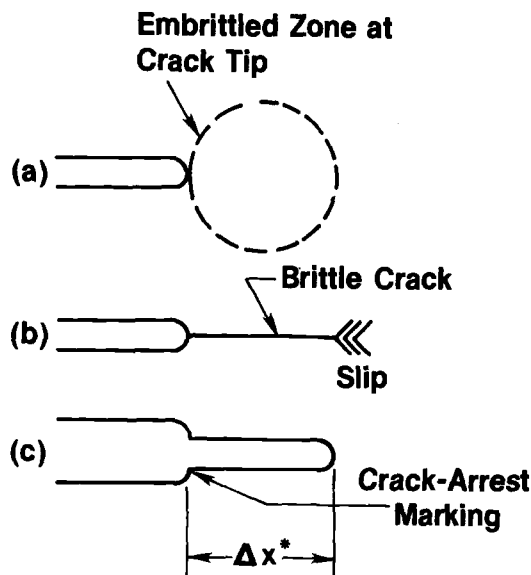


FIG. 2—Schematic illustration of the bulk-embrittlement model for transgranular SCC. It is thought that the lattice ahead of the stationary crack front becomes embrittled by interaction with the solution, thus permitting the initiation of a brittle crack. The latter is thought to be arrested by plastic deformation when it emerges from the embrittled region, producing a crack-arrest marking on the fracture surface. The crack is believed to propagate discontinuously by repeated cycles of this process.

ment [3,4]. This bulk-embrittlement model is illustrated schematically in Fig. 2. Under constant load or constant deflection, blunting of the crack by slip as it emerges from the embrittled zone and subsequent creep results in the formation of a marking on the fracture surface. However, such "crack-arrest" markings are resolvable with the SEM only under conditions in which appreciable blunting occurs, generally during constant-load tests at stresses approaching K_{Ic} or during certain slow strain-rate tests [2-4]. In deflection or load-pulsing experiments, sufficient additional strain is introduced to ensure the visibility of a crack-front marking under SEM examination. Crack-arrest markings and crack-front markings are thus similar in nature, since both result from the blunting of a sharp crack; they differ in that the latter are produced by an intentional small overload. It should be noted that, based on previous work [1,2], four or five crack-arrest markings would be expected to exist between each pair of crack-front markings in Fig. 1. It is thought that they are not visible because of the relatively small strains that occurred during crack arrest. This view is illustrated schematically in Fig. 3.

The present studies were carried out on polycrystalline specimens of annealed Type 304 and 310S stainless steels tested in tension in boiling aqueous magnesium chloride. One objective was to determine the generality of the findings for admiralty metal [1,2], but the primary aim was to use the method

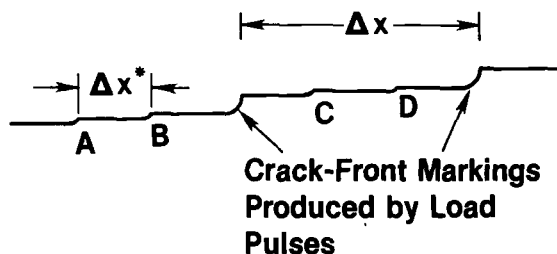


FIG. 3—Schematic drawing of a section of a transgranular stress-corrosion fracture surface, which illustrates the relationship between crack-arrest markings (A–D) and crack-front markings.

to investigate the mechanisms of SCC in this system. In particular, it was thought that the method might provide evidence for the still-controversial view that transgranular SCC is a discontinuous process. On the basis of the bulk-embrittlement model, it would be expected that a one-to-one relation would exist between the number of load pulses and the number of crack-front markings for the case where the time interval between pulses, Δt , is very much greater than the time, Δt^* , between crack-advance events under constant load. This was shown to be the case for admiralty metal [1, 2]. However, an interesting situation arises when Δt becomes smaller than Δt^* . If the bulk-embrittlement model is valid, it should be possible for several load pulses to be applied without the crack advancing; therefore, the distance between crack-front markings, Δx , would attain a limiting value, approximately equal to the crack-advance distance, Δx^* , under constant-load conditions. Note that if crack propagation were continuous as some workers propose, then Δx should decrease continuously to zero as Δt is decreased. Thus a major objective of the present work was to study the relationship between Δx and Δt , particularly at small values of the latter.

The fractography of transgranular stress-corrosion fractures in austenitic steels is similar to that shown for admiralty metal in Fig. 1. The orientation of the primary fracture facets is (100) for Type 310 steels [7, 8], but it has not been established for Type 304 steel. Crack-arrest markings have been reported on the fracture surfaces by several workers. Hänninen and Honkasalo [9] studied an 18–10 austenitic stainless steel tested in boiling aqueous magnesium chloride under a constant uniaxial tensile load. The markings were observed on the stress-corrosion fracture surface near the transition to ductile overload. They displayed the general characteristics of crack-arrest markings, that is, they were curved in some cases and were approximately perpendicular to the surface steps. But it was not demonstrated that they matched on the opposite fracture surfaces; therefore, some uncertainty exists in their interpretation. The spacing between the markings was approximately $1 \mu\text{m}$. Other workers have reported crack-arrest markings in Type 304 steel under slow strain-rate conditions [10, 11]. Vermilyea [10] carried out tests in pH 2.5 sulfuric acid at 289°C . Again the markings had the characteristics of crack-arrest markings,

but they were not shown to be matching. The spacing was approximately 0.2 μm . In recent work by Welch [11], precision matching of opposite fracture surfaces produced in hot (250°C) high-purity water established the existence of crack-arrest markings; the spacing was 2.5 to 3.0 μm .

A further objective concerns the relationship between transgranular and intergranular SCC. It has been suggested that the two forms of cracking occur through basically different mechanisms and that these mechanisms can be competitive in a single system [4, 12]. In Type 304 steels, for example, either mode of SCC can occur in aqueous magnesium chloride solutions, depending on specific conditions, and both can be produced in a single test [13]. The transgranular form is thought to occur by the bulk-embrittlement model, discussed earlier, and intergranular SCC by the classical film-rupture model [14, 15]. According to the latter model, a protective surface film is continually ruptured by localized plastic deformation at the crack tip, which permits rapid anodic dissolution of the exposed metal while the nondeforming crack walls remain protected by the film. Thus crack propagation is considered to occur continuously on a macroscopic scale [4], and, as noted before, the spacing between crack-front markings should then decrease continuously to zero as the time interval between load pulses is progressively decreased. Tests were conducted on Type 304 steel to investigate this prediction.

Experimental Procedures

Specimens used in this study were prepared from Type 310S and Type 304 stainless steels, kindly provided by J. A. Davis, Altech Specialty Steel. The compositions of the steels are given in Table 1. Strips of each alloy, 1.9 cm wide and 0.125 cm thick for Type 310S and 2.7 cm wide and 0.16 cm thick for Type 304, were annealed in argon for 2 h at 1050°C and water quenched, pro-

TABLE 1—Alloy compositions, weight percent.

Element	Type 310S	Type 304
C	0.054	0.057
Mn	1.90	1.71
P	0.022	0.025
S	0.004	0.01
Si	0.65	0.56
Cr	24.76	18.15
Ni	19.55	8.54
Al	0.01	0.007
Mo	0.11	0.18
Cu	0.17	0.45
N	0.049	0.051
Ti	0.012	0.01
Fe	balance	balance

ducing a grain size of ASTM 6 for both alloys. After heat treatment, the strips were cut into specimens of approximately 20 cm length, and a single-edge notch, approximately 0.7 cm deep, was cut into each by means of a jeweler's saw.

Stress-corrosion tests were carried out in a boiling solution of aqueous magnesium chloride, contained in a stainless steel beaker, that surrounded the notched region of the specimens. Approximately 300 ml of solution was used, and this was maintained at the boiling point ($154 \pm 5^\circ\text{C}$) by means of heating tape. The majority of the specimens were stressed under a constant tensile load using an MTS Systems Corp. closed-loop electrohydraulic testing machine. Preliminary experiments established that a load corresponding to a net-section stress of 220 MPa was suitable for the load-pulsing experiments and that an increment, ΔP , of 5 percent of the base load produced well-defined crack-front markings on transgranular stress-corrosion fractures. The magnitude of ΔP is not critical, but it should be noted that large values were found to cause excessive secondary cracking. In the load-pulsing tests, a function generator was used to produce the waveform shown in Fig. 4; the duration of the pulse was 2 s, and the interval between pulses, Δt , was varied from 5 to 1000 s. A limited number of specimens were tested under constant-extension-rate conditions using a Tinius Olsen testing machine (screw drive). In these cases, the specimens were loaded to a net section stress of 300 MPa, and the crosshead was kept stationary until cracking occurred. When the load dropped to give a net section stress of 250 MPa, the crosshead was turned on at a speed of 8.5×10^{-5} cm/s, and the specimen was strained to complete fracture.

To preserve detail on the fracture surface, failed specimens were quickly removed from the solution, rinsed with cold water, ultrasonically cleaned in acetone, and dried with an air blast. Fractographic examination was performed using Japan Electron Optics Laboratory Co. Ltd. (JEOL) scanning electron microscopes, Models JSM-U3, JEOL 25C, or JEOL 35C. The latter model was used for measurement of the spacing between crack-front mark-

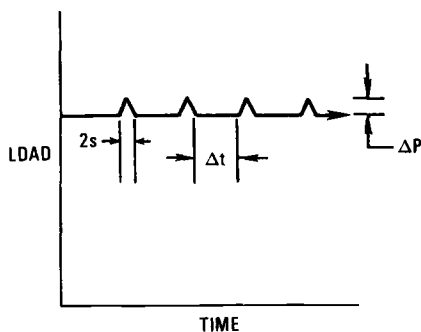


FIG. 4—Schematic drawing which shows a variation of load with time for load-pulsing experiments.

ings; an accelerating voltage of 25 kV was employed, and the position of the specimens was adjusted so that the fracture surfaces were in the eucentric position to ensure the accuracy of the magnification values.

Results

Type 310S Steel

Scanning electron microscope examination of fractured specimens indicated that failure did not result from the growth of a single crack from the notch. Rather, many stress-corrosion cracks were initiated at the free surfaces, and these grew until the central region of the specimens failed by ductile overload. Figure 5 illustrates a typical fractured specimen. It shows regions of ductile fracture and SCC on the primary fracture surface and numerous secondary cracks. The stress-corrosion cracks were almost entirely transgranular; a typical facet is shown in Fig. 6. As in other cases of transgranular SCC [3,4], cracking proceeds on a series of closely spaced, parallel crystallographic planes, separated by cleavage-like steps. The steps, which are serrated on a fine scale [16], can be seen to radiate from the initiation site at the free surface.

Several crack-front markings can be seen in Fig. 6, and these confirm the findings for admiralty metal [1,2] that the cleavage-like steps are approximately perpendicular to the crack front. Crack-front markings corresponding to a range of intervals between pulses, Δt , are shown in Fig. 7a through e. In addition to the crack-front markings, occasional slip steps, secondary cracks, and corrosion tunnels also are visible; each of these features is discernible in Fig. 7a. The spacings between crack-front markings, Δx , were measured in specimens tested at values of Δt ranging from 5 to 1000 s. A single specimen was tested at each value of Δt , and Δx was measured from fractographs of representative facets. It was found that Δx did not vary significantly in any single specimen, and therefore average values were obtained from a line drawn perpendicular to the markings and intersecting as large a number of markings as possible. Generally, a single such measurement was made on each fractograph. The data are presented in Table 2 and, in part, in Fig. 8a. The significant feature is that Δx did not continue to decrease as Δt fell below 15 s. Values of $\Delta x/\Delta t$ are also presented in Table 2 and graphically in Fig. 8b. A marked increase in $\Delta x/\Delta t$ can be seen for $\Delta x < 15$ s.

To determine whether a one-to-one correspondence exists between load pulses and crack-front markings, tests were conducted in which Δt was systematically varied. For example, in the case illustrated in Fig. 9, the interval between pulses was 100 s, except for every fourth interval, which was 200 s; the spacings between the crack-front markings can be seen to be consistent with this pattern. These experiments indicated that a one-to-one correspondence existed for $\Delta t > 15$ s but that there were fewer markings than pulses for smaller values of Δt .

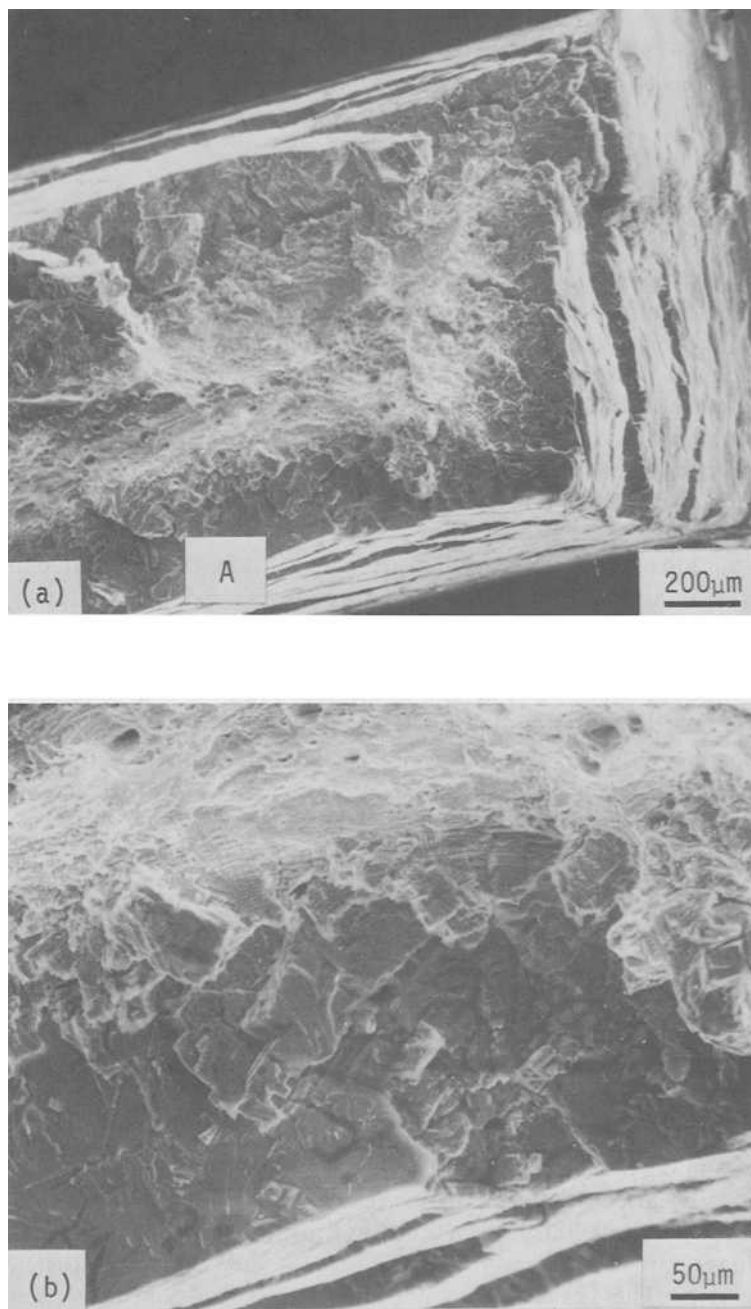


FIG. 5—Scanning electron photomicrographs of a typical fracture surface produced in Type 310S steel by load pulsing ($\Delta t = 100$ s) in boiling aqueous magnesium chloride. Region A of (a) is shown at higher magnification in (b).

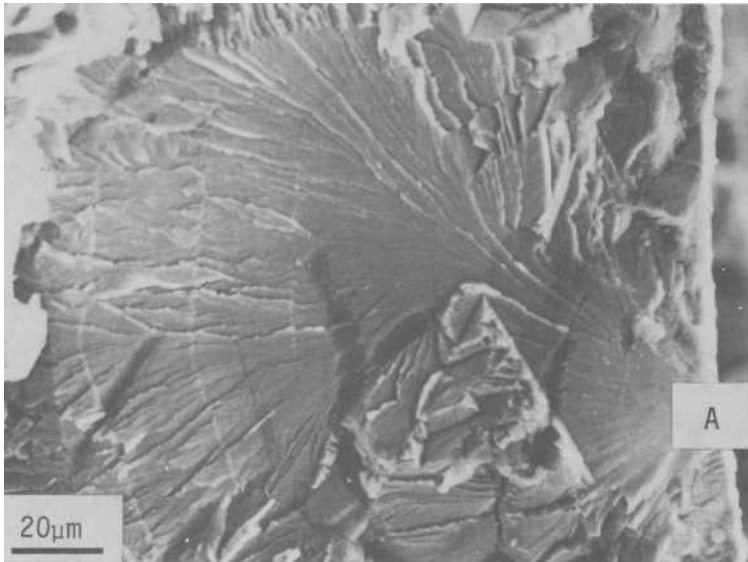


FIG. 6—Scanning electron photomicrograph of a typical transgranular stress-corrosion facet in Type 310S steel, load pulsed ($\Delta t = 1000$ s). Crack initiation occurred at A.

Attempts to identify crack-arrest markings were made in specimens tested under constant load and under constant-extension rate conditions. Many parallel markings were observed on the stress-corrosion facets, but comparison of the opposite fracture surfaces showed that these did not match, which indicated that they were slip steps. In the case of a specimen tested at an extension rate of 5×10^{-6} cm/s, markings having the correct characteristics were observed (Fig. 10), but it was not possible to compare the corresponding areas on

TABLE 2—Relationship of the time interval between load pulses, Δt , and the spacing between crack-front markings, Δx , for Type 310S steel.

Δt , s	Number of Measurements	Δx , μm	Standard Deviation, μm	$\Delta x/\Delta t$, $\mu\text{m/s}$
5	5	0.76	0.21	0.150
10	3	0.50	0.20	0.050
15	6	0.41	0.09	0.027
25	2	0.64	0.10	0.026
35	4	0.97	0.18	0.028
50	4	1.48	0.22	0.029
100	11	2.15	0.30	0.021
500	1	6.20	...	0.012
1000	1	17.90	...	0.018

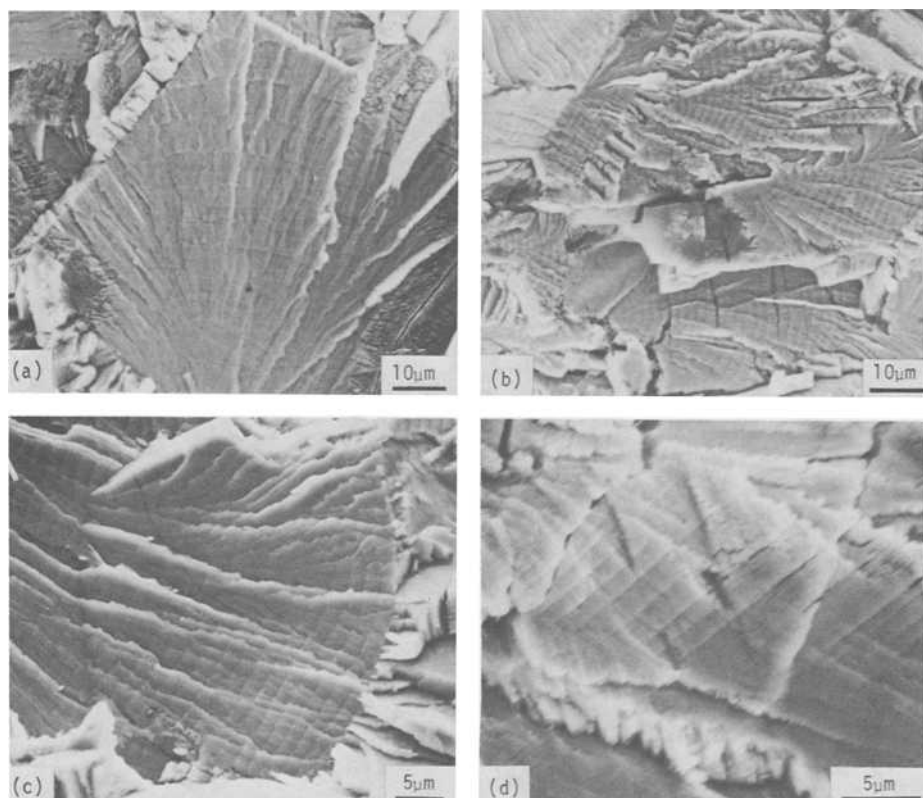


FIG. 7—Scanning electron photomicrographs of stress-corrosion fracture surfaces produced in Type 310S steel in load pulsing experiments. The values of Δt were (a) 500, (b) 100, (c) 50, and (d) 10 s.

the opposite fracture surface. The markings occurred in regions near the transition to ductile overload, where crack-arrest markings have been detected in other cases [3,4,9]. The spacing between these markings was approximately $0.8 \mu\text{m}$.

Type 304 Steel

Stress-corrosion cracking in these tests was predominantly intergranular, with relatively small amounts of transgranular failure. The significant feature of the fractographic observations was that well-defined crack-front markings were present on the transgranular fracture surfaces but none were detected on the intergranular facets (Fig. 11a). Tests were conducted at larger values of ΔP , but the markings were again detected only on the transgranular facets, as in Fig. 11b.

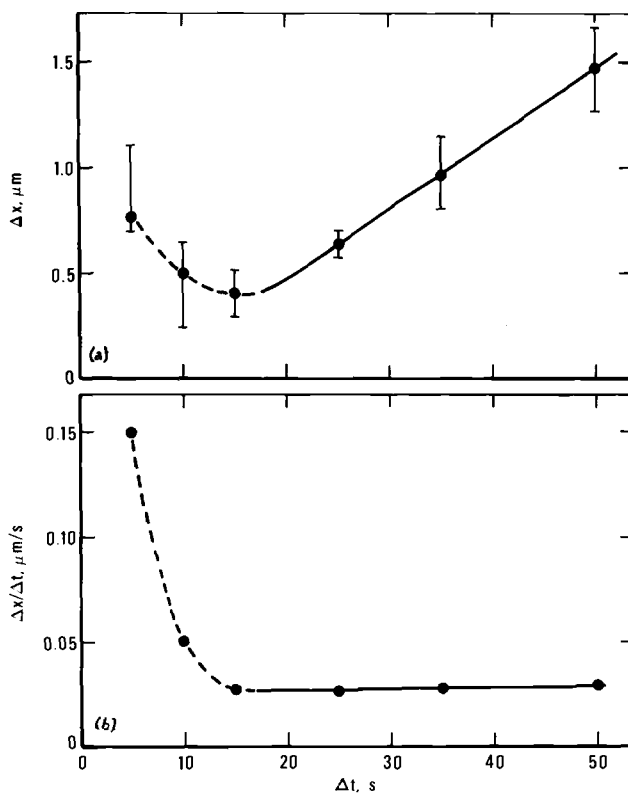


FIG. 8—The relationship of the time interval between load pulses, Δt , and (a) the spacing between crack-front markings, Δx , and (b) $\Delta x/\Delta t$. The points represent averages (see Table 2), and the bars indicate the highest and lowest values.

Discussion

The present studies established that crack-front markings can be produced readily on transgranular stress-corrosion fracture surfaces in austenitic stainless steels by the load-pulsing technique. The observations confirm the finding for admiralty metal [1,2] that the serrated steps that characterize transgranular fracture surfaces [3,4] are approximately perpendicular to the crack front and thus have the characteristics of conventional cleavage steps [4]. When Δt exceeded 15 s, a one-to-one correspondence existed between the number of markings and pulses, and in these cases the crack velocity is given by $\Delta x/\Delta t$. The velocity obtained in this way did not vary significantly with increasing crack length for a particular value of Δt , and this presumably indicates that cracking was in the stress-independent region of the v - K curve. The velocity also did not show a large variation with Δt (Table 2 and Fig. 8). The average value, 2×10^{-6} cm/s, is in good agreement with values reported in the literature [17-19].

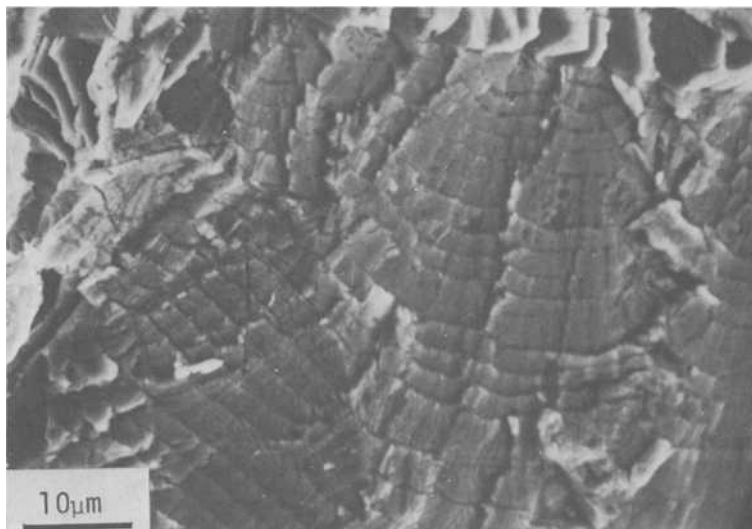


FIG. 9—Scanning electron photomicrograph of stress-corrosion fracture surface in Type 310S steel in which Δt was varied; the interval between pulses was 100 s, except for every fourth interval, which was 200 s.

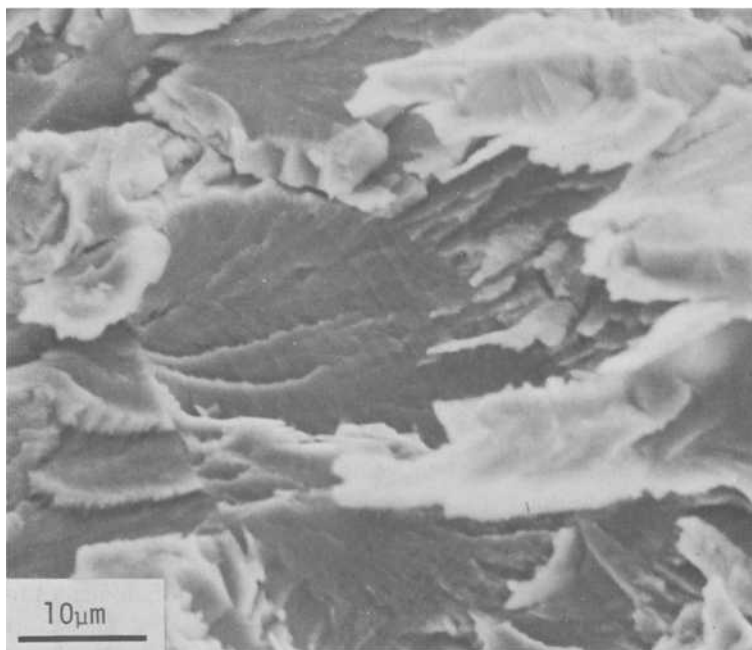


FIG. 10—Scanning electron photomicrograph of the fracture surface of a Type 310S specimen tested under a constant extension rate of 5×10^{-6} cm/s. The transition from SCC to ductile overload occurs near the right-hand edge.

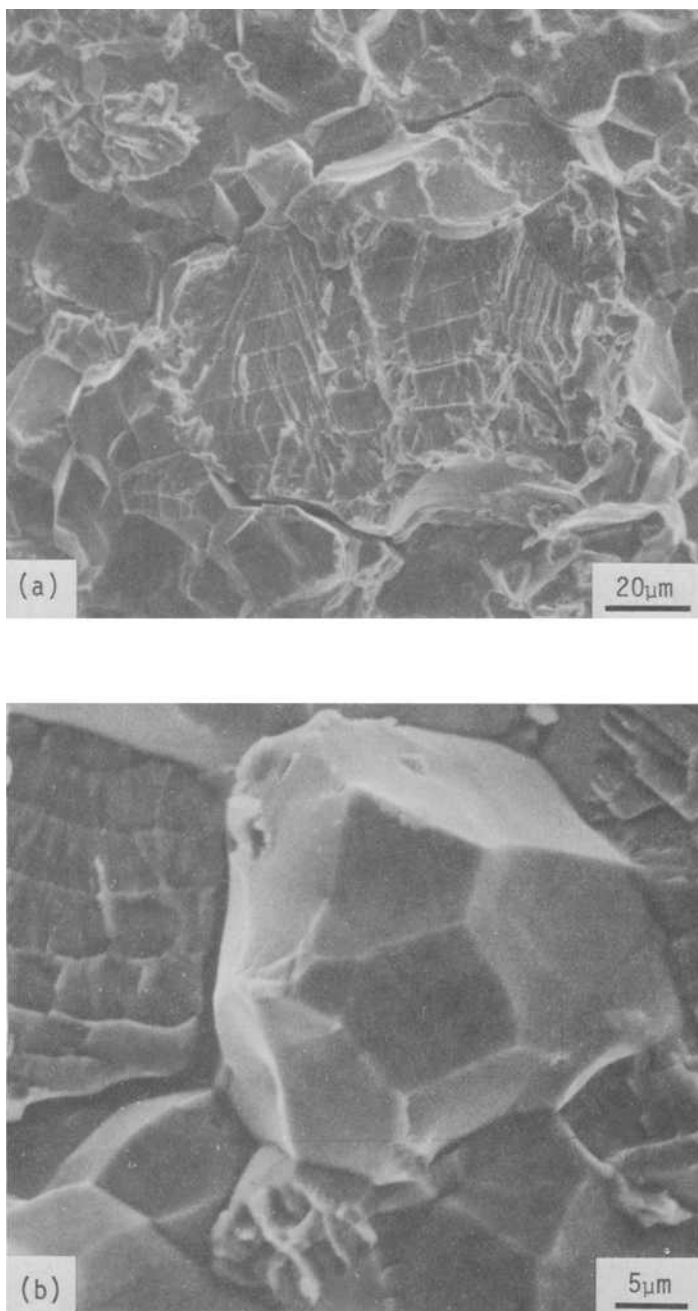


FIG. 11—Scanning electron photomicrographs of stress-corrosion fracture surfaces produced in Type 304 steel in load-pulsing experiments: Δt was 100 s in both cases, and ΔP was 5 percent in (a) and 10 percent in (b).

An important finding was that Δx for transgranular failures in Type 310S steel did not approach zero as Δt was decreased below 15 s (Table 2 and Fig. 8a) but remained at values between 0.4 and 0.7 μm . This behavior is fully consistent with the bulk-embrittlement model described in Fig. 2 and is considered to indicate that Δt falls below Δt^* at times of less than 15 s. These results suggest that Δx^* is approximately 0.4 to 0.7 μm ; Δx^* would be expected to be somewhat larger than this value, since it appears likely that the application of a load pulse would cause crack extension somewhat before this would occur under constant load. It was not possible to establish unambiguously the presence of crack-arrest markings, and therefore the value of Δx^* could not be established directly. However, it is probable that the parallel lines shown in Fig. 10 are, in fact, crack-arrest markings. It was noted that the spacing of these lines was approximately 0.8 μm . This value is also in good agreement with the value of Δx^* reported by Hänninen and Honkasalo for an 18–10 steel tested in boiling aqueous magnesium chloride [9]. The present studies do not permit direct determination of Δt^* . However, if Δx^* is taken as 0.8 μm and the crack velocity as 0.02 $\mu\text{m/s}$, then Δt^* becomes 40 s, which is in good agreement with Fig. 8.

The breakdown of the one-to-one correspondence between crack-front markings and load pulses for $\Delta t < 15$ s also supports the preceding conclusions. The observation that there were fewer markings than pulses for small values of Δt is considered to indicate that several pulses were applied without the crack advancing, because there was insufficient time for the necessary embrittlement to occur. This effect also explains the increase in $\Delta x/\Delta t$ shown in Fig. 8b, since this quantity does not equal velocity under these conditions.

The observations for the Type 304 steel that no crack-front markings were detectable on intergranular fracture surfaces were unexpected, but they are in accord with the view that the mechanism of cracking is basically different from that for transgranular SCC [4]. The absence of the markings can be rationalized in terms of the film-rupture model [14, 15] by considering that the crack is normally extremely blunt, and therefore the additional strain associated with the load pulse does not significantly increase the radius at the crack tip. The proposed cleavage-like nature of transgranular cracks at first sight is consistent with the occurrence of the markings in that case. However, although the crack is thought to advance in a brittle fashion (Fig. 2b), giving rise to the fine fractographic details, it is thought to become blunt as it is arrested by plastic deformation when it emerges from the embrittled zone and by creep during the period of arrest, which is approximately 15 s in this case. Slip-step emergence during this period would be expected to expose clean surfaces, so that appreciable crack blunting by dissolution would be expected—in effect, the film rupture mechanism would be expected to operate during this stage. The apparent absence of appreciable dissolution suggests that the rate of re-passivation is very much higher at the tip of transgranular cracks than in intergranular cracks.

A point not considered here is the possibility that load pulsing changes the mechanisms of cracking, specifically, that it may induce corrosion fatigue. This possibility cannot be ruled out, but it seems unlikely because of the small values of ΔP and because the application of the pulses does not change the basic appearance of the fracture surfaces.

Acknowledgments

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A Test Method to Determine the Degree of Embrittlement in Electrodeposited Copper

REFERENCE: Zakraysek, Louis, "A Test Method to Determine the Degree of Embrittlement in Electrodeposited Copper," *Fractography and Materials Science, ASTM STP 733*, L. N. Gilbertson and R. D. Zipp, Eds., American Society for Testing and Materials, 1981, pp. 428-439.

ABSTRACT: The brittle fracture of copper conductors in printed circuits is a severe problem in the electronics industry because such fractures will cause open-circuit conditions to occur in completed assemblies where the contributed value is high. In addition, repair possibilities may be limited due to the complexity of the assembly at the time that failures are discovered.

This paper deals with the development of a test method that is useful for the detection of an embrittled condition in foil prior to its application in circuits. Through the use of the mini-cup test method, a ductile-to-brittle transition was discovered, and, through further study of the transition phenomenon, procedures have been suggested for the characterization of electroplated copper foil.

KEY WORDS: copper foil, fractography, printed circuits, multilayer circuits, brittle fracture, ductile-brittle transition, foil cracking, materials, materials science

The problem of the brittle fracture of electrodeposited copper has become prevalent in the electronics industry. This problem manifests itself as an intermittent or open-circuit condition in assembled printed circuits after the imposition of certain processing or application stresses, especially thermally induced stress such as that encountered during soldering or thermal testing.

In Fig. 1 is the metallographic section of a 22-layer multilayer circuit board (MLB). As can be seen, this technology involves fabrication procedures of fairly high complexity. At higher magnification (Fig. 2), a fracture is evident in inner conductor layers after further processing. Also evident is the fact that a tedious and difficult routine is necessary to isolate a fracture in an assembly such as this. When circuit components have been attached to the MLB, the detection of a fracture can be even more difficult. In addition, the presence of cracks is certain to be quite costly.

¹ Manager, Physical Metallurgy, Electronics Laboratory, General Electric Co., Syracuse, N.Y. 13221.

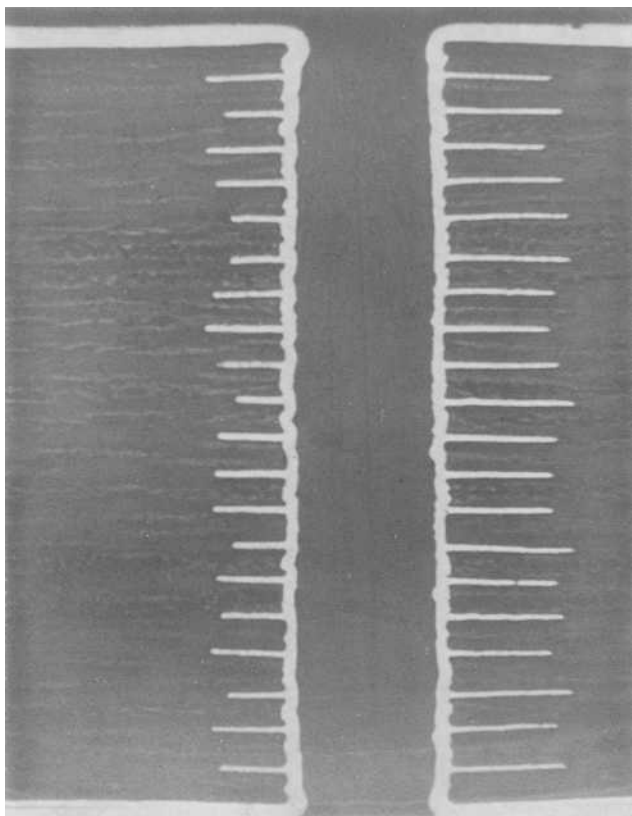


FIG. 1—*Metallographic section of a 22-layer multilayer electronic circuit board ($\times 40$).*

Fracture in electrodeposited foil has been attributed by Wild [1]² to embrittlement during the plating process. Mather [2] has investigated annealing as a method for preventing premature fracture. These studies indicate that fracture is due to the degradation of grain boundaries as a result of the codeposition of impurities. A study by Mathis and Zakraysek [3] has described the phenomenon as being due to the occurrence of a ductile-to-brittle transition as a function of temperature in the affected copper foil. Other suggestions for the solution of the fracture problem are based on plating process control [4] and on changing the substrate material to reduce stresses in an MLB [5].

Almost all the copper now used for conductors (foil or through-hole) in printed circuits for electronic assemblies is produced by electrolytic deposition from copper sulfate or copper pyrophosphate plating solutions. Smaller quantities of copper conductors are obtained through the use of copper fluo-

² The italic numbers in brackets refer to the list of references appended to this paper.

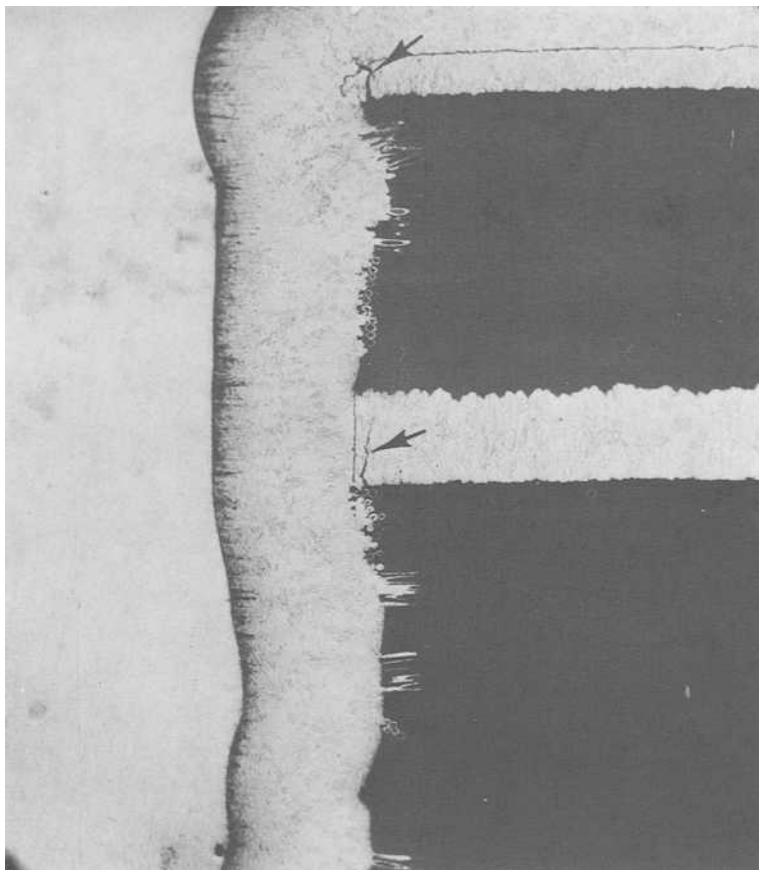


FIG. 2—*Inner conductor fracture* ($\times 250$).

roborate and copper cyanide solutions, and still less is produced by rolling foil from bar stock. The embrittlement phenomenon described in this study occurs only in the electrolytically deposited foil (not in rolled foil) and, to varying degrees, in foil from the different plating solutions.

Embrittlement in electrodeposited metals is a well-established phenomenon generally attributed to the presence of hydrogen, which is generated as a result of the electrochemical process. Less understood are the effects of the codeposition of organic additives such as brighteners. In either case, process control parameters, especially the current density, are known to modify the effects of hydrogen or of organics. In addition, the solubility of organic and other impurities in different plating solutions will vary, and this variation also changes the availability of the impurities for codeposition.

Embrittlement in electrolytically deposited metals occurs through the codeposition and inclusion of foreign materials, especially hydrogen, organ-

ics, and particulate fines in the deposit. The process controls and plating techniques for electrolytic copper are such that foil with tensile ductility of from <1 to 15 percent elongation is obtained when the foil is tested at room temperature. During this study, we have shown that this type of foil may exhibit ductile elongation at room temperature and brittle fracture at temperatures over 65°C when tested at a total strain equivalent to that imposed by forming a 6.3-mm hemisphere. When it occurs, the elevated temperature brittle fracture occurs by separation at grain boundaries. Although failures were induced by the addition of organic contaminants to the plating solutions and by the use of nonspecified process parameters, the exact failure mechanism has not, as yet, been defined, except that it is manifested as a brittle fracture surface, that is, with no visible deformation.

The specific problem that this study addresses is the development of a test method for the characterization of electrodeposited foil in terms of the presence, or absence, of a ductile-brittle transition. Given the nature of the foil-fracture problem, it will be extremely useful if a means is made available for detecting the potential for fracture prior to the use of foil in (MLB) assemblies.

Although tensile and elongation tests are used extensively for the determination of mechanical properties, these tests have some limitations [6] in the present instance. First, obtaining consistent results by the tensile testing of thin (20 to 40 μm) foil is not done without taking great care; second, embrittlement is not necessarily depicted through tests for elongation; and, most important, the embrittlement phenomenon is temperature dependent, that is, room-temperature testing is insufficient for foil characterization. Therefore, this investigation was made in search of alternative test methods.

Procedure

The presently suggested test method is based on a modification, actually a miniaturization, of the Olsen cup test [7] for sheet metal formability. One model of our test apparatus is shown in Fig. 3. In this equipment, a 6.3-mm diameter ball and a matching die cavity are used to form a hemispherical cup in the test specimen, as is shown in Fig. 4. The standard hardness test equipment was used to provide consistent strain rates and could be replaced by a similarly controlled arbor, since loads up to 10 kg will provide sufficient force to impose the desired strain on the foil specimen. Total strain is governed by the use of the standard 6.3-mm (0.25-in.) hardness indenter and a matching brass die cavity.

For each specimen, a test is made at desired temperature intervals to as high as 300°C. After the mini-cup is formed, each test piece is inspected visually at $\times 10$ to 20 magnification for evidence of fracture. When fracture occurs, the fracture surface is examined for evidence of brittle fracture. In this manner, a transition from ductile to brittle (if it exists) can be determined in a specimen of electrodeposited copper foil.

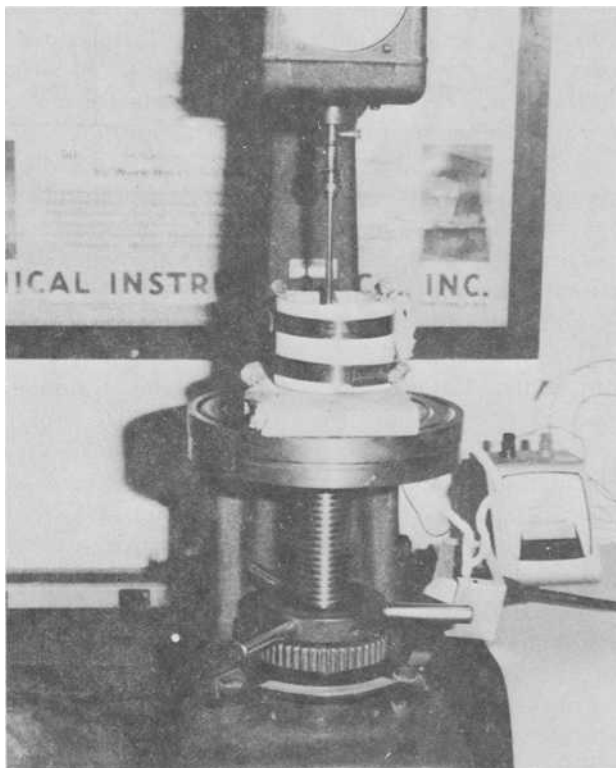


FIG. 3—Mini-cup ductility test apparatus.

Experimental Results

The mini-cup ductility test was developed as a method of stressing the thin foils at elevated temperature. To do this, a number of specimens were obtained for determining the effect of processing methods, commercial processing, plating procedures, and heat-treating processes on the quality of the copper foil. Each tested piece was examined visually and rated as either ductile or brittle according to the appearance of the cup formed as a result of the test; this is shown in Fig. 5 for one experimental series.

The Effect of Foil Processing

Tests were made of wrought copper foil and of foil electroplated from different types of copper plating solutions. Shown in Table 1 are the results of these tests. These results show that wrought foil is ductile over the entire test temperature range. This is as expected in foil made by casting and cold rolling.

The plated foil specimens were made by depositing copper from each of four plating solutions onto polished stainless coupons. After plating, the copper foil was peeled from the coupon and tested by the mini-cup method.

FIG. 4—Hemispherical cup formed in testing ductile foil ($\times 40$).

TABLE 1—Effect of foil processing.

Specimen	Bath Condition ^a	Foil Test Temperature, deg C	
		Ductile	Brittle
Wrought copper	. . .	290	. . .
Copper cyanide	new	290	. . .
	aged	290	. . .
Copper fluoroborate	new	RT ^b	65
	aged	. . .	RT
Copper pyrophosphate	new	230	290
	aged	. . .	RT
Copper sulfate	new	65	120
	aged	120	180

^a Bath conditions: new—freshly prepared, within the supplier's operating limits; aged—0.5 volume percent diethylene triamine added as a contaminant.

^b RT = room temperature.

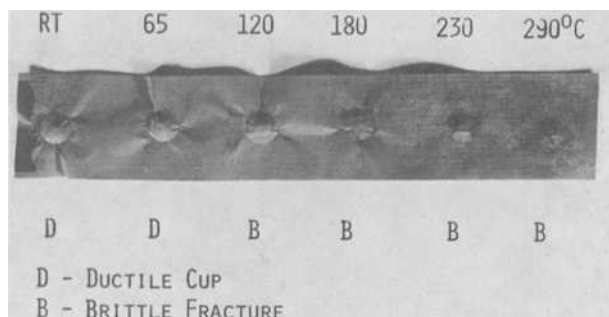


FIG. 5—Mini-cup test sequence Specimen No. 58B (about $\times 0.6$).

These results show that the copper from a cyanide bath is ductile and insensitive to the bath contaminant used in these experiments. The fluoroborate bath appears to be difficult to prepare and contaminant-sensitive in its deposition of embrittled copper. The copper from the pyrophosphate bath is ductile from a new bath and brittle from a contaminated bath. This indicates that the bath is easy to make up but quite sensitive to contamination. The copper sulfate bath seems to offer the potential for a ductile deposit but requires some control because of an apparent sensitivity to bath contamination.

The Nature of Commercial Foil

Foil for inner conductor layers in MLBs is purchased by all users from a few commercial suppliers. During this study, foil was obtained from three sources. The results of our tests on this foil are shown in Table 2. The tests were made by the random selection of test specimens from the available test lots. The data

TABLE 2—Commercial foil.

Source	No. Specimens	Foil Test Temperature, deg Celsius	
		Ductile	Brittle
A	16	65	120
	20	120	180
	8	180	230
	4	230	290
	2	290	...
B	1	120	180
	3	180	230
	2	230	290
C	1	room temperature	65

have been arranged, for convenience, in order of increasing transition temperature.

The results of these tests show that in a high percentage of all commercially available foil a ductile–brittle transition will occur with exposure to elevated temperature.

All of the tested foil is produced commercially through the use of proprietary electrolytic processes, so information on plating conditions is not readily available. However, these test results indicate that improvements are needed in each case.

Production Through-Hole Plating

By using the same specimen preparation technique as described earlier for evaluating processing effects, that is, deposition on a stainless steel coupon, production plating baths normally used for through-hole plating in MLB fabrication can also be monitored for deposit quality. Shown in Table 3 are the results of tests made on copper foil taken from a printed circuit production copper sulfate bath. This bath had been operated for 110 000 Ah when one set of specimens was made; then the bath was resampled after carbon treatment and filtration for the removal of organic contaminants.

These results show that the aged bath (over 100 000 Ah of production use) was beginning to produce copper with a decreasing ductile–brittle transition and that the treatment of the bath was effective in restoring ductility. These results indicate that this test procedure can be effective in monitoring the quality of the copper deposit obtained from production plating baths.

The Effect of Heat Treatment

Specimens that were found to exhibit a transition were annealed in order to determine whether this heat treatment could be used to eliminate the brittle condition. Shown in Table 4 are the results of these tests.

TABLE 3—*Production bath control.*

Source	Bath Condition ^a	Foil Test Temperature, °C	
		Ductile	Brittle
A	aged	180	230
	treated	230	290
B	aged	230	290
	treated	290	...

^a Bath conditions: aged—>110 000 Ah operation; treated—carbon treated and filtered.

TABLE 4—*The effect of heat treatment.*

Specimen	Heat Treatment		Foil Test Temperature, deg Celsius	
	Temperature, deg Celsius	Time, min	Ductile	Brittle
A	150	10	room temperature	65
	250	10	120	180
	350	10	180	230
	450	10	<290	...
	400	5	180	230
	400	10	230	290
	400	15	230	290
	400	20	230	290

These tests results show that an annealing treatment can increase the ductile–brittle transition in copper foil, and thereby eliminate the brittle condition. As can be seen, however, the annealing time and temperature have to be established properly for this treatment to be effective.

Discussion

The mini-cup ductility test appears to offer a simple and effective means for detection of the presence of grain-boundary degradation in thin electrodeposited copper foil. When this test is performed on copper foil, a visual examination is made of fracture surfaces. A typical comparison of tested specimens is shown in Fig. 6. These scanning electron microscope (SEM) views show the normal appearance of a ductile cup and its brittle fracture when tested above the ductile–brittle transition temperature. Keep in mind that these test pieces are from the same specimen of foil, but the testing temperature has been changed.

Because the evaluation after the cup test is visual, it is necessary to distinguish between ductile and brittle fractures since ductile fractures may also occur (especially in annealed foil). The SEM views shown in Fig. 7 are those of fracture surfaces as noted. As can be seen, the distinction between ductile and brittle fracture is readily discernible for each of the possible conditions encountered through the use of the mini-cup test. Therefore, the rating of tested specimens, although qualitative, is positive.

Conclusions

Embrittlement in electrodeposited copper is due to grain-boundary degradation, probably stemming from the codeposition of impurities during electroplating. Because that is a continuously variable process, the degree of embrittlement is also variable.

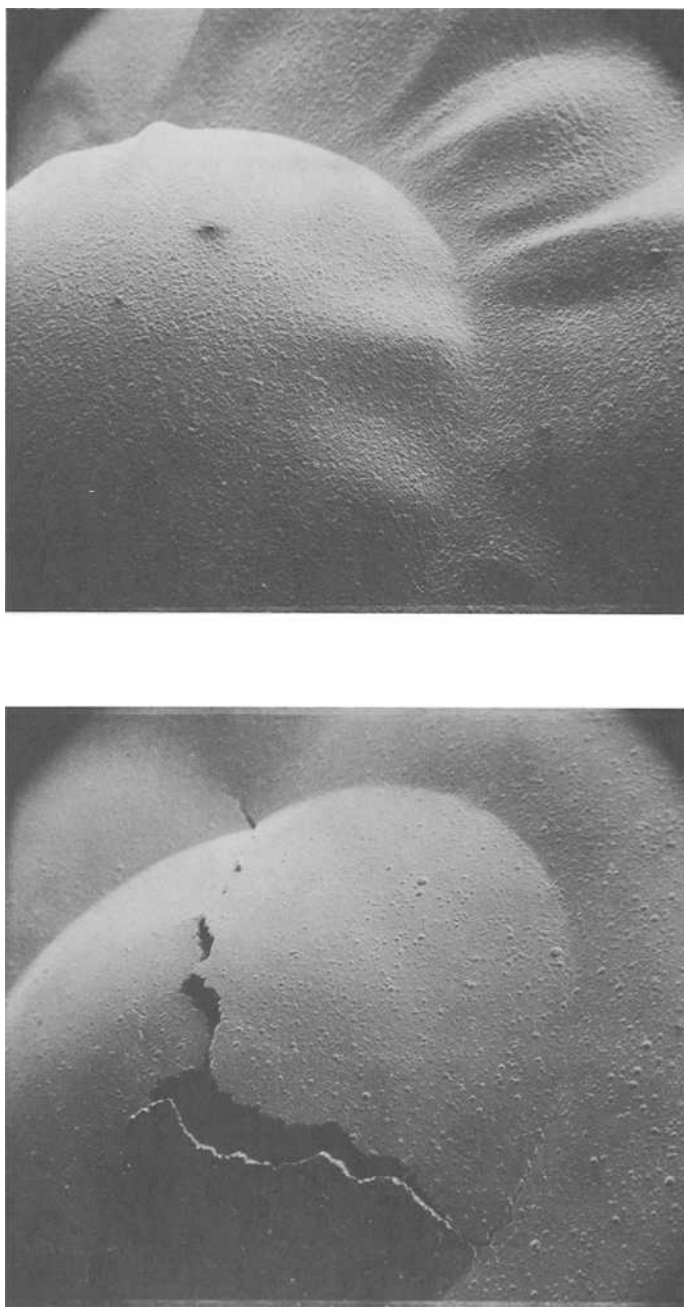


FIG. 6—(Top) *Specimen 11A, ductile cup, test at room temperature ($\times 17$)*; (bottom) *Specimen 11A, brittle fracture, test at 180°C ($\times 17$)*.

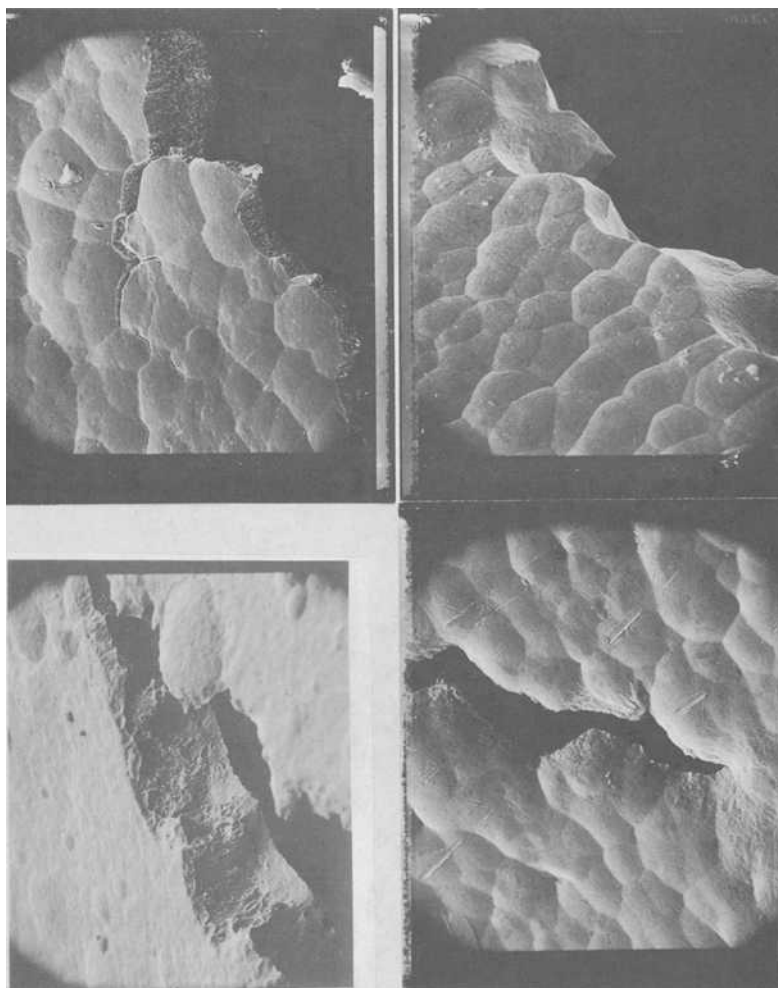


FIG. 7—(Top, left) ductile, electrolytic copper foil tested at 180°C; (top, right) brittle fracture, electrolytic copper foil tested at 180°C; (bottom, left) ductile, electrolytic copper foil tested at room temperature; (bottom, right) brittle fracture, electrolytic copper foil tested at room temperature (all at X260).

Testing for embrittlement requires stressing at elevated temperature since a ductile-brittle transition with temperature occurs in the electrodeposit. The location of the transition on the temperature scale is an indication of the degree of embrittlement; that is, the nearer to room temperature that brittle fracture occurs, the more brittle the material is.

The mini-cup ductility test is effective in detecting the transition temperature and, therefore, the degree of embrittlement.

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Summary

Summary

This volume is divided into four sections, dealing with the following areas in fractography and materials science: environment, microstructure and fatigue, nonmetallics and composites, and techniques.

The papers in the environment section illustrate that the fracture topography of the specimen is a synergistic combination of the microstructure, the chemical environment, and the loading condition. The paper by Ruppen and McEvily shows how the fracture topography of one alloy changes when different processing produces different microstructures. These authors also show that not only could the environment have an effect on fatigue fracture topography but different crack growth rates could change fracture mechanisms, which would result in different fracture topography. Meyn and Brooks, in their paper, present important evidence about a characteristic structure called a "flute," normally found in planar slip alloys. They report that in some titanium alloys greater numbers of "flutes" are associated with cleavage due to stress corrosion than are found with the "cleavage-like" structures that can result from fatigue. Fraser and Metzbower cover a broad range of treatments and alloys in high-strength steel, illustrating the interactions of microstructure with the mechanical and chemical environment. And Provenzano et al bring out the importance of microstructural contaminants in pressure vessel steels and their importance in stress-corrosion cracking.

Caskey, in his paper, uses yet another tool, X-ray diffraction, to bring out the importance of the crystal structure relationships in hydrogen-assisted fracture (HAF) of austenitic stainless steel. One of the most important parts of his paper is the evidence that the presence of martensite is not a prerequisite for cleavage in HAF of austenitic stainless steels. It was previously thought that only martensite lath structures were responsible for cleavage in HAF and stress-corrosion cracking of austenitic stainless steels.

The last paper in this section, by Mills, displays another important trend in evaluation of microstructures and fracture surfaces. Some of the significant microstructural features, particularly at grain boundaries, were too small to be resolved with conventional optical metallographic techniques. This work illustrates the trend toward using the scanning electron microscope and the transmission electron microscope (TEM) for examination of conventional

metallographic structures. Mills also uses the TEM to examine dislocation structures and relates the fracture topography to the dislocation structures.

In the microstructure and fatigue section, four papers are primarily concerned with the effect of microstructural variables on the fracture surface topography, and two papers deal with fatigue crack growth rate studies in aluminum and steel. Steele demonstrates the importance of understanding the relationship between fracture morphology and the underlying microstructure by studying crack initiation and propagation mechanisms in several ferrous alloys. The fractographic importance of microstructural parameters, such as prior austenitic grain size, microstructural phases, and mechanical twins, is demonstrated using examples.

The paper by Wilson investigates the influence of nonmetallic inclusions on fatigue crack propagation in three different plate steel grades. Fatigue studies were performed comparing steels with conventional steel-making inclusions to calcium-treated steels with controlled morphology inclusions. The results show that the calcium treatment of inclusions improves fatigue crack propagation resistance and minimizes anisotropy for all steel grades.

Metzbower and Moon reveal the fractographic features associated with single overload fractures in laser-welded steels and a titanium alloy. The significance of the microstructure in the base plate, heat-affected zone, and fusion area on the fracture topographs is clearly demonstrated by using examples.

The paper by Datta and Wood compare fractographic features of toughness specimens tested using different strain rates. The results show that Type 4340 steel, quenched and tempered to different strength levels, was characterized by the same fracture mechanism in the origin area for each strength level evaluated, regardless of the toughness test employed.

Albertin and Hudak have determined the effect of a negative R -ratio ($R = -1$) on the fatigue crack growth rate in Type 2219-T851 aluminum alloy and correlated the macroscopic growth rate with microfractographic observations. They conclude that fatigue striation spacings for negative R -ratios were a function of ΔK and that their measurement on fracture surfaces should be an extremely useful tool for failure analysis.

Au and Ke have studied the effect of R -ratio, frequency, environment, and carbon content on the fatigue crack growth rate in Type 9310 carburized steel and observed the microscopic features as a function of the macroscopic growth rate. They found that good correlation existed between fatigue striation spacings and the macroscopic growth rate in the intermediate growth range (10^{-4} to 10^{-3} mm/cycle).

The nonmetallics and composites section represents a more recent area of investigation using fractographic techniques. With increasing use of ceramics, composites, and plastics in structural applications, the understanding of the fracture behavior of these materials has grown in importance. Because of the greater difficulty in obtaining the equivalent of conventional metallographic microstructures with these materials, fracture surface evaluation is important

in helping to illuminate the actual structures of these materials. With ceramics in particular, fractography is a vital part of the evaluation of the fracture mechanics properties of ceramics.

The paper by Govila et al illustrates the use of fractography in interpreting mechanical test results of ceramics at high temperatures. These authors show that ceramics can also change in fracture mode with changing temperatures. Mecholsky and Freiman reveal how some fracture features are similar in several different polycrystalline ceramics. The paper by Abdel-Latif et al presents one point of view about the dynamic fracture of brittle materials. The authors try to relate current empirical brittle fracture equations to the multiple-mist phenomenon found in high-strength glass fractures. Adler and James, in their paper, present an interesting technique for the evaluation of impact damage to transparent materials.

Rimnac et al show the effect of molecular weight, plasticizer content, and residual orientation on the fatigue fracture topography of one particular polymer (polyvinyl chloride). These factors seem to affect the morphology and spacing of the discontinuous growth bands that are characteristic of fatigue fracture, although fatigue striations were sometimes seen at high ΔK levels.

The paper by Kim et al, dealing with an exotic metal matrix composite, introduces another useful tool in fracture analysis, Auger electron spectroscopy (AES). In these composites, subtle diffusion interactions detected by AES were shown to be responsible for brittle fracture of the tungsten fibers.

In the techniques section, many innovative techniques associated with fractography are presented by a variety of authors. Underwood and Chakraborty show how fatigue fracture mechanisms in titanium alloys are being studied using quantitative stereological analysis of the fracture surfaces. They demonstrate that the topographics associated with the fracture surfaces can be characterized in terms of different crack roughness parameters.

Passoja and Psioda apply Fourier transform analysis to crack profiles in Type 7075-T6 aluminum specimens, which were generated using cyclic loading conditions. They show that Fourier transform analysis reveals inherent periodicities in the fracture profile. These same statistical spacings were found in the microstructure of the aluminum alloy. This paper presents a new tool that may lead to a more quantitative evaluation of fracture surfaces.

The paper by Hahn et al reveals how the position of a stable propagating stress-corrosion crack in a stainless steel (and also in admiralty metal) can be monitored by applying a small load pulse to cause limited plastic blunting at the crack tip. The authors indicate that the markings associated with the blunting can be used to determine crack velocities or to investigate whether cracking is discontinuous or continuous.

In the electronic area, Zakraysek has developed a test method for determining the quality of electrodeposited copper used in printed circuit boards. He used a mini-cup ductility test to evaluate the intergranular embrittlement of copper in the temperature range of 22 to 300°C.

Two papers involve precision sectioning techniques, which allow direct correlation between the fracture surface topography and the underlying microstructure. Nenonen et al evaluate different steels that are characterized by a bainitic microstructure and have found that the bainite packet size and orientation are very influential in the fracture process. They also indicate that tear lines and holes on the fracture surface are due to the crack changing direction at high-angle packet boundaries. Krishnadev et al apply precision sectioning techniques to evaluate high-strength low-alloy steels and iron-base alloys with different microstructures under a variety of testing conditions to gain an understanding of fracture mechanisms. In their fatigue studies at various test temperatures, the results indicate enhanced fatigue lives at low temperatures. In their single-overload studies, the results show that chemical composition, temperature, strain rate, and mode of loading affect the fracture surface topography.

L. N. Gilbertson

Zimmer, U.S.A., Warsaw, Ind. 46580; symposium chairman and editor.

R. D. Zipp

International Harvester, Hinsdale, Ill. 60521; symposium chairman and editor.

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