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Development of fracture toughness of ultrahigh strength, medium carbon, low alloy steels for aerospace applications

Y. Tomita

Ultrahigh strength, low alloy, steels with a medium carbon (0.25–0.50 wt-%) content and various amounts of chromium, molybdenum, nickel, silicon, and vanadium have been needed for high performance aerospace parts. The steels can be successfully employed at yield strengths of ≥ 1400 MPa, but their commercial use is often limited in practice by their poor fracture toughness. The present review surveys studies carried out over about the past 10 years to improve the fracture toughness of ultrahigh strength, medium carbon, low alloy steels, and the present understanding of this property is outlined. Particular emphasis is placed on improving the fracture toughness by microstructural control via thermal and thermomechanical treatments, modification of sulphide inclusions, and new alloying design. The major metallurgical factors controlling the fracture toughness are discussed for each of these techniques.

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Introduction

Commercial low alloy steels have been used for many engineering components in quenched and tempered conditions. In particular, quenched and lightly tempered low alloy steels with medium carbon levels (0.25–0.50 wt-%) and various amounts of chromium, molybdenum, nickel, silicon, and vanadium are needed for high performance aerospace parts. These low alloy steels can be successfully employed at yield strengths of ≥ 1400 MPa and are designated as ultrahigh strength, medium carbon, low alloy steels (Table 1).^{1–3} To meet the requirements of high strength and stiffness, together with low volume and weight, the low alloy steels have been used during the past 40 years or so. For example, the landing gear on high performance, carrier based aircraft must meet unique, severe and conflicting requirements: the main

landing gear must withstand impact loads from sink rates (the vertical component of landing velocity) in excess of 12.86 m s^{-1} , while the nose gear must bear the entire force of catapulting the aircraft off the deck, however, once the aircraft is in flight, the landing gear represents parasitic weight.⁴ Thus, the weight and, more importantly, the volume the landing gear occupies when retracted, must be kept to a minimum.

The 4340 steel is a typical example of the ultrahigh strength, low alloy steels. This steel has been developed since 1950 and has been used for aerospace applications since 1955. It is usually austenitised normally, followed by oil quenching and subsequent tempering at or close to 473 K for use at 1790–1930 MPa tensile strength levels. In the 1960s, the 300M steel was introduced as a higher strength replacement for 4340 steel. It has a slightly higher carbon content than the 4340 steel, and silicon is added to allow it to be hardened to 1860–2079 MPa tensile strength levels with a tempering temperature higher than 473 K. Since 1966, the 300M steel has been extensively employed in critical components of aerospace vehicles. More damage tolerant steels such as silicon modified 4330 (4330Si), 4330VM, and HY-TUF have also been employed for aerospace applications at tensile strength levels of 1520–1650 MPa.

If strength were the only criterion, ultrahigh strength, medium carbon, low alloy steels would be used in preference to ultrahigh strength, medium and high alloy steels (e.g. HY180, AF1410, 15-5PH, and 18% maraging steels).³ In spite of the modification of the chemical compositions and well thought out procedures for quality assurance and control, the low alloy steels have often been limited by their poor fracture toughness. Catastrophic fracture has to be given serious consideration. In such situations and to minimise the risk of catastrophic failure, many manufacturers and designers have focused their attention on improving the fracture toughness of the low alloy steels in association with metallurgical control. Therefore, reviewing recent studies concerning the improvement in the fracture toughness published hitherto in the literature may provide helpful sugges-

Table 1 Chemical composition (wt-%) of typical AISI ultrahigh strength, medium carbon, low alloy steels for aerospace applications

Steel	C	Si	Mn	P,S	Cr	Mo	Ni	V
4340	0.38–0.43	0.20–0.35	0.60–0.90	0.010*	0.70–0.90	0.20–0.30	1.65–2.00	...
300M	0.40–0.45	1.45–1.80	0.60–0.90	0.010*	0.70–0.95	0.30–0.50	1.65–2.00	0.05–0.10
D6AC	0.42–0.48	0.15–0.30	0.60–0.90	0.015*	0.90–1.20	0.90–1.10	0.40–0.70	0.07–0.15
4330Si	0.27–0.33	0.40–0.70	0.60–0.80	0.015*	1.00–1.35	0.35–0.55	1.85–2.25	...
4330V	0.28–0.33	0.15–0.35	0.65–1.00	0.015*	0.75–1.00	0.35–0.50	1.65–2.00	0.05–0.10
HY-TUF	0.23–0.28	1.30–1.70	1.20–1.50	0.015*	0.20–0.40	0.35–0.45	1.65–2.00	...

* Maximum value.

tions for further developing low alloy steels for ultrahigh strength applications.

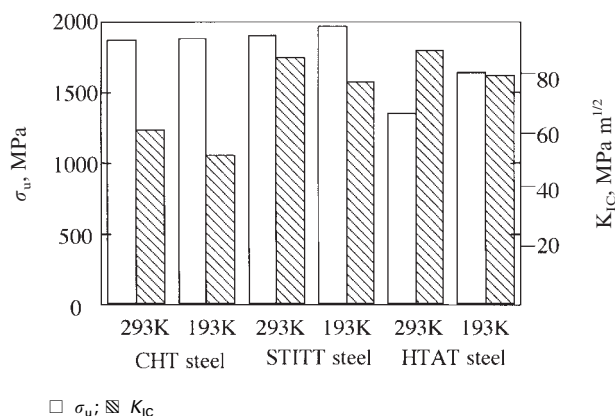
In the present review, the development of the fracture toughness of ultrahigh strength, medium carbon, low alloy steels over about the past 10 years is surveyed, and the present understanding of the property is outlined. Particular emphasis is placed on improvements obtained by microstructural control via thermal and thermomechanical treatments, modification of sulphide inclusions, and new alloying design. The major metallurgical factors controlling the fracture toughness are discussed for each of these techniques.

Improvement in fracture toughness by microstructural control via thermal treatments

The simultaneous attainment of high strength and fracture toughness is one of the more difficult problems in low alloy steel. The basic difficulty is that the mechanical properties cannot be improved simultaneously through conventional heat treatment. One of the potential ways of overcoming this problem is to develop steels having mixed structures in which separate constituents are responsible for the different property requirements. However, it has hitherto been reported that the mechanical properties of such steels were inferior to those of steels having conventional microstructures.⁵⁻⁸ Recent investigations⁹ have indicated that such mixed structure can significantly improve the mechanical properties if the ductile phase appears in a suitable morphology (size, shape, and distribution) in association with lightly tempered martensite. Therefore, mixed structures have recently been proposed in a deliberate attempt to improve the mechanical properties of ultrahigh strength, low alloy steels. Based on these experiments, short time, isothermal transformation treatment, and modified heat treatment, which utilise the beneficial properties of a mixed structure, have been suggested whereby the strength and fracture toughness of the low alloy steels can be dramatically improved. Another potential method for improving the fracture toughness of the steel is the duplex treatment, which consists of combinations of high and low temperature austenitisation, and high and low temperature tempering. The effect of the thermal treatments on the improved fracture toughness is discussed below.

Short time, isothermal transformation treatment

One potential approach to improve fracture toughness is to utilise the beneficial properties of a mixed



1 Effect of temperature on tensile strength σ_u and fracture toughness K_{IC} of various heat treated steels¹⁰

structure of martensite and lower bainite which is produced by a short time, isothermal transformation treatment (STITT) in the bainitic temperature range. Recently, Tomita¹⁰ improved the fracture toughness of 0.4C–Ni–Cr–Mo steel (4340 grade) through the STITT. The STITT produces a mixed structure of martensite and 25 vol.-% lower bainite. This is produced by isothermal transformation at 593 K just above the martensitic starting temperature M_s for a short time followed by water quenching (after austenitisation at 1133 K). The mechanical properties obtained by the STITT are compared with those obtained by the conventional heat treatment (CHT) and are summarised in Table 2. The tensile strength σ_u and fracture toughness K_{IC} data, which are noteworthy, are shown in Fig. 1. As can be seen from this table and figure, compared with the CHT, the STITT dramatically improved the K_{IC} and the 2 mm V-notch Charpy impact energy E_{ch} yielding higher strengths even at a temperature of 193 K. Recent investigations¹¹⁻¹⁵ have shown that a significant improvement in fracture toughness can be attained by high temperature austenitising treatment (HTAT), though the marked improvement of the fracture toughness is often not accompanied by an increase in blunt notch toughness such as the Charpy impact energy. Therefore, the results obtained by the STITT were also compared with those obtained by the HTAT. Compared with the HTAT, the STITT also improved the E_{ch} due to the significant increase in strength at similar K_{IC} levels at and above 193 K. Furthermore, Tomita¹⁶ suggested that the STITT is also attractive for ultrahigh strength applications of high carbon, low alloy steels such as 0.6C–Cr–Mo–Ni steel.

Table 2 Mechanical properties of short time, isothermally transformed (STITT) 4340 steel¹⁰

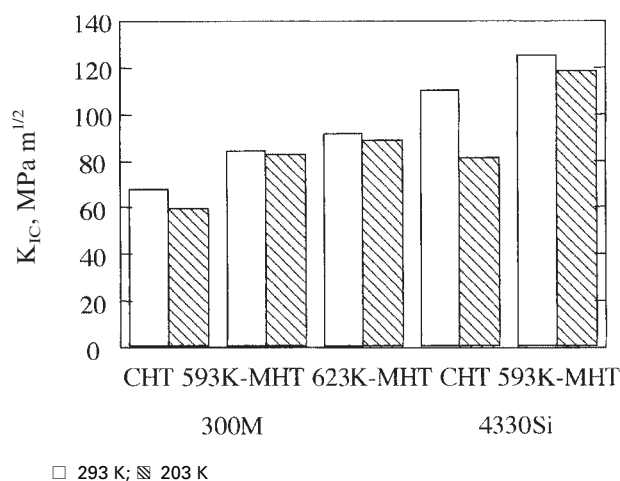
Designation of steel*	Test temp., K	0.2% offset yield stress σ_y , MPa	Ultimate tensile strength σ_u , MPa	2 mm V-notch Charpy impact energy E_{ch} , J	Fracture toughness K_{IC} , MPa m ^{1/2}
STITT	293	1490	1907	22.8	78.4
	193	1587	1971	15.6	71.0
CHT	293	1407	1873	18.3	54.4
	193	1523	1887	11.6	48.2
HTAT	293	914	1353	15.1	82.9
	193	1327	1643	10.5	73.4

* CHT = conventional heat treatment; HTAT = high temperature austenitising treatment; STITT = short time, isothermal transformation treatment.

From metallographic observations and the modified law of mixtures, the increase in strength and toughness by the STITT can be attributed to the following causes:^{9,10} (i) strength increases by the refinement of the substructure (i.e. packet size and lath width) in the matrix martensite and enhanced strength of lower bainite owing to higher plastic restraining of the bainite by the martensite; (ii) from true stress-strain analysis through modified Crussard-Jaoul analysis,¹⁷ the beneficial effect on ductility is due to full use being made of the superior mechanical properties of lower bainite because it can deform in association with the parent martensite as a result of plastic restraining by the martensite¹⁸⁻²⁰ – thus, excessive load is not applied to the bainite during plastic deformation; (iii) improved toughness through STITT can be attributed not only to the crack arresting effect of the bainite but also to the stress relieving effect of the bainite just ahead of the existing crack.

Modified heat treatment

Another possible approach for the improvement of the fracture toughness of low alloy steels is the use of the beneficial property of tough, ductile austenite, which results in increased cleavage resistance at low ambient temperature.²¹⁻²³ However, the conventional heat treatment of low alloy steels yields very little austenite. Fortunately, recent investigations²⁴⁻²⁷ have demonstrated that the presence of high silicon levels in steels that have been isothermally transformed in the bainitic temperature region encourages the retention of ductile high carbon austenite rather than the formation of brittle interlath cementite films, which have a detrimental effect on both the ductility and toughness. This occurs because silicon severely retards the precipitation of cementite from the residual austenite during bainitic transformation. However, improved mechanical properties of the steel by applying this principle depend on the premise that the dispersed retained austenite should be both thermally and mechanically stable.^{28,29} From this point of view, Tomita and co-worker³⁰⁻³⁵ suggested a modified heat treatment (MHT) whereby the fracture toughness of 300M and 4330Si steels is dramatically improved. The microstructure of the MHT steel consisted of triple phases of 50 vol.-% carbide-free upper bainite, martensite, and retained austenite. The microstructure was produced by partial isothermal transformation



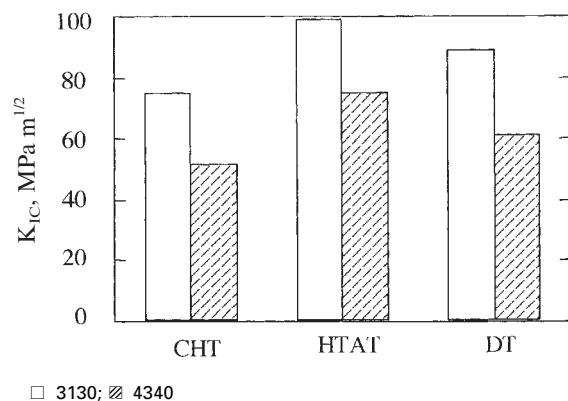
2 Effect of temperature on fracture toughness K_{IC} of various heat treated steels^{32,33}

for the required time at 593 or 623 K, followed by oil quenching and subsequent tempering at 473 K (after austenitisation at 1173 K). The results, which were compared with those obtained from the conventional quench and temper (CHT), are summarised in Table 3. The K_{IC} data, which are noteworthy, are shown in Fig. 2. Compared with the 300M-CHT steel, the 300M-MHT steel at 593 K (300M-593K-MHT) significantly improved the K_{IC} at similar tensile strength levels, due to the increased percent elongation over the temperature range 203–293 K. In particular, compared with the 300M-CHT, the 300M-MHT at 623 K (300M-623K-MHT) dramatically improved the K_{IC} over the temperature range 203–293 K; however, the strength was somewhat decreased. The MHT also lowered the ductile–brittle transition temperature (DBTT) in the 2 mm V-notch Charpy impact test independent of isothermal transformation temperature. Furthermore, the MHT improved the low temperature mechanical properties of the silicon modified 4330 (4330Si) steel. Compared with the 4330Si-CHT steel, the 4330Si-MHT steel at 593 K (4330Si-593K-MHT) improved the K_{IC} at similar tensile strengths and DBTT levels over the temperature range 203–293 K. The improvements in the fracture toughness can be attributed to the thin film type of austenite stabilised mechanically through MHT and the bainite ferrites whose individual ferrite plates are separated by the thin films of the austenite.³⁵

Table 3 Mechanical properties of modified heat treated (MHT) 300M and silicon modified 4330 (4330Si) steels^{32,33}

Designation of steel	Test temp., K	σ_y , MPa	σ_u , MPa	Total elongation, %	K_{IC} , MPa m ^{1/2}	DBTT*, K
300M-593K-MHT	293	1400	1975	10.8	84.0	223
	203	1519	1970	16.0	83.0	...
300M-623K-MHT	293	1204	1700	13.9	91.5	218
	203	1338	1718	17.9	88.6	...
300M-CHT	293	1585	1923	9.9	67.8	256
	203	1772	1911	10.2	59.3	...
4330Si-593K-MHT	293	1260	1550	12.4	125.4	163
	203	1305	1557	14.2	118.3	...
4330Si-CHT	293	1309	1588	12.4	110.6	168
	203	1372	1584	14.1	80.9	...

* Ductile–brittle transition temperature.



3 Fracture toughness K_{IC} of various heat treated steels³⁶

This occurs because the austenite and bainitic ferrite plates have high cleavage resistance and exhibit stress relieving and crack arresting effects on crack initiation and propagation at low ambient temperatures. The increased strength is attributed to the refinement of martensite by the bainite. However, the MHT steel suffers a loss of K_{IC} for temperatures at and below 183 K (Ref. 34). This is associated with the mechanical instability of the retained austenite at low ambient temperatures. The retained austenite is transformed into brittle martensite during deformation at and below this temperature, and the energy absorbed by the transformation is not compensated by the brittle nature of the transformed product. Thus, the MHT steel is attractive for low temperature, ultrahigh strength applications, but careful attention should be paid to the decomposition temperature of the austenite when the MHT steel is to be used for low temperature applications.

Duplex treatment

Based on the results of the significant improvement in fracture toughness by the HTAT,^{11–15} Cao and Lu³⁶ suggested the duplex treatment (DT) whereby both the fracture toughness and blunt notch toughness of 4340 and 3130 (0.3C–1.0Mn–2.5Ni–1.0Cr) steels can be improved simultaneously. The DT consists of a first cycle of austenitisation at high temperature, a tempering at a proper high temperature lower than the A_{C1} temperature of these steels after quenching from high austenitising temperature, and then a final austenitisation at conventional temperature followed by light tempering. Table 4 lists the mechanical properties produced by the DT, and the K_{IC} data are shown in Fig. 3 in which the results obtained by the DT are compared with those obtained for CHT and HTAT. The yield strength was estimated by $\sigma_{ys} = 1/3$

Vickers hardness (HV), and the apparent fracture toughness K_A (blunt notch fracture toughness) was calculated from $K_A = [(2/3)\sigma_{ys}\epsilon_F]^{1/2}\rho^{1/2}$ (Ref. 37). The results are summarised as follows: (i) a high K_{IC} can be obtained by the HTAT, but K_A is severely decreased for both steels; (ii) the K_{IC} of the DT steel is lower than that of the HTAT steel but higher than that of the CHT steel for both steels; (iii) the strength and the K_A of the DT steel are higher than those of the HTAT and CHT steels for both steels; (iv) thus, the DT steel has a good combination of yield strength, K_{IC} , and K_A . The authors suggested that the improvement in the mechanical properties of the DT steel could be due to the inheritance of the beneficial variations in fine scale microstructures, which occurred during heating at high temperature and to the depression of the memory effect of coarse austenite grain, which arose from high austenitising temperature.

Improvement in fracture toughness by microstructural control via thermomechanical treatment

A potential approach for developing low temperature fracture toughness at significantly increased strength level is the use of combined thermal and mechanical treatments, i.e. thermomechanical treatment. The thermomechanical treatment offers a potential technique for engineering of the mechanical properties as a result of microstructural refinement.^{38,39} Ausforming (low temperature thermomechanical treatment) in which high strength may be obtained by deformation of metastable austenite to produce martensite is particularly well known. However, application of ausforming to low alloy steels has been limited because it requires an extensive metastable austenite region. Therefore, thermomechanical treatment which involves deformation in the stable austenite region or before quenching to form martensite has been applied to medium carbon, low alloy steels. The effect of the thermomechanical treatment on the improvement in the fracture toughness is discussed below.

Quench and temper after controlled rolling

A thermomechanical treatment with the potential to improve the fracture toughness of low alloy steels is based on a controlled rolling process (CRP) in which the steel is deformed below 1173 K (austenite non-recrystallisation range) after hot rolling above 1273 K (austenite crystallisation range) followed by direct cooling. The CRP has been developed through an application of the technique for mass production of large diameter line pipe and steel plates used for arctic offshore structures.^{40–42} To date, this technique has also been extended to the production of high strength hull structural steel plate, which is used in lightweight ship building. A suitable combination of controlled rolling with accelerated cooling also makes it possible to produce rolled plates of great strength without deterioration of toughness.^{43,44} From this point of view, Tomita⁴⁵ studied the effect of quench and temper after CRP (CRP–QT) on the fracture

Table 4 Mechanical properties of steels processed by duplex treatment (DT)³⁶

Heat treatment	Rockwell hardness, HRC		Fracture toughness K_{IC} , MPa m ^{1/2}		Fracture strain ϵ_f	
	4340	3130	4340	3130	4340	3130
CHT	53.0	49.7	51.5	75.0	0.027	0.017
HTAT	52.3	47.9	74.9	99.1	0.022	0.027
DT	53.5	49.6	61.3	88.8	0.031	0.035

toughness of 4340 grade 0.4C–Ni–Cr–Mo ultrahigh strength, low alloy steel. The CRP–QT steel is produced by the following process: (i) austenitisation at 1423 K; (ii) 50% hot rolling at 1423–1273 K (austenite recrystallisation range); (iii) 50% hot rolling at 1173–1273 K (austenite non-recrystallisation range), followed by direct oil quenching and subsequent subzero treatment, and then tempering at 473 K. The results obtained by the CRP–QT are compared with those obtained by the CHT and are summarised in Table 5: the fracture toughness was estimated by the slow bend absorbed energy E_{sb} of a 2 mm V-notch Charpy specimen containing fatigue precrack. These data showed that the CRP–QT improved the low temperature fracture toughness in longitudinal orientation of the steel after tempering at a high strength level (473 K). The improvement in the fracture toughness at low ambient temperatures can be attributed to higher stress concentration at a crack tip being effectively relieved by mobile dislocations in the subcell introduced as a result of CRP–QT.⁴⁵ However, the CRP–QT steel suffers an abrupt loss of fracture toughness for a tempering temperature above 473 K, at which the properties become similar to those of the CHT steel followed by 473 K tempering independent of test temperatures. This can be attributed to the fine carbide precipitation that occurs on dislocations in the subcell, which promotes shear localisation and dimple fracture. Therefore, CRP–QT steel is attractive for improving low temperature fracture toughness. However, careful attention should be paid to the tempering temperature at which remarkable embrittlement occurs if the CFRP–QT steel is to be used for ultrahigh strength applications.

High temperature thermomechanical treatment

As mentioned above, CRP–QT is quite effective for improving the low temperature fracture toughness in the longitudinal orientation of ultrahigh strength, low alloy steels. However, the CFRP–QT significantly led to mechanical anisotropy of the steels. Therefore, the realm of high temperature thermomechanical treatment (HTMT) appears more attractive for the modification of low temperature mechanical properties.^{38,39} This process involves deformation at a temperature above the A_{c3} (stable austenite region) before quenching to low and medium alloy steels whose S -curve (time–temperature–transformation

diagram) significantly approaches the left. The strength increase obtained by HTMT is not as great as that with the ausforming, but the ductility and toughness may be significantly increased. Therefore, HTMT is expected to be one approach that greatly improves the low mechanical properties of ultrahigh strength, medium carbon, low alloy steels. However, because the deformation temperature is above the recrystallisation temperature, the best combinations of strength, ductility, and toughness resulting from HTMT depend on the ability to impart large deformation followed by quenching to prevent recrystallisation. From this point of view, the effect of HTMT on the low temperature fracture toughness of 4340 steel was investigated.⁴⁶ The results obtained by HTMT at 1177 or 1473 K (designated at 1173 K–HTMT or 1473 K–HTMT, respectively) were compared with those obtained by the CHT and are summarised in Table 6: the fracture toughness was estimated by the E_{sb} described above. When the 4340 steel was hot forged by 50% reduction using a forging machine with 1.47 MN capacity at 1177 K, followed by direct oil quenching and subsequent tempering at 423 K (1177 K–HTMT), the fracture toughness was significantly improved compared to that obtained by the CHT, with remarkably increased strength over the temperature range 77–293 K. Marked improvement in the fracture toughness was achieved due to a moderate increase in the strength over the temperature range 77–293 K, when the steel was deformed by 50% reduction at 1473 K followed by direct oil quenching and subsequent tempering at 453 K (1473 K–HTMT). The treatment also improved the upper shelf energy and DBTT in the Charpy impact test. The improved mechanical properties are attributed to the subcell structure introduced in austenite by HTMT processing and inherited by martensite.

Improvement in fracture toughness through modification of sulphide inclusions

Recently, there has been an increased demand for large sized, ultrahigh strength, low alloy steels and for the steels with isotropy regarding the mechanical properties for aerospace applications. Although the use of the previously mentioned mixed structure control and thermomechanical treatments are ben-

Table 5 Mechanical properties (longitudinal orientation) of 4340 grade steel processed by quenching and tempering at 473 K after controlled rolling (CRP–QT)⁴⁵

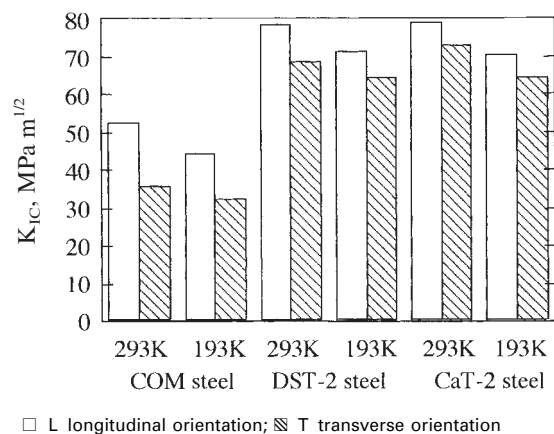
Designation of steel	Test temp., K	σ_y , MPa	σ_u , MPa	Total elongation, %	E_{sb}^* , J	E_{ch} , J
CRP–QT	293	1620	1890	13.2	1.9	15.2
	193	...	...	...	1.8	11.4
	77	...	...	...	0.5	5.7
CHT	293	1430	1790	7.2	0.9	10.5
	193	...	...	...	0.6	5.8
	77	...	...	...	0.3	2.6

*Slow bending absorbed energy tested using fatigue precracked specimens.

Table 6 Mechanical properties of high temperature thermomechanical treated (HTMT) 4340 steel⁴⁶

Designation of steel*	Test temp., K	σ_y , MPa	σ_u , MPa	Total elongation, %	E_{sb} , J	E_{ch} , J
1177 K–HTMT	293	1720	2200	9.8	2.6	13.5
	193	...	...	...	2.4	12.6
	77	...	...	...	1.4	4.8
1473 K–HTMT	293	1480	1900	10.4	2.6	16.8
	193	...	...	...	2.4	7.5
	77	...	...	...	1.3	4.0
CHT	293	1460	1850	5.8	0.8	10.8
	193	...	...	...	0.7	5.0
	77	...	...	...	0.7	3.5

* 1177 K–HTMT steel tempered at 423 K for 7.2 ks; 1473 K–HTMT steel tempered at 453 K for 7.2 ks; CHT steel tempered at 473 K for 7.2 ks.



4 Effect of temperature on fracture toughness K_{IC} of various heat treated steels⁵⁸

eficial for developing dramatic improvements in the fracture toughness of the ultrahigh strength, medium carbon, low alloy steels, they are not very promising for large sized and isotropic structural applications. A potential solution to the problem is the development of the fracture toughness through modification of sulphide inclusions. The key technologies are desulphurisation, calcium addition, and decreased hot rolling reduction. Therefore, the effect of desulphurisation, calcium treatment, and decreased hot rolling reduction treatment on improving the fracture toughness of the steel is discussed next.

Desulphurisation

In recent years, the theory and practice of desulphurisation employed to produce a reduced sulphur content have been significantly advanced, and this is reflected in the definite tendency towards a significantly low sulphur content in steels which are now produced in high quality grades. Experimental studies^{47,48} and the pioneering calculations of Rice and Johnson⁴⁹ suggested that the upper shelf fracture toughness of

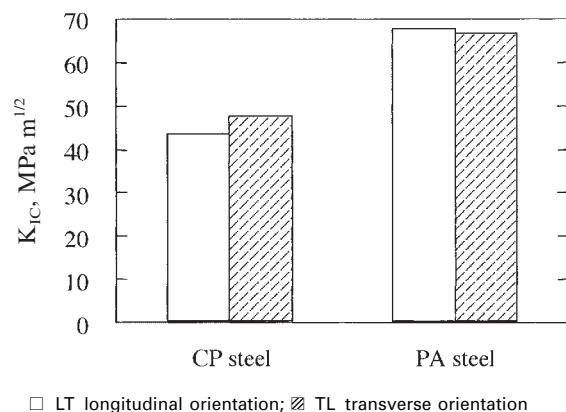
ultrahigh strength steels is sensitive to both inclusion spacing and inclusion volume fraction. Cox and Low⁵⁰ demonstrated that reducing the inclusion content increases the resistance to void nucleation and is also effective in improving the mechanical properties of 4340 steels. Leslie⁵¹ also suggested that reduction of the sulphur content might be a means of achieving reasonable fracture toughness of high strength steels. Knott and co-workers^{52,53} have also shown that crack opening displacement in Fe–C–Mn steel was significantly increased by the volume fraction of non-metallic inclusions containing manganese, titanium, silicon, and sulphur. From this point of view, Tomita^{54–58} investigated the effect of desulphurisation on the morphology of sulphide inclusions and the mechanical properties of 4340 grade 0.4C–Cr–Mo–Ni steels. The results, which are compared with those of steel containing sulphur at commercial levels, are summarised in Table 7. The significantly improved K_{IC} data are shown in Fig. 4. The results demonstrated that the steel containing sulphur at a commercial level (0.16 mass-%) (COM steel) exhibited remarkable anisotropy. This is because the stringy MnS inclusions that exist in COM steel cause brittle lamella fractures in association with the matrix martensite. Compared with COM steel, the desulphurised steel, which was reduced to a sulphur content of 0.0008 mass-% (DST-1 steel), dramatically improved the fracture ductility ϵ_f and Charpy impact energy E_{ch} as well as K_{IC} independent of the testing orientation at similar strengths at and above the 193 K level. Also, the desulphurising treatment significantly improved the isotropy regarding the $1/2 \Delta C_v$ and DBTT in the Charpy impact test. Most recently, Leap and Wingert⁵⁹ studied the effects of grain refining precipitates on the development of toughness in the 4340 steel. The results are summarised as follows (Fig. 5): (i) introduction of presolutional treatment (1523–1548 K) and subsequent subcritical annealing (973 K), before conventional austenitisation, produced grain refined precipitates

Table 7 Mechanical properties of desulphurised 4340 grade steels⁵⁸

Designation of steel*	Test temp., K	Tensile properties			K_{IC} , MPa m ^{1/2}	Charpy impact properties		
		σ_y , MPa	σ_u , MPa	$\epsilon_f \times 10^{-1}$		E_{ch} , J	DBTT (1/2 ΔC_v)†	
							J	K
COM-L (-T)	293	1485.2 (1483.4)	1820.4 (1818.4)	5.2 (1.0)	52.3 (35.8)	42.8 (13.2)	25.2 (8.8)	211 (-)
	193	1585.3 (1582.3)	1920.6 (1921.3)	4.3 (0.5)	44.3 (32.5)	24.4 (7.8)		
DST-1-L (-T)	293	1480.6 (1483.4)	1817.0 (1818.4)	4.7 (0.8)	51.2 (43.5)	34.2 (25.8)	20.8 (13.2)	241 (253)
	193	1570.1 (1569.2)	1928.6 (1930.5)	3.8 (0.5)	40.1 (38.2)	13.5 (11.2)		
DST-2-L (-T)	293	1485.9 (1488.7)	1818.0 (1828.0)	5.3 (3.8)	78.2 (68.4)	42.3 (34.8)	25.5 (18.8)	213 (223)
	193	1558.6 (1563.9)	1920.3 (1928.6)	5.1 (3.4)	70.9 (64.2)	24.5 (16.9)		

* COM = steel containing sulphur at commercial levels (0.16 mass-%); DST-1 = desulphurised steel with a sulphur content of 0.002 mass-%; DST-2 = desulphurised steel with a sulphur content of 0.0008 mass-%; L = longitudinal orientation; T = transverse orientation.

† Ductile brittle transition energy.



5 Effect of presolutional/annealing treatment on fracture toughness K_{IC} of 4340 steel;⁵⁹ CP conventionally processed steel, PA presolutional/annealing steel

(AlN and TiN) in the 4340 steel containing 0.006 wt-%Ti, 0.026 wt-%Al, and 70 ppm N; (ii) application of a presolution treatment and subcritical annealing to the desulphurised steel with sulphur content (0.002 wt-%) (PA steel) dramatically improved the K_{IC} and its isotropy compared with those of the conventionally processed steel with the same sulphur content (CP steel). However, when fine elliptical MnS inclusions appeared in the desulphurised steel with a sulphur content of 0.002 mass-% (DST-1 steel), intergranular failure often occurred, producing a very detrimental effect on the ductility and toughness. The MnS inclusions were observed along the grain boundaries. This is attributed to the overheating phenomenon^{60,61} in which manganese and sulphur, which are taken into solution in the high rolling temperature range, segregate to the grain boundaries and reprecipitate there as fine MnS inclusions. Therefore, careful attention should be paid to the removal of the fine elliptical MnS inclusions precipitated along the grain boundaries when the desulphurising treatment is applied to improve the

fracture toughness of ultrahigh strength, medium carbon, low alloy steels.

Calcium treatment

Application of chemical means is very attractive for the modification of sulphide inclusions. Among these chemical means are alkaline earth metals, e.g. calcium and magnesium, or rare earth metals, e.g. cerium and lanthanum, which are fed into molten steel in the ladle.^{62,63} It has been previously shown that, in principle, the addition of calcium or rare earth metals to steel has had remarkable effects on desulphurising, deoxidising, and controlling the morphology of non-metallic inclusions. Recently, the use of these elements has markedly advanced due to newer and more effective adding techniques.^{64,65} The effect of rare earth modification on the ductility of 4340 steel at various strength levels has been investigated in the laboratory.⁶⁶ However, for the treatment, some difficulty may be encountered in commercial steel-making practice: (i) the rare earth metals are liable to react with activated oxygen [O] in the molten steel, refractory materials, and atmosphere; (ii) the rare earth oxide and oxysulphide accumulate at the bottom of the ingot and cause the ductility and toughness of the steels to deteriorate. Fortunately, the calcium treatments have already been applied in cases where low temperature toughness is required or in deep water applications where high reliability is demanded,^{67,68} while there is a disadvantage in that calcium is apt to vapourise because of its low boiling point and poor solubility in the molten steel. From this point of view, Tomita⁵⁴⁻⁵⁸ investigated the effect of calcium treatments on the sulphide inclusions and mechanical properties of 4340 grade 0.4C-Cr-Mo-Ni steel. The results are given in Table 8. The remarkably improved K_{IC} data are shown in Fig. 4. The results demonstrate that the calcium treatment (calcium feeding (S/Ca = 3) using Ca-Si wire (Ca/Si = 60/30)) coupled with a low sulphur content of 0.002 mass-% (CaT-2 steel) produced fine particle inclusions con-

Table 8 Mechanical properties of calcium treated 4340 grade steels⁵⁸

Designation of steel*	Test temp., K	Tensile properties			K_{IC} , MPa m ^{1/2}	Charpy impact properties		
		σ_y , MPa	σ_u , MPa	$\epsilon_f \times 10^{-1}$		E_{ch} , J	DBTT (1/2 ΔC_v)	
COM-L (-T)	293	1485.2 (1483.4)	1820.4 (1818.4)	5.2 (1.0)	52.3 (35.8)	42.8 (13.2)	25.2 (8.8)	211 (-)
	193	1585.3 (1582.3)	1920.6 (1921.3)	4.3 (0.5)	44.3 (32.5)	24.4 (7.8)		
CaT-1-L (-T)	293	1476.9 (1479.9)	1820.6 (1825.3)	4.8 (4.5)	52.9 (40.2)	38.2 (18.2)	24.8 (12.8)	220 (-)
	193	1567.5 (1553.2)	1959.2 (1948.9)	2.8 (1.8)	46.3 (35.9)	18.2 (10.5)		
CaT-2-L (-T)	293	1478.3 (1475.9)	1820.2 (1818.1)	5.1 (3.7)	78.7 (72.6)	41.1 (34.8)	25.2 (18.2)	215 (221)
	193	1560.3 (1568.9)	1930.2 (1930.2)	5.3 (3.5)	70.1 (64.1)	24.2 (16.9)		

* CaT-1 = calcium treated steel containing sulphur at commercial levels (0.16 mass-%); CaT-2 = calcium treated steel containing sulphur content at 0.002 mass-%; COM = steel containing sulphur at commercial levels (0.16 mass-%).

Table 9 Effect of decreased hot rolling reduction (DHRRT) on microstructural parameters of 4340 steel containing MnS inclusions⁶⁹

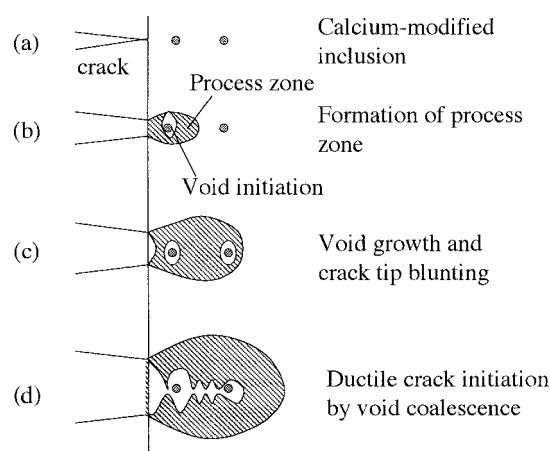
Steel	Prior austenite grain size d_{γ} , μm	Volume fraction of MnS	Arithmetic mean of			RA*, vol.-%
			Length, μm	Width, μm	Aspect ratio	
DHRRT	21.6	0.113	23.8	6.2	3.8	3.6
CHRRT	14.7	0.112	38.4	2.2	17.5	3.5

* RA = retained austenite.

sisting predominantly of CaS–CaO and a slight amount of Ti and Al. Compared with the steel containing sulphur at commercial level (0.16 mass-%) (COM steel), the CaT-2 steel dramatically increased the longitudinal and transverse K_{IC} and E_{ch} at similar strength levels at and above 193 K. Compared with the COM steel, the CaT-2 steel also remarkably improved the isotropy regarding the $1/2\Delta C_V$ and DBTT. The beneficial effect of the calcium treatment on the low temperature mechanical properties is attributed to the fact that the presence of the fine particle sulphide inclusions results in crack extension by a ductile fracture mode in which the main crack proceeds by initiation and coalescence of voids at the inclusion sites in the process zone (Fig. 6). These voids grow and eventually link up as a result of the rupture of intervening ligaments by localised shear. However, the calcium addition to the melt containing sulphur at a commercial level (0.16 mass-%) ($S/Ca > 3$) (CaT-1 steel) was not effective for modifying the morphology of the inclusions and, hence, improving the low temperature mechanical properties. This is because the calcium treatment produces two cluster types of composite inclusion: (i) CaS–CaO–MnS and (ii) CaS–CaO–TiS–TiO₂ which cause brittle fractures in association with the martensite matrix.

Decreased hot rolling reduction treatment

As noted above, the role of the modified sulphide inclusions to improve the fracture toughness and isotropy has been found to suppress the lamellae fracture as much as possible and then to result in the ductile mode fracture. Therefore, the other treatments

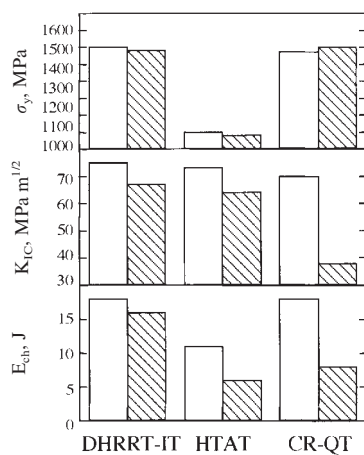


6 Schematic illustration of ductile transgranular fracture occurring in calcium treated steel coupled with low sulphur content of 0.002 mass-% (Ref. 58)

should cause the same effects, provided that they modify the morphology of the inclusions. The obvious alternative treatment is the modification of the sulphide inclusions through a decreased hot rolling reduction treatment (DHRRT). This has a great advantage in that the treatment is more economical in commercial practice. Also, there is an added advantage that, unlike chemical means such as calcium treatment, such mechanical treatments do not require highly developed techniques. From this point of view, Tomita^{69–72} investigated the effect of DHRRT on the modification of the shape of sulphide inclusions and the mechanical properties of several medium carbon, low alloy steels. The sulphide inclusion data obtained by the DHRRT of 4340 steels are compared with those obtained by conventional hot rolling reduction treatment (CHRRT) and are summarised in Table 9. It was observed: (i) the inclusions contained in the steels were mostly sulphide inclusions, MnS, while small amounts of the oxide inclusion, Al₂O₃, and cluster types of composite inclusions were also observed; (ii) decreasing the hot rolling reduction from 98% (CHRRT) to 80% (DHRRT) modified the shape of the MnS inclusions from stringy to ellipsoid, i.e. their length decreased from 38.4 to 23.8 μm , which produced a corresponding increase in the width from 2.2 to 6.2 μm ; (iii) the average aspect ratio of MnS inclusions decreased with hot rolling reduction; (iv) the prior austenite grain size obtained after the DHRRT is similar to that obtained after the CHRRT; (v) the texture of (111) pole figures having a weak pole density of a maximum 1.5 (ratio to α -Fe with random direction) was observed, independent of the hot rolling reduction ratio, and there was little difference in the texture between the steels. Table 10 gives the mechanical properties obtained for the same type of steel similarly processed.⁷⁰ The results obtained by the DHRRT are compared with those obtained by the CHRRT and are summarised as follows: (i) in the longitudinal testing orientation (L), the K_{IC} improved (by 20 MPa m^{1/2}) through DHRRT, whereas, only small differences in tensile properties and Charpy impact energy were obtained. This could be a result

Table 10 Effect of decreased hot rolling reduction (DHRRT) on mechanical properties of 4340 steel in longitudinal (L) and transverse (T) orientations⁶⁹

Steel	σ_y , MPa	Reduction of area, %	E_{ch} , J	K_{IC} , MPa m ^{1/2}
DHRRT-L	1475.6	40.7	26.3	64.7
DHRRT-T	1530.4	31.8	15.8	54.7
CHRRT-L	1486.9	38.3	26.7	44.3
CHRRT-T	1521.0	13.0	6.3	38.4



□ L longitudinal orientation; ▨ T transverse orientation

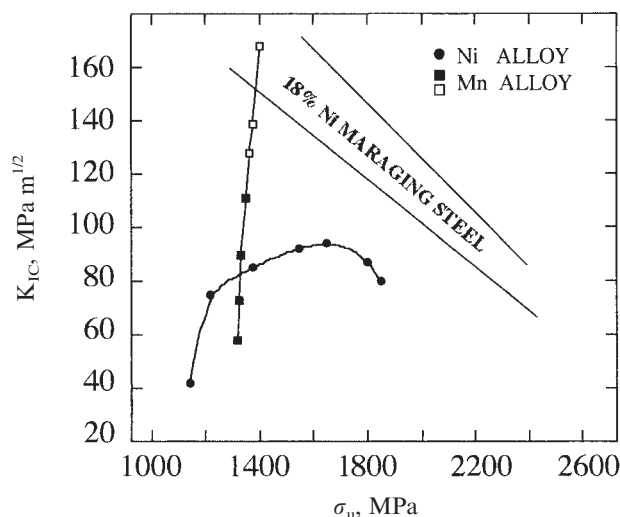
7 Effect of combined decreased hot rolling reduction and short time isothermal transformation treatment (DHRRT-IT), high temperature austenitising treatment (HTAT), and quench and temper after controlled rolling (CR-QT) on 4340 grade 0.4C-Cr-Mo-Ni steel⁷³

of the ellipsoid MnS inclusions separating from the matrix during plastic deformation thereby producing large voids which, during testing, act to blunt and arrest cracks propagating across the specimen that would otherwise cause failure; (ii) in the transverse testing orientation (T), the K_{IC} increased by 17 MPa m^{1/2} through DHRRT. The ductility and Charpy impact energy also increased through DHRRT, but there was little appreciable change of strength. This can be attributed to suppression of the lamella fracture, which occurs in a brittle manner along the interfaces of the MnS inclusion/matrix at the crack tip, as a result of the change in the shape of the MnS inclusions from stringy to ellipsoidal.

A new approach has been suggested by Tomita,⁷³ which consists of combining decreased hot rolling reduction and short time isothermal transformation (DHRRT-IT). Figure 7 shows the mechanical properties of 4340 grade 0.4C-Cr-Mo-Ni steel obtained using this approach; the results obtained by high austenitising treatment and CRP-QT are shown for comparison. The DHRRT-IT steel can be seen to have an improved combination of mechanical properties. From the results of metallography, X-ray measurements, and fractography, this improvement can be attributed to the cumulative effect of the modification of the shape of the MnS inclusions through DHRRT and the improved microstructure obtained via short time isothermal transformation.

Improvement in fracture toughness via new alloying design

As noted above, to meet the requirements of high strength and good fracture toughness, considerable research effort has been directed toward improving the fracture toughness of ultrahigh strength, medium carbon, low alloy steels as indicated in Table 1. In spite of the improvements achieved, there have recently been increased demands for even greater fracture toughness in severe environments. For



8 Relationship between tensile strength σ_u and fracture toughness K_{IC} of Fe-C-2Si base steel⁷⁶

example, manufacturers and designers have sought a material having a minimum K_{IC} of 110 MPa m^{1/2} at a tensile strength equivalent to that of 300M steel. Therefore, they have been interested in developing a replacement of alloy steel with improved fracture toughness for the ultrahigh strength, medium carbon, low alloy steels used traditionally (Table 1). Thus, improvement in fracture toughness by a new alloying design is discussed below.

Addition of manganese or nickel to Fe-C-2Si base steel

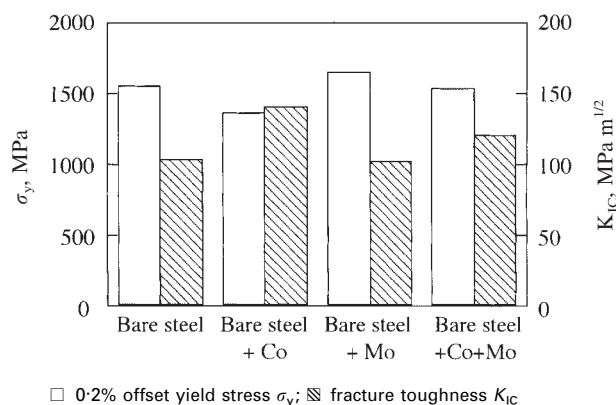
As mentioned above, a thin film of retained austenite contributes to the improvement of fracture toughness. Previous investigations²⁰⁻²⁵ have shown that the presence of silicon in low alloy steel isothermally transformed in the bainitic temperature region encourages the retention of a ductile high carbon austenite region (about 30 vol.-%) rather than the formation of brittle interlath cementite films. Mühkinen and Edmonds⁷⁴⁻⁷⁶ developed Fe-C-2Si base ultrahigh strength, low alloy steels by applying this principle. Their results, which they compared with those for commercial 18%Ni maraging steels, are given in Fig. 8. The addition of manganese to Fe-C-Si base steel (Fe-0.2C-2Si-3Mn steel) dramatically enhanced the fracture toughness (K_{IC} = 160 MPa m^{1/2}) at the 1440 MPa strength level, which was comparable with that attained for 18%Ni maraging steel. Nickel alloying and increased carbon content in Fe-C-2Si base steel (Fe-0.4C-2Si-4Ni steel) produced a good combination of strength and fracture toughness at strengths of 1500–1840 MPa. These authors demonstrated that the presence of ≤12 vol.-% retained austenite as a thermally and mechanically stable thin film has a beneficial effect on fracture toughness, but thermally and mechanically unstable blocky austenite, which is found for retained austenite content >12 vol.-%, has a detrimental effect on fracture toughness. To explain this detrimental effect, the authors proposed that the energy absorbed by the $\gamma \rightarrow \alpha'$ transformation cannot compensate for the brittle nature of the transformation products.

Additions of nickel and silicon to Fe-0.4C-1.0Cr-1.5Ni base steel

Silicon is also known to strongly inhibit the growth of transition carbides and the formation of Fe_3C during tempering, allowing the steel to be tempered at relatively higher temperature. Nickel is known to increase the resistance to the cleavage fracture of iron and causes a significant reduction in DBTT in the Charpy impact test. Thus, separate or combined additions of silicon and nickel would be expected to improve the fracture toughness of ultrahigh strength, medium carbon, low alloy steels. Garrison⁷⁷ has investigated this using 0.4C-1.0Cr-1.5Ni base steel. The results are given in Table 11. Separate addition of nickel and silicon did not significantly improve the Charpy impact energy or fracture toughness of the base steel, but their combined addition dramatically improved both properties. Garrison suggested that the improved toughness can be attributed to the increased sulphide spacing resulting from the effect of the combined silicon and nickel additions on the average sulphide size; however, no explanation was given for the increased sulphide size and spacing.

Addition of cobalt and molybdenum to Fe-C-Ni-Cr-Si base steel

Most recently, Malakondaiah and co-workers⁷⁸⁻⁸⁰ developed ultrahigh strength, medium carbon, low alloy steel with significantly enhanced fracture toughness by fundamental studies concerning the addition of combined cobalt and molybdenum to the Fe-C-Ni-Cr-Si base steel suggested by Garrison.⁷⁷ Table 12 gives the effect of the additions of cobalt and molybdenum on the mechanical properties of the base steel. The strength and K_{IC} data, which are noteworthy, are shown in Fig. 9. The results obtained are summarised as follows: (i) addition of cobalt to the base steel developed an optimum combination of strength and impact toughness and exhibited a K_{IC} value of $140 \text{ MPa m}^{1/2}$; (ii) combined additions of cobalt and molybdenum to the base steel dramatically increased



9 Effect on σ_y and K_{IC} of adding cobalt and/or molybdenum to Fe-Ni-Si-Cr base steel^{79,80}

the strength, while its K_{IC} and Charpy impact energy somewhat decreased compared with those obtained by only cobalt addition to the base steel. The improved strength and fracture toughness are due to the following causes: (i) cobalt additions enhance the fracture toughness by the segregation of carbon to the grain boundaries, thus increasing the cohesive strength of the grain boundary and rendering the crack initiation process much more difficult; and (ii) molybdenum additions increase the strength as a solid solution strengthener, while the fracture toughness is somewhat lowered as a result of the additions of molybdenum.

Conclusions

The present review describes the improvement in the fracture toughness of ultrahigh strength, medium carbon, low alloy steels over about the past 10 years. Obviously, the research presented here is primarily related to the manufacturing processes. However, it must be emphasised that studies on improving the fracture toughness of the steels have just begun. Most recently, there has been an increased demand for improvement of the stress corrosion cracking resistance of ultrahigh strength, medium carbon, low alloy steels at low ambient temperatures. The simultaneous attainment of high strength, cleavage resistance, and resistance to environmentally induced cracking appears to be a difficult problem, because these properties are incompatible in steels. One potential solution to the problem may be to produce a continuous network of thermally and mechanically stable austenite by mechanical alloying.⁸¹ Another solution to the problem may be to control the precipitates, which segregate to the grain boundaries. The key technology is thermomechanical treatments, which change the morphology of the grain boundary segregation of precipitates. Stress corrosion cracking is also exaggerated by the presence of the grain boundary embrittling elements, i.e. phosphorus. The most effective compound for the stable gettering of P in Fe-base alloys is LaPO_4 , and this phase should be accessible at high melt undercoolings achieved by rapid solidification.⁸² By the combination of centrifugal atomisation and late lanthanum additions to the melt, a very fine dispersion of LaPO_4 is obtained which gives the remarkable increase in K_{ISCC} .

Table 11 Effect on mechanical properties at room temperature of adding nickel and/or silicon to Fe-0.4C-0.5Mn-1.0Cr-1.5Ni base steel⁷⁷

Steel	σ_y , MPa	Reduction of area, %	E_{ch} , J	K_{IC} , $\text{MPa m}^{1/2}$
Base	1665	37	26	75
Base + Si	1772	41	30	70
Base + Ni	1565	48	39	79
Base + Ni + Si	1682	53	65	115

Table 12 Effect on mechanical properties of adding cobalt and/or molybdenum to Fe-Ni-Si-Cr base steel^{79,80}

Steel	σ_y , MPa	σ_u , MPa	Reduction of area, %	E_{ch} , J	K_{IC} , $\text{MPa m}^{1/2}$
Base	1550	1825	47	36	103
Base + Co	1360	1670	50	52	140
Base + Mo	1648	1904	48	35	102
Base + Co + Mo	1530	1890	48	34	120

(threshold stress corrosion cracking), which is a parameter indicating the resistance to the stress corrosion cracking, compared to conventional steels. However, there are insufficient data to develop these processes in commercial practice; nevertheless, it is hoped that a solution to the problems will be found from ongoing fundamental studies.

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