

Selected Technical Papers



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Fatigue and Fracture of **Medical Metallic Materials and Devices:** 3rd Volume

Editors:

M.R. Mitchell
Stephen W. Smith
Terry Woods
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Foreword

THIS COMPILATION OF *Selected Technical Papers*, STP1559, on *Fatigue and Fracture of Medical Metallic Materials and Devices: 3rd Volume* contains peer-reviewed papers that were presented at a symposium held November 14, 2012 in Atlanta, GA. The symposium was sponsored by ASTM International Committee E08 on Fatigue and Fracture and Subcommittee E08.05 on Cyclic Deformation and Fatigue Crack Formation.

The Symposium Co-Chairpersons and STP Co-Editors are M. R. Mitchell, Mechanics & Materials, LLC, Flagstaff, AZ, USA, Stephen W. Smith, NASA Langley Research Center, Hampton, VA, USA, Terry Woods, U.S. Food and Drug Administration, Silver Springs, MD, USA, and Brian T. Berg, Boston Scientific Corp., Saint Paul, MN, USA.

Contents

Overview	vii
The Effects of Test Speed on Fatigue Life of Nitinol Wire in Rotary Bend J. D. Weaver, S. Gupta, and T. O. Woods	1
The Effect of Inclusions on Fatigue Properties for Nitinol M. F. Urbano, A. Coda, S. Beretta, A. Cadelli, and F. Sczerzenie	18
Fatigue Crack Initiation in Superelastic Nitinol K. E. Perry and A. Teiche	35
In Vivo Strain Estimates for Medical Implants K. E. Perry, M. M. Quest, T. J. Johnson, and R. Conta	53
Literature Discrepancies in Biomechanical Loading of Orthopedic Trauma Devices Intended for Lower Extremities A. Whitten and J. Cartner	68
Bending Fatigue Characteristics of Orthopedic Biomaterials A. Whitten, J. Cartner, and R. Soileau	85
Ti-6Al-4V Fatigue Strength After Shot Peening: The Role of a Corrosive Environment R. Sonntag, J. Reinders, J. Gibmeier, S. Jaeger, and J. P. Kretzer	98
Evaluation of the Effects of Anodization on the Fatigue Performance of Titanium Alloy A. Whitten	109
Evaluation of Fatigue Performance and Residual Stress of Surface Modification Techniques for Ti-6Al-4V Medical Device Applications S. Terry, J. Rouleau, M. Dace, and M. Pelo	122
Fracture Toughness and Fatigue Crack Propagation Determination of Metal Matrix Composite Materials J. Popp and J. Lessar	141

Overview

This is the publication resulting from the *Third Symposium on Fatigue and Fracture of Medical Metallic Materials and Devices* that was held on November 14, 2012 in Atlanta, Georgia. Committee E08 on Fatigue and Fracture and Committee F04 on Medical and Surgical Materials and Devices sponsored the conference as a cooperative venture to further disseminate state-of-the-art technical information and methodologies regarding the fatigue and fracture of metallic materials employed in medical devices. Approximately 100 individuals were in attendance from the two committees and interactive discussions were quite apparent. This publication provides information on the fatigue behavior of nitinol SMA's and the influence of residual surface stress as well as the influence of inclusions and test speed on long-life performance. Additionally presented is information on biomechanical loading and fatigue characterization of orthopaedic devices. The influence of surface modifications, such as anodization and shot peening, on fatigue and corrosion behavior of Ti-6Al-4V and the fracture toughness of a Ti-MMC concludes the text.

M. R. Mitchell
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Brian T. Berg

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The Effects of Test Speed on Fatigue Life of Nitinol Wire in Rotary Bend

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ABSTRACT: In order to acquire durability data for medical devices in a reasonable amount of time, manufacturers often perform fatigue testing at an accelerated rate. However, the effects of increasing test frequency on the fatigue life of nitinol devices have not been thoroughly investigated. In order to better understand how test speed affects nitinol fatigue, superelastic nitinol wire was tested in body-temperature water in a free (unguided) rotary bend fatigue apparatus. Alternating strains of 0.9 %, 1.0 %, 1.1 %, 1.5 %, or 2.0 % were applied at rotational test speeds of 0.6, 6, or 60 Hz. Except for the highest strain level, results trended toward increased fatigue life at higher test frequencies. Fatigue life at 6 and 60 Hz was significantly greater ($p < 0.05$) than fatigue life at the slowest test speed for alternating strains of 1.0 %, 1.1 %, and 1.5 %. High-speed video of 1.0 % and 1.1 % alternating strain tests at 60 Hz revealed small vibrations that manifested as side-to-side rocking motion of the wire. Because of the unexpected vibrations seen in nitinol wire tests, supplementary testing of cobalt–chromium wire was conducted. High-speed video of cobalt–chromium wire at 0.5 % alternating strain and 333 Hz test speed showed a vertical bouncing motion that appeared to be a second vibration mode. To constrain motion and reduce vibration effects, wire

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³ASTM Third Symposium on *Fatigue and Fracture of Medical Metallic Materials and Devices* on November 14, 2012 in Atlanta, GA.

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guides were constructed and used in additional testing. Guided tests of cobalt–chromium wire showed reduced vibration, resulting in a lowered fatigue life that was nearer to values expected based on literature. Although further testing is needed, vibrations appear to artificially increase fatigue life by altering the imposed radius of curvature and reducing the amount of strain wires undergo in each cycle. Rotary bend wire fatigue testing remains a relatively simple and useful test; however, caution is recommended when the test is conducted without wire guides, as vibrations may affect fatigue life.

KEYWORDS: fatigue, nitinol, speed, frequency, rotary bend, wire

Introduction

Once implanted in the body, medical devices are subjected to a number of repeating mechanical loads. If devices are not designed to withstand these loads or the loads are greater than predicted during the design phase, this repeated mechanical loading can lead to fatigue fracture. Fracture of medical devices is not uncommon and can have severe consequences, especially in high-risk devices such as stents, endovascular grafts, inferior vena cava filters, and transcatheter heart valves. For example, a study of 80 patients with inferior vena cava filters found a fracture rate of 16 % [1]. In another study of femoropopliteal stenting in 93 patients, a fracture rate of 24.5 % was found, and stent fractures were associated with increased complication rates [2]. Manufacturers often conduct preclinical bench testing to demonstrate device durability and safety against fatigue failure. For instance, stents are typically tested according to ASTM F2477-07 to determine their ability to resist fracture when subjected to repeated radial pulsatile loading caused by diameter changes in vessels as the heart beats [3].

Rotary bend wire fatigue testing is commonly used to determine material-level fatigue properties. In this test, a straight wire is bent to a desired radius of curvature and is then rotated about its longitudinal axis, resulting in alternating tension-compression cycles [4–7]. Because of the simple test setup (a motor to spin the wire and a geometrical constraint to maintain the desired strain) and the number of medical devices made from wire, the rotary bend wire fatigue test has become a popular characterization method, and work is currently underway to standardize rotary bend wire fatigue tests.

One aspect of particular interest in fatigue testing in general and in rotary bend wire fatigue testing specifically is the speed (frequency) at which tests are conducted. Pulsatile loading in the arteries depends on the heart rate, which is typically around 70 beats per minute (1.2 Hz). However, commercially available wire fatigue testers from companies such as Valley Instruments and Blockwise Engineering typically run at speeds of 60 Hz or higher. Because faster tests equate to less time and money spent obtaining results, there is an inherent interest in running these tests at an accelerated speed. However, it is unknown exactly how test speed affects the results of these tests. Wagner et al.

found that the fatigue life of nitinol wires decreased with increasing test speed (0.6 to 13.3 Hz) when tests were conducted in air [8]. However, this effect was not present when tests were conducted in a silicon oil bath at constant temperature. Another study, conducted at room temperature and presumably in air, similarly showed a decrease in fatigue life with increased test speed (4.2 versus 7.6 Hz) [9]. Employing a damage-tolerance-based fatigue test configuration, Robertson and Ritchie investigated the fatigue crack growth rate of flattened nitinol tubing under a number of test conditions, including test speeds of 1 and 50 Hz in a simulated body fluid and of 50 Hz in air [10]. Interestingly, they found that the crack growth rate varied only slightly among the three test conditions and that the higher test speeds resulted in marginally faster crack-growth rates for a given stress intensity range. Given the lack of consensus on appropriate test speeds for conducting rotary bend wire fatigue experiments, in the present study we attempted to conduct a thorough examination of the effects of test speed on the fatigue life of nitinol wire in rotary bend. Nitinol was chosen because of its pervasive use in medical devices today, especially in catheter-based technologies that take advantage of the material's ability to recover strains as high as 10 % [11].

Methods

Nitinol Wire Fatigue Testing

Superelastic nitinol wire (Fort Wayne Metals) with an electropolished finish was subjected to fatigue in rotary bend (Fig. 1). The 0.5-mm-diameter nitinol wire (approximately 56 % Ni and 44 % Ti by weight) was provided by the manufacturer with an austenite finish (A_f) temperature between 10°C and 18°C. Wire fatigue experiments were conducted using the Rotating Beam Fatigue Tester Model FTXH (Blockwise Engineering). As seen in Fig. 1, the experiments were performed on free (unguided) wire that was unconstrained except at the ends, which were placed into the rotating chucks of the tester. All tests were performed in a bath of de-ionized water at $37^\circ\text{C} \pm 2^\circ\text{C}$ to mimic body temperature and ensure that the nitinol was in a fully austenitic state. Seven samples were tested at rotational speeds of 0.6, 6, and 60 Hz and alternating strains of 0.9 %, 1.0 %, 1.1 %, 1.5 %, and 2.0 %, for a total of 84 separate tests. All tests at 6 and 60 Hz were allowed to proceed until fracture occurred. Because of time constraints, 0.6 Hz tests were run until wire fracture or until reaching runout at 1×10^6 cycles. The alternating strain ε_a was calculated as

$$\varepsilon_a = \frac{d}{2R} \times 100\% \quad (1)$$

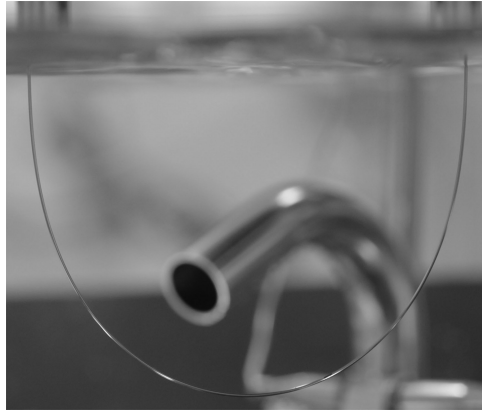


FIG. 1—*Photograph of the free (unguided) wire fatigue test setup. The bent wire can be seen in the water bath, and a portion of the rotating chucks is visible above the water at the top of the image. The final 0.25 in. of each wire end is held within the chucks by a compressive, inward radial force.*

where:

d = wire diameter, and

R = radius of curvature.

Note that the radius of curvature changes along the wire length, reaching a minimum at the midpoint and approaching infinity at both wire ends. For unguided wire fatigue tests, the wire is cut to a length L , given by

$$L = 2.19 \times C \quad (2)$$

C is the distance between the rotating chucks, as seen in Fig. 1. The minimum radius of curvature $R_{\min}$ is then given by

$$R_{\min} = 0.417 \times C \quad (3)$$

This result is based on a solution to the three-point bend problem for a beam that has been subjected to a central bending force that causes both wire ends to take a vertical orientation. In the present experiments, the desired alternating strain was imposed at the apex ($R_{\min}$) according to Eq 1.

Estimating Strain in Static Wire

High-quality images of static nitinol wires in the water bath at $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$ were taken at each alternating strain level. One image was selected at each

alternating strain level to estimate the strain in the wire. The (x,y) coordinates of points along the wires were measured using ImageJ (National Institutes of Health). An “instantaneous” radius of curvature was then estimated by calculating the radius of a circle composed of every three adjacent points along the wire length. The radius of curvature at each point was then used in Eq 1 to estimate strain along the wire length.

Microscopy and Fatigue Crack Area Estimation

Scanning electron microscope (SEM) images of fracture surfaces were taken at varying magnifications with a JSM-6390LV SEM (JEOL). Optical microscope images were taken with a KH-7770 digital microscope (Hirox). With the optical microscope, images of fracture surfaces were taken using an automated three-dimensional stepping method that combined images taken at varying distances from the sample into a single focused image.

Images taken with the optical microscope were used to estimate the relative size of the fatigue crack region. For this, the fatigue crack region was outlined and the area contained within was measured with ImageJ (National Institutes of Health). This area was then divided by the area of the entire fracture surface to obtain the fatigue crack area percentage of each fractured wire.

High-Speed Video and Alternating Strain Estimation

High-speed video of wires during fatigue testing was taken at each alternating strain level with a Motion Pro Y4-S1 (IDT). Video was captured at 100, 500, and 1000 frames per second for the 0.6, 6, and 60 Hz tests, respectively, with a light-emitting diode light source. Alternating strain was estimated for individual video frames using the method described above for estimating strain in static wire.

Cobalt–Chromium Wire Fatigue Testing

As a supplement to the nitinol wire testing described above, fatigue testing of FWM1058 (Fort Wayne Metals) cobalt–chromium wire was also completed. The 0.178-mm-diameter wire was tested in a bath of de-ionized water at $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$ at only one condition: 0.5 % alternating strain at a test speed of 333 Hz. Seven samples were tested in an unguided bend similar to that of the nitinol wire. Four more wires were tested under the same conditions, except that three soft plastic point-guides were placed adjacent to the wire to constrain wire motion, as seen in Fig. 2. High-speed video was taken of both the unguided and the guided tests in order to compare any differences in vibration between the two test setups.

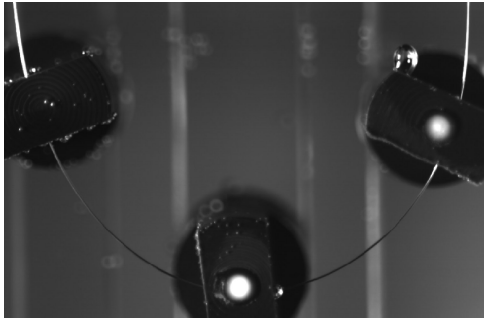


FIG. 2—*Photograph of a cobalt–chromium wire with three guides in place to restrict wire motion.*

Results

Nitinol Wire Fatigue Testing

Results of the nitinol wire fatigue testing are shown in Fig. 3 and Table 1. All tests were run until the wire fractured, except for three wires at 0.9 % alternating strain and 0.6 Hz test speed. These three samples ran out to 1×10^6 cycles, at which point the tests were stopped because of time constraints. For all alternating strain levels except 2.0 %, there was a general trend toward increased fatigue life as the test speed was increased. In particular, fatigue life at the faster test speeds of 6 and 60 Hz was significantly greater than that at the slowest test speed for alternating strains of 1.0 %, 1.1 %, and 1.5 % ($p < 0.05$).

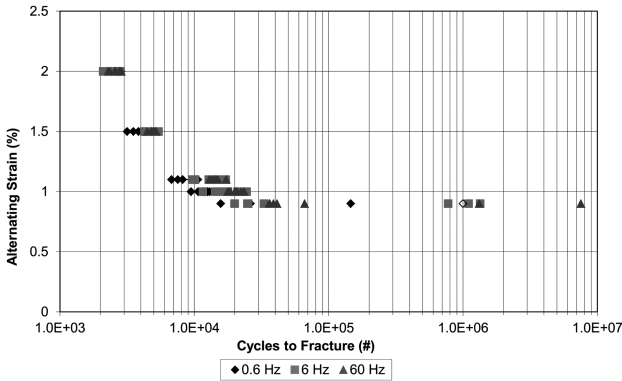


FIG. 3—*Strain-life diagram of all the nitinol wire tests. Each point represents wire fracture except for the open diamond, which represents runout to 1×10^6 cycles of three 0.9 % alternating strain samples at the slowest test speed of 0.6 Hz.*

TABLE 1—*Nitinol wire fatigue test results.*

Alternating Strain, %	Test Speed, Hz	Cycles to Fracture (Average $\pm$ Standard Deviation)
0.9	0.6	53 439 $\pm$ 61 814
	6	473 822 $\pm$ 582 440
	60	1 295 564 $\pm$ 2 790 627
1.0	0.6	11 573 $\pm$ 1192 ^a
	6	17 410 $\pm$ 4291
	60	20 474 $\pm$ 2008
1.1	0.6	9020 $\pm$ 1515 ^a
	6	12 534 $\pm$ 2869
	60	14 362 $\pm$ 1482
1.5	0.6	4011 $\pm$ 546 ^a
	6	4823 $\pm$ 445
	60	4935 $\pm$ 265
2.0	0.6	2598 $\pm$ 218
	6	2544 $\pm$ 253
	60	2600 $\pm$ 218

^aStatistically different from other test speeds at the same alternating strain ($p < 0.05$).

Estimating Strain in Static Wire

Except for 2.0 % alternating strain, all the measured strain values matched closely with the analytically predicted strain values in static wire (Table 2). For 2.0 % alternating strain, however, the measured strain was 2.5 %. For simplicity, and because the strain was not measured for each test, the predicted strain value is used throughout this paper, with the understanding that the measured strain might have differed slightly.

Microscopy and Fatigue Crack Area Estimation

SEM images for a 1.1 % alternating strain at a 0.6 Hz test speed and a 1.0 % alternating strain at a 60 Hz test speed are shown in Fig. 4. In general, fracture surfaces showed two distinct regions: a characteristic thumbnail-shaped fatigue

TABLE 2—*Methods of determining alternating strain.*

Predicted Strain, %	Measured Strain, %	Chuck Distance, mm
0.9	0.9	66.61
1.0	1.0	59.95
1.1	1.0	54.50
1.5	1.5	39.96
2.0	2.5	29.97

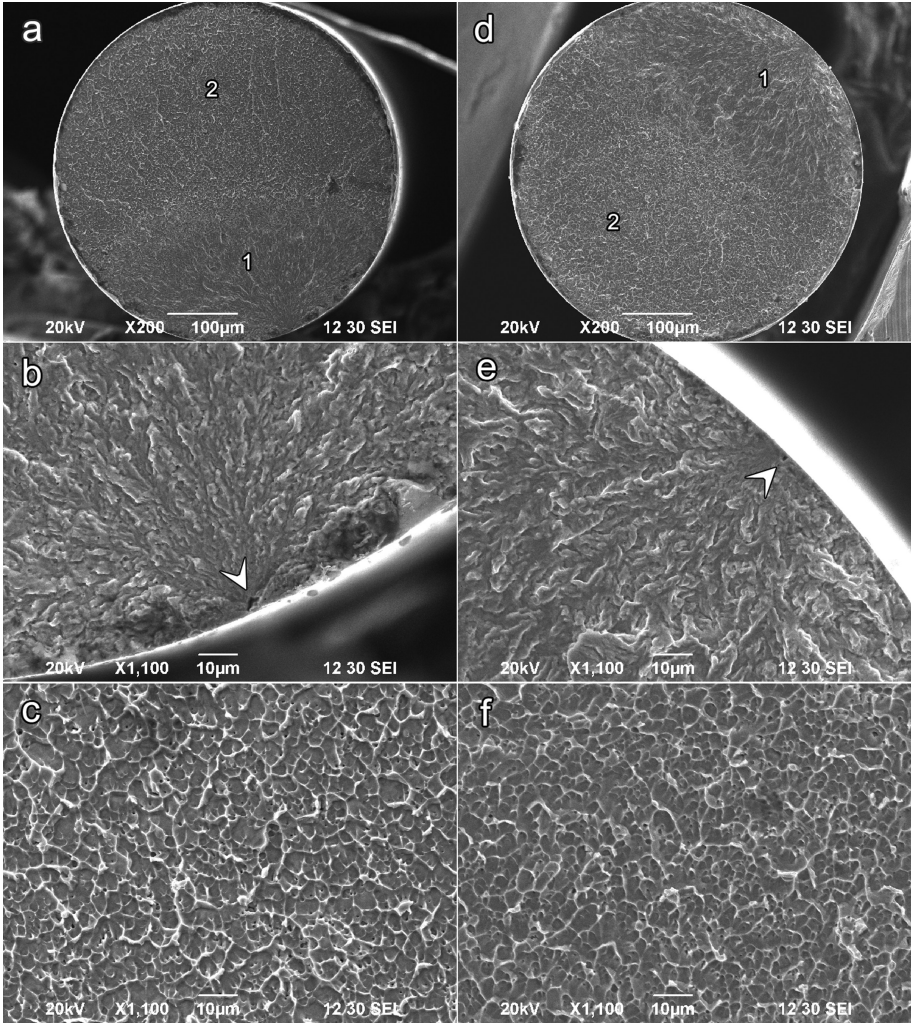


FIG. 4—SEM images of fracture surfaces. (a)–(c) 1.1 % alternating strain test conducted at 0.6 Hz. (d)–(f) 1.0 % alternating strain test conducted at 60 Hz. Panels (a) and (d) show the entire fracture surface; the fatigue crack region is labeled “1,” and the ductile fast fracture region is labeled “2.” Panels (b) and (e) show close-up views of the crack initiation point; small inclusions, indicated by arrows, can be seen in each image. Panels (c) and (f) show the ductile fast fracture regions.

crack region, and a region of ductile tearing or fast fracture. No differences in the morphology of the fracture surface were noted across test speeds for any alternating strain. In many cases, a small inclusion could be found near the crack initiation site.

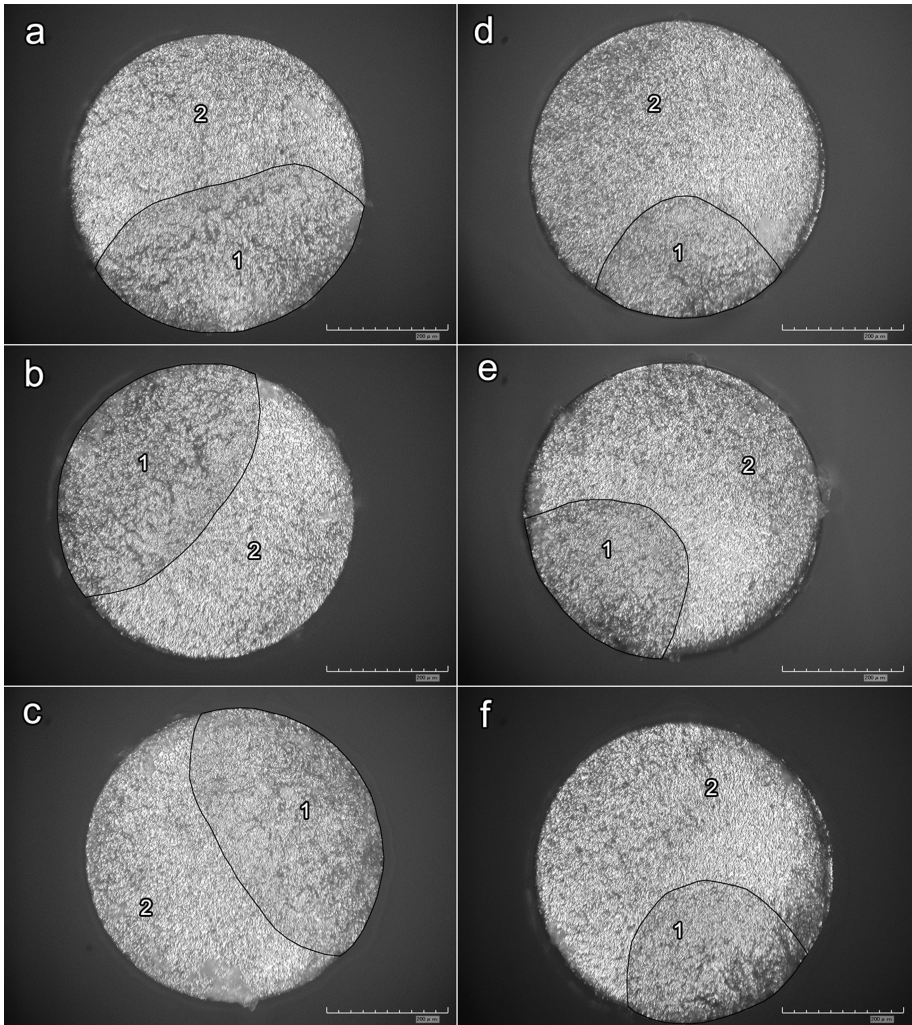


FIG. 5—Digital optical microscope images of fracture surfaces with the fatigue crack areas outlined. The fatigue crack region is labeled “1,” and the ductile fast fracture region is labeled “2.” Panels (a), (b), and (c) show 0.9 % alternating strain tests at 0.6, 6, and 60 Hz test speed, respectively. Panels (d), (e), and (f) are 2.0 % alternating strain tests at 0.6, 6, and 60 Hz test speed, respectively.

Representative fracture surfaces of 0.9 % and 2.0 % alternating strain specimens imaged with the optical microscope are shown in Fig. 5, with the fatigue crack region outlined. The crack shape differed across alternating strain levels, with the 0.9 % alternating strain exhibiting an elliptical crack shape and the 2.0 % alternating strain exhibiting a semicircular crack shape. Intermediate

TABLE 3—*Fatigue crack area as a percentage of cross-sectional area.*

Alternating Strain, %	Test Speed, Hz	Percentage of Cross-Sectional Area (Average $\pm$ Standard Deviation), %
0.9	0.6	45.9 $\pm$ 2.6
	6	44.2 $\pm$ 4.4
	60	45.6 $\pm$ 3.3
1.0	0.6	41.2 $\pm$ 3.8
	6	42.4 $\pm$ 0.9
	60	43.0 $\pm$ 1.9
1.1	0.6	38.9 $\pm$ 1.4
	6	40.3 $\pm$ 1.9
	60	39.8 $\pm$ 2.9
1.5	0.6	28.1 $\pm$ 1.3
	6	31.3 $\pm$ 2.0
	60	30.1 $\pm$ 3.1
2.0	0.6	25.6 $\pm$ 1.5
	6	25.9 $\pm$ 2.0
	60	27.7 $\pm$ 6.7

strain levels exhibited fatigue crack shapes that were between elliptical and semicircular. No change in crack shape was noted across test speeds. The fatigue crack area percentage for all fracture surfaces is given in Table 3. In general, the crack area percentage decreased with increasing alternating strain, as expected. Across different test speeds at the same alternating strain, however, no distinct trends in crack area percentage could be found.

High-Speed Video and Alternating Strain Estimation

High-speed video of the 1.0 % and 1.1 % alternating strain tests at 60 Hz revealed a side-to-side rocking motion as the wire rotated. Two frames of this rocking motion have been superimposed on each other in Fig. 6 to illustrate the extent of the lateral motion. No strong rocking motion was observed in high-speed video of 1.0 % and 1.1 % alternating strain at 6 Hz or 1.5 % alternating strain at 6 and 60 Hz, even though significant differences in fatigue life were observed relative to the slowest test speed of 0.6 Hz. Additionally, upon close examination, some very slight lateral motion could be seen in the high-speed videos of nearly all the test conditions; these small and nearly imperceptible motions did not correlate with the number of cycles to fracture.

In order to determine whether the rocking motion led to changes in the radius of curvature that affected the imposed alternating strain, image analysis of two video frames from each of the 1.0 % and 1.1 % alternating strain conditions at 60 Hz was conducted to estimate the strain. Neither condition revealed



FIG. 6—Two superimposed frames from a high-speed video of a nitinol wire illustrating the lateral rocking motion. The alternating strain was 1.0 %, and the test speed was 60 Hz.

a measurable difference in strain between the two extremes of the lateral motion. Figure 7 illustrates the relatively minor differences obtained in the strain estimations for the 1.0 % alternating strain case at 60 Hz.

Cobalt–Chromium Wire Fatigue Testing

As seen in Table 4, unguided tests of the cobalt–chromium wire resulted in an average fatigue life of $119\,948 \pm 20\,127$ cycles for the seven samples tested. The addition of wire guides resulted in fatigue lives that varied from 51 583 to 110 971 cycles. As is noted in Table 4, in guided tests 1 and 4, which ended in fracture after a similar number of cycles as in the unguided tests, the test wire appeared to be bouncing off the wire guides during all or a portion of the test.

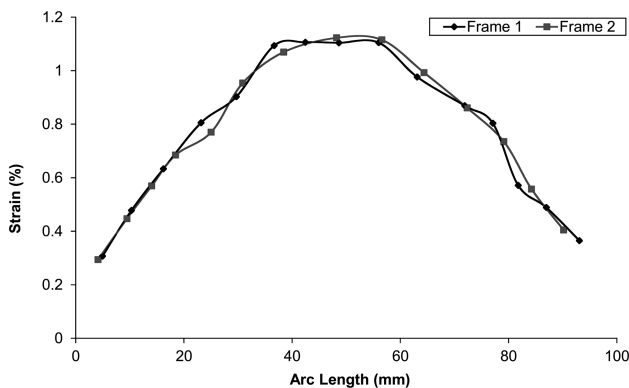


FIG. 7—Alternating strain versus arc length for two frames from a high-speed video of a nitinol wire with an alternating strain of 1.0 % and at a test speed of 60 Hz. The two frames were taken from the two extremes of the rocking motion seen in Fig. 6.

TABLE 4—Cobalt–chromium wire fatigue test results.

Test Description	Cycles to Fracture
Unguided average $\pm$ standard deviation ($n = 7$)	119 949 $\pm$ 20 127
Guided 1	110 971 ^a
Guided 2	54 844
Guided 3	51 583
Guided 4	97 807 ^b

^aWire may have been bouncing on the guides.

^bGuide positions were readjusted in the middle of the test after the wire was observed to be bouncing on the guides.

In contrast, guided tests 2 and 3 did not show obvious vibratory motion during testing and resulted in a lower fatigue life.

High-speed video of an unguided cobalt–chromium wire fatigue test showed the wire undergoing a vertical bouncing motion in addition to the expected rotation about its longitudinal axis. This bouncing motion appeared to be a different vibration mode than that observed with the nitinol wire at 1.0 % and 1.1 % alternating strain at 60 Hz test speed. Figure 8 shows an overlaid image of the high and low points of the bouncing motion. When the three guides were in place, as shown in Fig. 2, high-speed video of the cobalt–chromium wire showed very little or no motion aside from the wire rotation.

Discussion

The present work demonstrates that test speed can indeed affect the results of rotary bend wire fatigue testing and leads us to advise caution in the selection of an appropriate test speed, as faster test speeds trended with artificially increased fatigue life for all alternating strain levels except 2.0 %. Fatigue life was found to be significantly greater in unguided 1.0 %, 1.1 %, and 1.5 %

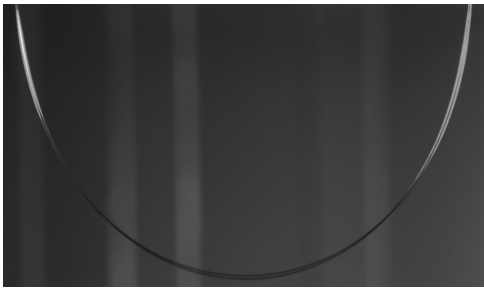


FIG. 8—Image showing two superimposed frames from a high-speed video of a cobalt–chromium wire illustrating the vertical bouncing motion. The alternating strain was 0.5 %, and the test speed was 333 Hz.

alternating strain tests conducted at the faster test speeds of 6 and 60 Hz than in tests at the slowest test speed of 0.6 Hz. Imaging of the fracture surfaces did not reveal differences in fracture morphologies or mechanisms underlying the observed differences in the number of cycles to fracture. In contrast, high-speed video of wires during testing did show some differences between fast and slow test speeds. High-speed videos of 1.0 % and 1.1 % alternating strain tests at 60 Hz showed a side-to-side rocking motion. This rocking motion might have led to changes in the radius of curvature along the wire length that resulted in lower strains than expected being applied to the wire during each loading cycle and thus a greater number of cycles to failure. Interestingly, high-speed video revealed strong rocking motions at only the 1.0 % and 1.1 % alternating strains at 60 Hz test speed. As mentioned previously, even though there were significant differences in fatigue life for 1.0 % and 1.1 % alternating strain at 6 Hz and 1.5 % alternating strain at 6 and 60 Hz (relative to the slowest test speed of 0.6 Hz), no strong rocking motion was observed in high-speed video of these test conditions. It is therefore important to note that although high-speed video can be a useful tool for examining what is happening during a wire fatigue test, the fatigue life of wires may be affected by small motions that are not obvious in high-speed video. However, it should also be noted that some very slight lateral motion could be seen in the high-speed video of nearly every test condition. These small and nearly imperceptible motions did not correlate with the number of cycles to fracture and appear to be very different from the obvious rocking motion shown in Fig. 6. Given the challenges in observing and interpreting wire motion, it can be difficult to interpret results of unguided high-speed wire fatigue tests.

In an effort to better understand the underlying fatigue behavior of the nitinol wire, power law models for fatigue life were determined for each of the three test speeds between alternating strains of 1.0 % and 2.0 %. The model for the 60 Hz experimental data is given by

$$\varepsilon_a = 25.07 \times N^{-0.33} \quad (4)$$

where:

ε_a = alternating strain, and

N = number of cycles to fracture.

Data from 0.9 % alternating strain tests were excluded as the material entered the high-cycle fatigue regime at this point, leading to large scatter in the experimental data. The power law model fit the experimental data well, with all three models having an R^2 value greater than 0.94.

Using Eq 4, the predicted fatigue life for 1.0 % alternating strain would be calculated as 19 503 cycles, which is in good agreement with the experimental data (20 474 cycles). As a point of comparison, a fatigue life of 11 573 cycles

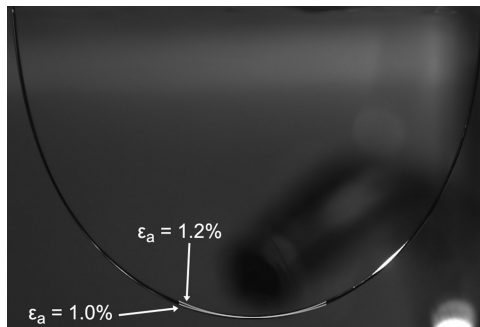


FIG. 9—*Photograph of a 1.0 % alternating strain nitinol wire test with two arcs superimposed to show the radius of curvature required in order to impose a 1.0 % and a 1.2 % alternating strain at the wire apex.*

(the experimental value obtained at 1.0 % alternating strain and 0.6 Hz test speed) would be caused by an alternating strain of 1.2 % according to the 60 Hz model. In order to provide a visual comparison of the different radii of curvature that would give rise to 1.0 % and 1.2 % alternating strain, both radii are overlaid on a picture of a wire with a strain of 1.0 % in Fig. 9. In the image, the arcs that illustrate the two radii are nearly identical at the wire apex. Practically speaking, it would be very difficult to distinguish between the two. Because of this, it might be difficult to measure accurately with the methods described herein the changes in alternating strain resulting from small wire motions.

In general, the predicted strain was comparable to the measured strain, except for the 2.0 % predicted strain. In this case, the measured strain was higher at 2.5 %. This divergence may be attributed to the stress-induced phase transformation from austenite to martensite that takes place once the material has been strained to 2.0 %. Because the austenite and martensite phases have different elastic moduli, the initial assumptions underlying the analytical (predicted) strain value no longer hold, and the radius of curvature at the apex of the wire is smaller than predicted. This behavior is not unexpected and was observed in a previous study on the bending of nitinol wire [12]. Therefore, the actual strain induced in the 2.0 % case was likely closer to 2.5 %.

Unguided cobalt–chromium wire tests revealed what appeared to be a second vibration mode in which the wire bounced in a vertical motion (Fig. 8). It was easier to see how the wire apex flattened slightly as it bounced in these tests than with the rocking motion observed for the nitinol wire. It seems likely that this vertical wire motion led to an increase in the radius of curvature that artificially increased the fatigue life, especially given the dramatic change in fatigue life seen once the guides were put in place. When the guides were properly positioned (as in guided tests 2 and 3), the fatigue life dropped from around 120 000 cycles to around 53 000 cycles. Additionally, this lower fatigue

life is more in line with fatigue data extrapolated from an earlier study of cobalt–chromium wire [13], leading to more confidence in results from the guided tests. During guided test 1, the wire appeared to be bouncing against the guide, and the fatigue life was essentially unchanged from that observed in the unguided tests. During guided test 4, the wire bounced against the guide during a portion of the test. The test was stopped so adjustments to the guides could be made, and the resulting fatigue life fell between results from guided and unguided tests. These results demonstrate that guides can reduce vibrations but must be positioned properly in order to do so. A better method to eliminate vibration effects might be to create a guide with an arc shape that follows the wire along its entire length, thus eliminating user variability in the placement of the guides.

One drawback to using wire guides is that they could introduce frictional forces or additional wear that could accelerate the fatigue process. However, these effects can be mitigated by the use of a soft plastic material with a low coefficient of friction that is unlikely to scratch the sample surface. Furthermore, wire fatigue testing with guides is relatively common, and no issues related to excessive friction have been reported with the technique [5,7,14]. Given the difficulties presented here regarding the interpretation of results from unguided wire fatigue tests at high speed, guided-bend tests might be preferable.

One of the limitations of the present study is that it was conducted in de-ionized water, rather than in a simulated body fluid such as saline. Although testing in saline would have been more physiologically relevant, we believe the results remain valid given the electropolished surface of the nitinol wires. However, future work will include the addition of a simulated body fluid in order to assess the effects of a corrosive environment in addition to test speed.

Although the work presented here was conducted at relatively high alternating strains that might not typically be imposed on implanted medical devices [15], vibration issues could be a concern at lower alternating strains as well. If vibrations were present but not detected, researchers might overestimate fatigue lives, leading to medical devices that were not designed sufficiently to withstand fatigue failure. Overall, researchers and designers conducting high-speed wire fatigue testing and interpreting the results of those tests should be aware of the possible effects of test speed and take caution when designing and conducting experiments.

Conclusions

Test speed can affect results in free (unguided) rotary bend wire fatigue experiments. Vibration effects appear to alter the radius of curvature in the wire, resulting in a lowered alternating strain that leads to an artificially high fatigue

life. High-speed video can be helpful in identifying these vibration effects, but fatigue life may be affected even if vibration is not observed in the high-speed video. Wire guides, when properly placed, appear to reduce the dependence of fatigue life on test speed, although further work is needed to determine the best way to use guides. Overall, rotary bend wire fatigue testing remains a useful and relatively simple means of determining the fatigue properties of a material, but caution must be used when conducting such tests in order to minimize the effects of the test speed.

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The Effect of Inclusions on Fatigue Properties for Nitinol

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ABSTRACT: ASTM F2063-05 covers microcleanliness in wrought nickel-titanium shape memory alloys for medical devices and surgical implants. Microcleanliness is evaluated in mill products with section sizes of between 6.3 and 94 mm in diameter, thickness, width, height, wall thickness, and so forth. These dimensions exclude most of the finished components for biomedical applications. Thus, the inclusion assessment is conducted at an intermediate manufacturing process step. To our knowledge, no rigorous method has been developed to correlate the inclusion dimensions in the final product to the dimensions in precursor semi-finished materials. Moreover, the method of inclusion analysis must be based on a robust statistical approach in order to define accurately the inclusion content. Finally, specifications about microcleanliness should rely on studies explaining the correlation between inclusion size and material performance, in particular fatigue. We propose to consider extreme-value inclusion rating, already applied to rate inclusions in hard steels, for shape memory alloy. To this end, a study of the effect of inclusions on nitinol fatigue was undertaken. Here we present the results of the first part of the study, concerning rotating-bending fatigue on 0.3-mm-diameter superelastic wires. Materials from seven ingots were

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tested. A comprehensive experimental campaign was conducted involving tests at different strain amplitudes. The fracture surfaces of failed wires were analyzed through scanning electron microscopy, and data regarding the presence of particles and their morphology were recorded. Collected data were subsequently analyzed. By means of a simple interpretative model, we compare the fatigue performances of four different processes and show that they are different. Then we show qualitatively how inclusion dimensions affect fatigue. Finally, by means of extreme value statistics, we rate the particle dimensions of the four processes and correlate them to fatigue performance. An outline of ongoing and future work is also provided.

KEYWORDS: extreme value inclusion rating, nitinol, superelasticity, bilinear uniform scatter band

Introduction

Starting with the studies of Melton and Mercier in 1979, significant efforts have been devoted to the study of the fatigue behavior of nitinol. The reader can refer to the review by Adler et al. from 2007 [1] and a more recent paper by Pelton [2] for an overview of fatigue studies on nitinol and the difficulties related to this peculiar material; the type of annealing, the nickel/titanium composition, and the co-presence of martensite and austenite are just some of the many parameters potentially affecting the fatigue behavior of nitinol that have been considered to date.

To our knowledge, however, no systematic research has been performed to study the possible effects of inclusions on the fatigue performance of nitinol, and only a few papers treat this issue. In Ref 3, the rotary beam fatigue testing (RBT) of 267- μm -diameter wires obtained from different suppliers and characterized by different maximum inclusion size dimensions are compared, but no conclusive statements are provided in the paper. In Ref 4, the nucleation of cracks is considered, and inclusions, scratches, and surface defects are recognized as nucleation sites of fatigue cracks. In Ref 5, inclusions are recognized as key contributors to failure for as-drawn or electro-polished nitinol wires. In contrast, for wires undergoing a grinding process, surface defects are the key contributors to failure; ground wire exhibits far better fatigue performance as a result of either compressive surface stresses induced by the process or surface inclusion removal.

The limited knowledge about the effect of inclusions is reflected in the regulations pertinent to the characterization of inclusions in nitinol. The inclusion rating is covered only in ASTM F2063-12 [6], which requires the evaluation of microcleanliness in wrought nickel-titanium shape memory alloys for medical devices and surgical implants. In this standard, microcleanliness is evaluated in mill products with section sizes between 6.3 and 94 mm in diameter, thickness, width, height, wall thickness, and so forth. These dimensions exclude most of the finished components for biomedical applications. Thus, the inclusion assessment is conducted at an intermediate manufacturing process step.

Quite recently, a standard practice for the statistical analysis of nonmetallic inclusions in steels was introduced [7]. The aim of the practice is to estimate the expected largest inclusion in a determined volume of material. The need for a thorough method for the analysis of inclusions followed the studies reported in Ref 8, focused in particular on ball bearing steels, that demonstrate the negative effect of inclusions on the fatigue behavior of hard steels and the need to adopt the *extreme value inclusion rating*.

In this work, we present the first part of a research program aimed at verifying that the findings of Refs 7 and 8 are applicable to nitinol. We have proceeded as follows: We have selected lots of superelastic wires manufactured with differing melting techniques and hot processing steps but presenting almost equal thermo-mechanical properties. The process differences are supposed (this is verified a posteriori) to influence the inclusion size and nature of superelastic wires. Wires underwent RBT at several strain levels, and fracture sections were observed via field emission scanning electron microscopy, at which time we measured the dimensions of the inclusions present at the fracture nucleation sites. At this stage one can note (at the moment only qualitatively) the negative effect of inclusions on fatigue, which is independent of the kind of melting process. The whole set of fatigue data collected for each process was analyzed through an interpretative model, namely, the *bilinear uniform scatter band model*. This analysis allows us to appreciate quantitatively how different processes influence the fatigue behaviors, in particular in the low-strain/long-life region. Similarly, the statistical distribution of the recorded inclusion sizes for each process is considered. The distributions were found to follow the Gumbel distribution and to be different, confirming the initial assumption. Moreover, the size of extreme inclusions for the different materials is correlated to fatigue limits obtained in the experimental tests.

Experimental

Materials

The samples for this study were from seven Ni-44.0Ti wt. % ingots and eight lots of wires manufactured by means of four different processes, referenced as P1, P2, P3, and P4. The processes differed in the melting, through varied combinations of vacuum induction melting and vacuum arc re-melting, and in the hot-processing steps. Cold drawing, interpass annealing, and final straight annealing were carried out according to a standard recipe. The final surface finish was amber oxide for all the wires.

Every material was characterized at the ingot level in terms of gas impurities (C, O) using a LECO analyzer (ASTM F2063). Differential scanning calorimetric analysis was performed on both coil and annealed wires according to

TABLE 1—*Chemical and mechanical properties of the tested wires.*

TTR										
Material	Impurities, ppm		Coil	Wire		Mechanical Properties (37 °C)				
Heat	Carbon	Oxygen	A _s	A _f	A _f ^{BFR}	UTS, MPa	ε _{max}	ε _{res}	UPS, MPa	LPS, MPa
P1,1	265	211	−11	12	1	1151	10.9	0.15	553	338
				15	6	1165	9.6	0.11	557	373
P2,1	206	215	−22	12	2	1158	10.1	0.16	585	395
P2,2	269	254	−18	14	0			0.16	593	370
P2,3	261	266	−15	12	2			0.16	583	368
P2,4	270	254	−13	14	5			0.17	580	360
P3,1	250	250	−9.5	16	4	1158	10.7	0.22	573	372
P4	<20	210	−18	13	2	1138	10.9	0.19	569	365

ASTM F2004 [9]. Bend-free recovery and tensile tests were performed on straight annealed wires according to ASTM F2082 [10] and F2516 [11]. The results are summarized in Table 1. For this experimental campaign, we selected wire lots having similar thermo-mechanical properties in order to minimize the effects of their variability on fatigue.

Metallographic longitudinal sections of wires from the four different processes were prepared according to standard practice and analyzed via scanning

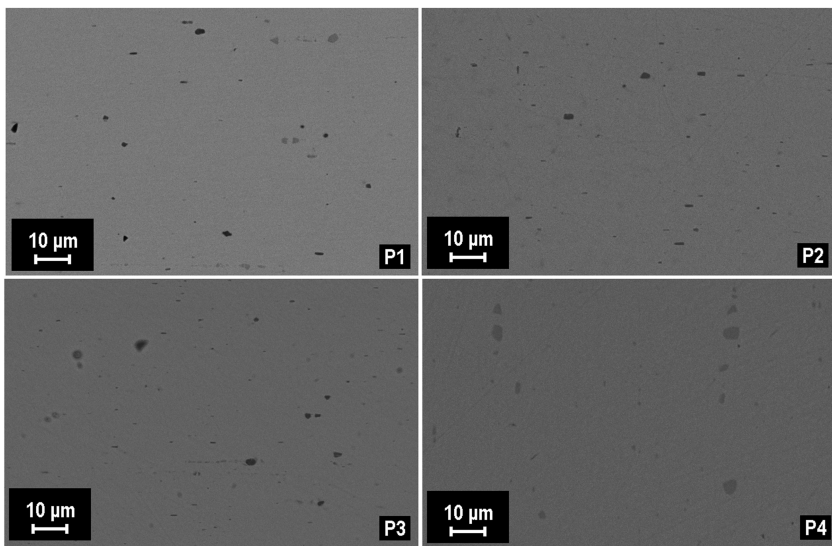


FIG. 1—BSE SEM images of longitudinal metallographic sections of wires from processes P1 to P4. Black particles are titanium carbide inclusions, and gray particles are intermetallic oxides.

TABLE 2—Inclusion contents of the four examined nitinol wires.

Wire	Maximum Area Fraction, %		Maximum Inclusion Size, μm	
	Carbides	Oxides	Carbides	Oxides
P1	0.73	0.15	6.33	7.18
P2	0.47	<0.01	7.07	<0.1
P3	0.73	0.04	14.06	3.83
P4	0.02	0.91	2.65	34.31

electron microscopy (SEM). In Figures 1(a)–1(d), back-scattered secondary electron (BSE) SEM images of the four materials are reported. The main differences are in the nature of inclusions. Material P1 shows a mix of carbides (in black) and oxides (in gray); in materials P2 and P3, only carbides were detected, and in material P4, mainly oxides were present. Although all wires underwent the same cold drawing and annealing process, it is not possible to exclude completely other microstructural differences, such as different grain sizes.

A preliminary inclusion-size analysis was conducted according the internal procedure described in Ref 8. Table 2 summarizes the results in terms of maximum area fraction and maximum inclusion size for both carbides and oxides.

Fatigue Tests

Almost 500 samples were fatigue tested by means of RBT at different alternating strain amplitudes. A Blockwise rotating beam wire fatigue tester (model FTX) was used for all experimentation. All tests were performed at 1000 rpm in a temperature-controlled water bath at 37 °C. Between 5 and 15 tests were repeated for each deformation, and the run-out criterion was fixed at 1×10^6 cycles. Some tests, however, were further extended, with run-outs fixed at up to 24×10^6 cycles.

More than 100 failed wires were analyzed via SEM. In observing the fracture surfaces, it was possible to recognize whether an inclusion caused the nucleation of the crack, and when present, the dimensions were measured. The nature of inclusions (carbide or oxide) was also identified and recorded based on dimensions, morphology, and different gray scales of BSE imaging, or by using an EDX microprobe. In Fig. 2, a typical fractographic section is shown in which a particle at the fracture origin, a fracture propagation, and a collapse region can be distinguished. In Fig. 3, different types of inclusions observed are shown. The particle's distance from the surface and its shape have been taken into account in order to properly compensate for the particle area, as recommended in Ref 7.

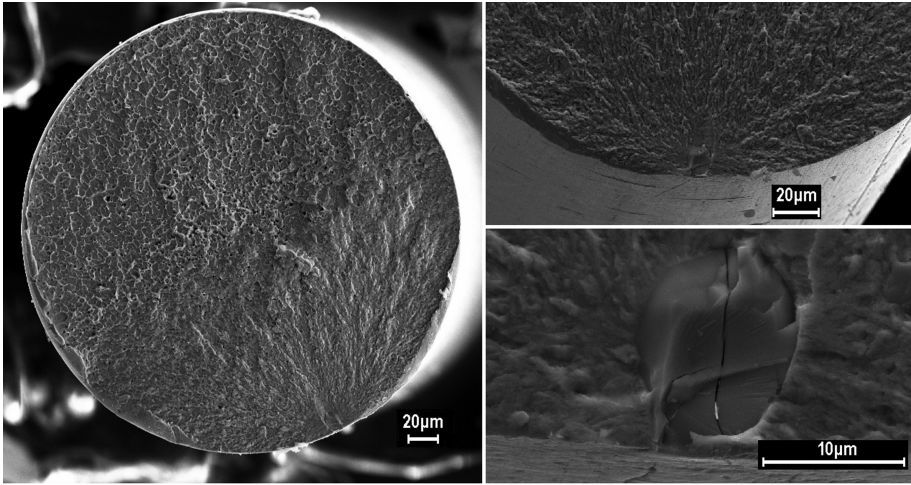


FIG. 2—Results of fatigue tests: (a) a typical fracture surface with details (b, c) of the initial propagation region, where a $Ti_xNi_yO_z$ particle can be seen at the fracture origin.

Rotary Beam Fatigue Testing Result Analysis

The typical results of rotating bending fatigue tests can be plotted in $\epsilon-N$ diagrams, similar to the $S-N$ diagrams usually adopted in fatigue. Considering that the testing plan was aimed at determining the fatigue limit in the long-life region, a statistical analysis able to describe both fatigue regions (namely, “finite life” and “fatigue limit”) was adopted.

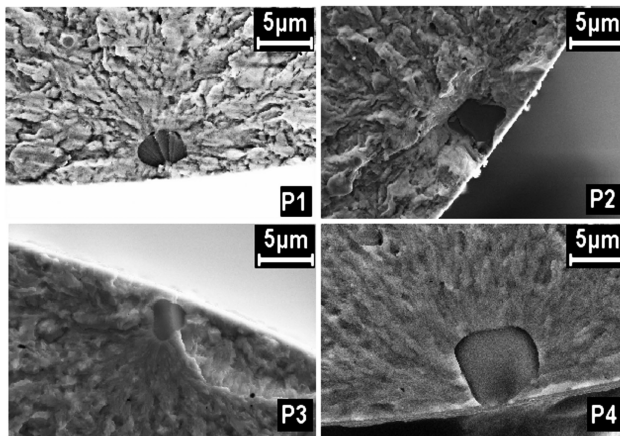


FIG. 3—Typical inclusions observed at the crack origin; inclusions differ in size, position, and nature (oxides or carbides).

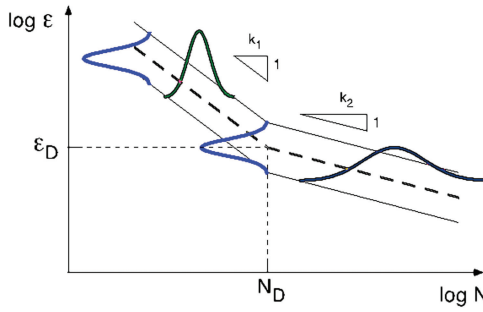


FIG. 4—Concept of the uniform scatter band applied to wire rotating bending fatigue data.

In particular, we used the model of the *bilinear uniform scatter band* (first proposed by Haibach [9]), which is common in the elaboration of design curves for components subjected to fatigue (see, for example, Eurocode 3 curves for welded joints). The simple assumption behind the method is that in a log S –log N diagram, a fatigue response can be described by means of a statistical model with constant dispersion along the log S direction. The same concept was adopted for the analysis of the present ϵ – N data; consequently, the following hypotheses (which are consistent with the ASTM E739 recommendations [12]) were included in the statistical model (see Fig. 4):

- Fatigue life—that is, the dependent variable in the fatigue experiments—is assumed to be log-normally distributed.
- The dependence of fatigue life on ϵ is modeled as

$$\begin{cases} \mu_{\log N} = \log N_D + k_1 \cdot (\log \epsilon - \log \epsilon_D) & \text{for } \epsilon > \epsilon_D \\ \mu_{\log N} = \log N_D + k_2 \cdot (\log \epsilon - \log \epsilon_D) & \text{for } \epsilon < \epsilon_D \end{cases} \quad (1)$$

where N_D and ϵ_D identify the “knee” in the fatigue response.

- In order to model the uniform scatter in the log ϵ direction, the standard deviation of log N is modeled as

$$\begin{cases} \sigma_{\log N} = \sigma_{\log \epsilon} \cdot k_1 & \text{for } \epsilon > \epsilon_D \\ \sigma_{\log N} = \sigma_{\log \epsilon} \cdot k_2 & \text{for } \epsilon < \epsilon_D \end{cases} \quad (2)$$

The analysis of the fatigue data for the different wire batches was conducted using the maximum likelihood method [10], maximizing the log-likelihood of the data for a given sample.

$$\ell = \sum_{i=1}^n \log(f(y_i)) + \sum_{j=1}^u \log(1 - F(y'_j)) \quad (3)$$

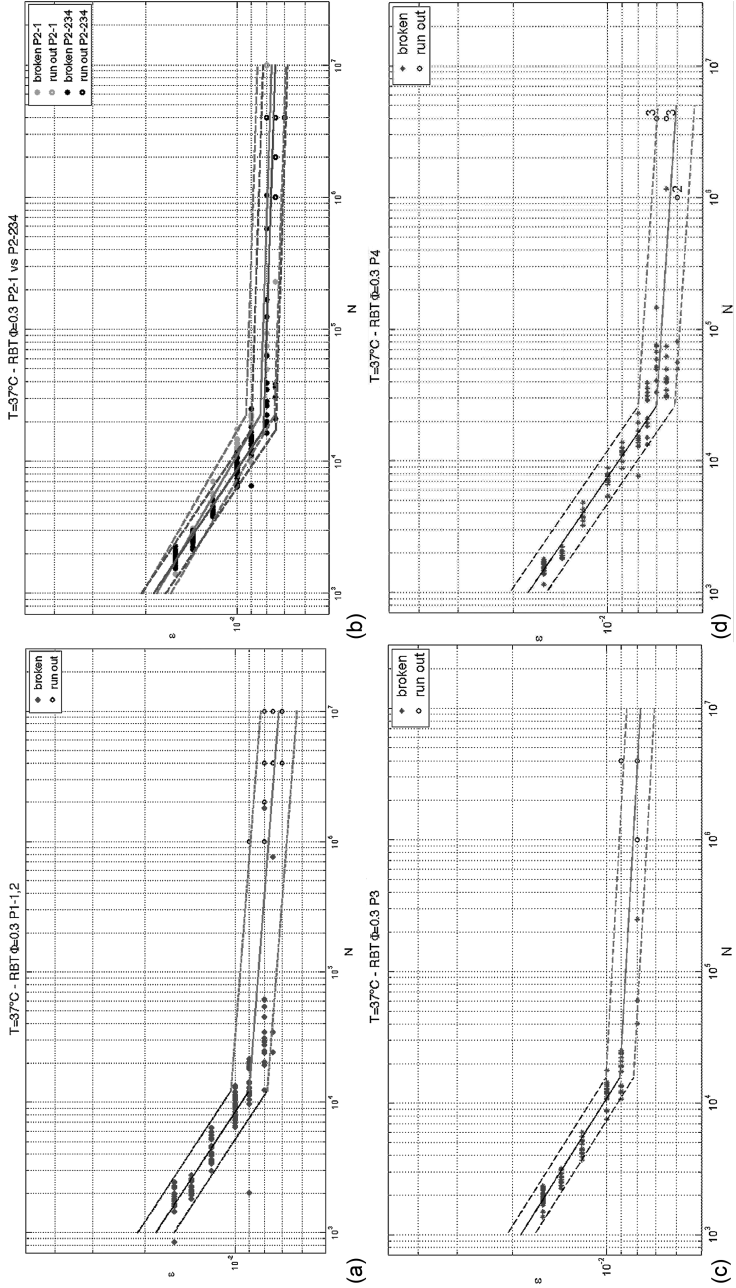


FIG. 5—Interpolation of RBT fatigue data on nitinol wires: (a) P1 data for H1–H2; (b) P2 data (the three heats); (c) P3 data; (d) P4 data.

TABLE 3—Best-fit parameters of the bilinear uniform scatter band models and fatigue limit corresponding to strain amplitudes for 50% failure probability at 10^6 cycles.

Material	k_1	k_2	ε_D , %	N_D	ε_{av} (at 10^6 cycles), %	ε_{inf} (at 10^6 cycles), %
P1	−3.46	−24.64	0.90	11 900	0.75	0.66
P2	−3.60	−57.89	0.84	17 900	0.78	0.70
P3	−3.78	−42.18	0.91	15 600	0.82	0.74
P4	−3.46	−36.08	0.70	26 000	0.63	0.55

where:

$f(y)$ and $F(y)$ =respectively, the probability density and the distribution function of the fatigue life y , which is assumed to be a log-normal distribution whose parameters are described by Eqs 1 and 2, and

y_i ($i = 1, \dots, n$) and y'_j ($j = 1, \dots, u$)=a complete test (failure) and a suspended test (run-out), respectively.

The results of such analyses for the four materials are shown in Fig. 5, where it can be seen that the analysis with the bilinear uniform scatter band was able to provide a complete picture of the fatigue behavior of the wires. The fatigue properties that were considered relevant are the strain corresponding to 50 % failure probability at 10^6 cycles, ε_{av} , and its lower bound (which was calculated as the 5 % percentile), ε_{inf} . A series of statistical likelihood ratio tests [10] showed that there was no significant difference between the two lots of P1. Similarly, the statistical tests indicated no significant difference between the four heats of P2; accordingly, data were interpolated by a single curve for each process. A comparison of the model parameters for the four processes is shown in Table 3; it can be noticed that the wires with the lowest fatigue limit were the P4 wires, and the best fatigue limit corresponds to P3 wires. In the following sections, we investigate whether there is a correlation with the size of inclusions.

Effect of Inclusions on Fatigue

Experimental Evidence

Several inclusion observations at the origin of the fracture surface were available for the first lot of P1. The dimensions of the inclusions (expressed in terms of the parameter $\sqrt{\text{area}}$) are reported in relation to the fatigue response in Fig. 6; it can be seen that at strain levels near the fatigue limit (e.g., strain levels 0.008 and 0.009), the fatigue life is undoubtedly inversely correlated to the defect dimensions. Alternatively, most of the data points lying on the 5 % line represent the largest inclusions. This fact illustrates clearly the detrimental

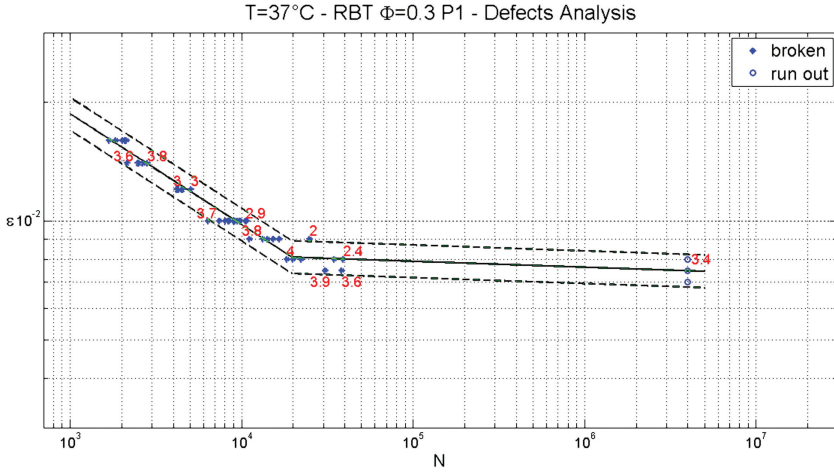


FIG. 6—Empirical correlation between test results and inclusion dimensions for the first lot of P1 wires. The inclusion size in microns (expressed in terms of $\sqrt{\text{area}}$) is reported in the plot with the relative ϵ - N value for most fatigue data.

effect of inclusion size on the fatigue limit of nitinol wires, the main topic of the present research.

Models in the Literature

The fatigue strength of a component containing defects or inhomogeneities subjected to axial or bending forces can be predicted on the basis of the models of Murakami and Endo [13]. They showed that at the fatigue limit, small non-propagating cracks are always present at a defect tip. Because of this fact, the inhomogeneities can be treated as cracks, and accordingly the fatigue limit σ_{lim} has to be calculated as the cyclic threshold stress at which the cracks emanating from the defects do not propagate.

This type of analysis firstly implies the need to calculate the stress intensity factor for a crack with an irregular shape. Murakami suggested that the maximum stress intensity for a crack with an irregular shape could be calculated easily with the simple equation

$$K = 0.65\sigma\sqrt{\pi\sqrt{\text{area}}} \quad (4)$$

where $\sqrt{\text{area}}$ is the square root of the defect area projected on a plane perpendicular to the applied stress. Because of this model, in the following description the inclusions size d is expressed in terms of $\sqrt{\text{area}}$. Prediction of the fatigue limit would therefore be possible considering:

$$\Delta\sigma_{\text{lim}} = \frac{\Delta K_{th}}{0.65 \cdot \sqrt{\pi \sqrt{\text{area}}}}. \quad (5)$$

However, the application of Eq 5 is not straightforward because the fatigue threshold is a function of crack or defect size [11], and only for long cracks does it tend to have a constant value, $\Delta K_{th,LC}$. A conservative approach, proposed by Robertson and colleagues [14], is to estimate the ΔK_{th} for small cracks in terms of a lower bound of the da/dN curves obtained on standard CT specimens. A better alternative is to model the $\Delta K_{th}-d$ (where d is the defect/crack dimension) relationship (that is, the so-called Kitagawa diagram) via experimental tests [11] or theoretical models (see Ref 15 for a review). In both approaches, the result will be a relationship of the type

$$\Delta\sigma_{\text{lim}} = \frac{C_1}{d^{1/n}} \quad (6)$$

where d can be expressed in terms of Murakami's $\sqrt{\text{area}}$ parameter and $n > 2$ (this condition is necessary for describing the tendency of $\Delta\sigma_{\text{lim}} \rightarrow \Delta\sigma_{wo}$; see Fig. 7). According to Murakami and Endo [16], the parameter C_1 depends on the metal matrix hardness HV (or, in other words, the tensile properties of the material) and $n = 6$ (whereas for Robertson, $n = 2$).

Considering that the strain amplitudes that correspond to fatigue lives greater than 10^5 are in the region of a nominally elastic response for nitinol ($\Delta\epsilon_{\text{lim}} \propto \Delta\sigma_{\text{lim}}$), it can be concluded that in the region of the fatigue limit, a relationship of the type

$$\Delta\epsilon_{\text{lim}} = \frac{C_2}{\sqrt{\text{area}}^{1/n}} \quad (7)$$

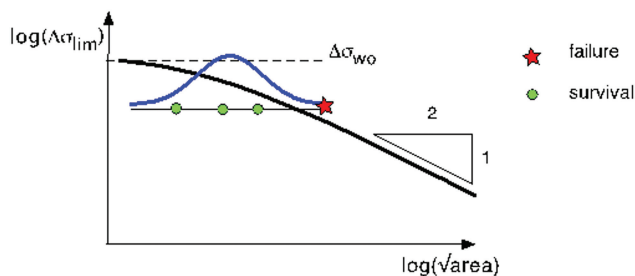


FIG. 7—Schematic of the Kitagawa diagram. For tests at a given stress (or strain) level, only the maximum inclusion present in the material will lead to failure.

should be valid. In the following, the existence of such a relationship is shown; in particular, because the parameter C_2 should be a function of the tensile properties, the present investigation was conducted on wires from different metallurgical processes (namely, P1, P2, P3, and P4) that had very similar tensile properties (UTS for the four wires is in the range of 1135 to 1165 MPa).

Estimation of Extreme Inclusions

The effect of a prospective population of inclusions/defects in a material subjected to a given stress level is shown in Fig. 7. In particular, failure of the material will occur only if the maximum inclusion is above the $\Delta\sigma_{\text{lim}}-\sqrt{\text{area}}$ relationship. Because of this fact, the fatigue limits are correlated not to the average inclusion content but to the size of the extreme, or maximum, inclusion. A number of successful applications support these concepts [12,16,17] which have been condensed into a simple recommendations for extreme value inclusion rating, ASTM E2283 [18].

The estimation of maximum defects is conducted with statistics of extremes; in the most simple version, it is based on block maxima sampling, in which only the maximum defects detected in a given area (or volume) are recorded and then analyzed with a type 1 extreme value (or Gumbel) distribution [14]. This sampling strategy can be applied very simply to the inclusion detected at the origin of fatigue failures, because the wires in the central part of the specimen (where they are bent with a constant radius of curvature) are subjected to a constant stress/strain, and we expect that failures will have occurred at the maximum inclusion present in the most stressed volume. In particular, we can assume that inclusions at the fracture origin are the largest particles in a cylinder with an annular section with diameters equal to the wire diameter and 0.9 times the wire diameter, and with a length equal to the central portion of the specimens under an RBT test rig.

According to extreme value statistics, the cumulative probability of the particle diameter for each wire batch should follow the Gumbel distribution function [13],

$$F(x) = \exp \left[-\exp \left(-\frac{x - \lambda}{\delta} \right) \right] \quad (8)$$

where λ and δ , respectively, are the location (63.2 percentile) and scale parameters.

Using Eq 8, data can be plotted on a Gumbel probability plot in which the defect inclusions are plotted against the reduced variate $-\ln(-\ln(F_i))$, where F_i are the empirical cumulative probabilities for the different data points (ASTM E2283). Such a plot is shown in Fig. 8 for extreme inclusions in the

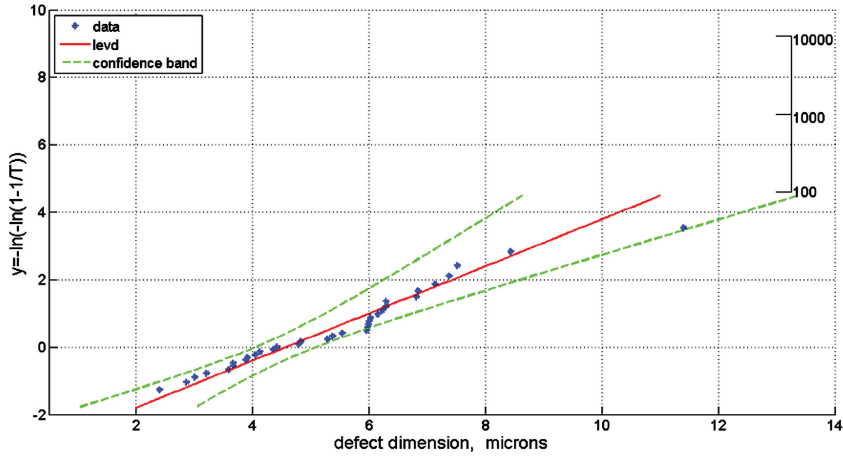


FIG. 8—*Gumbel probability plot for the extreme inclusions in batch P4.*

wires of the P4 process; the best fit distribution, whose parameters were calculated with the moments method [13], is plotted on the same graph, together with 95 % confidence bands. It can be seen clearly that the Gumbel distribution describes the data quite well. The parameters for the distribution of extreme inclusions for the four different wires are reported in Table 4.

The prospective size of the distribution with a return period T can then be calculated as

$$d_{\max} = \lambda + \delta \cdot \left(-\log \left(-\log \left(1 - \frac{1}{T} \right) \right) \right) \quad (9)$$

We have also collected the maximum inclusions at the fracture surface of RBT specimens; a $d_{\max}$ with a return period T corresponds to a defect size that, on average, exceeded every T specimen. We then calculated the prospective size of maximum defects for $T = 10\,000$ (obtaining the defects that are exceeded once every 10 000 specimens); in this way, we estimated extreme defects that should be correlated to the lower bound of the fatigue limit.

TABLE 4—*Fitted parameters of the Gumbel distribution for the four materials and expected particle size for a return period $T = 10^4$.*

Material	λ , mm	δ , mm	$d_{\max}$, mm
P1	2.73	0.68	8.79
P2	2.61	0.58	7.94
P3	1.83	0.48	6.25
P4	4.57	1.43	17.76

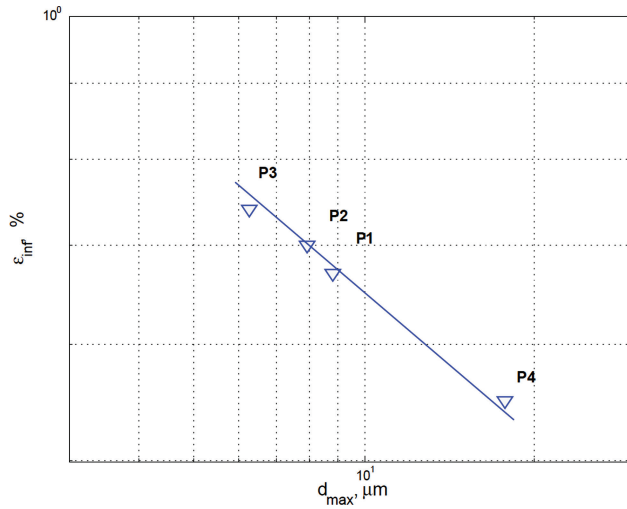


FIG. 9—Dependence of ε_{inf} on extreme inclusions for the nitinol wires.

Correlation Between Fatigue Threshold and Inclusion Dimension Distributions

The inclusion dimension distribution can be related to the fatigue performance of different materials by plotting the expected maximum diameter ($T = 10\,000$) against the fatigue limit, as in Fig. 9. According to this picture, extreme inclusion dimensions and fatigue limit are negatively correlated, which confirms Eq 6. Moreover, it can be added that samples obtained from materials characterized by different inclusion distributions behaved differently in fatigue testing. This result supports the concept of extreme value inclusion rating for the quality control of nitinol materials [19–24].

Conclusions and Future Work

In this work, we report the results and analysis of RBT on 0.3-mm-diameter superelastic NiTi wires obtained with four different processes. Cycles to failure of about 500 samples taken out of the four different materials were recorded for different deformation levels, and a descriptive model of ε – N behavior has been proposed and utilized to compare different process materials. Fracture sections were inspected via SEM, and inclusions in the crack nucleation region were measured, classified, and analyzed with the concept of “statistics of extremes.”

Our results indicate that the maximum inclusion dimension in the highly strained volume of a fatigue test contributes to a decrease in the fatigue

performance. Moreover, we have shown that the statistical distribution of the maximum inclusion dimension in lots of materials that can be considered homogeneous as far as fatigue mechanics and fatigue properties are concerned follows the Gumbel distribution. Lots characterized by Gumbel distributions predicting smaller inclusions provide better fatigue performance. Thus, this statistical distribution is a good candidate for specifying the relevant inclusion information of a material.

Additional research is in progress to extend the statistical data presented in this paper and further investigate fatigue behavior in nitinol. In particular, non-zero average strain fatigue behavior will be explored. Particle size distributions derived via fracture analysis will be compared with distributions obtained through metallographic section observations not only on wires, but also at intermediate process steps.

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Fatigue Crack Initiation in Superelastic Nitinol

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ABSTRACT: Phase shifted moiré interferometry is used to study fatigue initiation in superelastic Nitinol. Notched four-point bend coupon samples were fatigue tested according to a multi-level amplitude test protocol tailored for medical implant applications. Fringe patterns of local strain measurements are presented for three loading sequences based on the type of loading and range of anticipated strains in service. The effect of an initial series of pre-load cycles is examined and resulting life data is accompanied by local strain results highlighting the unique mechanisms associated with the initiation of fatigue cracks and the development of effective test methods and protocols.

KEYWORDS: nitinol, fatigue, crack, initiation, local, strain, gradient, phase transformation, multiaxial, constraint, plasticity, mean stress, dynamic, moiré interferometry

Introduction

Fatigue failure is a common problem for repetitively loaded engineering structures [1], resulting primarily from the effects of local plastic strain. Fatigue crack initiation is generally described by strain life equations that relate the alternating strain amplitude to the number of cycles to crack initiation. The

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deformation behavior of superelastic Nitinol is complicated by a number of factors related to the unique phase transformation of the material, leading to difficulties with quantifying the role of such factors as mean strain.

The fatigue life of metals is determined by assessing the relationship between local plastic strain amplitude and the number of cycles to crack initiation. These strain-life equations must account for the effects of mean strain, multiaxiality, and cumulative damage [1]. These factors complicate fatigue life prediction for many engineering materials, especially for superelastic Nitinol.

Tabanlı et al. [2] were the first to explore the role of mean strain and demonstrate how it complicates the design of fatigue experiments for superelastic Nitinol. Pelton [3] provided an excellent review of the microstructure and fatigue mechanisms in superelastic Nitinol. The author concludes that the cyclic transformation between austenite and martensite is more complicated than elastic crystallographic theories would predict and emphasizes that optimized, thermomechanically treated microstructures yield more stable (and predictable) behavior than annealed ones.

As described in Ref. [4], equivalent thermomechanical processing can be used to reproduce the desired material response from samples scaled to different physical dimensions. This approach has marked advantages: it equalizes both the material and process histories in addition to being a scaled representation of at least one mode of the structure's anticipated *in vivo* loading.

Most implants experience mixed-mode physiological loads *in vivo* and multiaxiality complicates establishing comprehensive fatigue models based on uniaxial material data. Runcimen et al. [5] recently combined strain-life data from different loading modes using a modified Coffin–Manson approach where the number of cycles to failure was related to an equivalent alternating transformation strain. Data from different loading modes and testing conditions were combined. The approach performed well for low mean strains but was not satisfactory for conditions of high mean strain.

Cumulative damage (mentioned above) refers to the effects of load sequence and inelastic deformation history, local to a potential site for fatigue crack initiation. It is typical for medical implants made from superelastic Nitinol to experience one or more high amplitude loading cycles prior to low amplitude fatigue loading. Components may also have loading histories that intermix high-amplitude with low-amplitude cycles throughout the service life. The sequence of events in a component's loading history matter because of the different mean strains that can result in a transfer sequence from compression to tension and vice versa [6].

The ongoing challenge is to design robust superelastic Nitinol implant components and develop effective test methods to guarantee they will resist fatigue. Obtaining the appropriate cyclic stress strain behavior is necessary to relate applied strain test data to crack initiation periods in fatigue life models;

however, this is complicated in superelastic Nitinol by local material phase transformation and plasticity.

A key element of our approach is to observe and characterize local notch root strain fields during fatigue. In this work, we further extend the experimental technique of phase shifted moiré interferometry [7,8] to get in situ, full-field, local strain fields under controlled and instrumented cyclic load conditions, prior to and during fatigue crack initiation.

We use a specimen geometry optimized for superelastic Nitinol material and component studies (material, process, and loading history). A single edge-notch at the center of the beam provides a strain concentrator, and non-zero (tensile-tensile) bending fatigue experiments are performed for samples, with and without initial high-amplitude pre-load cycles.

Materials and Methods

Specimen Geometry, Material and Loading Fixtures

A schematic of the specimen geometry is shown in Fig. 1. The same specimen geometry has been designed and optimized for both axial and four-point bend fatigue loading. This configuration has been shown to achieve uniform tensile loading and pure bending over a large range of deformations.

The primary modification from Ref. [7] was the addition of a 0.3 mm notch at the center of the beam to provide a strain concentrator. As can be seen in the lower left of Fig. 1, the redundant beam that would not be observed during the experiment has been removed.

The incoming material was seamless drawn superelastic SE-508 NiTi tubing from Nitinol Devices and Components, Inc., with an outer diameter of

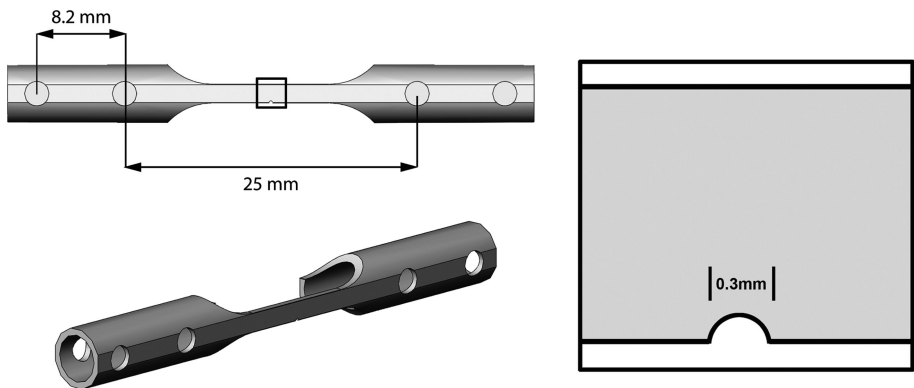


FIG. 1—Schematic diagram of the test sample.

5 mm and a 0.6 mm wall. Two pairs of 1 mm diameter holes, spaced 8.2 mm and producing a 25 mm span were laser cut in the ends of the samples and then carefully honed to insure precision pin loading of the test samples. The pins in the outer holes of the sample were free to slide laterally in slotted holes of the lower portion of the fixture. The pins for the inner holes were attached via linkages to the upper portion of the fixture.

The gage section of the samples were EDM'ed to produce perpendicular edges, maximizing the amount of material in the gage section and ensuring uniform behavior in bending. Slow feed, low power, and multiple passes were used to minimize adverse effects on the bulk material properties. A flat surface was carefully ground on one face of the test samples to provide an optically flat surface for the grating replication process. The height of the gage section after grinding and polishing was 1.75 mm from top to bottom. At the neutral axis, the samples were 0.4 mm thick and at the outer fibers the thickness was 0.5 mm.

The sample was further ground and mechanically polished in the shoulder regions to eliminate strain concentrators that could compete with the notch. The tubing samples were thermomechanically processed with a single heat treatment at 500°C resulting in an active Austenite finish temperature (A_f) of 5°C. A final chemical polish consistent with typical medical implant processing specifications resulted in a uniform, reproducible surface finish for the test articles.

Crossed diffraction gratings were epoxy replicated from 300 l/mm holographic diffraction grating plates using conventional techniques [7,9] with Micromasurements (Raleigh, NC) M-Bond AE-10 adhesive kit. The final grating thickness of all of the samples was minimized and estimated to be 5–20 μm .

A grating density of 300 l/mm was chosen to maximize measurement sensitivity for the anticipated range of deformations. This resulted in a fringe order sensitivity of 1.67 $\mu\text{m}/\text{fringe}$.

The custom four-point bend fixture used for the experiments is shown in Fig. 2. The fixture utilized smooth, hardened steel pins secured with o-rings (shown removed) for attachment of the sample. Slots and the independent inner elements of the fixture minimize over-constraint of the sample and encourages a symmetrical deformation.

Loading Sequences and Fatigue Test Parameters

Three loading sequences were used to explore the fatigue initiation behavior of the four point bend samples. The first of these sequences, designated 'Ia', was a simple sinusoidal cycle with no pre-load. Samples tested according to this sequence were loaded from zero and cycled between the specified maximum and minimum fatigue displacement levels for that particular sample.

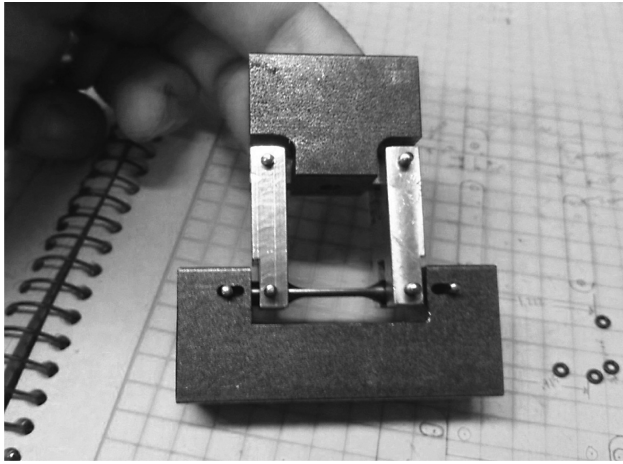


FIG. 2—Precision machined four point bend fatigue test fixture.

Samples tested according to the loading sequences designated “IIa” and “IIb” were also cycled between the maximum and minimum fatigue displacement levels, but received an initial ten pre-load cycles. The difference between these two cases is the transfer sequence leading into the high cycle fatigue portion of the loading history.

For the case of IIa, the component is unloaded from peak applied loading directly into high cycle fatigue cycles. For the case of a IIb loading sequence, the sample is fully unloaded after the initial ten pre-load cycles and high cycle fatigue cycles begin from an unloaded condition. Figure 3 illustrates the three loading sequences and defines the relevant loading levels.

To isolate the effects of the different loading sequences, it was important to utilize consistent testing parameters. Preliminary observations were used to select values for the peak pre-load level, the mean applied displacement, and the number of pre-load cycles to be performed were established.

The load/deflection behavior of several samples was observed in conjunction with live moiré fringe patterns. A peak pre-load displacement of 0.6 mm was chosen because it was high enough to clearly initiate phase transformation on the free surface of the specimen under observation. Ten cycles at this fixed displacement amplitude did not produce obvious damage at the notch were applied for samples fatigue tested with initial high-amplitude cycles.

A mean displacement level sufficiently high to induce a mixed phase condition was desired. A minor but consistently detectable change in the global load-deflection behavior for all of the samples was observed by 0.40 mm and a value of 0.45 mm was selected for the mean displacement level. This mean applied displacement level corresponded to a mean force of $18\text{N} \pm 1\text{N}$ for the sample during the initial loading cycle.

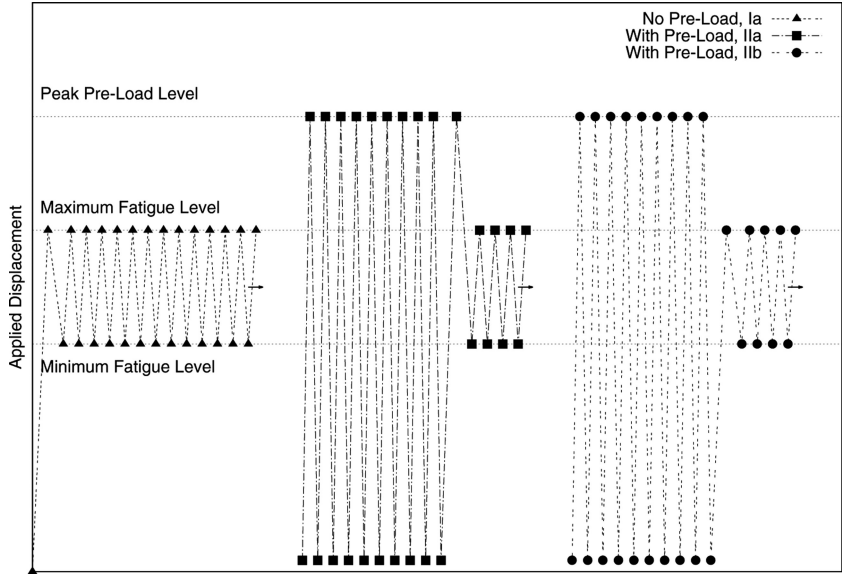


FIG. 3—Schematic diagram of the three loading sequences.

A first set of experiments was performed at the 0.45 mm fixed mean displacement to identify the 1 M cycle applied strain amplitude for a 50 % fracture rate and the associated standard deviation. Eighteen samples were fatigue tested without pre-load cycles with cyclic amplitudes selected using a D-optimal algorithm [10]. These data (not included herein) informed the selection of four discrete strain amplitudes for fatigue testing with high-amplitude pre-load cycles. Cyclic displacement levels of 0.8, 0.6, and 0.4 mm were chosen for the pre-load fatigue study. Table 1 summarizes the test conditions for each loading sequence.

Fatigue Test System and Data Acquisition

The samples were fatigue tested with a Bose 3230 ElectroForce test system (Eden Prairie, MN). Global measurements of the applied load were made using

TABLE 1—Summary of fatigue loading conditions.

Loading Sequence	Pre-Load Details	Mean Fatigue Loading Level	Samples Tested
Ia	No pre-loading	0.45 mm (18 N)	14
IIa	Ten cycles, 0.0–0.6 mm cycled from peak load	0.45 mm (18 N)	10
IIb	Ten cycles, 0.0–0.6 mm cycled from zero load	0.45 mm (18 N)	10

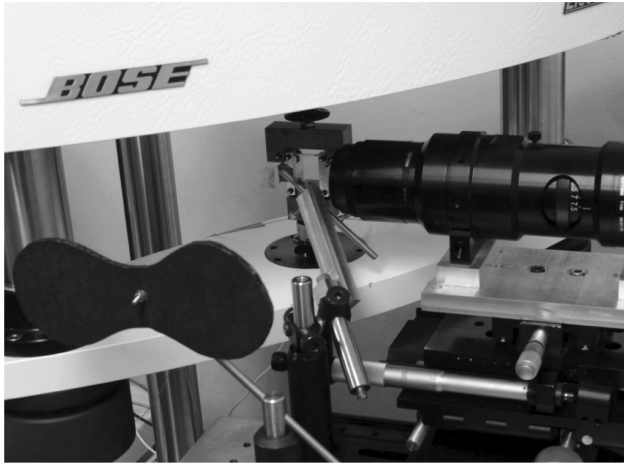


FIG. 4—Sample and fixture installed in the Bose 3230 dynamic test machine.

a 450 N range load cell and the crosshead transducer was used for displacement sensing and control. Figure 4 shows a photograph of the four point bending fixture mounted in the load frame. Calibration for all sensors was performed to ensure accurate measurements.

Displacement control was used for all loading conditions with amplitude compensation to maintain the specified limits during a test. Testing was conducted at a maximum of 20 Hz. Full field data was available at the maximum resolution of 1360 by 1024 up to 20 Hz cycling. Higher speeds can be achieved either by decreasing the resolution or taking data every N th loading peak instead of every peak.

A thermocouple on the back side of the sample was used to monitor temperature of the samples during the experiments. This data was recorded along with load and deflection for both timed data and peak valley acquisitions. All samples were tested under ambient conditions and confirmed to remain at 24°C , $\pm 1^{\circ}\text{C}$ throughout all of the testing.

The experiments employed two personal computers, one each for the Bose test system and phase shifting moiré interferometer system. The first computer was responsible for controlling the load frame, and the acquisition of all non-moiré data. Load, cross-head deflection, cycle count, and thermocouple measurements were digitized to 16 bits and routinely recorded at appropriate intervals during testing for all of the samples. A second computer manages the moiré interferometer, phase shifter, high speed camera, and the control interface described above.

Local Strain Measurement

Moiré interferometry provides in-plane displacement data in the form of sinusoidally modulated fringe patterns. The general technique is described in detail

in Ref. [11]. Phase shifting extends the basic moiré interferometry technique [12] by temporally oversampling the interference patterns and introducing a known phase shift into one of the beam paths. The sampled intensities and the known phase shift are used to create a significantly noise reduced “wrapped” fringe pattern with no loss of spatial resolution.

The interferometer used in these experiments was custom built with an integrated fiber optic splitter and phase shifter to provide exceptional stability and configurability [13]. The four beam interferometer head mounted on the optical table is shown in Fig. 5. The horizontal pair of beams was used to produce the U-field fringe patterns and the pair of vertical beams was used for the V-field.

Digital images were recorded using the Qioptiq T-100 Optem telecentric 10x zoom lens and an Allied Vision Technologies, Canada, Inc. (Burnaby-BC) GC1380H Prosilica GC high speed camera. The specialized equipment has a range of high performance spatial resolution and frame rate combinations from 1360 by 1024/30 fps to 170 by 128/120 fps, both at 12 bit gray scale resolution.

A custom interface was written to integrate the cameras capabilities into the phase shifted moiré interferometry [12] software system. The interface integrates control of the camera and fiber phase shifter with two digital control lines from the dynamic test machine. The analog actuator output signal is rectified and used to hardware-trigger the camera, allowing frames to be captured at the minimum and maximum loading conditions. A user-controllable digital output is used to allow the fatigue testing control software to trigger

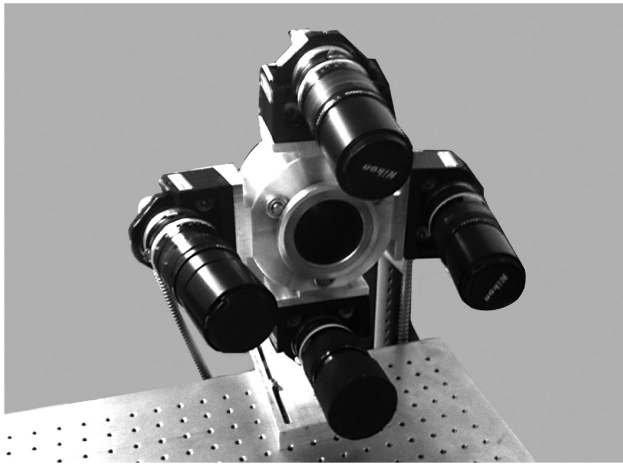


FIG. 5—*Four beam phase shifted moiré interferometer mounted on the optical table.*

taking phase shifted moiré interferometry data while the fatigue test machine is paused. Using the block configuration mode within the fatigue test machine software, the machine could fatigue the sample for a given time, pause the machine, start phase shifting and acquiring image data, and automatically restart cycling after the data was recorded. To permit fully automatic data collection, the software includes an algorithm to estimate the signal to noise ratio of a wrapped image and reject those that fall below a quality threshold.

Maximum/minimum data was obtained one field at a time. A beam block, shown in Fig. 4, was mechanically moved to isolate the individual fields. Five phase shifted, wrapped images were captured for each local displacement field measurement. Each image must go through an unwrapping process, which reconstructs a smooth representation of the underlying continuous displacement fields. After unwrapping, each field goes through a repair process where any pixels that failed to be unwrapped due to artifacts on the diffraction grating are filled in based on the values of the pixels around them. This prevents inaccurate data from propagating beyond the single pixel during the derivation stage. Before and after derivatives are taken a variable amount of smoothing is applied (but kept consistent within each data set). For any given load state, the process was repeated for both fields.

Results

Local Deformation and Strain Behavior

Figure 6 shows a series of images of a sample with a single edge notch loaded in tension. Wrapped fringe patterns of the U-field displacements on the left and surface images on the right are shown for increasing applied load up to the third row, which corresponds to the peak pre-load. The fourth row shows the sample fully unloaded after the first pre-load cycle.

The caustic forming ahead of the notch root by the third row indicates regions of the sample surface experiencing high local strain. Phase shifting of the fringe patterns recovers enough signal such that fully resolved and differentiable strain fields were obtained even without the use of carrier fringes [7].

The loading depicted in Fig. 6 is characteristic of the complete loading range for all of the samples tested. The applied displacement in the second row of Fig. 6 was 0.45 mm and corresponds to the mean fatigue displacement level. There is some evidence of non-uniform local deformation in the surface image of the second row, but it is readily recovered upon unloading.

A contour plot of the maximum principal strain at the peak pre-load level for this sample is shown in Fig. 7. Local strain as high as 3–3.5 % is visibly

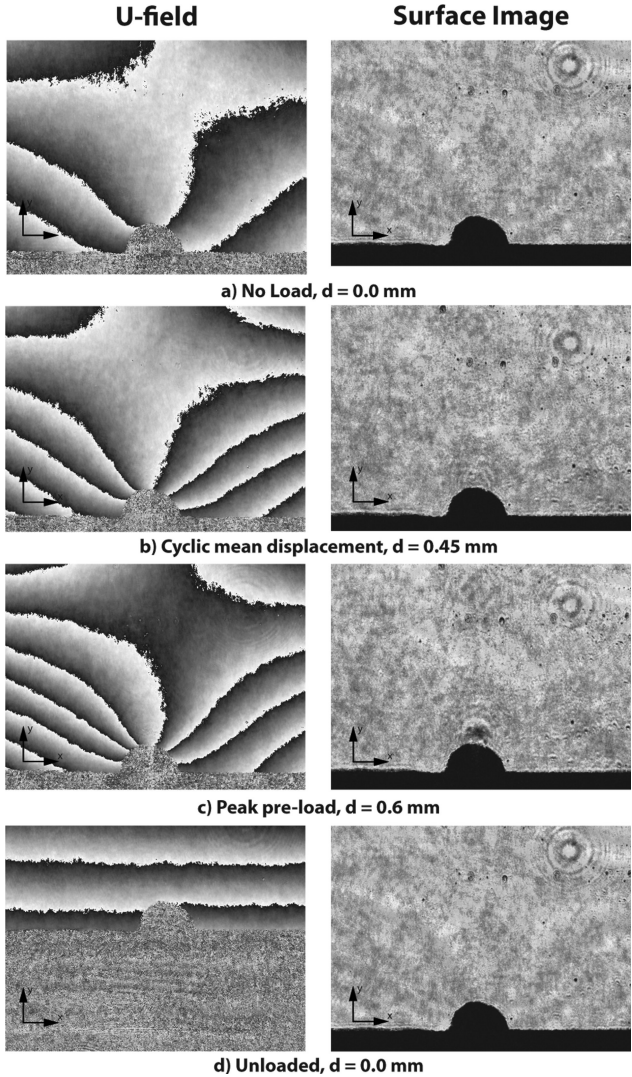


FIG. 6—Sequence of U-field fringe patterns (left) and surface images (right) for applied loading up to peak pre-load (third row) and subsequently fully unloaded (fourth row) for the first pre-load cycle.

concentrated on either side of the notch root centerline. The peak strain and general distribution compares favorably with results produced using finite element analysis (not included here).

In the final row, the sample has been fully unloaded. The horizontal fringes indicate uniform (zero) strain due to a uniform rigid body rotation. The shadow at the notch root can no longer be seen in the surface image, as the

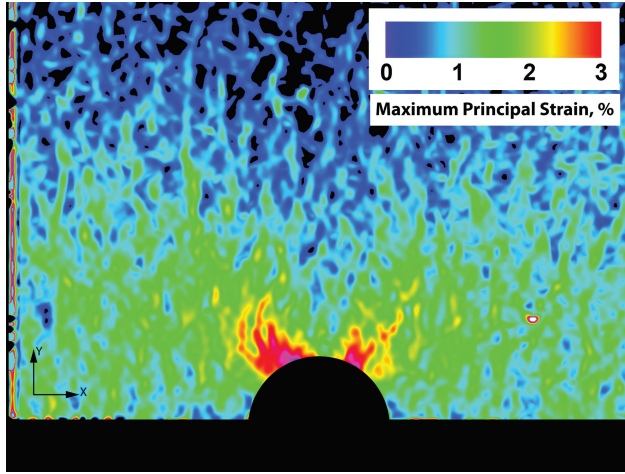


FIG. 7—Contour plot of the max principal strain acquired at the peak pre-load level.

local deformation has been fully recovered and there are no signs of local residual deformation or damage on the surface of the sample.

Cyclic Amplitude and Crack Initiation Periods

Fourteen samples were fatigue tested without pre-load cycles around the nominal mean fatigue level of 0.45 mm. The median cyclic amplitude for 1 M cycle survival was estimated to be 0.04 mm. Based on this value, a 1 % fracture rate

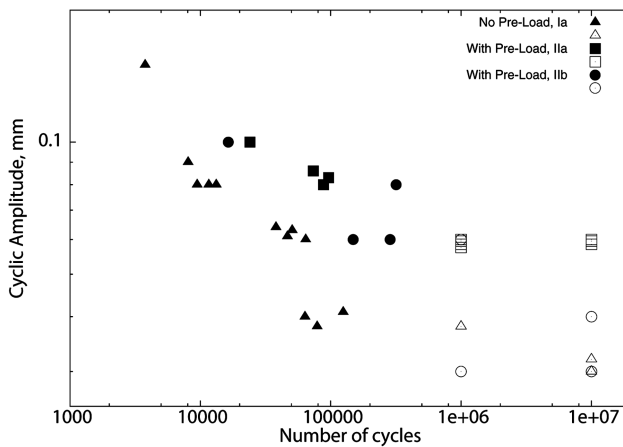


FIG. 8—Cyclic amplitude/fatigue crack initiation period data for the three loading sequences. Open symbols indicate sample run out.

would be expected for a cyclic amplitude of 0.036 mm, with 99 % of samples expected to initiate a fatigue crack at cyclic amplitudes greater than 0.044 mm.

Fatigue crack initiation periods for samples with pre-load cycles are compared in Fig. 8 to the baseline data established for samples without pre-load cycles. All the data points were tested at the nominal mean fatigue level of 0.45 mm at the cyclic amplitudes indicated.

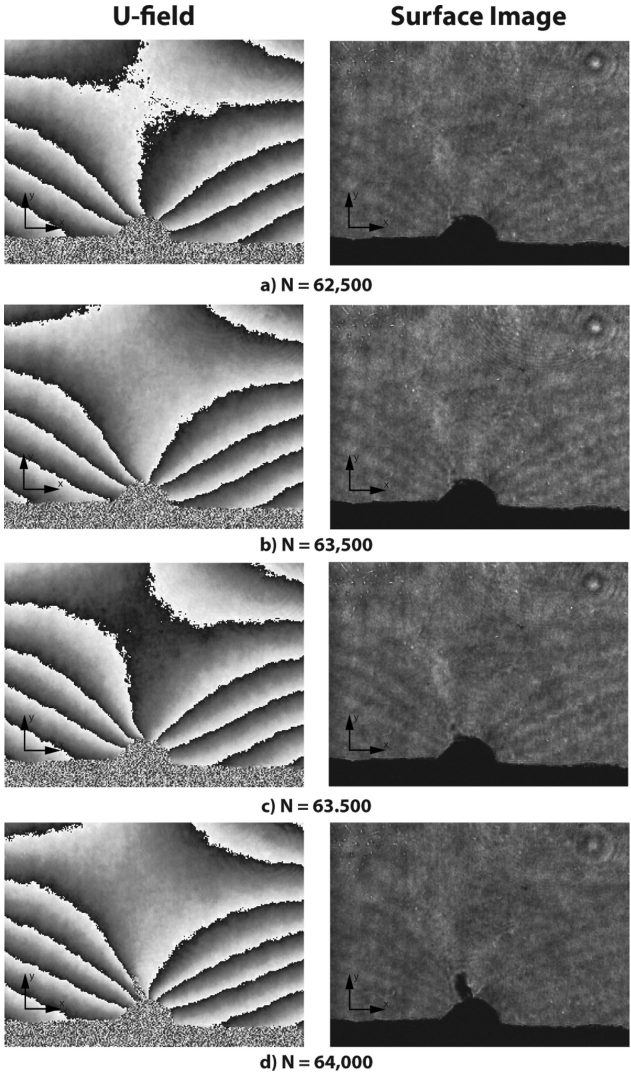


FIG. 9—Series of U-field fringe patterns (left) and surface images (right) acquired in situ at the max fatigue level during crack initiation for a Ia loading sequence.

Initiation periods for samples tested with high-amplitude, pre-load cycles are shown as filled squares and circles in Fig. 8. Initiation periods for samples with pre-load cycles were longer than those without. Samples fatigue cycled after unloading from the peak pre-load showed the strongest effect, while initiation periods for those unloaded to zero first were more variable.

Fatigue Crack Initiation Without Pre-Load Cycles

For samples without high amplitude loading cycles, the deformation ahead of the notch was minimal. In many cases, there were no visible signs of damage or localization until just before a fatigue crack would appear at the notch. Initiation occurred directly at the center of the notch, or slightly off to one side.

Figure 9 illustrates a fatigue crack initiating for a sample without pre-load cycles during a fatigue test conducted at 20 Hz. Phase shifted fringe patterns were acquired every 500 cycles at both the maximum and minimum fatigue levels of the U-field displacements to observe the initiation of a fatigue crack. This sample was tested at a cyclic amplitude of 0.04 mm. Top row images correspond to a cycle count of approximately 62 500. Preceding image data was nearly identical, with uniform deformation and no localization on the face of

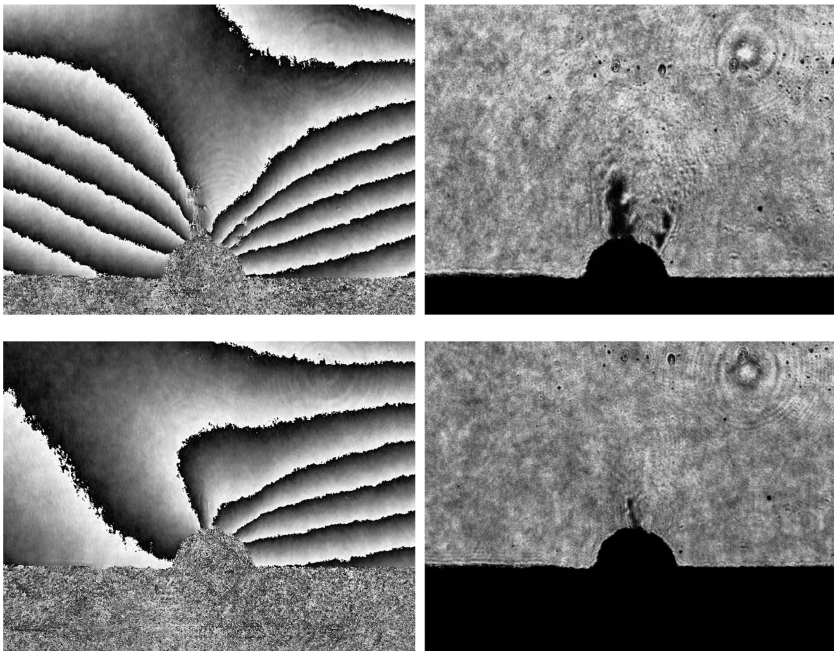


FIG. 10—*High resolution U-field fringe pattern (top) and surface image (bottom) for a typical fatigue crack.*

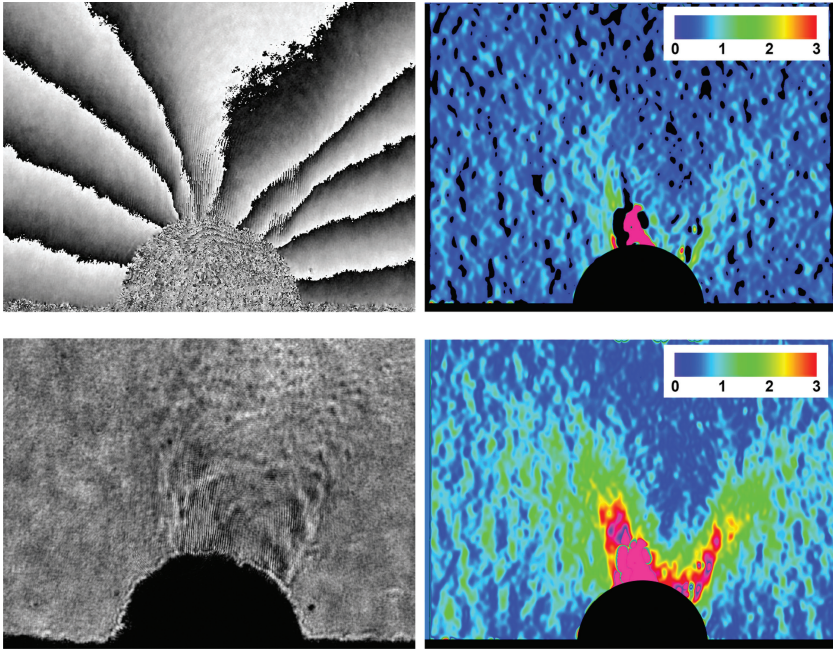


FIG. 11—*U-field fringe patterns and surface images at the max and minimum fatigue levels during initiation for a IIa loading sequence.*

the sample. Beginning in the second row, two caustics appear on either side of the centerline. By row three, the initiation site eventually favors the left side.

High resolution image data of a typical fatigue crack initiating from the notch is shown in Fig. 10. This image was taken at the maximum fatigue level where five and one half fringes separate either side of the fatigue crack, corresponding to $9.2\text{ }\mu\text{m}$ of displacement along the edge of the notch.

The local deformation becomes more complex ahead of the notch with the appearance of a secondary pattern of high frequency fringes and mixed material phases. Distinct boundaries between different material phases can be seen in the corresponding surface image. The two sharp interfaces bifurcate from the midline of the fatigue crack at angles of 53° and 58° from the loading axis.

Fatigue Crack Initiation With Pre-Load Cycles

Figure 11 shows the fatigue crack initiation of a sample at 102 000 cycles, tested with pre-load cycles at a cyclic amplitude of 0.08 mm. The top row was acquired at the maximum and the bottom row at the minimum fatigue level during fatigue cycling at 20 Hz. The surface images reveal the nature and

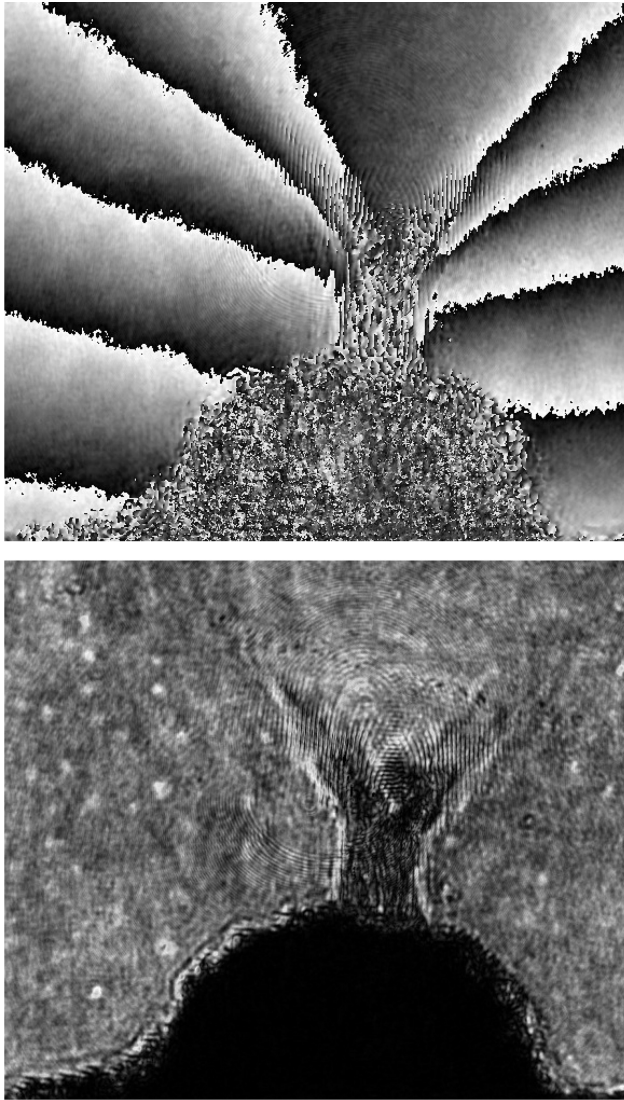


FIG. 12—High resolution U-field fringe pattern and surface image at the max fatigue level during initiation for a IIa loading sequence. Contour plots of alternating (top) and mean strain (bottom) for this fatigue cycle are shown on the right.

extent of local phase transformation around the notch throughout the loading cycle.

Additional data was taken at full optical magnification for this condition. Figure 12 (left) shows the zoomed wrapped fringe pattern and surface image for the maximum fatigue level for a typical IIa initiation. Contour plots (right)

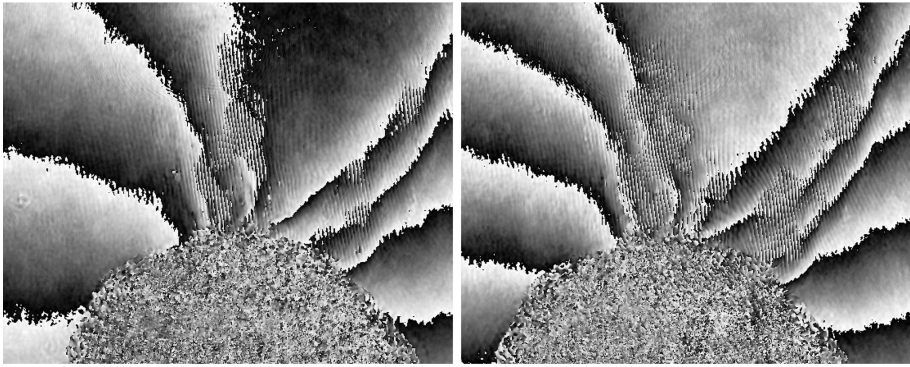


FIG. 13—High resolution *U*-field fringe patterns at the minimum (left) and max (right) fatigue levels during initiation for a IIb loading sequence.

show the maximum principal alternating (top) and mean (bottom) fatigue strains.

Figure 13 features high resolution *U*-field fringe patterns at minimum (left) and maximum (right) fatigue levels during initiation for a IIb loading sequence. Figure 14 is a high resolution surface image at the maximum fatigue level during initiation for a IIb loading sequence. The fatigue crack is initiating along the edge intersecting the notch indicated by the arrow.

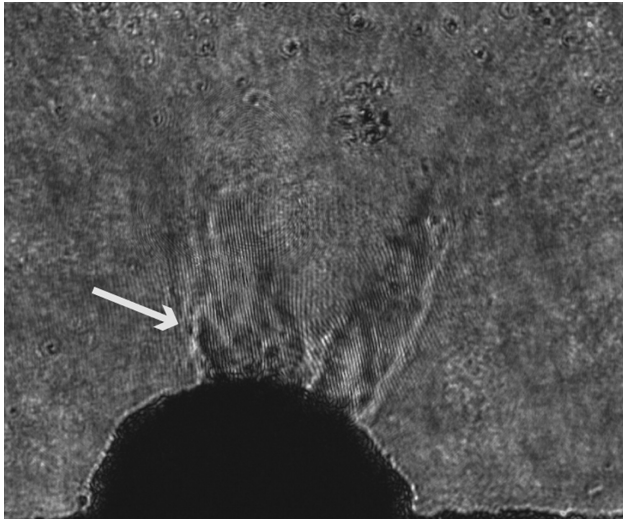


FIG. 14—High resolution surface image at the max fatigue level during initiation for a IIb loading sequence.

Conclusion

We have demonstrated an experimental technique for measuring local strain in superelastic Nitinol enabling us to quantify the effects of important factors relevant to the prediction of fatigue crack initiation periods for superelastic Nitinol components. The technique of phase shifted moiré interferometry was extended to acquire in situ fatigue data and shown to provide exceptional spatial and temporal resolution over a wide range of deformations.

We observed that the local strain fields at a notch in superelastic Nitinol are complex and evolve with applied loading. The effect of constraint establishes strong local stress gradients, leading to phase transformation and complex interactions between mixed phases of the material. Moving phase boundaries with singular stresses at the free surface of the specimen interact with the microstructure of the material in the notch root. This complicates the traditional methods for modifying the strain life equations to account for factors such as mean strain, multiaxial loading, and the consequences of different loading amplitudes and histories that are inevitably experienced in vivo.

For samples fatigue tested with high amplitude pre-load cycles, higher cyclic amplitudes were required to initiate fatigue cracks compared to samples that did not receive pre-loading. This effect was particularly noticeable for samples that were fatigue cycled immediately after the peak application of the high-amplitude cycles. Samples that received initial high-amplitude cycles but that were fully unloaded prior to high cycle fatigue had longer lives than the baseline; however, there was more variation in the fatigue crack initiation periods.

These results were selected as a case study to show the extensibility of our approach to the general opportunity to generate data and improve understanding of the factors affecting the fatigue crack initiation in superelastic Nitinol. By utilizing samples produced from tubing, we have demonstrated a technique that has great utility for medical devices and their applications.

The design of a single sample geometry capable of being tested in both simple uniaxial tension and pure four point bending enabled us to explore differences between alternative loading configurations with a high degree of consistency.

We anticipate the application of our approach to both general studies and to improve end-use material characterization, numerical modeling, design optimization, and validation.

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In Vivo Strain Estimates for Medical Implants

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ABSTRACT: Anticipating the in vivo loading conditions is a challenging aspect of validating the design and durability of cardiovascular implants. Complex implant geometries, multi-axial displacements, device/tissue interactions, and time-dependent factors can make it difficult or impossible to identify a single loading cycle that is representative of the full range of conditions. Computational modeling is often used to reduce a large number of complex conditions into representative alternating and mean strains at locations of maximum strain. A novel approach for estimating the alternating and mean strains during in vivo loading will be presented. The method determines local strains by deforming a finite-element model of an implant into a three-dimensional geometry reconstructed from two-dimensional images of a physically deformed implant. The method is then extended to in vitro fatigue experiments. Two examples are given of a structural heart implant.

KEYWORDS: computational models, experimental mechanics, inverse methods, medical devices, imaging, nitinol

Introduction

During the design verification and validation process, in vitro tests are used to determine whether a medical device performs according to specifications and

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to help predict *in vivo* performance. Many medical devices undergo cyclic loading throughout their lifecycle; thus, fatigue testing is an important component of verification testing. Regardless of the fatigue test paradigm being followed (test-to-success or test-to-fracture), it is necessary to characterize, parameterize, and bound the anticipated cyclic loading for use in durability specifications.

The fatigue performance of deformable structures is directly related to the deformation and motion amplitudes the structures experience. Therefore, determining the physiologic conditions that a cardiac implant encounters in the body is critical to device lifetime prediction and demonstration. Results from fatigue tests are typically used in statistical models, and therefore, the test conditions must be precise, well-defined, and controllable to minimize the variability in test results. Ideally, test conditions should be developed from well-known, precise, and representative measures of the *in vivo* conditions.

However, acquisition of representative *in vivo* data can be difficult for medical devices that are unique or placed in novel areas of the body. This is particularly true for cardiac implants placed in locations where the dynamic properties of the surrounding anatomy are not well known, cannot be measured directly, and can be altered by a device *in situ*. Static deformations can be directly measured in an animal model (e.g., after sacrifice), but it is challenging to map the motions associated with a device in a beating heart. In addition, animal models do not always accurately represent the conditions found in comparable human anatomy. The amount of deformation or motion can be substantially different in healthy animals versus sick patients, because healthy tissue can interact differently with an implanted device. Measurements are further confounded by the potential for the measurement method itself to interact with the device or tissue and reduce the fidelity of the measurement.

Preferably, a non-contacting, non-invasive measurement method should be used for measuring deformation and motion of a medical device *in situ*. Ideally, the data from this non-contacting method should be incorporated into the durability test development as early as possible. Advances in medical imaging and computational methods have made CT, MRI, and radiography powerful tools for investigating three-dimensional motion and deformation [1], but the data must often be further processed to create quantitative parameters for use in durability tests.

We have developed a technique that allows quantitative analysis of both *in vivo* and *in vitro* conditions with a single method. It is based on biplane cine-fluoroscopy and allows virtual reconstruction of an implanted medical device, thereby allowing precise quantitative measurements of *in vivo* deformations and motions. These measurements may then be used to reduce the number of parameters used for rigorous *in vitro* fatigue tests. Finally, the reconstruction method used for *in vivo* conditions may also be applied to *in vitro* devices. Both *in vitro* and *in vivo* computations can then be compared and contrasted to

verify the assumptions and simplifications that were required to reduce the complex, multivariate in vivo conditions to those suitable for a fatigue test. Fatigue results may be directly linked to in vivo conditions because equivalent computational methods can be used for both.

Methods and Materials

Cardiac Implant

The XE2 implant of the CARILLONTM Mitral Contour SystemTM (Cardiac DimensionsTM, Kirkland, WA) has been evaluated in the TITAN clinical study [2]. The XE2 has been developed to treat functional mitral regurgitation and is delivered percutaneously through a 9Fr catheter to the coronary sinus (CS) and great cardiac vein (GCV). The distal end of the implant is deployed and anchored within the GCV, proximal traction is applied to reshape the mitral annulus and then the proximal end of the implant is deployed and anchored into the CS.

The XE2 implant is constructed from superelastic NiTi wires. The flexible NiTi wires that form the endoluminal anchors deform during deployment to exert anchoring force against the CS/GCV lumen. The wires then move as the heart beats (see Fig. 1).

In Vivo Imaging

Cinefluoroscopy provides excellent spatiotemporal resolution throughout the cardiac cycle. The pixel size of cinefluoroscopic images is often smaller than 0.2 mm. In addition, the 15 to 30 frames/s frame rate of cinefluoroscopy provides an accurate radiographic snapshot without the motion artifacts that may cause blurring and reduced resolution in CT. Therefore, cinefluoroscopy is a valuable platform for measuring minute displacements of small structures, particularly in applications wherein the structures of interest are moving rapidly (e.g., a cardiac implant moving during the cardiac cycle). However,



FIG. 1—The CARILLON Mitral Contour System XE2 implant. The proximal end of the implant is shown on the right with the region of interest evaluated in this paper circled.

single-plane cinefluoroscopy is limited in application because of inaccuracies associated with view-dependent parallax and cosine errors that can be present.

Synchronous biplane cinefluoroscopy (SBC) is an imaging modality that can reduce the parallax and cosine errors inherent in single-plane fluoroscopic images. SBC imaging systems acquire single-plane cinefluoroscopic images from two distinct directional views with individual frames that are captured at nearly the same time. By analyzing the geometric information from both views, the geometry of the implant can be resolved in 3D and foreshortening errors eliminated. The high spatiotemporal resolution of SBC provides a precise method for resolving small displacements (e.g., under 2 mm) and structures (e.g., stent struts or small diameter wires).

Imaging data of the implanted device were collected from multiple fluoroscopes at various European clinical sites used in the clinical evaluation of the XE2 implant. SolidWorks (Dassault Systmes S.A., Vlizy, France), a 3D solid modeling (CAD) program, was used to perform the post-processing of SBC image data to reconstruct virtual devices using the method described in the section below.

Biplane Reconstruction

First, each analyzed SBC frame in a given DICOM image dataset was exported as separate left and right images that could be individually imported into CAD (see Fig. 2). CAD models were then created for each analyzed frame using the following procedure. Planes for each of the SBC images were created utilizing the image orientation information embedded in the DICOM dataset (see Fig. 3). Typically, the two images were captured from approximately

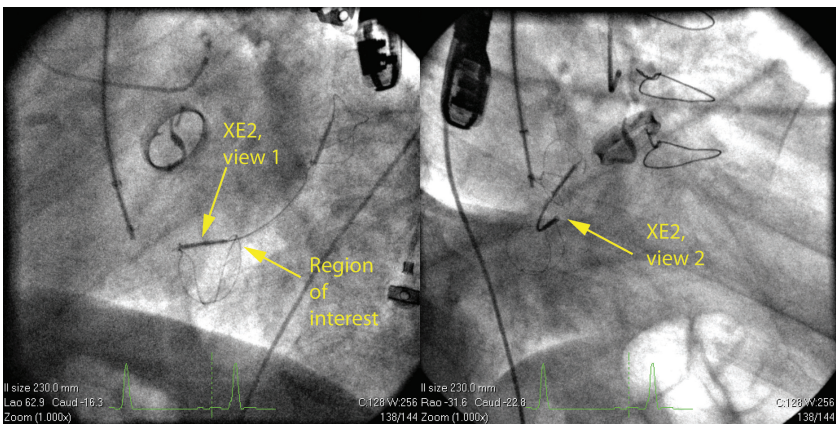


FIG. 2—A representative SBC frame showing the synchronized left and right images. Note the image orientation embedded in the lower left of each image.

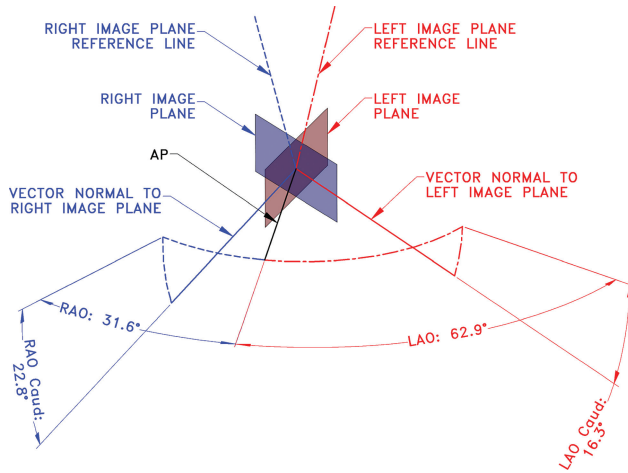


FIG. 3—Setup of SBC image planes in CAD. The angles used to set up the planes are taken from the left and right images and are relative to the AP direction. Reference lines placed in the left and right image planes are used to orient the images.

60°LAO/–15°Caudal and –30°RAO/–15°Caudal, but the image projections varied and images from any two projections may be utilized.

Reference lines were placed on both the left and right image planes, again using the orientation data from the DICOM dataset, to allow the images to be rotated to compensate for rotation of the fluoroscopic imager (as shown in Fig. 3).

The two images were then placed on the appropriate plane and aligned with the appropriate reference line. To ensure that the two images were scaled and vertically aligned with one another, a series of horizontal lines were constructed using reference features on the implant in one of the images, whereas the other image was scaled and moved vertically until all of the reference features were aligned. Vertical alignment is critical to ensure that features intersect when projected into 3D space. However, horizontal alignment is unimportant because of the way the planes are aligned in CAD; horizontal displacement of an image only changes the position of the resulting geometry in 3D space and does not affect the shape. Typically, between four and five reference features were utilized for each frame to ensure that only one image scaling and alignment solution existed.

Once the images were properly scaled and aligned to each other, two non-deformable components on either end of the implant were reconstructed and their lengths measured in pixels. These measured lengths were later used to scale deformation measurements taken in the resulting 3D CAD model from pixels to inches. The non-deformable components were reconstructed by first

sketching their axes on the left and right images. These 2D axes were then projected normal to their respective sketch planes creating surfaces. Lines were created at the intersections of these surfaces to form the component axes in 3D space (see Fig. 4). The lengths in pixels of the two lines were then measured in the reconstructed CAD model.

Once the rigid component lengths were measured, other non-deformable features were reconstructed through ratiometric scaling in CAD and compared to the original cine image. For example, the unscaled diameter of the rigid components in pixels could be determined by multiplying the measured length in pixels by the known ratio of diameter to length in inches. This enabled a qualitative confirmation for how well the reconstructed model fit the imaging dataset.

Deformable implant features (e.g., the anchor wires) were also reconstructed using this methodology. After all relevant features had been reconstructed, coordinate system origins were placed on and aligned to the non-deformable components. Measurements in pixels were taken from these origins to fiducial features placed on the deformable implant components.

Whereas the above procedure is described sequentially, it was an iterative process. A complete cardiac cycle typically consisted of 12 to 15 cine frames. Initially, only three to five frames were analyzed with the left to right image scaling and frame scale factors derived from the two rigid components evaluated for each frame. The image scaling was then adjusted slightly until a single image scaling factor was identified that provided a good fit across all

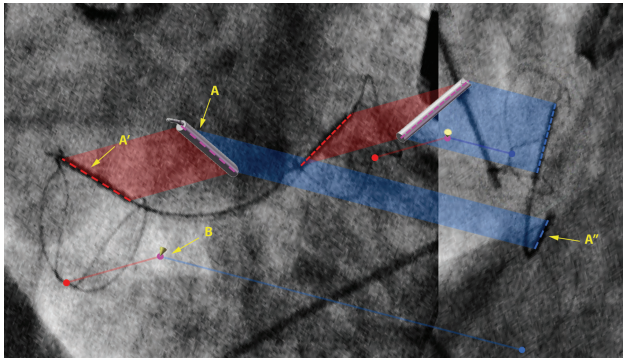


FIG. 4—A typical reconstruction showing the two non-deformable components (silver cylinders) e.g., (A) used to scale the resulting CAD model. The feature axes are shown on the left (red lines), e.g., (A') and right (blue lines), e.g., (A'') images; the resulting 3D axes are shown in purple. Also shown are the two fiducial points at the apex of the implant that were used for deformation measurements (B).

frames. Once the image scale factor was identified, additional frames were analyzed.

Following the creation and analysis of all of the CAD models for a patient dataset, deformation measurement scaling from pixels to inches was completed using Excel (Microsoft, Redmond, WA). A device scale was determined by dividing the non-deformable component lengths by their known lengths. Each of the unscaled measurements in pixels was then converted into a scaled inch measurement by multiplying the unscaled value by the device scale.

Checks and verifications were performed to ensure the reconstructed model accurately represented the in vivo condition. Multiple reference features were used to align the left and right images. These same features were used successfully across multiple frames in each dataset. A single image scaling factor was identified for each DICOM dataset. In addition, the frame scale factors derived from the length of the two rigid components were independently determined across frames and matched closely (to within 3 %). The reconstructed model compared favorably to images of deformed implants. Finally, the overall length of the implant was measured in the reconstructed model and, in all cases, the measured value was within the design specification for this dimension. Taken together, all of these factors indicate that the resulting reconstructions are accurate representations of the deformations imposed by the in vivo environment.

Finite-Element Models

A global finite-element model was created for the proximal anchor of the XE2 implant in a relaxed state using a coarse mesh of linear elements and is shown in Fig. 5. The mesh was constructed by sweeping a meshed cross section of the wire comprised of 12 four-noded elements along the full length of the wire-form. The global model required approximately 12 000 eight-noded brick (C3D8) elements with approximately 1000 nodes along the centerline and a spacing of about 0.15 mm.

A coarse mesh was used for the global model to avoid overconstraint and convergence issues and also to increase computational efficiency and solution speed. Mesh density and other model studies were performed to optimize the choice of node spacing along the centerline of the global model and the mesh density through the cross section of the wire. Boundary conditions from in vivo medical imaging data were applied at all of the centerline nodes of the global model.

Continuum, three-dimensional elements were chosen for the global model over beam elements because these simpler element formulations lack adequate contact capabilities. The XE2 proximal anchor has several regions such as the apical twist that involve self-contact and/or contact with other portions of the implant. It was found that these interactions played a significant role in the response of the XE2 and needed to be included in the model.

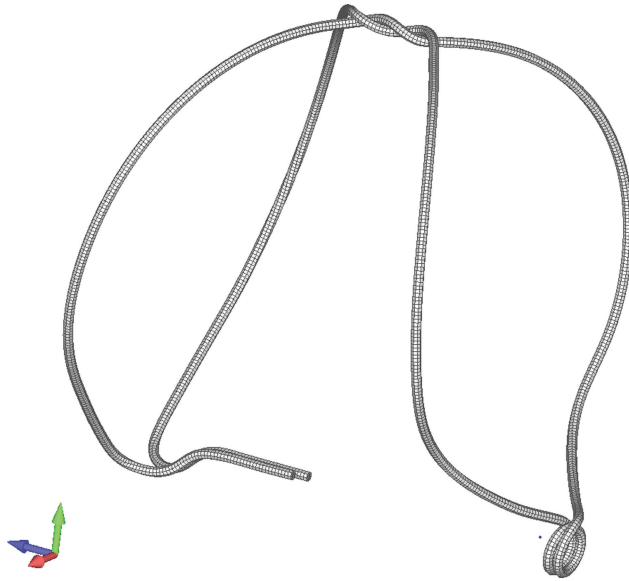


FIG. 5—*Finite-element global model used to link the reconstructed displacements and the deformation of the XE2.*

A sub-model approach was adopted to study fatigue strain history in specific regions of interest. Figure 6 is a sub-model of the region of interest of the proximal anchor of the XE2. The sub-model utilized the same model properties as the global model and provided much finer resolution for subsequent stress/strain calculations.

The sub-model was coupled to the global model at cross section/planes shown in Fig. 6. Constraints were also used for nodes within and in contact with other non-deformable components, represented in Fig. 6 as three planes.

Linear, eight-noded bricks were again used, but for the sub-model, there were 60 elements in the cross section and over 100 000 elements for the region

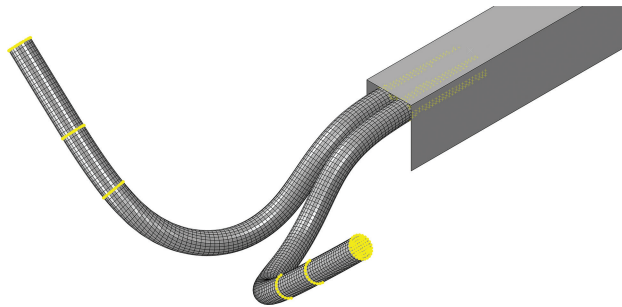


FIG. 6—*Finite-element sub-model of the region of interest on the proximal anchor of the XE2.*

of interest. Fully integrated, three-dimensional continuum solid elements were used and a local material coordinate system was used for fatigue post-processing.

Rigid bodies were included in both of the models to interact with the deforming wireform and represent non-deforming physical geometry. Surface-to-surface solid contact was enforced throughout both the global and sub-model analyses. Material properties for the wireform and other essential aspects of the model, such as boundary conditions and contact parameters including a friction coefficient of 0.1, were chosen based on validation experiments not discussed herein.

All analyses were performed using ABAQUS/Standard 6.10 (SIMULIA Providence, RI).

Motion and Strain Analysis

Analysis of measurements that were taken throughout the cardiac cycle allowed quantification of the in vivo mean and alternating deformations of the device. Mean deformations were calculated by taking the difference between the in vivo reconstruction values and measurements made from the same origins on undeformed devices. Motion amplitudes were determined by taking the difference between the minimum and maximum deformation values. Two frames (and the correlated 3D CAD models) that corresponded to the minimum and maximum deformation conditions were chosen as inputs to the computational model. These two frames had the largest displacement of a fiducial feature at the apex of the implant, which was found in previous analyses to have the largest motion in frame-by-frame inspections across a larger number of patients.

The parametric spacing of the tabulated coordinates for the in vivo reconstruction centerlines were adjusted by fitting tensioned splines [3] to the raw data and interpolating at uniform increments to correspond to the centerline node locations of the global model shown in Fig. 5. In general, reconstructed data was not completely available along the entire centerline of the wireform. In such cases, each segment for both frames was carefully registered and aligned to the undeformed global model in an iterative process to ensure an accurate translation.

Special care was taken to ensure that the individual segments of the resampled curves produced results that matched the original data, especially in the region of interest. Any gaps or discontinuous segments were identified and either eliminated or replaced at this step in the process. The location and curvature of the interpolated data points were carefully checked to preserve the original continuous wireform shape, particularly at free ends and where segments are joined. Consistent with previous efforts, the nominal wireform length was

maintained by using it as a fitting parameter for application to the deformed model.

Results

In Vivo Reconstruction

Implant loading conditions were created for two patients, A and B. Each set was reconstructed in CAD according to the methods described above and tabulated centerline data were used to drive the global model for the proximal anchor of the XE2. To qualitatively assess the fidelity of the computational results, the deformed finite-element models were compared to the original SBC images as shown in Fig. 7 for patient A. The resulting computational models produced high-quality reproduction of the deformation in the region of interest.

Comparison of Different Patient Loading Conditions

The virtual reconstructions and centerline data for minimum and maximum deformation (based on the relative displacement of the fiducial described above) were developed for patients A and B. The maximum and minimum deformed global model centerline nodes for each patient are shown in Fig. 8.

The nodal centerlines for the maximum and minimum fatigue cycle positions are clearly distinguished in Fig. 8. Gaps in the centerline data exist and

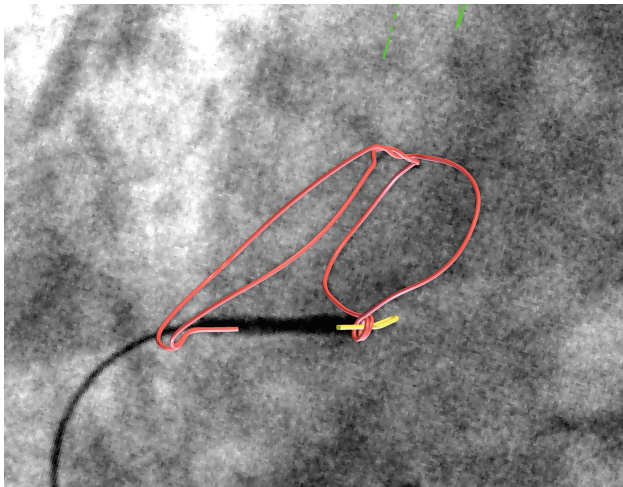


FIG. 7—Comparison of the deformed FEA model to the original cine frame for patient A. The deformable NiTi proximal anchor wireform is shown in red and a non-deformable ratiometrically scaled feature is shown in yellow.

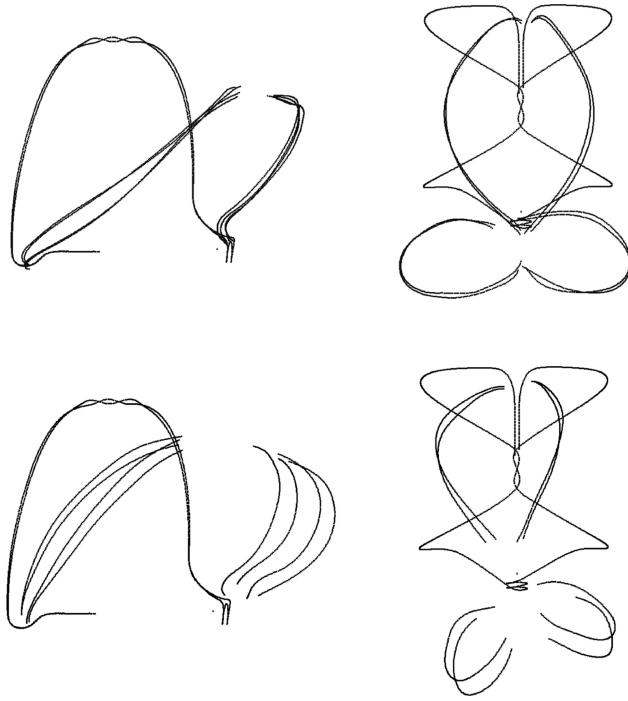


FIG. 8—Comparison of *in vivo* deformations for patients A (top) and B (bottom).

were discussed above. The deformation of the proximal anchor for patient A is seen to be more symmetrical than that for patient B.

In Vivo Fatigue Strain Results

Sub-models of the region of interest were run for patients A and B, and a fatigue analysis was performed to estimate the applied mean and alternating strain. Contour plots of alternating fatigue strain for the two patients are shown in Figs. 9 and 10 is a plot of the alternating versus the mean strain.

Even as the gross deformation for patients A and B are seen to be different, the alternating and mean fatigue strains in the region of interest have similar magnitudes and occur in generally the same locations.

In Vitro Reconstruction

Reconstructions of an *in vitro* condition were also created using the methodology described above, but using photographs of the implant taken from two angles while on test. A bench model similar to that used for fatigue testing the XE2 is shown in Fig. 11 (lower left). This bench model was designed to hold the proximal anchor and pull on the fiducial feature at the apex to replicate the

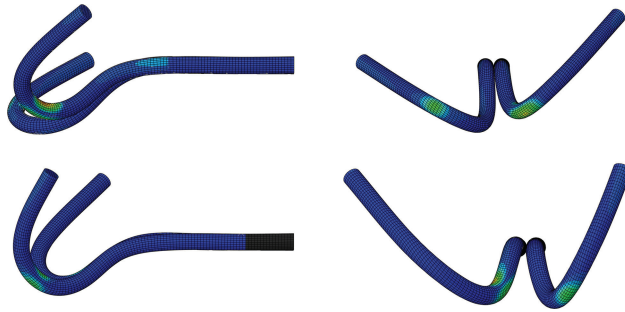


FIG. 9—Contour plots of alternating fatigue strain for patients A (top) and B (bottom).

deformations observed in the clinical images, shown in the upper left of Fig. 11. The location and amplitudes of the strains compared favorably at the region of interest for both in vitro and in vivo models suggesting that the bench model provides a reasonable representation of in vivo strains.

Discussion

Virtual reconstructions derived from precise 4D image data enable quantitative validation of the reconstruction method, computational model, and strain calculations by comparing models based on in vivo and in vitro data. Because the data is quantitative, the associated uncertainty of the measurements can be measured with statistical methods to demonstrate statistically valid boundary conditions. Whereas engineering judgment may still be required because small, practicable sample sizes will have large confidence intervals, the accuracy of

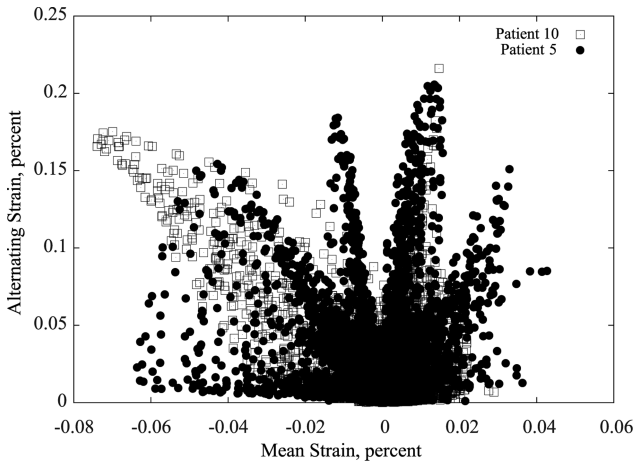


FIG. 10—Alternating versus mean strain for patients A and B.

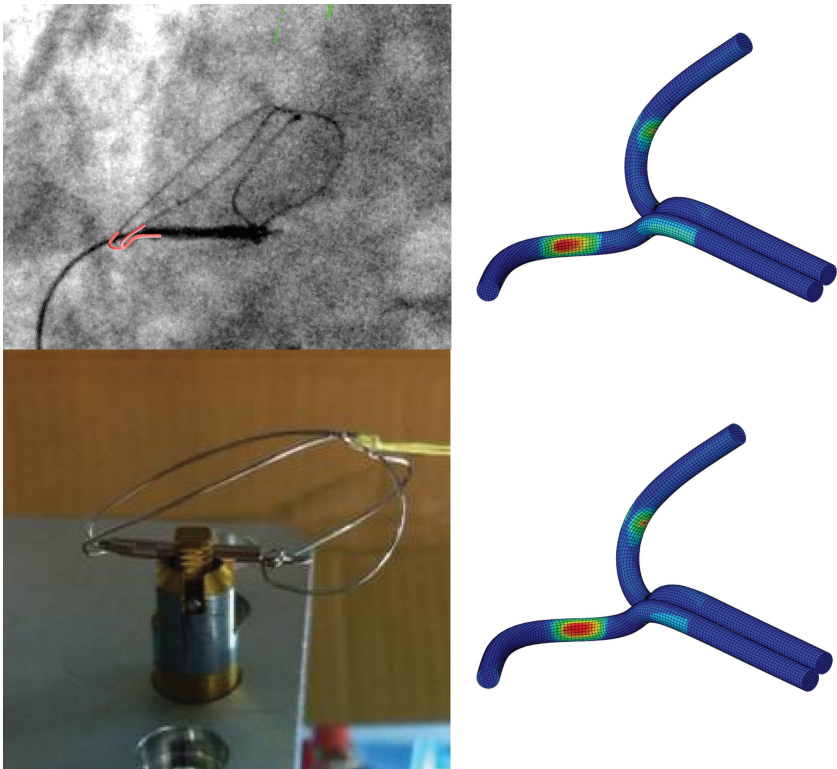


FIG. 11—*Deformation and alternating fatigue strain results for simulations of in vivo (top) and in vitro (bottom) loading of the XE2.*

the underlying cyclic loads can be greatly improved. Additionally, quantitative data can be applied directly to empirical tests or used in computational models. For imaging modalities that allow observation in 3D, fiducial features may be identified and the motion of those features evaluated. This evaluation can be used to reduce the number of parameters to those that are considered important and to create a physiologic envelope that bounds multiple axes of motion.

When FEA is based on clinical imaging data, computational models become more independent tools for use in both validation and prediction. The fatigue model does not need to rely on boundary condition inputs that may not be relevant to the design or conditions. Portions of a device may be analyzed using inputs derived from known clinical conditions, and the calculated strains can be calibrated with test-to-fracture results from fatigue conditions near those observed in vivo. Gaps may exist in the clinical imaging data where the high strain locations are hidden or too small to acquire precise local deformation estimates. These gaps are not critical because computational models that utilize a fully reconstructed 3D model as input may rely on extrapolation from remote locations where the conditions are well known. Sensitivity analyses may be

used to validate and revalidate the assumptions made when the number of parameters is reduced to apply the conditions on test, and the limits may be derived from a 3D envelope of conditions for a small sub-model or portion of the device.

Furthermore, the calculated strains can be validated by deforming a device *in vitro*, measuring the strains on the device with an alternate method (e.g., strain gages, digital image correlation, etc.), photographing the silhouette of the deformed device from two views, reconstructing the deformation and motion, and then comparing the strains calculated in the computational model to those measured in the physical device using the alternate method. The deformations for the *in vitro* test are selected to be qualitatively similar to that observed *in vivo*, so the measured strains are anticipated to have a strong correlation to the computed values. Because the same methodology can be used for *in vitro* and *in vivo* strain calculations, the model derives consistent strain measures for both environments that can be correlated.

The methodology was demonstrated above by using geometry derived from SBC in computational models of a cardiac implant to calculate *in vivo* mean and alternating strains. The technique can potentially add freedom to the design and evaluation of implantable structures. For example, worst case conditions can be estimated from statistical models of reconstructed image data, applied in fatigue tests, and then validated through the computational model in a more traditional fashion. Statistical models of both the fatigue strength and physiologic conditions can be decoupled, refined independently, and then recombined to estimate the probability of fatigue fracture, resulting in a quantitative measure of risk.

By decoupling the analysis of the physiologic loading conditions from the fatigue strength (the two are coupled in a test-to-success), the device design may be incrementally improved by decreasing the alternating strains where fractures occur in the test-to-fracture and then comparing the result to models of the physiologic conditions. These models of the physiologic conditions may have increasing confidence if they continue to be refined in parallel with the device design. The design modifications should be limited to relatively small spatial regions of high strain because the transfer function between motion and alternating strain will be correlated to the interaction between the global device geometry and the anatomy.

Conclusion

A single methodology can be used to effectively compute strains for a device *in vitro* and *in vivo*. Quantitative 3D reconstructed models can be built from two viewing angles. The 3D reconstructions provide spatial data that can be applied directly in a fatigue test, without requiring a computational model to

derive the conditions. The definition of the in vivo physiologic envelope allows early determination of an empirical fatigue strength design margin. This method can confirm that the simplifications applied early in the durability test development are appropriate, enabling early test-to-fracture along the most common or largest axes of motion for the device.

The use of high-resolution 4D imaging emphasizes the need for clinical data as early as possible for use in the design and development of high reliability medical devices. As more clinical data is collected, a statistical model of the physiologic parameters can be refined independently of the device and the risk-profile updated accordingly.

High-resolution finite-element models, combined with in vivo imaging and their associated measurements, aid in understanding the boundary conditions of relevant fatigue parameters. The computational models based on reconstructions (in vivo and in vitro) allow direct comparison of in vivo and in vitro mean and alternating strains, potentially shortening design cycles and improving the value of in vitro tests for medical devices.

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Literature Discrepancies in Biomechanical Loading of Orthopedic Trauma Devices Intended for Lower Extremities

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ABSTRACT: No standards presently exist for loading magnitudes employed in biomechanical studies of fracture fixation. Thus, the purpose of this investigation was to analyze the loading magnitudes utilized in published technical papers on biomechanical fracture fixation investigations. The hypothesis was that loading magnitudes used in biomechanical testing are not standardized, are inconsistent, and are poorly documented. English-language medical literature was searched for references to lower extremity biomechanical testing related to fracture fixation that utilized body weight as the loading scenario. Included articles were categorized based upon weight bearing (WB) protocol, implant type, and anatomic region and were compared to reported body weights published by the U.S. Department of Health and Human Services Centers for Disease Control (CDC). There were 5289 publications identified as potentially applicable; 54 articles met all included conditions. The majority of articles attempted to mimic full WB conditions. Others utilized half body weight, partial WB protocols, crutch-assisted WB protocols, and toe-touch only. A range of biomechanical testing forces from 31 to 980 N was reported, depending upon the desired percentage of WB that corresponded to a mass range of 3 to 100 kg. The CDC reported mean body masses for U.S. females and males, across all races and ethnicities, of 75 kg (736 N) and 88 kg

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(863 N), respectively, for an averaged total of 82 kg (804 N). In comparison to the report from the CDC, the literature underestimated mean body weight. Consistent loading parameters among biomechanical tests are not well documented. Biomechanical testing results should support clinical experience, and in this study a discrepancy was observed between biomechanical test parameters that might ultimately influence the orthopedic standard of care. Based on the literature search and reported CDC findings, a force of 804 N or greater might be most appropriate for simulations of full WB protocol.

KEYWORDS: body weight, orthopedics, trauma, biomechanics, lower extremity

Introduction

Bridging the gap between biomechanical research and clinical orthopedic care must be approached with caution. Loading scenarios used in biomechanical testing are often limited to constrained monotonic axial compressive forces. Clinical loading is known to be more complicated because of the three-dimensional forces and moments generated about a particular joint [1]. Recently, the use of multiplanar repetitive forces (simultaneous cyclical axial compression bending and torsional moments) has been shown to better simulate clinical failure of fixation constructs than the more often utilized constrained monotonic axial compression forces [2].

From a device commercialization perspective, consensus standards can be used reliably to evaluate new devices. If an evaluation compares a new device to a predicate device in a standardized test, it might not be necessary to use a test force that is based on a body weight estimate. For those tests not covered by consensus standards, a review of the biomechanical literature can yield test methods that have been adapted to the specific device being evaluated. These nonstandard test methods usually define four key points: device construct type, failure criteria, loading time (cycles), and force.

Outcome parameters of biomechanical tests such as stiffness and fatigue survival are dependent upon the chosen force and are, at best, estimates of clinical behavior. Furthermore, authors have mentioned anywhere from 562 N [3] to 980 N [4] as the force used for full body weight protocols. Some orthopedic reconstruction testing consensus standards do provide testing forces for the evaluation of a medical device. ASTM F2068-09 [5] and ISO 7206-4 [6] provide testing forces for hip implants, and ASTM F2083-11 [7] and ASTM F1223-08 [8] provide testing forces for knee implants. Interestingly, none of these values are said to be based on body weight.

The consensus standards for orthopedic trauma devices can be divided into three categories: plates and screws (ASTM F382-99 [9] and ASTM F384-12 [10]), intramedullary nails (ASTM F1264-03 [11]), and external fixation (ASTM F1541-02 [12]). None of these standards provide recommendations for loading with regard to body weight. It should be noted that osteosynthesis

standards provide test methods intended to compare one device to another in a consistent manner. Specific details of an individual test are determined based on multiple factors that include material choice, implant design, and intended use.

Orthopedic trauma devices can be broken into two most basic categories: internal fixation and external fixation. Internal fixation devices are used to achieve definitive fixation in fractured bones. Plates and screws and intramedullary nails are considered internal fixation devices that are intended to treat a wide range of fractures in bones from long bones to extremities such as phalanges. External fixation devices are often used as a temporary means of achieving fracture fixation in cases where severe tissue damage does not allow for immediate surgical treatment. Circular fixation and unilateral fixation devices often incorporate half pins or wires to achieve the desired bone stability. External fixation can also be used for deformity correction and limb-lengthening cases.

The purpose of this investigation was to analyze the loading magnitudes utilized in published reports of biomechanical fracture fixation studies, specifically those related to orthopedic traumatology, and to provide a systematic categorization methodology such that future studies would have a basis for choosing an appropriate loading scheme for comparative analyses.

Hypothesis

Our hypothesis was that loading magnitudes are not well documented, consistent, or standardized for biomechanical studies of fracture fixation constructs.

Methods

The following peer-reviewed medical journals were searched for articles published between January 2000 and July 2009 that included a reference to biomechanical testing and utilized “body weight” (keyword) as an orthopedic loading scenario: *Clinical Biomechanics*, *Clinical Orthopaedics and Related Research*, *Injury*, *Journal of Biomechanics*, *Journal of Bone and Joint Surgery—American*, *Journal of Bone and Joint Surgery—British*, *Journal of Orthopaedic Research*, *Journal of Orthopaedic Trauma*, *Journal of Trauma*, and *Medical Engineering & Physics*. The number of hits per journal was recorded, and the percentage of included articles was calculated. Only lower extremity, weight-bearing anatomical regions were included. Any publications that did not specifically pertain to orthopedic traumatology were excluded. Table 1 shows a list of the included criteria.

Included articles were categorized based upon weight bearing protocol, implant type (intramedullary nails, plates and screws, or external fixation), and anatomical region. Results from the literature search were compared to

TABLE 1—Inclusion criteria used in the selection of journal articles to identify body weight.

Journal Article Inclusion Criteria
• Lower extremity
• Weight-bearing anatomical regions
• Orthopedic traumatology

reported body weights published by the U.S. Department of Health and Human Services Centers for Disease Control and Prevention and National Center for Health Statistics [13].

Results

Literature Search Results

With the given search criteria, 5289 publications were identified as potentially applicable. Of these, only 54 articles (1.0 %) met all inclusion conditions and were included in final analyses [3,4,14–65]. Table 2 shows the breakdown between the number of hits per journal and the number of pertinent articles that met all inclusion criteria. The *Journal of Orthopaedic Trauma* had the highest percentage of pertinent articles at 26.9 %.

The majority of articles (81 %) utilized full body weight as a justification for the chosen loading scenario. The remainder of the publications referenced half body weight (8.6 %), partial weight bearing (5.2 %), crutch-assisted weight bearing (3.4 %), or toe-touch only weight bearing (1.7 %) in their force justifications. Regarding implant type, intramedullary nails were cited 47.1 % of the time, plates and screws were cited 41.2 % of the time, and external fixation was cited 11.8 % of the time. There were eight distinct anatomical regions

TABLE 2—Number of hits per journal.

Journal	Number of Hits	Pertinent Articles	Percentage
<i>Clinical Biomechanics</i>	706	9	1.3
<i>Clinical Orthopaedics and Related Research</i>	416	4	1.0
<i>Injury</i>	246	5	2.0
<i>Journal of Biomechanics</i>	1444	3	0.2
<i>Journal of Bone and Joint Surgery—American</i>	950	9	0.9
<i>Journal of Bone and Joint Surgery—British</i>	341	0	0.0
<i>Journal of Orthopaedic Research</i>	508	1	0.2
<i>Journal of Orthopaedic Trauma</i>	67	18	26.9
<i>Journal of Trauma</i>	320	4	1.3
<i>Medical Engineering & Physics</i>	291	1	0.3
Sum	5289	54	1.0

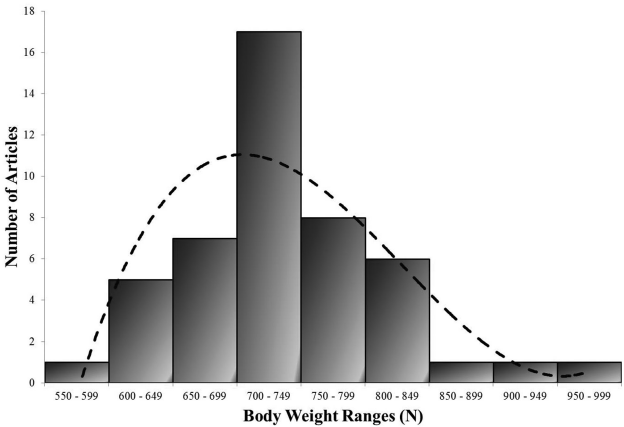


FIG. 1—*Histogram of those articles in the medical literature referencing some quantity for body weight as a force justification for a biomechanical test.*

cited: proximal femur (24.4 %), femoral shaft (2.4 %), distal femur (12.2 %), proximal tibia (9.8 %), tibial shaft (7.3 %), distal tibia (17.1 %), foot and ankle (22 %), and pelvis (4.9 %).

Figure 1 shows a histogram of the body weight ranges versus the number of citations. For all loading scenarios, the mean force applied during testing was 645 ± 197 N (range: 31 to 980 N), indicating minimal consensus among the included articles.

Analyses Based on Type of Weight Bearing

In the articles utilizing full body weight justifications, a mean force of 729 ± 79 N was reported; the range spanned over 418 N. Figure 2 shows the

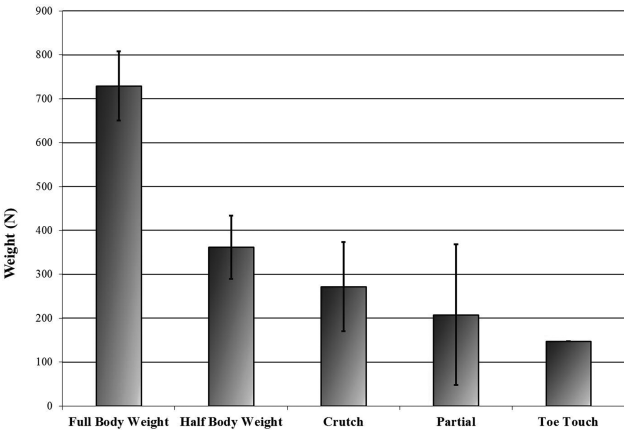


FIG. 2—*Distribution of body weights by weight bearing type.*

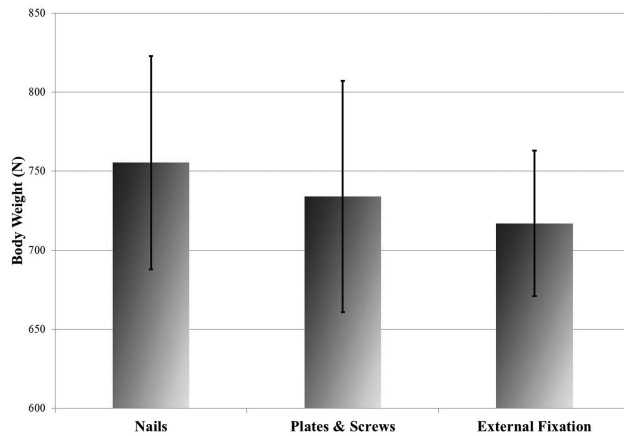


FIG. 3—Distribution of body weights by implant type, when full body weight was referenced.

distribution of cited body weights among the five loading scenarios. Half body weight had an average loading force of 362 ± 72 N. Crutch-assisted loading had an average loading force of 272 ± 101 N. Loading utilizing partial weight bearing had an average force of 208 ± 160 N. Toe-touch weight bearing was cited only once, with a force of 147 N.

Analyses Based on Implant Type

Articles citing intramedullary nails gave an average force of 755 ± 67 N. Articles citing plates and screws gave an average force of 734 ± 73 N. An average force of 717 ± 46 N was given for external fixation studies. Of those studies citing full body weight as a force justification, there was no statistically significant difference between implant types ($p = 0.24$ to 0.77 , depending on comparison), although external fixation studies trended toward citing lower body weights. Figure 3 shows the distribution of cited body weights among the three implant categories when full body weight was referenced.

Analyses Based on Anatomic Location

Foot and ankle studies utilized the least average force (665 ± 54 N), whereas proximal tibia studies implemented the greatest average force (776 ± 50 N). Only one citation for the femoral shaft was discovered, and it gave a force of 750 N. The proximal femur studies used an average force of 763 ± 116 N. Distal femoral studies used an average force of 710 ± 35 N. Tibial shaft studies used an average force of 745 ± 43 N. An average force of 745 ± 50 N was used for distal tibia studies. Studies that focused on the pelvis used an average force

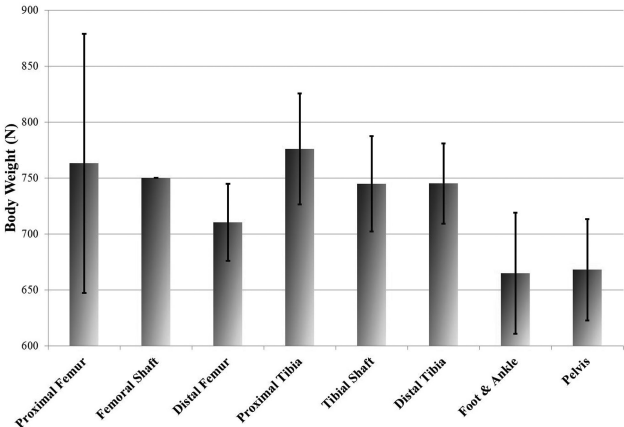


FIG. 4—*Distribution of body weight by anatomical region, when full body weight was referenced.*

of 668 ± 45 N. Figure 4 shows the distribution of cited forces among the eight anatomical regions when full body weight was referenced.

Body Weight Relative to Population

The most commonly cited mass associated with body weight was 70 kg; this was denoted in only 12 % of the articles. Relative to the report from the U.S. Department of Health and Human Services Centers for Disease Control and Prevention (CDC) and National Center for Health Statistics, the biomechanical studies found in this search underestimated mean body weight. The CDC reported a mean body mass for U.S. females between 20 and 80+ years old across all races and ethnicities of 75 kg (736 N). The CDC reported a mean body weight of 88 kg (863 N) for U.S. males across all races and ethnicities; their ages also ranged between 20 and 80+ years. This resulted in an averaged total across both groups of 82 kg (804 N). The 95th percentile for this average was 119 kg (1167 N). A difference of 75 N between the average full body weight loading level cited for testing justifications (729 N) and the body weight of the average U.S. citizen (804 N) was seen. Interestingly, a review of historical CDC data also showed trends of increasing body mass for the U.S. population. These numbers are 2 kg (20 N) greater than those in a previous report that was published only three years prior [66].

Discussion

Engineering analyses of the multifactorial physiological realities of fracture repair constructs are limited by the choice of test parameters. In this literature review, we found that an overwhelming majority of publications of

biomechanical tests did not identify the justification for the test force used. In the articles that did quantify loading parameters, there was low interstudy consistency in terms of weight bearing type, anatomical region, or fixation method.

Based on the results of this study, relative to the findings from the CDC, the current literature underestimates the average full body weight by 75 N. Weight bearing is often prescribed clinically as a certain percentage of body weight. As this study shows, weight bearing definitions and body weight definitions are often ill defined. Clinical studies have shown that the weight bearing protocol can have ramifications on outcomes [67–70]. Rubin et al. even went as far as calling for a need to standardize weight bearing orders for patients [70].

Another concern to be considered is the growing trend of increased weight gain among the U.S. population. Williamson et al. estimated that the average U.S. adult body weight is increasing at approximately two pounds per year [71]. As stated previously, the CDC showed an average increase of 2 kg (20 N) from data reported between 2005 and 2008. As average body weight continues to increase, material choice and design will become more critical in evaluations of the safety and efficacy of orthopedic implants, highlighting the need to consider the intended use and population when defining testing protocols.

Obesity is defined by the calculation of a patient's body mass index (BMI), that is, the patient's weight in kilograms divided by the square of his or her height in meters. A patient with a BMI between 25 and 30 kg/m² is considered overweight, one with a BMI between 30 and 40 kg/m² is considered obese, and one with a BMI over 40 kg/m² is considered morbidly obese [72]. Multiple studies pertaining to fractures of the pelvis and acetabulum have demonstrated complications when treating obese and morbidly obese patients [72–74]. Similarly, complications due to patient obesity have been shown in the syndesmotic fixation of ankle fractures [75].

Unlike reconstruction surgeries of the hip and knee, orthopedic trauma surgeries are rarely elective. There is little time to prepare the patient for fracture repair; there is also little control over patient physiology. The patient population can vary from young, healthy teenagers to elderly, osteoporotic adults. Along with this, patient body weight can also vary widely.

Anthropomorphic data obtained from multiple patient-based studies [76–79] can be found in Table 3, including mean age, mean body weight, mean height, and mean BMI. These patients varied in age, sex, and health status, giving a wide sampling of the population. The body weight ranged from 558 to 818 N, corresponding to an average BMI of 23.9 ± 1.8 kg/m². The body weight from these studies fell within the range of estimated body weights we found (562 to 980 N).

Although not fully inclusive of all trauma patients, the patient population for this subset of studies was shown to be non-obese. This implies that although

TABLE 3—*Anthropomorphic data from various chair-rise studies.*

Author	<i>n</i>	Age, years	Body Weight		Height, m	BMI, kg/m ²	Comments
			N	kg			
Gross et al. [76]	26	70.1	606.5	61.9	1.62	23.6	Healthy elderly
	12	24.2	568.3	58.0	1.65	21.3	Women
Schenkman et al. [77]	11	28.9	590.0	60.2	1.64	22.4	Young adults
	10	67.3	694.8	70.9	1.72	24.0	Older adults
Alexander et al. [78]	16	81.9	634.1	64.7	1.64	24.4	Training group, men and women
	14	84.4	611.5	62.4	1.59	24.9	Control group, men and women
Alexander et al. [79]	7	26.4	705.6	72.0	1.78	22.7	Young males
	10	21.0	605.6	61.8	1.68	21.9	Young females
	9	71.6	818.3	83.5	1.75	27.3	Old, able males
	14	72.4	628.2	64.1	1.59	25.4	Old, able females
	11	84.4	557.6	56.9	1.50	25.3	Old, unable females
	Average	57.5	638.2	65.1	1.7	23.9	
	Standard deviation	26.4	75.4	7.7	0.1	1.8	

the estimated body weights found in the literature may be representative of the non-obese clinical population, they might not include representation of the obese to morbidly obese segment of the population; this is one limitation of this study. The more obese patient populations would allow for an even greater worst-case loading scenario if incorporated into biomechanical testing.

The CDC body weight of 804 N corresponds to an average BMI of 28.4 kg/m², which is considered overweight; the 95th percentile body weight of 1167 N corresponds to a BMI of 40.1 kg/m², which is considered morbidly obese. These values were for all race and ethnicity groups ranging between 20 and 80+ years old and were an average of males and females. As there was a 75 -N difference between the estimated body weight found in the literature and the values obtained from the CDC, biomechanical studies should always seeks to provide a rationale for the chosen estimated body weight utilized. One reason for the discrepancy could be the population sampling. Data from the literature might have been chosen from a more limited patient population that was not as representative of the overall patient population. The CDC data might be a better representation of the potential patient population for orthopedic trauma surgeries.

Additionally, the effect of this discrepancy on medical device strength and/or longevity is unknown. Future work could include finite element analysis of various implant designs, evaluated at the different body weight levels, to

illustrate the differences in peak stresses found in the implants due to the differing loads. Mechanical testing could perhaps illustrate the differences in implant strength based on the biomechanical body weight found in literature versus the greater average body weight given by the CDC. In a cyclic fatigue model, the greater forces would lead theoretically to earlier construct failure, and could even change the failure mode observed in the tested construct.

Conclusions

Biomechanical testing should always support clinical data. In this study a discrepancy between biomechanical test force parameters was observed, indicating that comparisons between biomechanical studies might be difficult, especially when extrapolating results used to influence clinical care. Researchers should clearly identify the weight bearing force parameters to be simulated in each biomechanical testing protocol. Consideration should also be given to the intended use of the device being evaluated in a study. Underestimating the forces used to evaluate medical devices might result in false positive test results. A device might be shown to pass a predetermined acceptance criterion, yet in reality the device might not be suited to accept full clinical loading. The opposite is also true—overestimating the force used to evaluate medical devices could lead to a false negative. A device might be shown to be inadequate, when in reality it might perform as needed in a clinical situation.

Based on our literature review, researchers chose body weight as 729 N on average. From the reported CDC findings used in this study, a loading scheme of 804 N or greater might be most appropriate for simulations of full body weight. When testing medical devices for safety and efficacy purposes, it is often the goal of biomechanical testing to provide a worst-case loading condition. The average 95th percentile of body weight was found to be 1167 N. Thus, in biomechanical testing, a recommended force for body weight within the range of 804 to 1167 N might be most appropriate.

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Bending Fatigue Characteristics of Orthopedic Biomaterials

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ABSTRACT: The overall mechanical properties of titanium and stainless steel biomaterials are well known, but a review of the literature indicates that comparing their biomechanical fatigue performance may be application dependent and is often evaluated at high cycle counts up to 10×10^6 cycles or more. The purpose of this study was to utilize a standardized methodology to compare the bending fatigue performance of 316L stainless steel (SS) and Ti-6Al-4V (Ti) orthopedic trauma implants in a trauma device lifecycle scenario of no more than 1×10^6 cycles. 1×10^6 loading cycles represents approximately 1 year in vivo that is the typical utility life of a trauma device, because healing would be expected in this time frame. Cannulated rods, made of either SS or Ti, were prepared, all of which incorporated a transverse hole that simulated the distal geometry of an intramedullary nail. Based on ASTM F1264-03 Standard Specification and Test Methods for Intramedullary Fixation Devices, the specimens were tested by applying multiple force levels using four-point bending, sinusoidal-wave, cyclical fatigue until failure, or run-out at 1×10^6 cycles occurred. Various force levels were utilized during testing to evaluate the trend between stress levels (range: 480–2200 MPa) versus fatigue life. On average, the SS specimens survived longer than the Ti specimens at greater stress levels and the Ti specimens survived longer than the SS specimens at lesser stress levels. This study shows that fatigue

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life comparisons among trauma intramedullary nails may not only be design dependent, but is also governed by the relationship between expected in vivo lifetime and stress level. Therefore, understanding the clinical indications and intended uses are critical in the decision to use either material for orthopedic trauma applications.

KEYWORDS: biomechanics, trauma, stainless steel, titanium, mechanical testing

Introduction

The mechanical properties of stainless steel (SS) and titanium (Ti) are well known. Biomechanical fatigue performance of orthopedic trauma implants is material and application specific. The fatigue performance of SS and Ti are typically evaluated at lives of 10×10^6 cycles [1,2]. Few data exists in the literature for low-cycle fatigue performance of biomaterials up to 1×10^6 cycles. 1×10^6 cycles is representative of approximately 1 year in vivo that is considered the life expectancy of trauma devices [3].

The merits of SS and Ti have been discussed repeatedly throughout the literature [4–7]. The choice of material for trauma fracture fixation devices is complicated given the wide variety of intramedullary (IM) nails, plates and screws, and external fixation options. The various material types and properties available, as well as differences in device geometry, can play an important role in clinical outcome.

Some of the most commonly used steels for trauma implants include 316L, 316LVM (vacuum re-melted), 22-15-5, and BioDur 108 [8]. Titanium and its alloys are also widely used, including commercially pure Ti (CP-Ti), Ti-6Al-4V, Ti-6Al-4V ELI, Ti-6Al-7Nb, and Ti-15Mo [9,10].

Stainless steels, such as 316L, are frequently used as biomaterials for internal fixation devices because of qualities such as good mechanical properties, corrosion resistance, cost effectiveness, nonmagnetic response, and biocompatibility [8]. An alternative to 316L has been recently introduced. BioDur 108 is a nickel-free austenitic stainless alloy with a high nitrogen content (Carpenter Technology Corporation, Reading, PA) that has greater tensile and fatigue performance as compared to nickel-containing alloys such as 316L. BioDur 108 exhibits superior resistance to pitting and crevice corrosion as compared to 316L SS and is non-magnetic [11]. Titanium and titanium alloys such as Ti-6Al-4V have become an increasingly popular option for internal fixation devices. Titanium is biocompatible, exhibits excellent corrosion resistance, shows fewer resultant magnetic resonance imaging (MRI) and computed tomography (CT) scan artifacts as compared to SS, and has a modulus of elasticity closer to that of bone [7,9,10].

The purpose of this study was to evaluate the fatigue performance of Ti-6Al-4V as compared to SS in a manner that is relevant to trauma devices.

Specifically, a comparison between Ti-6Al-4V and 316L evaluated the fatigue limit of both materials at 1×10^6 cycles, which is relevant to trauma devices. A secondary comparison between Ti-6Al-4V, 316L with various percentages of cold working, and BioDur 108 was also performed. This secondary comparison evaluated the fatigue performance of each material at one load level. The overall goal of the study is to provide information to assist in material selection for both design and use of orthopedic trauma devices. Depending on the intended use of the device, alternate material choices may be made to improve the design and provide the best possible healing outcome for the patient.

Materials and Methods

Specimen Preparation

Test specimens were prepared using Ti-6Al-4V, which met the requirements of ASTM F1472-08e1 [12], and 316L that met the requirements of ASTM F138-08 [13]. Test specimens were also prepared using BioDur 108. Specimens were cannulated rods with a transverse hole, and were designed to simulate the same cross-sectional geometry and perpendicular locking screw hole diameter as a 13-mm-IM nail. The specimens were 17.8 cm (7.0 in.) in length, had a 13 mm (0.51 in.) outer diameter, an 8 mm (0.33 in.) inner (cannulation) diameter, and a 5 mm (0.21 in.) transverse screw hole diameter. The transverse screw hole was added at the midpoint of each test specimen, in line with the loading direction. Flats were machined 25.4 mm (1.0 in.) from either end of the cannulated rod. The flats prevented the specimen from rotating during the test. Figure 1 shows a representative photo of the test specimen.

The cannulated rods were tested at nominal gross section stresses that were calculated using the formula:

$$\sigma = \frac{M(c)(K_t)}{I} \quad (1)$$

where:

σ = stress, psi or Pa,

M = bending moment, lbf · in. or N · m,

c = distance from neutral axis to outermost fiber, in. or m,

K_t = stress concentration factor, and

I = second moment of area, in.⁴ or m⁴.

The stress concentration factor, K_t , at the edge of the transverse screw hole, was calculated by extrapolating data from Peterson's stress concentration factors [14] for a round shaft in bending with a transverse hole, and was determined to be 3.3.

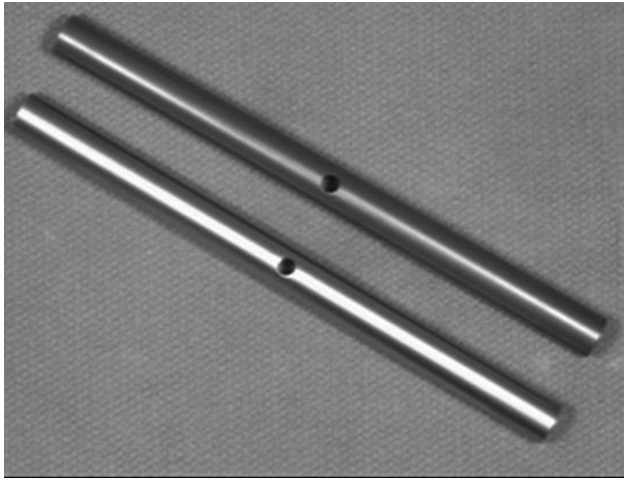


FIG. 1—Representative photo of the 13 mm OD test specimen.

Mechanical Testing

Four-point bending fatigue testing was performed based on the recommendations of ASTM F1264–03 [3]. A universal joint was added to the top (loading) fixture, and was locked such that it could only move in the plane of the cannulated rod being tested. The addition of the universal joint ensured proper loading during each test. A four-point bend fixture was secured to a mechanical testing frame. Top (loading) and bottom (support) fixtures were aligned to ensure proper load application to the test specimen. The top fixture had a loading span of 50.8 mm (2.0 in.) and the lower fixture had a support span of 152.4 mm (6.0 in.). The loading and support fixtures incorporated roller bearings and 9.53 mm (0.375 in.) loading rollers. Figure 2 shows a photo of the test setup.

Cannulated rods were tested in a sinusoidal cyclic manner with an axial compressive load at a frequency of 3 Hz. Tables 1 and 2 show the forces used and number of specimens for the Ti-6Al-4V and 316L test specimens, respectively. An R ratio of 0.1 was used. Test specimens were tested in air at room temperature ($22 \pm 1^\circ\text{C}$). Fatigue testing was performed until cannulated rod failure or completion of 1×10^6 cycles without failure (run-out). Displacement limits were set to ± 0.51 mm (± 0.02 in.) beyond the initial peak/valley displacement values for each nail coupon, based on the output from the test frame controller. Force limits were set to ± 89 N (± 20 lbf) beyond the initial peak/valley force values for each nail coupon. The number of cycles to failure, as well as failure location, was recorded for each specimen. The fatigue limit for each group was determined when five samples in each group achieved run-out of 1×10^6 cycles.

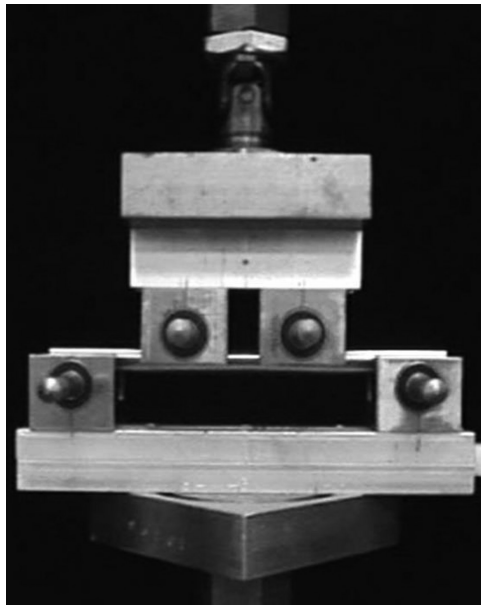


FIG. 2—Four-point bending fatigue test setup with the 13 mm OD test specimen.

For the BioDur 108 evaluation, the test setup was the same as stated previously, with one exception. Instead of testing at multiple load levels, one load level was chosen; 2002 N (450 lbf) was chosen based on previous testing experience. At this force, all test specimens were expected to fracture. Included in the evaluation of BioDur 108 were test specimens manufactured from

TABLE 1—Forces and number of specimens used for the Ti-6Al-4V test specimens.

Force		No. of Samples
(N)	(lbf)	
1223	275	5
1334	300	5
1446	325	5
1557	350	5
1735	390	4
1846	415	4
2002	450	10
2224	500	4
2669	600	4
3336	750	4
4003	900	4

TABLE 2—*Forces and number of specimens used for the 316L test specimens.*

Force		No. of Samples
(N)	(lbf)	
890	200	5
1001	225	5
1112	250	5
1223	275	5
1334	300	5
1446	325	5
1557	350	5
1668	375	5
1779	400	4
1868	420	3
2002	450	10
2224	500	3
2669	600	3
3336	750	3
4003	900	3

Ti-6Al-4V, as well as two groups of 316L of various cold working percentages. The first group (cw1) had a minimum ultimate tensile strength (UTS) of 1103–1241 MPa. The second group (cw2) had a minimum UTS of 862 MPa.

Statistical Analysis

For the 316L versus Ti-6Al-4V evaluation, a log transform was performed on the cyclic data. Only those load level groups that contained only fractured specimens were evaluated. The results were compared with an ANOVA single-factor statistical method; to make multiple comparisons, the Tukey method was used post hoc ($\alpha = 0.05$).

For the BioDur 108 evaluation, a log transform was performed on the cyclic data. The results were compared with an ANOVA single-factor statistical method; to make multiple comparisons, the Tukey method was used post hoc ($\alpha = 0.05$). Only the data that resulted in a fracture was used for the comparison; run-out data points were not included.

Differences from ASTM F1264

This study deviated from the recommendations of ASTM F1264-03 [3] in four ways. First, a transverse screw hole was added to the midpoint of the cannulated rods used for testing. The intent of the standard is to evaluate the shaft portion of an IM nail. A transverse screw hole was added to better simulate the

proximal or distal portion of an IM nail, locations at which a fracture is more likely to occur [7,15,16]. Second, anti-rotation flats were added at both ends of the cannulated rod. The addition of flats differed from the recommended guide shoe method, as outlined in section A3.8.5 of the standard. The use of the flats was necessary to maintain the desired hole orientation and did not appear to have an effect on the bending fatigue performance of the test coupons, as none of the samples fractured outside of the loading span. Third, a universal joint was added to the upper loading fixture. The universal joint was added to alleviate potential misalignment issues present in the setup. Finally, the load and support spans chosen for this test were different than those given in section A3.8.3.

Results

Ti-6Al-4V versus 316L SS Evaluation

All test specimens that fractured did so through the tensile side of the nail coupon at the transverse screw hole. In general, the Ti-6Al-4V test specimens fractured at lower stress values when compared to the 316L test specimens at the same cycle counts. The fatigue limit at 1×10^6 cycles was found to be 664 MPa (96 ksi) and 483 MPa (71 ksi) for the Ti-6Al-4V and the 316L test specimens, respectively. Figure 3 shows a graphical representation of the fatigue results, in the form of an S-N plot, which gives stress in MPa, versus cycle count, plotted in log scale.

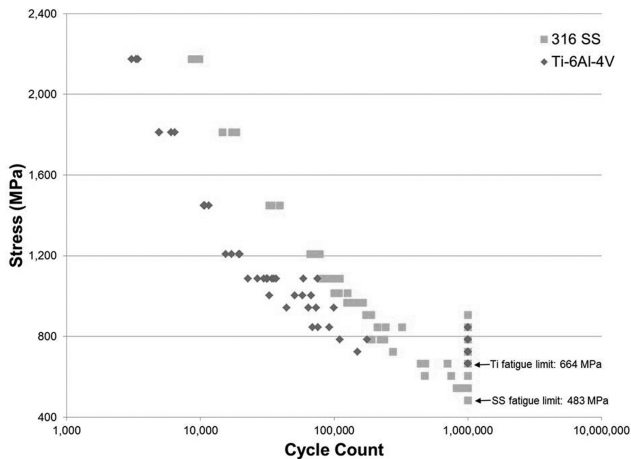


FIG. 3—Standard S-N curve for the bending fatigue test.

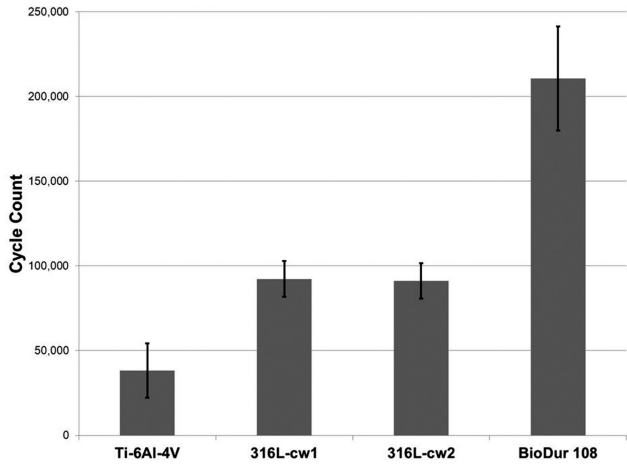


FIG. 4—Results of the bending fatigue evaluation of BioDur 108.

Statistical analysis was performed on those data sets that did not contain run-out values, which corresponded to load levels of 4003 N–1735 N (900–390 lbf). For these load levels, there was a statistically significant difference ($p < 0.05$) in the fatigue performance of the 316L test specimens as compared to the Ti-6Al-4V test specimens; the 316L test specimens had the higher fatigue performances at each load level. Below 1735 N, statistical analysis was not performed as the groups contained a mixture of fractures and run-outs.

BioDur 108 Evaluation

All of the test specimens in the Ti-6Al-4V group, in the 316L-cw1 group, and in the 316L-cw2 group fractured at the transverse hole. The BioDur test group had five of 16 samples achieve run-out of 1×10^6 cycles; the remaining 11 samples fractured at the transverse screw hole. Of those specimens that fractured, the average fatigue performance of BioDur specimens was 450 % higher than Ti-6Al-4V specimens ($p < 0.05$), 128 % higher than 316L-cw1 specimens ($p < 0.05$), and 131 % higher than 316L-cw2 specimens ($p < 0.05$) (Fig. 4).

Discussion

Ti-6Al-4V versus 316L SS

At higher stress values, SS test specimens had higher average fatigue lives as compared to Ti-6Al-4V test specimens. However, at lower stress values, Ti-6Al-4V demonstrated a higher fatigue performance as compared to SS, and as indicated by the higher fatigue limit Pohler showed a similar trend with SS

as compared to CP-Ti [10]. In his study, at greater stress levels, SS had a greater average fatigue performance as compared to CP-Ti; at lower stress values, CP-Ti had an increased fatigue performance as compared to SS.

Ti-6Al-4V test specimens had a fatigue limit of 664 MPa at 1×10^6 cycles. Ti-6Al-4V fatigue limits have been reported to be 240 MPa for notched specimens [1], 510 MPa for un-notched specimens [1], and ranging from 620 to 724 MPa for specimens with unknown conditions [17]. In each of these cases, the fatigue limits were reported at 10×10^6 cycles. The results of our study fell within the ranges found in the literature.

Fatigue limits at 10×10^6 cycles for 316L range from 269 MPa (unknown condition) [1] to 1379 MPa (annealed) [2]. Our 316L fatigue limit at 1×10^6 cycles of 483 MPa fell within this range.

The reported fatigue limits for Ti and SS are given typically at 10×10^6 cycles. While it is a limitation of this study that we chose to compare our fatigue limit values with the reported literature, the goal of this paper was to provide information on these materials at a run-out limit of 1×10^6 cycles. While orthopedic reconstruction devices such as hips and knees are designed to last 10–15 years, orthopedic trauma devices are not. Their intended life span is approximately 1 year, as healing should have occurred by then, barring any unforeseen complications [3].

A limitation of the S-N data provided in this study is that these values were specific to the test specimen geometry, in this instance a simulated IM nail. The fatigue performance of the chosen materials may be affected by differences in design and testing conditions.

BioDur 108

While our bending fatigue results illustrate an increased fatigue performance of BioDur 108 as compared to Ti-Al-4V and 316L, it should be noted that these results are specific to this test and these geometric conditions. Differences in implant design and component surface finish may influence the results of the test more than material properties alone. Other considerations for this material include higher cost and decreased machinability [2]. The modulus of elasticity of BioDur 108 (200 GPa; 29 Msi) is almost twice as high as Ti-6Al-4V (1140 GPa; 16.5 Msi) [17]. Stiff, rigid constructs have been shown to be problematic during fracture healing, resulting in stress shielding and poor callus formation [18–22].

One limitation to the BioDur 108 testing is that the coupons were evaluated only at a single force level, unlike the earlier presented information for 316L versus Ti-6Al-4V. Single force level testing is often performed for comparison purposes between devices [6,7,16]. It is also recommended, again for comparison purposes, in testing standards for IM nails and bone screws [3] and bone plates [23,24].

SS versus Ti in the Literature

A review of the literature with regard to biomaterial type and orthopedic trauma implants was also performed. A wide variety of results were found, some of which are given in the following paragraphs. The literature search further supports the results of this study, in that while biomaterial choices can have an influence in the device performance, other factors such as intended use and implant design should be considered as well.

Mahar et al. compared the static and fatigue performance of SS and CP-Ti flexible nails in a simulated femoral fracture [4]. Flexible nails are most commonly used in pediatric and adolescent patients. CP-Ti nail constructs were superior to SS nail constructs when range of motion, axial stiffness, and shortening of the construct during fatigue testing were considered. The superior CP-Ti construct results were explained by illustrating the greater elastic limit of CP-Ti as compared to SS. The greater elastic limit of CP-Ti likely contributed to the superior performance of the CP-Ti constructs, because of the nails better conforming to the bone canal.

Pieske et al. compared Ti and SS pins utilized as fracture fixation devices in a distal radius fracture model [5]. SS pins were shown to have a greater rate of pin loosening. SS pins were also shown to have a higher rate of premature removal because of severe pin-track infection. Micromotion can cause pin-related complications such as loosening that can lead to infection. In this study, it was anticipated that the greater modulus of SS would help prevent undesired micromotion. However, Ti pins equalized the effects of increased micromotion because of lower elastic modulus along with superior osseointegration. Pieske et al. commented on an increased cost of Ti pins as compared to SS pins [5]. Because of this, they still recommended the use of SS pins, as the results did not show a significant reduction in complications surrounding external fixation about the wrist to justify the higher cost of Ti pins.

Hoffmeier et al. evaluated the effects of material and working length (the distance between the first locking screws from each side of a bridged fracture) on the mechanical properties of locked distal femur plating constructs [6]. SS and CP-Ti plates of three different working lengths were evaluated using both static and fatigue mechanical testing. SS plate constructs had a higher fatigue life as compared to CP-Ti constructs. SS plate constructs showed no correlation between working length and fatigue life; the CP-Ti plate constructs did show an increasing trend between working length and fatigue life of the constructs.

Hsu et al. evaluated the static and bending fatigue performance of IM nail coupons and screws manufactured from SS and Ti [7]. In their study, they varied the inner diameter and root diameter of the screws; for the IM nail coupons, the outer diameter and the transverse screw hole diameter both varied. Titanium screws had superior static and fatigue performance as compared to SS screws. However, SS nail coupons had superior static and fatigue performances

as compared to Ti nail coupons. These differences were attributed to the higher notch sensitivity of titanium.

Conclusions

As shown in this study, as well as throughout the literature, material choice can have a wide variety of effects that may influence the overall performance of orthopedic trauma implants. Fracture fixation should be tailored to fracture pattern, fracture location, bone quality, and the location of the implant as dictated by the anatomy. All of these factors combine to form an array of considerations when choosing an implant.

When choosing 316L as an implant material, many factors should be considered including elastic modulus, a range of material strengths afforded by various levels of cold working, and higher average fatigue performance at higher stress values as compared to Ti-6Al-4V. Stress shielding should also be guarded against; a stiff construct can impair callus formation.

As compared to 316L, Ti-6Al-4V provides a lower elastic modulus that more closely matches the modulus of bone. Titanium exhibits improved fatigue performance at lower stress values as compared to 316L. However, Ti-6Al-4V does exhibit notch sensitivity, which can influence fatigue performance, and has a higher cost as compared to SS. For this study, 316L showed a statistically significantly higher fatigue performance as compared to Ti-6Al-4V at load levels ranging between 4000 and 1735 N.

As shown in this study, and in a review of the literature, material choice alone should not be the deciding factor for orthopedic trauma devices. Device geometry, intended use, and patient comorbidities should also be considered when designing a new device, or choosing between existing devices when treating patients. While both SS and Ti have their advantages and disadvantages, both have been used successfully to treat a wide array of fracture types with a variety of medical devices. Through careful consideration of material choice, fracture fixation can be optimized to provide the patient with the best possible standard of care.

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Ti-6Al-4V Fatigue Strength After Shot Peening: The Role of a Corrosive Environment

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ABSTRACT: When used as implant material in joint arthroplasty or trauma surgery, titanium and its alloys are subjected to dynamic in vivo loading conditions. In the case of an inappropriate design or an unforeseen loading event, breakage of the metal component might occur. This is a rare (<1 % in total hip arthroplasty) but serious complication, and it requires special attention for revision surgery. Titanium and its alloys, such as Ti-6Al-4V, which was used in this study, can be considered as the most corrosion resistant of the orthopedic metals used today. However, the influence of a corrosive medium on fatigue strength during cyclic loading has not yet been fully investigated. In order to enhance the fatigue strength, mechanical surface treatments have been applied to some implant designs, in particular revision hip systems. Therefore, specimens that had been shot peened in a two-step procedure (high-intensity cut wire peening and low-intensity cleaning by glass beads) were used for fatigue testing to a maximum of 10×10^6 cycles at 10 Hz ($R = 0.1$) under two conditions: (a) in a dry atmosphere (room conditions) and (b) circulated by a corrosive saline solution. Notable increases in fatigue strength were found in both cases after shot peening relative to untreated (annealed) specimens, and this has been associated with the

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introduction of high residual stresses at the surface. Even though no differences were seen at 10×10^6 cycles, the influence of a corrosive environment might be relevant at higher loads.

KEYWORDS: Ti-6Al-4V, corrosion, fatigue failure, arthroplasty, trauma surgery, shot peening

Introduction

In medical engineering, titanium and its alloys are used as standard materials for load-bearing cementless total joint prostheses and implants in trauma surgery such as bone plates, screws, or intramedullary nails. This is mainly because of the metals' excellent mechanical properties, including adequate tensile and fatigue strength and an elasticity that is closer to that of bone than those of other medical metals. This is a particularly important issue for total joint replacements (TJR), as bone is continuously adapting to mechanical changes in its environment in accordance with Wolff's law [1]. A stiff structure that "splints" the bone and keeps mechanical stimuli to a minimum will result in an effect that is also known as *stress shielding* and can be the cause of considerable hypertrophy and mechanically induced osteolysis [2]. In addition, titanium and its alloys are well known for their good biocompatibility, allowing the bone to grow toward the surface thanks to the formation of a bio-tolerant titanium dioxide layer (TiO_2) under a physiological environment [3]. That TiO_2 layer also serves as a protective coating (passivation) preventing further corrosion of the reactive substrate. Titanium was clinically introduced in the 1950s [4] with very good in vitro and long-term results in patients and has more than proven itself in the clinical routine [3]. Today it is the material of choice in many cases.

Despite these advantages, several case reports have recently been published on titanium (alloy) components that suffered early mechanical failure [5–9], as a result of either inadequate designs or an unexpected corrosive attack in situ under high loading conditions (Fig. 1). From an engineering point of view, the size of a TJR or trauma implant is predetermined by the surrounding bone structures. Titanium and its alloys are known to be sensitive to critical notches (i.e. sharp edges and small radii), which may be used for functional details. However, today, a trend exists toward small and minimal invasive implant components that allow greater intra-operative flexibility when using modular systems [10]. Under cyclic loading conditions or micromotions in the interface of assembled components, high local stresses are produced that can lead to crack initiation and propagation, and these are thought to be the main cause of early fatigue fracture. Furthermore, concerns have recently been raised about adverse reactions to corrosive metal debris [11,12].

Exact failure rates are not reported in the literature, but national registers give a good overview of implant fracture as a reason for revision after total hip arthroplasty (Table 1) [13,14]. However, registers barely differentiate between the single components or materials that led to fracture. Nevertheless, according

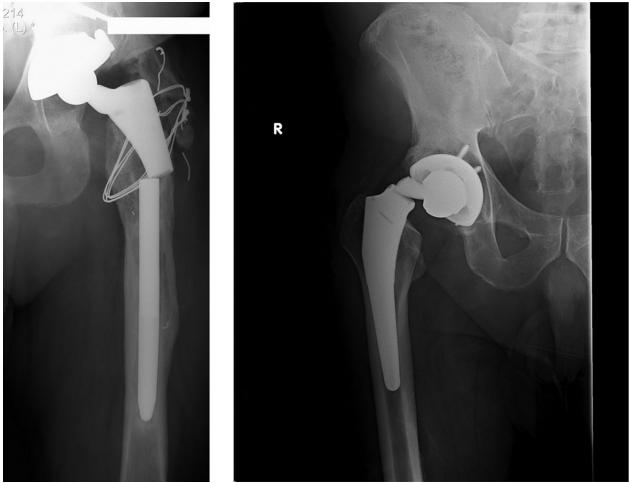


FIG. 1—Examples of failed titanium alloy components. Left: Reprinted from Ref 5 with permission from the *Journal of Bone and Joint Surgery*, American Volume. Right: Reprinted from Ref 6 with permission from Elsevier.

to these data, the number of revisions due to fracture of a metallic component is less than 1 %. In other fields of joint arthroplasty or trauma surgery, data on fracture incidence are not available. However, component failure is known to occur after cyclic loading. It is a rare but serious problem and is a challenge for the surgeon who performs revision surgery.

Metal components that are intended to temporarily or permanently replace load-bearing functions are subjected to extensive external loads that might not have been anticipated. Bergmann et al. used instrumented TJR systems in order to quantify the loads that act on TJRs [15]. They measured forces in the hip joint of up to 250 % of the patient’s body weight (%BW) during normal walking and up to 320 %BW when the patient was walking downstairs. Maximum loads of up to 870 %BW have been measured when a patient stumbles during level walking [16]. Comparable forces have been reported in the knee joints [17]. For non-load-bearing joints such as the shoulder, forces of up to 150 %BW have been measured during daily activities (e.g., lifting a 2 kg weight to head height) [18]. Those high forces are mainly the result of compensating muscle forces that compress the joint in addition to the acting gravitational

TABLE 1—Register data from the Swedish and Australian national hip joint registries [13,14].

Register	Reason for Revision	Rate Relative to All Revisions, %
Swedish	Implant fracture	1.5
Australian	Implant breakage, acetabular	0.9
Australian	Implant breakage, stem	0.8

forces. In that context, realistic loads in a worst-case range (high activity, heavy patient, etc.) should be taken into account for preclinical fatigue testing of implant components [19].

Implant breakage is therefore still an issue in implantology and can be attributed to the implant's design, the implant material, and the external load conditions. As the design is very much linked to the function and the external loads that are given by the patient's activity, new approaches aim to improve the fatigue strength of a given design by modifying the properties of the material used. One approach is to replace the titanium with polyether ether ketone, which is currently being used in clinical studies [20]. Mechanical surface treatments as part of this study also might be an appropriate alternative. They have been used for a long time in applications in aeronautics and the automotive industry and, to some extent, in revision arthroplasty [21,22]. The best-known treatment is shot peening, in which a blasting material (cut wire, glass, ceramic, etc.) is accelerated toward the surface to be treated. In the course of the impact, the substrate is plastically deformed at the surface, resulting in compressive residual stresses (Fig. 2). Studies on titanium and its alloys have shown that fatigue strength may be significantly improved by this technique [23].

In a physiological environment, a load-bearing component is always working in a corrosive medium that could attack the substrate and have a direct effect on the mechanical outcome. Under static conditions, titanium is able to easily passivate by building up a thin and fine-pored TiO_2 layer that protects the substrate from further corrosion. However, under dynamic loading, which occurs during daily activities, the oxide layer might fail locally because of a difference in elasticity relative to the bulk titanium and high tensile stresses under bending conditions. A crack can be initiated, followed by repassivation of the cleared substrate (Fig. 3). This effect, together with the notch sensitivity of titanium, might further promote metal fatigue failure in a corrosive medium.

Therefore, the aim of this study was to determine the influence of a corrosive environment on the fatigue strength of a Ti-6Al-4V component following

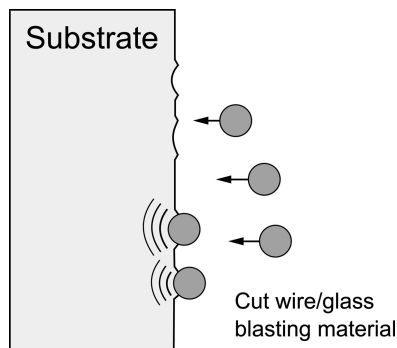


FIG. 2—*Principle of shot peening.*

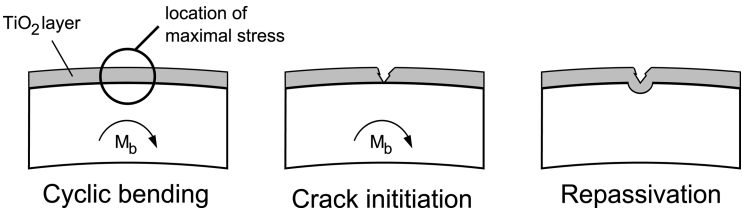


FIG. 3—Scheme of crack initiation and passivation of a dynamically loaded titanium part.

a shot peening process. This effect is currently not addressed when fatigue testing is performed in a dry environment.

Materials and Methods

Bone-shaped fatigue specimens made of a standard Ti-6Al-4V alloy (DIN ISO 5832-3 [24]) were used with a relieved middle part and a minimal sectional diameter of 10 mm (Fig. 4).

All specimens were cleaned and subjected to an initial annealing treatment in an oven at 620°C for 10 h while surrounded by an inert gas (nitrogen) in order to remove all residual stresses before testing. After annealing, the specimens were etched in a 20 vol. % HNO₃ bath at 60°C overnight to detach the residual blushing layers. Additionally, all specimens were subjected to a two-step shot peening treatment (description/size/Almen intensity/coverage): (1) high-intensity cut-wire peening (stainless steel cut wire 0.4/d = 400 μm/0.012 in. A/200 %) followed by (2) a low-intensity cleaning process employing glass blasting medium to remove contaminations from the cut wire (GP 60 glass beads/d = 60 to 150 μm/0.006 in. N/125 %).

Residual stress depth measurements on one specimen before shot peening (as annealed) and one specimen after shot peening were performed using the $\sin^2 \psi$ method [25]. The depth profile was realized via iterative electrochemical layer removal and X-ray diffraction stress analysis on the α titanium phase ({114} planes) using Fe-filtered Co K α radiation (XRD3000 PTS, Rich. Seifert Ltd., Germany). In addition, the arithmetic roughness (R_a) was measured in

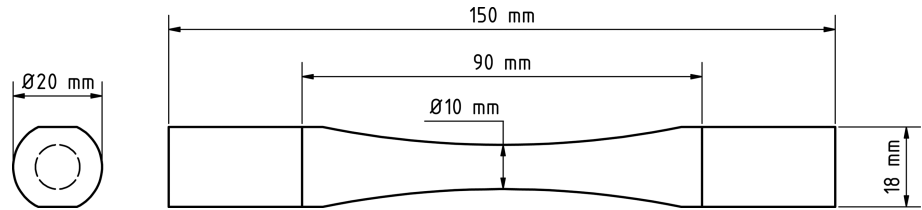


FIG. 4—Ti-6Al-4V fatigue specimen.

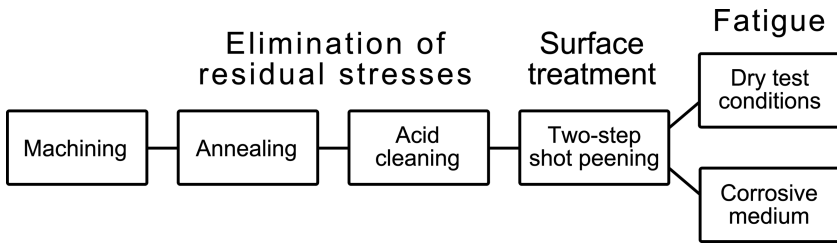


FIG. 5—Protocol of the study.

both cases (Mahr Perthometer M2, Germany). Figure 5 gives an overview of the study protocol.

Finally, mechanical fatigue testing was performed under two different conditions (Fig. 6):

- (a) in a dry atmosphere ($n = 8$), and
- (b) with samples surrounded by a corrosive 0.9 % saline (NaCl) solution circulated using a peristaltic pump at a total volume of 1000 ml (volume in the tube: 42 ml) ($n = 6$).

It has been shown that a physiologically relevant corrosive environment can generally be simulated by a saline solution at 37°C; this is also addressed in relevant ASTM and ISO standards [26]. All specimens were tested at 10 Hz under pulsating compressive stress ($R = 0.1$) until fracture occurred or the maximum of 10×10^6 cycles had been reached.

The residual stress distribution and fatigue performance were then compared to those of specimens that had not undergone additional shot peening treatment (as-annealed specimens), which served as a reference.

Results

Figure 7 shows the residual stress profiles prior to and after the shot peening treatment. As expected, no residual stresses were detected after the annealing

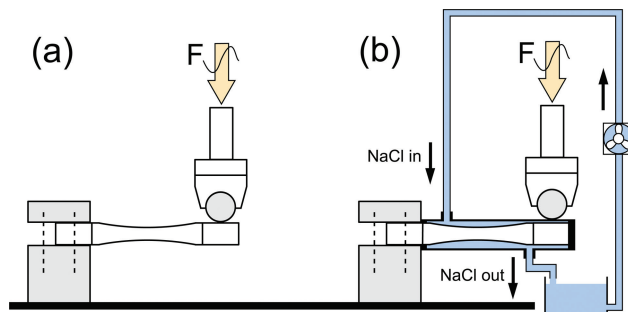


FIG. 6—Fatigue testing setup: (a) dry conditions; (b) circulating corrosive medium.

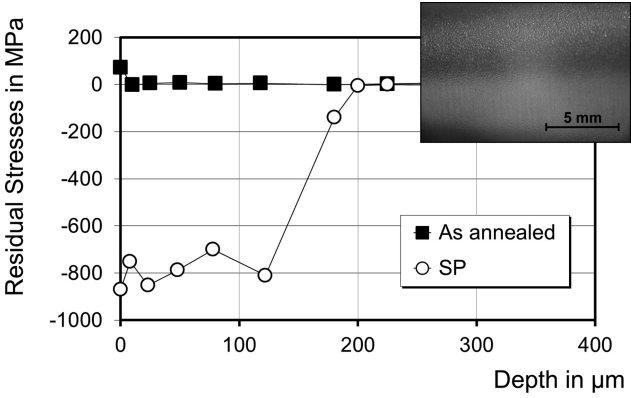


FIG. 7—Residual stress profile. Upper right: surface after shot peening (SP).

process (as annealed), whereas shot peening introduced high compressive stresses of up to -870 MPa at the surface and up to a depth of $200\text{ }\mu\text{m}$.

The roughness increased after shot peening from $R_{a,\text{annealed}} = 0.34 \pm 0.20\text{ }\mu\text{m}$ to $R_{a,\text{shot peened}} = 2.02 \pm 0.16\text{ }\mu\text{m}$ ($p < 0.01$). The indentation marks of the blasting medium are visible in Fig. 7 (upper right).

No differences in fatigue strength were found between the treated specimens under dry conditions and those in the saline solution (Fig. 8). However, the specimens seemed to fail somewhat earlier under higher loads in the NaCl solution, even though the difference is low and not statistically meaningful. As expected, shot peening led to a considerable increase in fatigue strength in both cases.

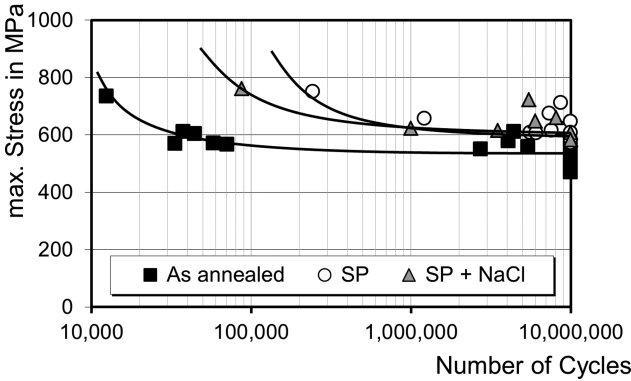


FIG. 8—Woehler curves. SP: Shot peening with testing under dry conditions; SP + NaCl: Shot peening with testing in saline solution.

Discussion

Based on these results, no influence of the corrosive medium is seen in the Woehler curves above 10^6 cycles. However, it seems that the fatigue strength might decrease at higher loads, resulting in a shorter time to failure in a saline solution than in the dry conditions. A significant difference has been reported for pure titanium and Ti-6Al-4V rods in a synthetic saliva environment and under seawater conditions [27,28]. This might be due to higher stresses at the passivated surfaces where the oxide layer may fail and the substrate is exposed to the corrosive medium. The effect of crack propagation may then be promoted by corrosion, leading to an earlier failure in saline solution, as the substrate might not have the time to repassivate to the same extent. However, these findings need to be replicated in a larger number of specimens in order to produce statistically relevant data.

In the modular connection of a TJR, destruction of the oxide layer is known to be the result of not only the stresses induced by high external loads, but also the micromotions between the connected components. In that case, the pH value may drop within the crevice as the oxonium ion content (H_3O^+) increases locally [8]. As a result of this, the oxygen content decreases, and repassivation might be inhibited. This effect is not considered in the present model, as fretting is not addressed and the saline solution is continuously circulated. It is thus assumed that the pH value is not changed locally to a significant extent.

Today, mechanical surface treatments are rarely applied to medical implants. However, they seem to have a significant benefit for the fatigue performance of a Ti-6Al-4V component and should be considered as a constructive option [29]. In this study, an increase in fatigue strength of over 12 % relative to the as-annealed specimens is reported for the final two-step shot peening treatment (cut wire + glass beads). Other surface treatments (e.g., roller burnishing or laser shock peening) might be worth considering as well. They could be applied to critical regions of TJRs, such as stemmed components or modular connections, or to implants in trauma surgery such as intramedullary nails.

Conclusion

The circulation of a NaCl solution around the specimen to be tested does not have a long-term, direct impact on the fatigue strength of Ti-6Al-4V components after shot peening. Based on the data, pure fatigue testing under dry conditions seems to be reasonable, at least at lower loads and a high number of cycles. However, when testing under fretting conditions (e.g. modular systems in TJRs), other effects, such as pH modification within the crevice, might occur, and the corrosive environment might change the outcome relative to the

dry conditions. This needs to be considered in future studies so that a better understanding of the fracture mode of Ti-6Al-4V can be gained.

Acknowledgments

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Evaluation of the Effects of Anodization on the Fatigue Performance of Titanium Alloy

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ABSTRACT: Titanium is a widely accepted metal used to manufacture orthopaedic trauma implants that are typically anodized. Type II anodization potentially improves fatigue strength and provides an increased resistance to galling and fretting wear. Color anodization (Type III) imparts a color to the titanium surface, providing aesthetic appeal and aiding in identification. The purpose of this study was to compare the fatigue performance of Ti-6Al-4V alloy test specimens using both anodization processes. Simulated intramedullary nails and commercially available cortical screws were both evaluated. Cannulated rods with a transverse hole were used to simulate the distal geometry of an intramedullary nail; as-machined rods, color anodized rods, and Type II anodized rods were evaluated. For the cortex screws, one group was color anodized, the other was Type II anodized. Four-point bend fatigue testing based on the recommendations of ASTM F1264-03 was employed. The results showed that when the cross-sectional geometry of the test specimens are the same, as with the cannulated rods, there was a statistically significant difference ($p < 0.05$) between as-machined and Type II anodized rods. There were not statistically significant differences ($p > 0.05$) between either as-machined and color anodized rods, or between color anodized and Type II anodized rods. In the case of the cortical screws which had cross-sectional differences, the color anodized screws with the larger minor diameter showed an improved fatigue performance, at multiple force levels, over the Type II anodized screws, which had a smaller minor diameter. These results indicate

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that surface finish and design both play roles in the fatigue performance of trauma implants.

KEYWORDS: titanium, anodization, biomechanics, trauma, mechanical testing

Introduction

For decades, stainless steel (SS) was the most prominent material of choice for orthopaedic trauma implants. Titanium and titanium alloys have become an increasingly popular option. Titanium is biocompatible, exhibits excellent corrosion resistance, shows fewer resultant magnetic resonance imaging (MRI) and computed tomography (CT) scan artifacts as compared to SS, and has a modulus of elasticity closer to that of bone [1–3].

Titanium and its alloys are known to be notch sensitive [1,3]. With regard to Ti-6Al-4V, notch sensitivity is primarily due to its $\alpha + \beta$ microstructure. Notch sensitivity can be an issue with orthopaedic trauma implants such as intramedullary nails with transvers holes, or locking plates with threaded screw holes. The anodization process has been shown to reduce the effects of design and manufacturing related stress risers in titanium. This has been observed under magnification, whereas “scars from machining and/or deburring are ‘leveled’ into a continuous, smoother, gray-colored surface” [4]. The two most popular forms of anodization for titanium are Type II and color, which is also known as Type III.

Type II anodization is a standardized anodic treatment of titanium performed using ASM 2488 [5]. The surface coating is created using various electrolytes. The device is made anodic, with a cathodic terminal attached to a DC power supply. The Type II anodization process results in a grey color imparted on the specimen. Some of the benefits of Type II anodization include anti-galling effects, resistance to wear, reduced fretting, increased fatigue strength, corrosion resistance, and a smoother surface finish [4]. However, Type II anodization of devices adds additional cost to the manufacturing process and may be prohibitive depending on the intended use or intended implantation region for the trauma device.

Color anodization has not been standardized; numerous proprietary processes exist and are specified by individual companies. The anodization process is possible due to the reaction titanium has when excited by heat or electricity when in an electrolyte. This reaction creates a thin oxide layer at the surface of the device on the order of 500–1000 Å. The oxide layer is seen as color due to an interference phenomenon. Voltage applied to the device through an electrolytic bath controls the color; not all colors of the light spectrum are produced during this process. Various electrolytes are used to create the oxide layer. The specimen is made anodic with a corresponding cathodic terminal attached to a DC power supply. While the oxide layer is harder than the underlying titanium,

it does not show good abrasion resistance due to the thinness of the film. Type III anodization is hypoallergenic, biocompatible, causes no dimensional change in the device, and is useful for identification purposes [1,5].

Many orthopedic trauma companies offer titanium products, including intramedullary (IM) nails, plates, screws, and external fixation devices. While many titanium products are color anodized for identification purposes, the prevalence of Type II anodized implant systems is growing. Multiple commercially available IM nail systems incorporate Type II anodization in their nail designs. The addition of Type II anodization can improve the surface finish of a titanium device by reducing surface imperfections, and may improve the fatigue performance, which is advantageous to an implant subjected to loading throughout its expected life.

The purpose of this study was to evaluate and compare the fatigue performance of Ti-6Al-4V alloy simulated IM nails that had been anodized using Type II or Type III anodization processes in a clinically relevant, standardized test method. In addition to the IM nail study, a secondary study is presented that evaluated bending fatigue performance of Type III and Type II anodized commercially available 5.0 mm screws.

Materials and Methods

Simulated IM Nail Specimen Preparation

Simulated IM Nail test specimens were prepared using Ti-6Al-4V alloy that met the requirements of ASTM F1472-08^{e1} [6]. Cannulated rods with a transverse hole designed to simulate a similar cross-sectional geometry and perpendicular locking screw hole diameter as a 13 mm IM nail were custom manufactured. The specimens were 17.8 cm (7.0 in.) in length, had a 13 mm (0.51 in.) outer diameter (OD), a 8.38 mm (0.33 in.) inner (cannulation) diameter, and a 5.34 mm (0.21 in.) transverse screw hole diameter. The transverse screw hole was added at the midpoint of each test specimen, oriented in line with the loading direction. Flats were machined 25.4 mm (1.0 in.) from both ends of the cannulated rod to prevent rotation during testing.

There were three test groups: (1) as-machined, un-anodized specimens, (2) specimens color anodized (gold) per Smith and Nephew proprietary specifications, and (3) specimens that were Type II anodized (grey) per AMS 2488 [4]. A minimum of 10 specimens were tested per group. Figure 1 shows representative Type III and Type II anodized test specimens.

Mechanical Testing

Four point bend fatigue testing was performed based on the recommendations of ASTM F1264-03 Annex A3 [7]. A universal joint was added to the top



FIG. 1—Representative photos of the color anodized (bottom) and Type II anodized (top) 13 mm OD test coupons.

(loading) fixture and was locked such that the universal joint could only move in the plane of the cannulated rod. Adding a universal joint ensured proper loading during testing. Four-point bend fixtures were then secured to the mechanical testing frame. The top (loading) and bottom (support) fixtures were aligned to ensure proper force application to test specimens. The top fixture had a loading span of 50.8 mm (2.0 in.), and the lower fixture had a support span of 152.4 mm (6.0 in.). Loading and support fixtures incorporated roller bearings and 9.53 mm (0.375 in.) loading rollers. Figure 2 shows a picture of the test setup.

Cannulated rods were tested in a sinusoidal cyclic manner with an axial compressive force of 2002 N (450 lbf) at a frequency of 3 Hz. An R value of 0.1 was used. Test specimens were tested in air at room temperature ($22 \pm 1^\circ\text{C}$). Fatigue testing was performed until specimen failure or completion of 1×10^6 cycles without failure (run-out). Failure was defined as fracture of the test specimen between the span of the loading rollers. Displacement limits were set to ± 0.5 mm (± 0.02 in.) beyond the initial peak/valley displacement values for each test specimen, based on the output from the test frame controller. Force limits were set to ± 89 N (± 20 lbf) beyond the initial peak/valley force values for each test specimen.

Specimens were tested at a nominal stress of 284.6 MPa (41,274.1 psi). This stress was calculated using the formula

$$\sigma = \frac{M(c)(K_t)}{I} \quad (1)$$

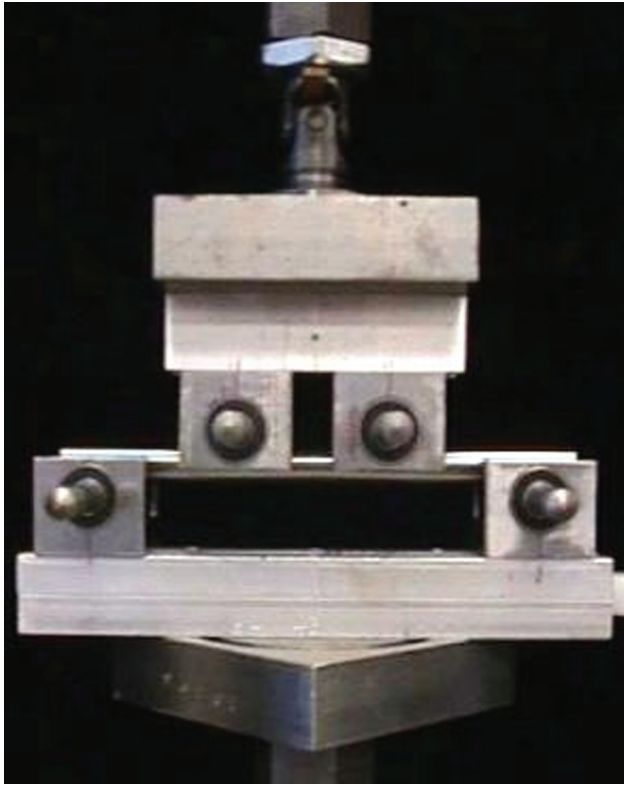


FIG. 2—Four-point bend fatigue test setup for the simulated IM nails with a 13 mm OD.

where:

σ = stress, psi or Pa,

M = bending moment, lbf·in. or N·m,

c = distance from neutral axis to outermost fiber, in. or m,

K_t = stress concentration factor, and

I = second moment of area, in.⁴ or m⁴.

The stress concentration factor, K_t , at the edge of the transverse screw hole was calculated by extrapolating data from Peterson's Stress Concentration Factors [8] for a round shaft in bending with a transverse hole, and was determined to be 3.3.

Differences from ASTM F1264

This study deviated from the recommendations of ASTM F1264-03 [7] in four ways. First, a transverse screw hole was added to the midpoint of the

cannulated rods. The intent of the standard is to evaluate the shaft portion of an IM nail. A transverse hole was added to better simulate the proximal or distal portion of an IM nail, where fracture is more likely to occur [3,9,10]. Second, anti-rotation flats were added at both ends of the cannulated rod to maintain the desired hole alignment. The flats differed from the recommended guide shoe method, as outlined in section A3.8.5 of the standard. The use of the flats did not appear to have an effect on the bending fatigue performance of the test coupons since none of the samples fractured outside of the loading span. Third, a universal joint was added to the upper loading fixture, which was done to alleviate potential misalignment issues present in the setup. Finally, the force and support spans chosen for testing were different than those recommended in section A3.8.3.

Data Collection and Statistical Analyses

Number of cycles to failure and failure location were recorded for each test specimen. Cycle count data was given in terms of average $\pm$ standard deviation for each group. A log transformation was applied to the cycle count of each specimen in order to normalize the data. An ANOVA single-factor statistical method was used to analyze the fatigue data; in order to make multiple comparisons, the Tukey method was used post hoc ($\alpha = 0.05$).

Commercially Available Screws

Commercially available color anodized Ti-6Al-4V 5.0mm cortical screws were compared to commercially available Type II anodized 5.0mm locking screws. The Type III, color anodized screws were anodized gold; the Type II anodized screws displayed the typical grey color resultant from the Type II process. It should be noted that there are differences between the minor diameters and thread forms between each screw. Color anodized screws had a minor diameter of 4.3 mm (0.168 in.); Type II anodized screws had a minor diameter of 3.98 mm (0.157 in.). Both screws are used in IM nail fixation, and have similar indications for use.

The screws were tested per ASTM F1264-03 Annex A4 [7]. A universal joint was added to the top (loading) fixture, and was locked such that the universal joint could only move in the plane of the screw being tested. The additions of a universal joint helped to ensure proper loading during each test. The four-point bend fixture was then secured to the mechanical testing frame. The top (loading) and bottom (support) fixtures were aligned to ensure proper load application to the test specimen. The top fixture had a loading span of 20.3 mm (0.8 in.), and the lower fixture had a support span of 60.7 mm (2.4 in.). The

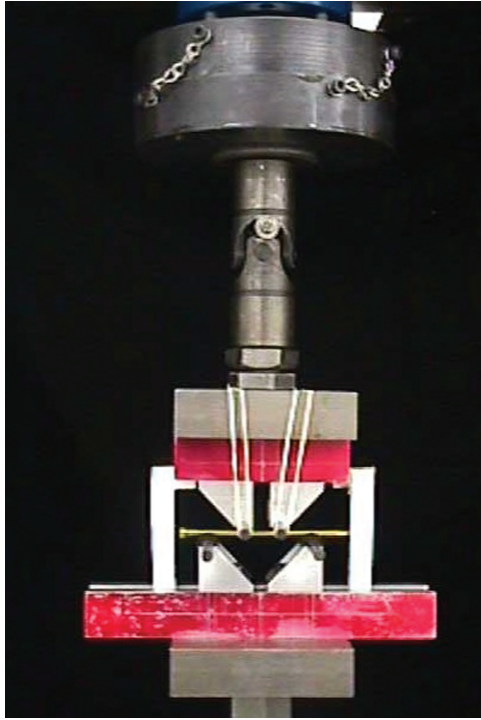


FIG. 3—*Four-point bend fatigue test setup for the commercially available screws with a 5.0 mm OD.*

loading and support fixtures incorporated 6.4 mm (0.25 in.) loading rollers. Figure 3 shows a picture of the test setup.

Screws were tested in a sinusoidal cyclic manner with an axial compressive load at a frequency of 3 Hz. Four force levels were used to evaluate the fatigue performance: 489 N (110 lbf), 534 N (120 lbf), 623 N (140 lbf), and 667 N (150 lbf). An R value of 0.1 was used. Specimens were tested in air at room temperature ($22 \pm 1^\circ\text{C}$). Fatigue testing was performed until screw failure or completion of 1×10^6 cycles without failure (run-out). Failure was defined as fracture of the screw between the span of the loading rollers. Displacement limits were set to ± 0.5 mm (± 0.02 in.) beyond the initial peak/valley displacement values for each test specimen based on the output from the test frame controller. Force limits were set to ± 89 N (± 20 lbf) beyond the initial peak/valley force values for each test specimen. A minimum of three specimens were tested in each group.

Number of cycles to failure and failure location were recorded for each test specimen. Cycle count data was given in terms of average $\pm$ standard deviation for each group.

Results

Simulated IM Nails

All specimens fractured on the tensile side at the transverse hole prior to run-out (Fig. 4). Figure 5 shows a graphical representation of the fatigue performance of the three IM Nail test groups. As-machined test specimens had an average fatigue life of $38\,246 \pm 16\,127$ cycles (4.6 ± 0.2 log cycles). Color anodized test specimens had an average fatigue life of $67\,195 \pm 73\,284$ cycles (4.7 ± 0.3 log cycles). The average fatigue life for Type II anodized test specimens was $123\,656 \pm 113\,407$ cycles (5.0 ± 0.3 log cycles).

When compared to as-machined specimens, Type II anodized specimens had a 223 % higher average cycle count; there was a statistically significant difference between the log of the cycle counts ($p < 0.05$). Type II anodized specimens had an 84 % higher average cycle count as compared to color anodized specimens; there was not a statistically significant difference between the log of the cycle counts ($p > 0.05$). Color anodized specimens had a higher average cycle count as compared to as-machined specimens; this result, however, was not statistically significant ($p > 0.05$).

Commercially Available Screws

The average bending fatigue performances for color anodized screws at the four force levels were $1\,000\,000 \pm 0$ cycles (489 N), $1\,000\,000 \pm 0$ cycles (534 N), $365\,343 \pm 438\,787$ cycles (623 N), and $132\,652 \pm 163\,340$ cycles (667 N), respectively. Of the Type III anodized screws that fractured, each did

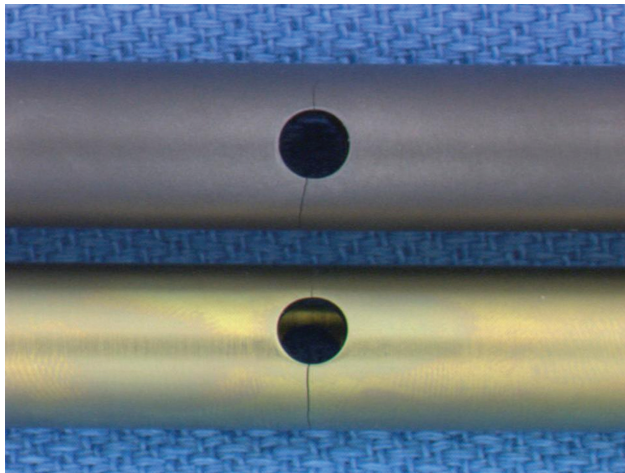


FIG. 4—Fractures through the transverse holes in the Type II anodized 13 mm OD test coupon (top) and the color anodized 13 mm OD test coupon (bottom).

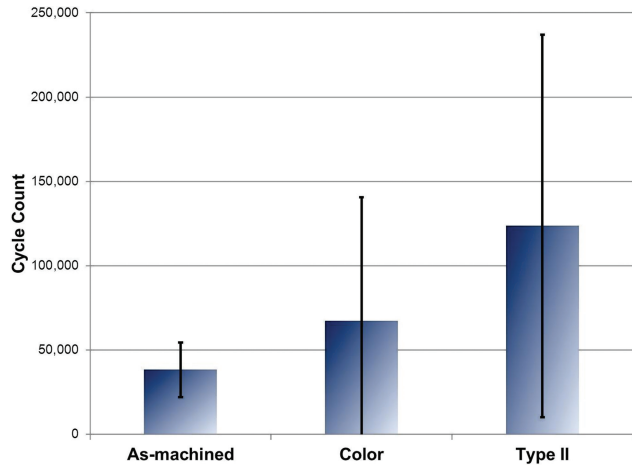


FIG. 5—Graphical representation of the fatigue performance for the three groups of Ti-6Al-4V test specimens.

so through the minor diameter of the screw between the threads. The average fatigue performances of the Type II anodized screws at the four load levels were $375\,891 \pm 411\,200$ cycles (489 N), $641\,002 \pm 480\,278$ cycles (534 N), $29\,922 \pm 6622$ cycles (623 N), and $42\,091 \pm 27\,031$ cycles (667 N), respectively. Each of the Type II anodized screws fractured through the minor diameter of the screw, between the threads. Figure 6 shows a graphical representation of the bending fatigue results for the commercially available screws. For each of

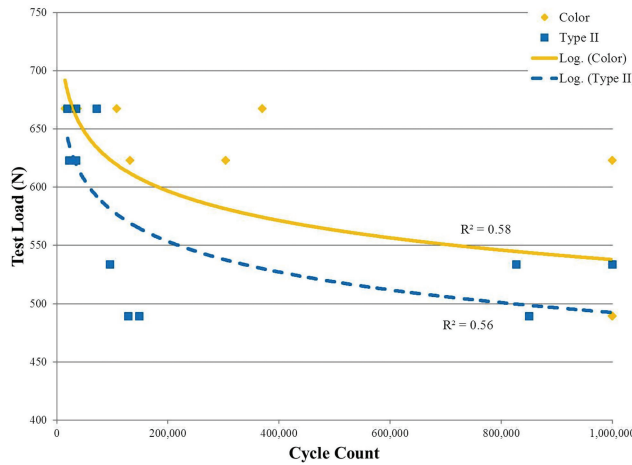


FIG. 6—Graphical representation of the bending fatigue results for the color anodized screws as compared to the Type II anodized screws.

the load levels, color anodized screws had a higher average fatigue life as compared to Type II anodized screws.

Discussion

Simulated IM Nails

Type II anodization provided increased fatigue performance as compared to color anodized and as-machined IM Nails. Color anodized specimens had similar bending fatigue performance as compared to as-machined specimens.

It should be noted that these conclusions were drawn under the assumption that the test conditions were all the same for this study; the material and cross-sectional geometry of the test specimens were the same across each group. When testing conditions are varied, such as differing cross-sectional geometries, the improvement to fatigue performance by Type II anodization alone may not be the deciding factor in the testing results.

A review of the literature showed work performed by another company that supported the results seen in the simulated IM nail testing. TIODIZE Co., Inc. performed rotating beam testing on notched specimens at 30 Hz and a stress value of 414 MPa (60 ksi) [11]. While multiple anodization and surface conditions were tested, two groups of Ti-6Al-6V-2Sn test specimens were of interest. As-machined specimens ($n=6$) and “TIODIZED” Type II anodized specimens ($n=3$) were evaluated. The “TIODIZED” specimens had a higher average fatigue life as compared to as-machined test specimens ($p=0.003$).

The results provided by the TIODIZE Co. show that Type II anodization does appear to provide improved fatigue performance as compared to as-machined components. However, test conditions, material, and cross-sectional geometry were the same for each group. Changes in test setup or differences in specimen design may influence results more so than the addition of Type II anodization alone.

Commercially Available Screws

Evaluation of the 5.0 mm color anodized screw as compared to the 5.0 mm Type II anodized screw showed that anodization alone was not the deciding factor in determining fatigue performance. Differences in screw design were likely contributing factors of the color anodized screw having a higher average fatigue performance; the color anodized screws had a larger minor diameter. Hsu et al. demonstrated that titanium screws showed an increasing trend in fatigue performance as the minor diameter was increased and the major diameter remained the same [3]. The graphical representation of the screw fatigue testing results showed, in general, that the larger diameter Type III anodized screws were able to withstand greater loads for longer cycles as compared to

the smaller diameter Type II anodized screws. The difference in minor diameter between the two screws represented a 22.5 % decrease in bending stress for the Type III anodized screw as compared to the Type II anodized screw.

In both the cannulated rod test and the cortical screw test, large standard deviations were noted. One explanation for this could be the inherent nature of titanium itself. As stated previously, Ti-6Al-4V is notch sensitive. Titanium is also prone to scatter (larger standard deviation) during fatigue testing; therefore the large standard deviation seen in this test is not uncommon.

In fatigue crack propagation measurements of titanium, there can be large amounts of scatter in the data. Part of the scatter may be due to test reproducibility, as well as material variability; this is why more than one sample is tested. This material variability may be due to small material or processing inhomogeneities. Variations in chemistry, microstructure, and texture effects may also influence data scatter [12].

In the case of the simulated IM nails, a notch was intentionally introduced into the test sample, which could have contributed to small differences at the transition and which could influence the fatigue performance. With regard to the screw testing, it is possible the loads used for the fatigue testing of the screws were near the fatigue limit of the material, that would result in a mixture of run-outs and failures of the screws.

Osseointegration

One issue commonly observed with the use of titanium as an orthopaedic implant material is osseointegration. As summarized by Pohler et al. [2] and Bose et al. [13], titanium is a bioinert material and as a result does not become encapsulated by fibrous tissue after implantation. The presence of a naturally occurring oxide layer on the surface of titanium and titanium alloys provides “a bone-bonding ability, called osseointegration, a process whereby bone is apposed to the implant without an intervening fibrous capsule” [13]. Close adhesion to or bone covering over titanium implants can cause difficulty upon implant removal. Reports of bone ongrowth onto screw heads, or covering the head of IM nails are common [2,14–17].

Type II anodization is claimed to prevent or reduce bone integration or tissue adhesion [18]. The electrolyte typically used with Type II anodization is sulfuric acid, which has been shown to increase bone integration of Titanium implants [18]. When using hyaluronic acid as an electrolyte, a decrease in osteoblast integration was noted; this may prevent the undesired integration of bone onto the titanium surface [18,19].

Conclusions

Device longevity is important in the race to callus formation and eventual healing, when considering orthopaedic trauma devices. As shown in this study and

corroborated in the literature, Type II anodization provides increased fatigue performance as compared to as-machined and color anodized devices when comparing devices of similar cross-section. When the cross-section of the devices is not the same, the effects of anodization alone may not be the deciding factor in fatigue performance. Other variables such as material choice, implant geometry, intended use, implant design, and cost should also be considered when surface modifications are desired.

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Evaluation of Fatigue Performance and Residual Stress of Surface Modification Techniques for Ti-6Al-4V Medical Device Applications

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ABSTRACT: A variety of surface modification techniques exist that offer the potential to enhance mechanical fatigue performance of medical device components, yet these technologies are not widely applied across the industry. As implantable device designs are continually miniaturized, components are often subjected to higher stresses, requiring improvements in material properties, such as fatigue strength. This paper describes an evaluation of three different surface modification techniques for their potential to enhance the fatigue resistance of Ti-6Al-4V anterior cervical plates: shot peening, laser shock peening, and electropolishing. The primary evaluation criterion was high-cycle bending fatigue testing following ASTM F1717-09, 2009, "Standard Test Methods for Spinal Implant Constructs in a Vertebrectomy Model," *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA. The relative roles that compressive residual stress and surface roughness play in determining fatigue performance are examined by the measurement of residual stress depth profiles (by the hole-drilling method) and surface roughness. Whereas similar studies have been reported in the literature in the context of engine components, the smaller size of implantable medical device components makes this work unique. There is concern that

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thin cross sections may give rise to significant sub-surface compensatory tensile stresses when techniques, such as shot peening or laser peening, are used to impart near-surface compressive residual stresses. Results from this study are somewhat counterintuitive: the deeper compressive residual stress profiles of laser shock peening are not necessarily beneficial when they come at the expense of less compressive peak residual stress. This reflects the importance of surface and very-near-surface residual stresses under bending conditions for thin plates. Although increasing shot peen intensity yielded relatively deeper *and* more compressive residual stress profiles, these higher-intensity peening conditions resulted in lower fatigue performance compared to lower-intensity peened samples (which, in turn, were not conclusively better than the as-fabricated baseline). Electropolishing is shown to be beneficial alone, as well as when combined with prior shot peening.

KEYWORDS: cervical plate, residual stress, fatigue, surface modification, peening, roughness, hole drilling

Introduction

Many common implantable medical devices are subjected to high cycle fatigue conditions that arise from their *in vivo* environment. For example, cardiac devices, such as implantable heart valves or arterial stents, must withstand hundreds of millions of pulsatile loads inherent to the circulatory system. The motivation for this work originates from implantable devices used in spinal discectomy and fusion procedures. These procedures are performed to relieve pain by removing deteriorated intervertebral discs (discectomy). Restoration of the structural integrity of the spine is achieved by packing bone-graft material in the resulting gap. During a postoperative period, normal biological healing processes join the adjacent vertebrae and graft material into a single rigid bony mass (fusion).

Anterior Cervical Plates

Spinal fusion surgery outcomes are improved by limiting relative motion between the fusion segments through internal fixation or external bracing. Cervical (neck) discectomy and fusion procedures frequently use a permanently implanted internal fixation device called an anterior cervical plate (ACP) [1]. Anchored to vertebrae adjacent to the discectomy site via bone screws, the ACP minimizes motion across the fusion segment by transferring load from the superior to inferior vertebra, helps to contain the bone graft, and maintains the correct local alignment of the cervical spine during fusion (Fig. 1). Once fusion is complete (typically between several weeks and several months postoperative), the physiologic loads carried by the ACP are significantly lower because the fused segments can now participate in transferring load across the spine. Accordingly, the primary cyclic fatigue loads that spinal fixation devices, such as ACPs, must resist occur over a number of weeks and months, rather than over a number of years for an implantable cardiac device. Nevertheless, even

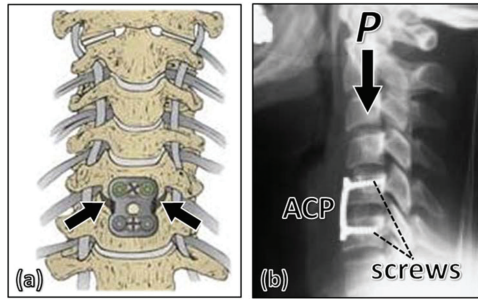


FIG. 1—(a) An anterior cervical plate reduces relative motion between the superior and inferior vertebrae adjacent to the gap (arrows) left behind after cervical disc removal (discectomy). This improves the likelihood for successful postoperative fusion of the vertebrae with the bone-graft material packed into the intervertebral space after discectomy. (b) The primary physiological load P is along the longitudinal axis of the vertebral bodies (arrow), which is eccentric to the ACP shown in profile view in this lateral plane x ray of the cervical spine.

the relatively short period of cyclic loading for spinal fusion fixation devices necessitates deliberate design against high cycle fatigue (HCF) because of the frequency of load cycles from normal daily activities, such as walking. ASTM F1717-09, Standard Test Methods for Spinal Implant Constructs in a Vertebrectomy Model, conservatively sets the number of cycles corresponding to the maximum run out load for fatigue testing to be 5 000 000 cycles [2].

A common, albeit typically minor, complication of anterior cervical discectomy and fusion (ACDF) with an ACP is postoperative dysphagia (painful or difficult swallowing). Whereas the factors causing dysphagia in this context are not well understood, a potential factor relates to ACP design [3,4]. Because the ACP protrudes from the anterior side of the cervical vertebrae, axial rotations of the head cause the esophagus to wipe back and forth across the ACP. ACP manufacturers have traditionally sought to minimize the thickness (profile) of their plates to minimize potential irritation of the esophagus and dysphagia. Limited results reported by Lee et al. support this approach [5].

A better understanding of the implications of reduced profile thickness can be obtained by considering a simplified description of the expected physiologic loading of the simplest representation of an ACP, a flat plate of thickness (profile) h , width (transverse to the spine) b , and length (parallel to the spine) l .

A simplified physiologic loading can be represented by subjecting a beam (having the geometry of the aforementioned flat plate) to pure bending (Fig. 2). Internal plate stresses (for example, stress in the plane of bending, σ_x) associated with the bending moment M_z can be calculated as

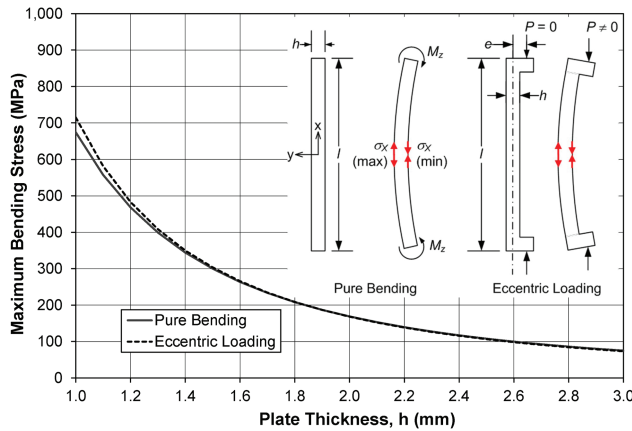


FIG. 2—Comparison of calculated max bending stress, σ_x , under pure bending (Eq 3) and eccentric loading conditions (Eqs 4 and 5) for a plate of dimensions $b=10\text{ mm}$ and $l=35\text{ mm}$. For the eccentric loading calculation, the load $P=35\text{ N}$ (8 lb), the eccentricity is 30 mm (prescribed by ASTM F1717 bending fatigue test method), and E is taken to be 115 GPa (typical for Ti-6Al-4V alloy). For the pure bending calculation a bending moment M_z of $35\text{ N} \times 30\text{ mm} = 1.05\text{ N}\cdot\text{m}$ is assumed. The max bending stress is located on the surface of the plate ($y = h/2$).

$$\sigma_x = \frac{-M_z \times y}{I_z} \quad (1)$$

where y is the distance from the neutral axis of the plate and I_z is the area moment of inertia about the z axis of the plate cross section. The area moment of inertia can be expressed as

$$I_z = \frac{b \times h^3}{12} \quad (2)$$

The bending stress has its maximum tensile value at the anterior surface ($y = h/2$). Combining Eqs 1 and 2 shows that maximum surface stresses on a plate for a given physiological load have an inverse-square dependence on plate thickness

$$\sigma_x = \frac{-6 \times M_z}{b \times h^2} \quad (3)$$

A more rigorous analysis of the loading condition treats the plate as an eccentrically loaded beam-column with pinned fixation and results in the following relationships [6]:

$$\sigma_x = \frac{P}{A} + \frac{-M_z^{\max} \times y}{I_z} \quad (4)$$

$$M_z^{\max} = P \times e \times \sec \left(\left(\frac{P}{E \times I_z} \right)^{1/2} \times \frac{l}{2} \right) \quad (5)$$

where P is the compressive force (a negative number), A is the cross-sectional load-bearing area, $M_z^{\max}$ is the maximum moment, E is modulus of elasticity, and e is the distance between the load axis and plate (load eccentricity). Figure 2 shows that for values of the aforementioned variables relevant to this work, the inverse-square relationship (Eq 3) is a fair approximation of the more rigorous but less intuitive beam-column analysis (Eqs 4 and 5). Furthermore, Fig. 2 indicates that decreasing the profile of this simple plate design from 1.6 mm (one of the thinner commercially available ACPs) to, say, 1.3 mm increases the maximum tensile bending stress by about 50 %. The increase in tensile bending stress can be expected to reduce the fatigue performance of plate. For example, because the number of cycles to failure, N_f , is usually an inverse power law function of the elastic alternating stress amplitude during HCF, the fatigue test run out load will be significantly lower for the thinner plate profile.

Profile Reduction Entitlement by Surface Modification

Medical device designers seek improvements to the fatigue performance of biomedical materials that would allow further device miniaturization, such as profile reduction in ACPs. Surface modification technologies are of interest because they offer the potential to improve fatigue performance of legacy alloys whose suitability for long-term in vivo use has already been established at great time and expense. The surface modification technologies considered in this work seek to improve fatigue life by either imparting a compressive residual stress in the near-surface layers or reducing surface roughness.

Shot peening and laser shock peening use the mechanical impact of steel shot or laser-induced shock waves, respectively, to produce a thin layer of plastic deformation in the surface and near-surface region of a component. The constraint provided by the deeper, un-deformed material sets the surface and near-surface layers in a state of compressive residual stress. This compressive residual stress, superimposed on an applied stress, lowers the net mean stress during fatigue. Per the widely used Goodman relationship, the lower mean stress (σ_m) increases the alternating stress (σ_a) leading to failure for a given number of cycles in a material characterized by a fatigue limit (σ_f) and tensile strength (σ_{TS})

$$\sigma_a = \sigma_f \times \left(1 - \frac{\sigma_m}{\sigma_{TS}} \right) \quad (6)$$

Electropolishing is an electrochemical method by which the roughness of non-planar metallic surfaces can be reduced. Surface roughness can be detrimental to HCF life because the topological valleys can function as stress concentrators and initiate flaws from which fatigue cracks can grow. The fatigue performance benefit from either surface roughness reduction or compressive residual stresses is expected to be greatest under HCF conditions. The higher stresses characteristic of low cycle fatigue (LCF) minimize the relative importance of fatigue crack initiation to overall lifetime (N_f) and tend to plastically relax residual stresses imparted by surface modification.

Materials and Methods

Test Specimen

A test specimen representing a simplified ACP (Fig. 3) was designed for the purposes of evaluating surface modification technologies for ACPs. It is machined from rectangular drawn Ti-6Al-4V ELI (ASTM F136-08e1 [7]) bar stock with an annealed equiaxed alpha-beta microstructure. Typical ultimate tensile strength and 0.2 % yield strength for this material is 990 and 917 MPa, respectively. All machining is performed using the same high-volume manufacturing processes used to produce commercial ACP product. The resulting surface contains a surface texture and residual stress profile that determines the baseline mechanical fatigue performance of the test specimen. Because commercial ACPs frequently contain features, such as fixation screw holes near the center of the plate, that can behave as stress concentrators, a centrally located hole with spherical countersink was included in the test specimen design. This provides a net section stress concentration of bending stresses tangential to this hole, estimated by elasticity, to be approximately $K_t = 2.4$ for the test specimen geometry [6].

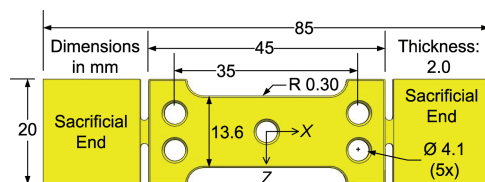


FIG. 3—Ti-6Al-4V test specimen geometry designed for surface modification studies. The sacrificial ends provide material for gripping by fixtures during subsequent surface modification. These unmodified ends were removed prior to any testing where their presence would complicate measurement (fatigue testing, for example).

Surface Modification

All shot peening (SP) was performed by Medtronic Spinal and Biologics Warsaw Operations facility using a highly automated and process validated direct pressure shot peening system [8]. Saturation curves were developed to establish proprietary process recipes for low, medium, and high Almen intensities. Both sides of the test specimens were peened by rotating the specimens about their longitudinal axis before a single robotic nozzle.

Laser shock peening (LSP) was applied by an external provider using a nominal intensity estimated by the vendor to be appropriate for this combination of alloy type and plate thickness. The general approach in this work was to evaluate surface modification processes applied to the entire plate because this is generally the most straightforward approach for these processes. An exception was allowed in the case of LSP where the peening was confined to an annular pattern around the central fixation hole of the test specimen (on both the anterior and posterior sides of the plate). Because LSP is applied on a point by point basis as the laser beam is rastered over a component surface, the cost of LSP is proportional to the area treated. Furthermore, a unique feature of LSP is the ease with which it can be selectively applied; thus, using it for full-surface coverage when this coverage is unnecessary undermines part of the reasoning to consider LSP in the first place. Test specimens intended exclusively for residual stress measurement received LSP treatment over a square area between the central fixation hole and one of the ends so that measurement location would be consistent with that used for SP specimens.

An electrolyte for electropolishing (EP) was selected from ASTM E1558-09, Standard Guide for Electrolytic Polishing of Metallographic Specimens [9], specifically a 15.7 ± 1 volume ratio mixture of perchloric acid to glacial acetic acid (class II-1 electrolyte per ASTM E1558-09). The use of perchloric acid required special safety precautions including a dedicated wash down fume hood. Additional safety precautions included careful temperature monitoring to keep the electrolyte temperature below 29°C and always discarding (never storing) solution after use.

EP was carried out by the author by submersing a test specimen (the positively biased anode) into a 1.1-l, 10-cm-diameter stainless steel beaker (the cathode) containing a 800 ml acetic acid and 51 ml perchloric acid solution stirred by a PTFE-coated magnetic stir bar. The beaker sat on top of a submersible stir plate inside a recirculating, chilled, thermostatically controlled water bath. A high-power switching DC power supply supplied 55 VDC power to the anode and cathode. Typical material removal rates were approximately 5–7 $\mu\text{m}/\text{min}$.

Certain regions on the posterior side of the test specimens were protected from EP by prior application of a lacquer mask (MICCROStop stop-off lacquer, Tolber Chemical Division, Hope, AR) resistant to this EP process. Limiting EP to the anterior face of the test specimens that would be subjected to

tensile stresses during bending fatigue testing minimized heating and consumption of the electrolyte, as well as current draw from the power supply.

A stress relief anneal was performed on some test specimens to observe the effect of removing residual stress from the samples. A high-temperature diffusion-pumped high-vacuum furnace was used to treat the samples for 1 h at 600°C, a standard stress relief heat treatment for this alloy [10]. The heating rate was 15°C/min and the sample was furnace cooled in vacuum to 90°C, at which point argon gas was admitted to the chamber to accelerate further cooling.

Residual Stress and Surface Roughness Measurement

Measurement of residual stress as function of depth below the test specimen surface was made by American Stress Technologies, Inc. (Philadelphia, PA) using their Prism hole-drilling residual stress measurement system. This system is unique for its use of an optical strain measurement technology (electronic speckle pattern interferometry) in place of strain gauges used in traditional hole-drilling measurement systems. Residual stress measurement was performed incrementally at ten depths below the surface starting at a minimum nominal depth of 0.030 mm and stopping at a maximum nominal depth of 0.35 mm by pecking a single blind 0.794-mm-diameter hole with a two-fluted TiN-coated drill. Stresses were calculated using the integral method [11] for an assumed non-uniform stress distribution. The hole-drilling with optical strain measurement approach was chosen over another frequently used method, $\sin^2(\psi)$ x-ray diffraction (XRD) [12], because of the significantly greater time and labor to do the latter when depth profiles are desired. The $\sin^2(\psi)$ XRD requires a time-consuming iterative process of x-ray measurements followed by electropolishing layer removal for each depth measurement. One advantage of the $\sin^2(\psi)$ XRD method, however, is its ability to measure residual stress at the surface, which is not possible with hole-drilling.

Surface roughness was measured with a white-light interferometry optical profilometer. This instrument has height resolution on the order of nanometers. Lateral resolution, determined by the image sensor resolution and the objective lens magnification, changed depending on the sample being imaged but was typically between 0.4 and 2.2 μm . The size of the area imaged was typically 2 mm $\times$ 2 mm. A second-order plane fit was subtracted from the raw data to remove the underlying form, if any, of the specimen surface. High-frequency noise in the data shorter than 10 μm in wavelength was filtered by a low-pass fast Fourier transform algorithm. The reported values for R_a roughness are based on areal data.

Mechanical Fatigue Testing

Compressive fatigue testing of all specimens generally followed ASTM F1717-09. Although the loads applied by the force actuator are compressive

(load ratio $R = 10$) the eccentricity of the load axis to the plane of the plate results in cyclic tensile stresses on the anterior surface of the plate where the load ratio is approximately $R = 0.1$. One notable deviation from ASTM F1717-09 was that the cyclic load frequency used in this work was increased from 5 to 10 Hz. Four-mm-diameter $\times$ 17-mm-long fixed-angle ACP bone screws were used to attach the test specimens to the UHMWPE test blocks. Specimen failure was defined to occur as soon as either of the two following conditions occurred: (1) displacement peak/valley change detected with 0.25-cm sensitivity and tolerance relative to the steady-state peak/valley values recorded after the initial 200 cycles, or (2) force peak/valley change detected with 5-lbf sensitivity and tolerance relative to the set point maximum and minimum compressive loads. A servo-hydraulic dynamic axial test system and 1-kN dynamic load cell were used to conduct the fatigue tests.

Results and Discussion

As-Fabricated Baseline

A plot of maximum compressive load versus N_f is shown in Fig. 4 for the baseline condition, an as-fabricated test specimen. The run out load was established to be -41 lbf (-182 N). Subjecting the baseline specimens to a stress relief

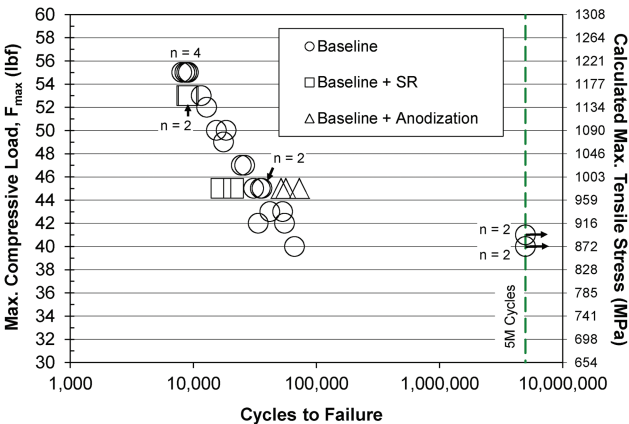


FIG. 4—Baseline compressive fatigue performance for the as-fabricated test specimen, after stress relief (SR), and with anodization. Where partially overlapping data symbols occur, the number of overlapping symbols, n , is labeled for clarity. The second y axis indicates the max tensile bending stress at the test specimen surface for a given max compressive load calculated per Eqs 4 and 5. The dashed vertical line locates the number of cycles for run out (5×10^6).

thermal anneal resulted in a reduction in cycles to failure (Fig. 4). This effect implies the presence of compressive residual stresses, even in the as-fabricated (baseline) test specimens, in the surface and near-surface layer where bending stresses are high. The presence of a “native” compressive residual stress profile is typical of components subjected to machining or surfacing operations. Impact-based surface modification techniques must impart compressive residual stresses of greater magnitude and/or to greater depths relative to the native compressive residual stress profile to improve fatigue performance. This underscores the importance of fabricating baseline specimens with relevant manufacturing processes.

The specimens failed by a loss of load carrying capability from the presence of cracks on the front (anterior) face of the test specimen. These cracks always originated at or near the intersection of the central fixation hole with the front face of the specimen and the z axis and grew roughly parallel to the z axis (lateral mid-line of the plate).

Commercial titanium alloy ACPs typically have a Type II anodization coating (SAE, AMS2488 [13]) as a cosmetic feature. Although the interaction of this coating with surface modification was outside the scope of this work, a limited assessment of its impact (slightly positive) on fatigue performance is shown in Fig. 4. Others have associated anodization with a reduction in Ti-6Al-4 V fatigue performance [14].

Laser Shock Peening (LSP)

Figure 5 summarizes fatigue performance of test specimens subjected to LSP surface modification. The fatigue performance of the eleven low intensity LSP specimens was significantly worse than baseline across the range of maximum compressive loads, most noticeably under HCF conditions (less compressive loading) where the low intensity LSP run out load was < -26 lbf (-116 N) versus -41 lbf (-182 N) baseline run out load.

Residual stress measurements on the baseline and LSP specimens (Fig. 6) help explain the failure of the LSP surface modification to improve fatigue performance in this study. As discussed earlier, compressive residual stresses are beneficial to fatigue performance. The near-surface compressive residual stresses in the LSP specimens are *lower* in magnitude than the baseline compressive residual stresses until approximately 0.05 mm below the surface. Beyond the 50- μ m depth, the LSP residual stresses are more compressive (favorable) compared the baseline specimen residual stresses. However, tensile bending stresses are greatest at the surface of the plate and inversely proportional to depth below the surface (recall Eq 1), thus the residual stress profile imparted by LSP may not be suitable for this situation. Additionally, the LSP treatment generated increased the R_a roughness value by a factor of

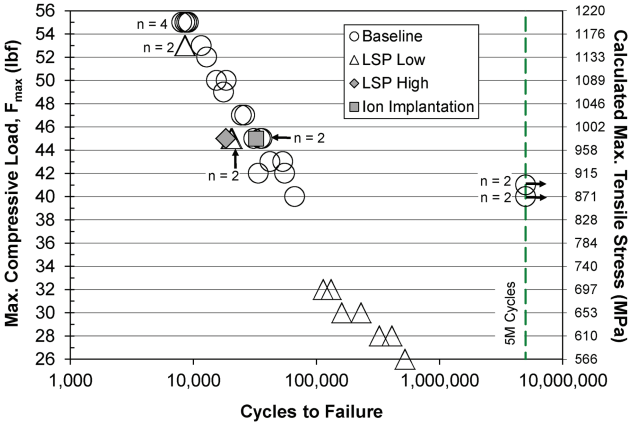


FIG. 5—Neither laser shock peening (LSP) surface modification nor ion implantation improved on the baseline LCF performance. LSP resulted in lower run-out load (<26 lbf) compared to the 41 lbf run-out load for the as-fabricated test specimens.

approximately 10 over the baseline specimen and this can lead to reduced fatigue performance as well.

As an example of when residual stress evaluation can serve as a proxy for actual mechanical testing in determining the potential benefit of a specific surface modification process, fatigue performance and residual stress for an ion

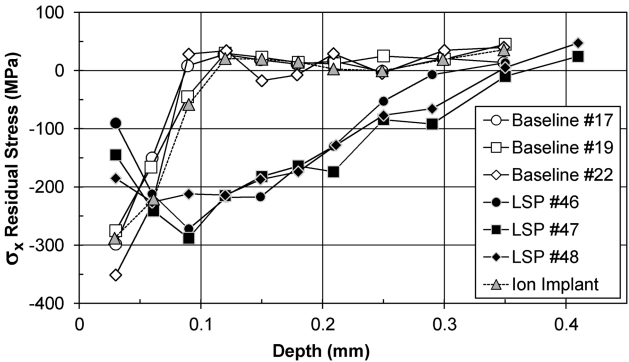


FIG. 6—Residual stress in the x axis (the direction of the peak bending stresses during fatigue testing per Fig. 2). Note that whereas the LSP compressive residual stress profile extends deeper below the plate surface, it is less compressive than the baseline profile within the first approximately $50 \mu\text{m}$ below the surface. Measured in-plane shear stresses (τ_{xz} , not shown) were near zero at all depths for all measurements reported in this work so the σ_{xx} stress is approximately equivalent to the uniform in-plane biaxial stress.

implanted test specimen is shown in Figs. 5 and 6, respectively. Ion implantation surface modification seeks to impart compressive residual stress in the near-surface region of a component without changing the surface roughness [15]. This experiment showed no significant change in the residual stress profile after ion implantation. Therefore, it is not surprising that preliminary (single specimen) mechanical testing (Fig. 5) does not indicate a substantial change in fatigue performance.

Shot Peening (SP)

Fatigue performance of the SP test specimens compared to the baseline results is summarized in Fig. 7. The highest intensity SP treatment significantly underperformed the baseline fatigue performance with a run out load of -28 lbf (-125 N), 13 lbf (58 N) lower than the baseline run out load. The lowest intensity SP treatment fatigue performance was similar to the baseline results but had a slightly inferior run out load of -39 lbf (-173 N), 2 lbf (9 N) lower than the baseline run out load. The failure of the high- and low-intensity SP treatments to increase fatigue performance motivated testing of intermediate intensity SP specimens. The data for this treatment condition is limited but suggests

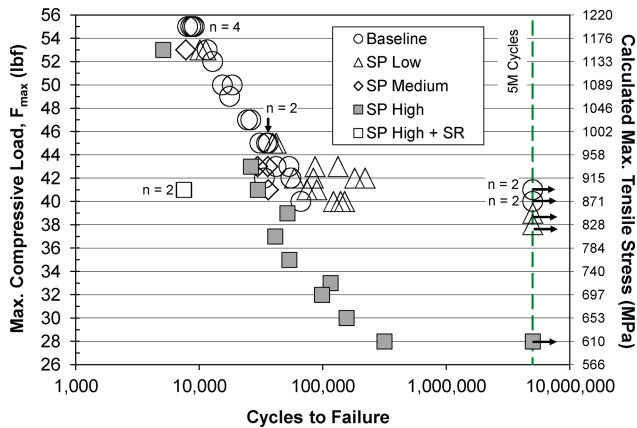


FIG. 7—The effect of shot peening (SP) surface modification on the as-fabricated test specimen fatigue performance is dependent on the relative SP intensity (low, medium, or high). The run-out load for the low-intensity SP was only slightly lower than the baseline value (39 lbf versus 41 lbf, respectively). The high-intensity SP fatigue performance was considerably lower than baseline (28 lbf versus 41 lbf run-out load, respectively). Medium-intensity SP specimens were not tested to run out but their performance appears to fall between the low- and high-intensity SP specimens. Performing a stress relief heat treatment on two high-intensity SP specimens resulted in further reduction in fatigue performance at the baseline run out load value.

the medium intensity treatment provides fatigue performance intermediate to the lower- and higher-intensity treatments. In summary, fatigue performance improved with decreasing SP intensity, but never exceeded the baseline performance.

None of the SP treatments, or the LSP treatments, changed the location of fatigue crack initiation on the specimen relative to that described for the baseline condition. Exemplary macrographs of the failed specimens are shown in Fig. 8.

Residual stress measurements performed on SP specimens (Fig. 9) were consistent with the expectation that increasing SP intensity leads to more compressive and deeper residual stresses. Even the lowest intensity SP treatment yielded a more compressive residual stress profile within the first 100 μm below the surface. Nevertheless, these compressive residual stress profiles failed to improve fatigue performance relative to the baseline condition.

One concern with surface modifications that rely on imparting near-surface compressive residual stresses is that these compressive stresses must be balanced by compensatory tensile stresses deeper within the component to satisfy static equilibrium. These deeper compensatory tensile stresses have the potential to facilitate sub-surface fatigue crack initiation. The greater the near-surface compressive residual stresses, the greater the compensatory tensile stresses. For example, in Fig. 9 the high-intensity SP specimens can have 200 MPa tensile residual stresses within 0.3 mm of the surface. Nevertheless, relieving the residual stresses from high-intensity SP specimens through stress relief heat treatment resulted in far *worse* performance (Fig. 7). This strongly suggests that the fatigue performance of the high-intensity SP samples was not compromised by their residual stress profile (specifically, by the higher compensatory tensile residual stresses deeper below the surface). In fact, the compressive region of this residual stress profile was mitigating some of the deleterious effects of the rougher surface that is generally the result of higher intensity SP. For example the roughness of the SP specimens, normalized by the baseline roughness, was measured as $R_a = 3.5$, 13.0, and 24.5 for low-, medium-, and high-intensity SP specimens.

A sequential combination of medium intensity SP and electropolishing (EP) provided an improvement in fatigue performance relative to the as-fabricated specimens (Fig. 10). As shown in Fig. 11, EP reduced the increased roughness associated with the medium-intensity SP by about 38 %, allowing the enhanced compressive residual stresses to extend fatigue life. The run out load for the medium-intensity SP + EP specimens is better than -47 lbf (-209 N), an increase of 15 % over the baseline run out load of -41 lbf (-182 N). It is possible that higher-intensity SP (greater and/or deeper compressive residual stress) combined with more aggressive EP (greater reduction in surface roughness to retard fracture initiation) could provide further fatigue performance benefits, however, additional factors, such as manufacturability,

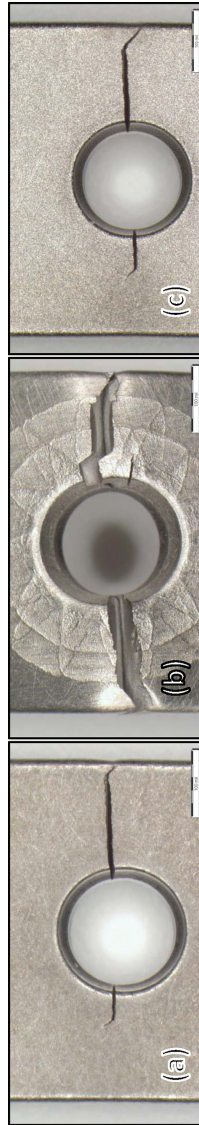


FIG. 8—Macrographs of exemplary baseline (a), laser shock peened (b), and shot peened (c) specimens fatigue tested to failure under 45-lbf max compressive load. Additional images captured during earlier testing cycles indicate crack growth originates on the face of the plate adjacent to the centrally located hole. The length of the scale bar is 2.5 mm.

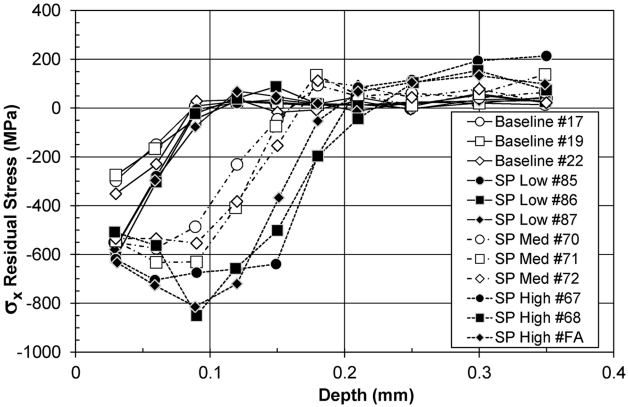


FIG. 9—Residual stress profiles of specimens subjected to three different intensities of shot peening (SP), as well as the as-fabricated (baseline) measurements for comparison. The increasingly compressive peak residual stresses are balanced by increasingly tensile residual stresses at deeper depths. In general, these results follow the expected trend of higher intensity SP leading to deeper and more compressive residual stress.

come into play that complicate the evaluation criteria for what would be considered optimal.

Figures 10 and 11 also describe the performance of EP surface modification in absence of SP. The performance of the EP-only specimens is impressive

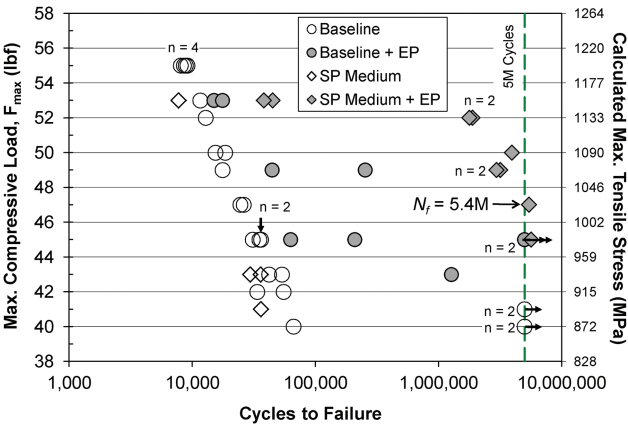


FIG. 10—Electropolishing (EP) improves fatigue performance relative to the baseline as-fabricated test specimen condition, and to an even greater extent when combined with medium-intensity shot peening (SP). The run-out load for baseline + EP condition is 45 lbf, whereas the run-out load for medium intensity SP + EP is greater than 47 lbf.

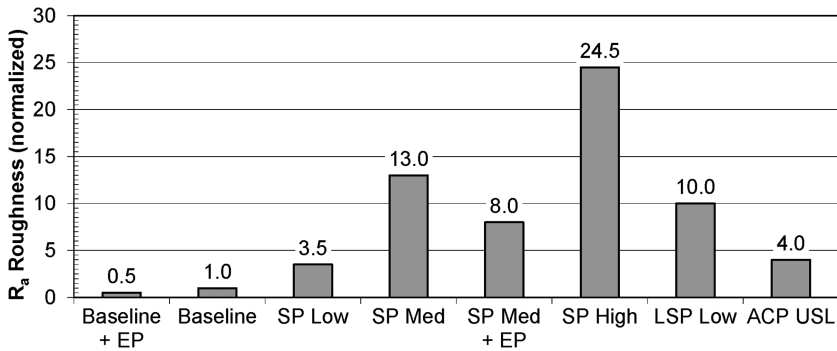


FIG. 11—Surface roughness (R_a) normalized by the baseline value for different surface modification treatments including shot peening (SP), laser shock peening (LSP), and electropolishing (EP). The upper specification limit (USL) for a commercially available ACP is included for comparison.

[run out of -45 lbf (-200 N)] and additionally attractive from the standpoint of eliminating the additional SP manufacturing steps. However, to achieve this level of fatigue performance without the advantage of additional compressive residual stress, these specimens rely on a very smooth surface with an R_a value eight times lower than the upper specification limit for some commercial ACPs. This fatigue benefit may be undone by the generation of small scratches during downstream manufacturing processes or during the implant procedure itself. In contrast, plates benefiting from an SP + EP surface modification approach may be less sensitive to small scratches because of their inherently higher surface roughness and the enhanced compressive residual stress.

Conclusions

The loading conditions for ACP tested by the ASTM F1717-09 compressive fatigue method are approximated well by a simple model of a beam subjected to pure bending. This means that peak stresses vary inversely with the square of the plate profile and that the bending stress decreases linearly with depth below the surface, where it is maximal. These insights have implications for the use of surface modification as a means to gain a fatigue entitlement to reduce ACP profile thickness for greater patient comfort:

- LSP (and other surface modification technologies, such as deep rolling and roller ball burnishing) have evolved as a means to drive compressive residual stresses deeper below a component surface than can be typically achieved by SP. In absence of subsurface crack initiation, the added value of a deep compressive stress for a plate subject to bending is questionable, especially when it involves a tradeoff with maximum compressive stress as it did in this study.

- The thin material cross sections associated with many implantable medical devices, compared to those in the aerospace and automotive industries, for which SP and LSP have been developed, make the potential for negative effects on fatigue performance from compensatory tensile residual stresses a concern. This negative effect did not materialize in this study because the measured subsurface reversal in residual stress from compression to tension was gradual and deep (Figs. 6 and 9) and the service loads are greatest at the surface.

Whereas LSP and ion implantation did not enhance fatigue performance in this work, this study should not be viewed as an indictment of their potential. The intensity of these surface modification technologies (and the profile of the compressive residual stress and/or surface roughening they impart) is widely variable and this work did not necessarily evaluate the optimal intensities. Fatigue performance enhancement has been reported by others for ion implantation [15] and LSP [16].

Mechanical fatigue testing is a time-consuming aspect of evaluating and developing surface modification technologies to improve fatigue resistance. Residual stress depth profile characterization and surface roughness measurement can weed out specific process recipes that do not improve upon the residual stress profile or surface roughness of a baseline component without the need for mechanical testing. The hole-drilling method used here provided depth profiles that would be very time-consuming to obtain using XRD $\sin^2(\psi)$ measurements followed by layer material by electropolishing in an iterative manner. However, given the importance of the surface residual stress in this application, surface XRD $\sin^2(\psi)$ measurements would provide useful information complementary to the hole-drilling depth profiles.

Notch-sensitive materials, such as the titanium alloys frequently used for implantable medical devices, present special challenges for surface modification technologies that seek to improve fatigue performance by imparting compressive residual stresses and are accompanied by an increase in surface roughness. Electropolishing appears to be an effective method to reduce this roughness without losing the compressive residual stress benefit.

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Fracture Toughness and Fatigue Crack Propagation Determination of Metal Matrix Composite Materials

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ABSTRACT: Metal matrix composite materials comprised of hard carbide particles in various titanium alloy matrices have been developed which may be utilized for biomedical applications requiring high-wear-resistance materials (i.e., spinal disc replacement). Two matrix materials, an alpha-beta and a beta titanium, were evaluated with varying carbide contents ranging from 0 % to 15 % by volume. In general, the alpha-beta titanium matrix yielded higher fracture toughness and resistance to crack propagation compared to the beta matrix composites. This matrix also showed less sensitivity to wet testing conditions over the beta matrices, retaining its increased toughness with increased carbide content. The addition of carbide particles generally reduced the fracture and fatigue properties; however, the effect was variable. It was observed that, of the composite materials, the 10 % carbide in an alpha-beta titanium matrix composite yielded the highest fracture toughness (30.7 MPa·m^{1/2}) and fatigue crack threshold (2.2 MPa·m^{1/2}) and was the most stable with respect to environment.

KEYWORDS: metal matrix composite, fracture toughness, fatigue crack propagation

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Introduction

The human spine is organized of alternating rigid vertebral bodies and soft disks. The rigid bodies allow for structure and support of adjacent members, such as the head and ribs. The disk acts as the hinge between the rigid segments, as well as a form of shock absorption [1–3].

Multiple remedies exist for medical ailments associated with spinal disk degeneration, ranging from physical therapy to surgery and complete stabilization. Whereas each has their distinct advantages and disadvantages, some dynamic disc replacement implants have been developed to take the place of such disks and retain local motion [4].

The forces on biomedical implants are not trivial. Depending upon implant location, the weight of the upper body, head, and arms all go through the spine [3,5]. These forces, combined with the local movement of the vertebral bodies, result in high requirements for wear and strength, in addition to biocompatibility. To accommodate both, a novel metal matrix composite material has been developed in which hard carbide particles are embedded in a titanium alloy matrix.

Carbide materials comprise some of the hardest materials known, in addition to being very chemically stable [6]. This makes them optimal biomedical implant materials in wear applications. However, because of their high hardness, the manufacturing process may be extremely difficult or costly.

Titanium alloys have been used as a medical implant material for many years in spinal, neurological, and cardiovascular applications [7,8]. It is a proven biologically favorable material that is easily machined. Unfortunately, with a relatively low hardness compared to carbide-based materials, these alloys may not be suitable for a wear-type application [9].

When a titanium alloy is impregnated with a compatible carbide material, the positive properties of both may be realized [9]. As the matrix material makes up a significant portion of the resulting composite, an implant can be manufactured from barstock material, with a surface containing both the carbide particles and titanium alloy matrix. Upon further refinement, the soft titanium alloy matrix is removed, further exposing the carbides above the matrix. When in contact with a mating part of similar material, the carbide particles sustain the contact forces, insulating the matrix from the interface. With their high wear resistance, they allow for minimal contact of the titanium alloy matrix and reduce material loss over time.

As the carbide content is increased, concerns arise regarding any negative properties that may become apparent, such as decreased fracture toughness. Carbide particles are inherently very strong; however, they are not ductile. Increasing their concentration may result in material that is very low in toughness relative to other metallic materials. Thus, a balance must be maintained between required wear properties and desired toughness or machinability.

It could be expected that the medical implants that could be made with this material would be relatively small to fit within the human cervical spine. Thus, even small cracks can be relatively large in comparison to the wall thicknesses. Because of the cyclic loading of the device and high reliability requirements of medical device implants, it is desired to understand the fracture toughness and fatigue crack propagation rates of the material as it varies with matrix alloy and carbide concentration. In addition, before moving to device-level testing, which incorporates a greater detailed evaluation of biological implications, a preliminary evaluation of the materials' sensitivity to the environment is desired. This will ultimately provide a framework to choose implant materials and quantify any perceived risks.

Fracture Toughness

The work of Griffith [10] and Irwin [11] is considered the foundation of fracture mechanics theory. From this body of work, a stress-intensity factor, K , has been derived that describes the local stress intensity increase caused by an existing crack. Today, the common representation of this work is described by relating the crack size (a), applied stress (σ), and crack geometry (Y) to the resulting stress intensity in mode I (pure tensile) conditions.

$$K = Y\sigma\sqrt{\pi a} \quad [12] \quad (1)$$

In mode I conditions, the minimum fracture toughness is referred to as mode I critical fracture toughness (K_{IC}). Although many medical implants may operate in the plane stress and mixed mode conditions, determining the actual fracture toughness becomes non-trivial. Plane strain fracture toughness, however, can be used to determine a conservative estimate of this material property.

In addition, the surrounding environment may play a role in crack propagation. For example, a chemical species may disassociate at the crack tip, be absorbed into the bulk ahead of the crack, and reduce local material plasticity. The end result is increasing the ease at which the crack grows and reducing the measured fracture toughness. Because of the various chemical species present in the human body, this may be a concern for biomedical applications.

Fatigue Crack Propagation

In addition to crack propagation during monotonic loading, cyclic loading is a concern for many biomedical implants. Even at small load amplitudes, the local stress concentration resulting from a crack may result in local plastic deformation. Over the course of many cycles, the material's plasticity is fully accounted for, resulting in crack propagation.

TABLE 1—*Metal matrix compositions.*

Alpha/beta Titanium Matrix	Beta Titanium Matrix
0 % carbide	0 % carbide
10 % carbide	7.5 % carbide
15 % carbide	

Cracks will propagate at different rates in different materials, depending upon the applied stress intensity. Generally, the higher the stress intensity, the faster the crack grows. Material performance is typically represented as crack advancement per cycle (da/dN) as a function of cyclic stress intensity (ΔK).

There does exist a threshold below which cracks will not likely propagate, referred to as the stress intensity threshold (ΔK_{th}) [12]. As with fracture toughness, the local environment may affect the performance of the material. However, if the loading conditions and flaw sizes are well defined, one can mathematically determine whether the component will likely survive indefinitely. This is a valuable tool for predicting component reliability and product performance.

Materials

Five different material combinations were evaluated, shown in Table 1. Two different matrix materials were used, one a commercially available alpha/beta alloy and the other a custom beta alloy. Both were infused with varying levels of carbide contents to determine the relative effect of concentration on fracture and fatigue properties. The alpha/beta titanium alloy material was manufactured using commercial titanium extrusion techniques. All others were manufactured with a powdered metallurgy process [9]. All materials were provided in 1-in.-diameter rod samples in the as-drawn condition. The mechanical properties of each are provided in Table 2.

TABLE 2—*Composite material mechanical properties.*

Material	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Modulus (GPa)
Alpha/beta titanium	992	923	113
10 % carbide	1075	1013	134
15 % carbide	1096	1082	155
Beta titanium	1461	1447	111
7.5 % carbide	972	875	119

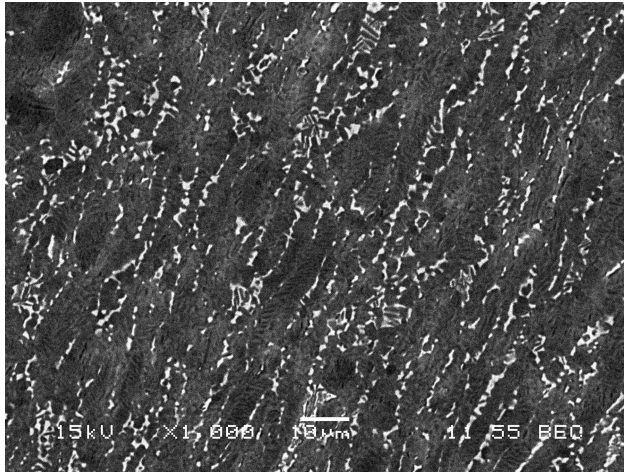


FIG. 1—*Alpha/beta titanium, no carbide.*

Microstructure

Prior to mechanical testing, metallurgical cross sections were prepared of all five materials in both the longitudinal and transverse directions with respect to the extruded bar axis. Scanning electron micrographs (SEM) of the representative microstructures (longitudinal cross-sections) are shown in Figs. 1–5. SEM images were taken in a backscattered mode, which highlights compositional variations with contrast differences. For the alpha/beta matrix materials, the slightly more atomically dense beta titanium appears brighter than the darker alpha. As the carbide inclusions contain carbon that significantly less dense

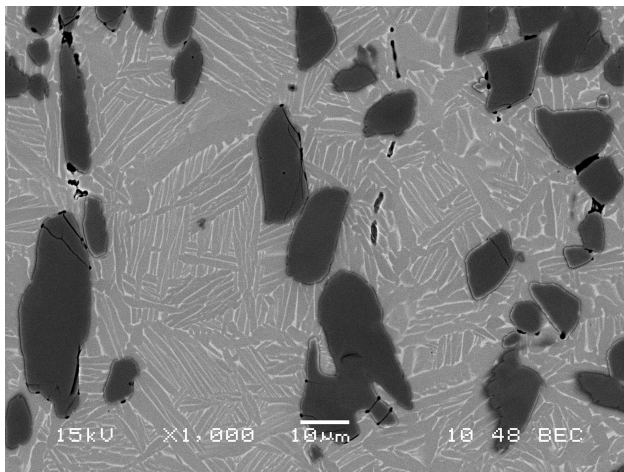


FIG. 2—*Alpha/beta Ti, 10 % carbide.*

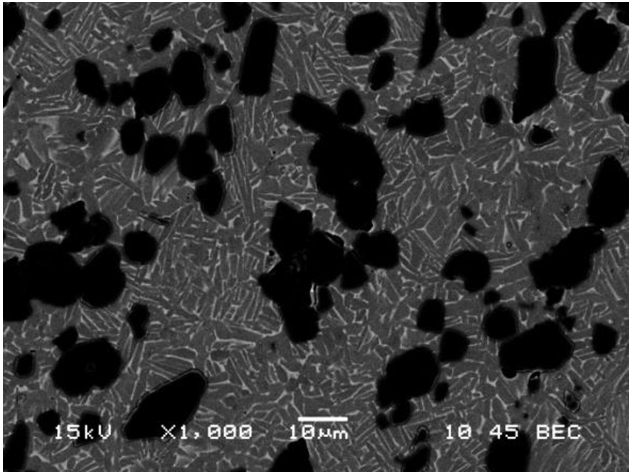


FIG. 3—*Alpha/beta Ti, 15 % carbide.*

than titanium, it appears darker yet. Voids, containing no material, appear black. These contrast differences are highlighted in Fig. 2.

The alpha/beta matrix material with no carbides contained an elongated alpha phase with intergranular beta. The 10 % carbide material contained a coarse acicular alpha grain structure with intergranular beta and carbide inclusions loosely aligned along the rod axis. The axial alignment of the carbides is a result of the extrusion process used to manufacture the rod material. These large aspect ratio inclusions (up to approximately 5/1) also appeared to have some associated voids, again postulated to be a result of the extrusion process. These voids were observed in both the longitudinal and transverse directions.

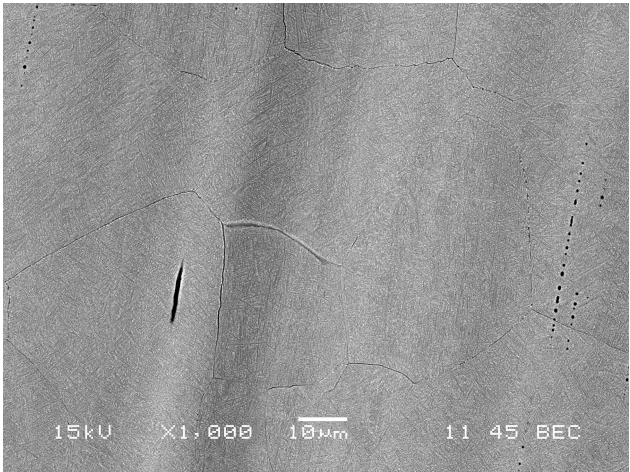


FIG. 4—*Beta Ti, no carbide.*

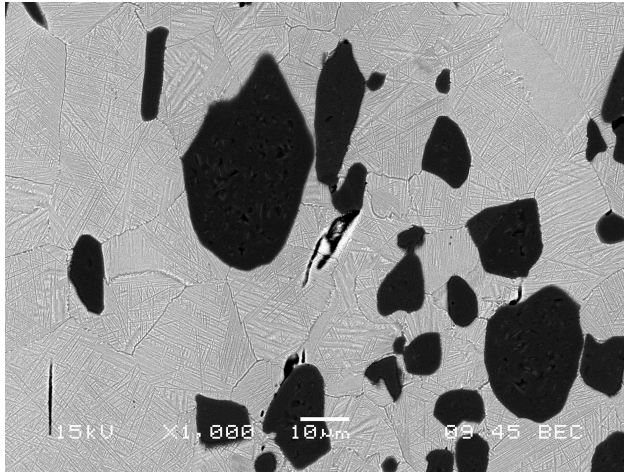


FIG. 5—*Beta Ti, 7.5 % carbide.*

The 15 % carbide material was similar; however, the matrix material appeared to have a more coarse, equiaxed appearance.

The beta matrix material (no carbide) contained equiaxed grains with a very fine acicular substructure. There also appeared to be small strings of voids extending parallel to the rod axis, as well as beta-rich phases visible under SEM observation. These phases also ran parallel to the rod axis.

The 7.5 % carbide material contained carbide inclusions larger than those in the alpha/beta matrix material and appeared orientated along the longitudinal axis when examined at low magnification. A stabilized beta phase with very thin acicular alpha phases present was found. It should be noted that the alloying element used to stabilize the beta phase was varied slightly in this lot of material, thus increasing the presence of the alpha phase.

Experimental

As the material is provided in the extruded condition, round compact tension specimens were electro-discharge machine (EDM) cut from the 1-in.-diameter bar stock. Two orientations were evaluated according to the ASTM designations: C-R and R-L. R-L samples were made by cutting a flat bar out of the center of the rod. Round compact tension specimens were then cut from this flat bar in a geometry consistent with the C-R orientation. Graphical representations of these components are provided in Figs. 6–8.

Based upon preliminary estimates of the loads required for testing and the equipment available, a sample thickness (B) of 4.7 mm was chosen that corresponded to a width (W) of 18.8 mm. This aligns with the ASTM E399-90 [13]

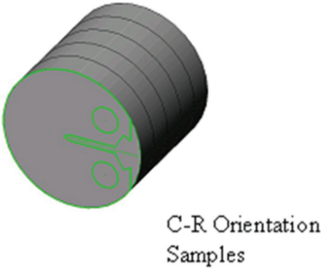


FIG. 6—*C-R orientation samples.*

and E647-00 [14] requirements of a width (W) to thickness (B) ratio equal to 4 for this sample configuration [13,14].

Concerns existed regarding the effect of the EDM recast layer on crack propagation rates for both orientations. Therefore, this layer was removed on the major face of all samples using silicon carbide sandpaper under a water coolant, similar to metallographic preparation. Each sample was then individually measured for dimensions prior to testing.

Fracture toughness was determined in accordance with the ASTM E399 standard [13]. A minimum of three samples were tested in both orientations. To evaluate the material under wet conditions, deionized water was allowed to drip through a tube in a controlled manner from an elevated reservoir. The distal end of the tube was connected to a syringe needle placed directly adjacent to the EDM cut notch. Upon supply of the water, it naturally wicked into the pre-cut crack, wetting the surface. This water was present during both the fatigue pre-crack, as well as the fracture toughness test to ensure all aspects of environmental effects were taken into account.

Applied stress intensities for fatigue pre-crack generation were chosen based upon initial testing upon the alpha/beta matrix material with no carbides,

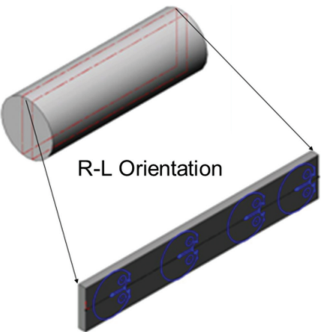


FIG. 7—*R-L orientation samples.*

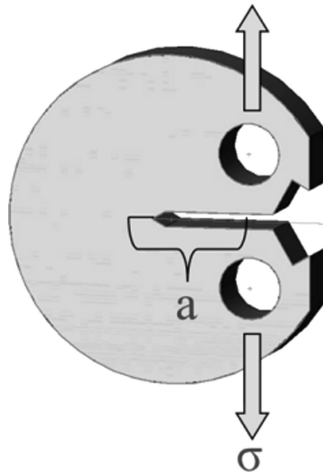


FIG. 8—Round compact tension specimen.

as well as an estimation based upon the ASTM E399 [13] Appendix A2 guidelines. A detailed list of test parameters is provided in Table 3.

Fatigue crack propagation tests were conducted in accordance with ASTM E647 [14]. Samples, orientations, and preparations were consistent with those used for the previously described fracture toughness test. Again, deionized water was present at the EDM cut notch during the duration of the test (pre-crack and crack propagation).

Five samples were evaluated for crack propagation rates. Depending upon the material, one to two samples for each material were evaluated at decreasing stress intensities until the stress-intensity threshold parameter could be determined (target crack growth rate of 0.3 nm/cycle). It should be noted that a stress-intensity threshold parameter lower than what is suggested in the ASTM standard (1 nm/cycle) was utilized. It was chosen to evaluate variability in the fatigue crack-propagation rate at near threshold stress intensities. Once the stress-intensity threshold could be determined, the same sample was tested at gradually increasing stress intensities using a constant load amplitude test. This

TABLE 3—Fracture toughness test parameters.

	Alpha/beta Titanium	10 % Carbide	15 % Carbide	Beta Titanium	7.5 % Carbide
Pre-crack cycle frequency (Hz)	20	20	20	20	20
Notch length (a_0) (mm)	7.6	7.6	7.6	7.6	7.6
Final crack length (mm)	9.1	9.1	9.1	9.1	9.1
Pre-crack starting stress intensity ($\text{MPa}\cdot\text{m}^{1/2}$)	21	15	15	13	15
Pre-crack final stress intensity ($\text{MPa}\cdot\text{m}^{1/2}$)	16.4	12	12	9.9	11
Ramp rate (mm/min)	1.3	1.3	1.3	1.3	1.3

TABLE 4—Fatigue crack propagation test parameters.

	Alpha/beta Titanium	10 % Carbide	15 % Carbide	Beta Titanium	7.5 % Carbide
Pre-crack cycle frequency (Hz)	25	25	25	25	25
Notch length (a_0) (mm)	7.6	7.6	7.6	7.6	7.6
Final pre-crack length (mm)	9.1	9.1	9.1	9.1	9.1
Pre-crack starting stress intensity (MPa·m ^{1/2})	21	15	15	13	15
Pre-crack final stress intensity (MPa·m ^{1/2})	16.4	12	12	9.9	11
Fatigue cycle frequency (Hz)	25	25	25	25	25
R factor (unitless)	0.6	0.6	0.6	0.6	0.6
Normalized K gradient (1/mm)	K increasing	0.2	0.2	0.2	0.2
	K decreasing	−0.2	−0.2	−0.2	−0.2
	Constant stress intensity	0	0	0	0

is recommended within the ASTM standard to determine whether the plastic zone in front of the propagating crack influences the crack propagation rate when nearing the threshold stress intensity. With the advancing crack stressed to constant load amplitudes, the stress intensity increased until sample fracture.

In addition to the samples determining stress-intensity threshold parameters, one was cycled at a constant stress intensity to evaluate the variability of the crack propagation rates. Finally, two samples were tested at gradually increasing stress intensities until fracture of the sample. All data was then agglomerated together to form a full da/dN representation as a function of cyclic stress intensity.

Fracture Toughness Results

Fracture toughness results of all materials, orientations, and environments evaluated are presented in Figs. 9 and 10. In the C-R orientation, the 10 % carbide material was very similar to the control material. However, when the carbide concentration was increased to 15 %, additional material variability was observed, with some samples exhibiting a fracture toughness greater than the control material. In the R-L orientation, the 10 % carbide material was tougher than the 15 % carbide, however, not as tough as the control material with no carbide. The beta titanium material exhibited the lowest fracture toughness of all materials evaluated. This trend was not surprising based upon its elevated tensile strength noted previously in Table 2. Surprisingly, it was found that the dry 7.5 % carbide material, with relatively small amounts of alpha titanium, appeared to exhibit the highest fracture toughness of the metal matrix composite materials evaluated here in both orientations under dry conditions.

The addition of water at the crack tip had varying results, depending primarily upon the matrix material. For most alpha/beta titanium matrix materials,

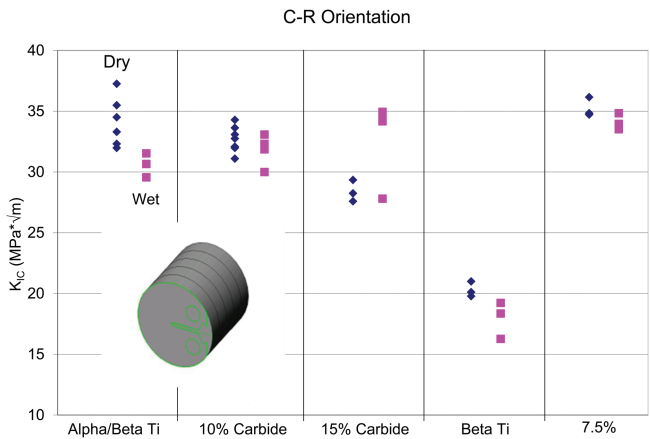


FIG. 9—*C-R fracture toughness, dry and wet.*

it appeared to minimally shift the mean fracture toughness results down. For the 15 % carbide material tested in the C-R orientation, the mean appeared to actually increase; however, there was significant spread in the data.

The presence of water also appeared to have an effect on the beta matrix material. In this material with no carbides, the fracture toughness values were already reduced compared to the other test conditions. With the presence of water, these values shifted slightly lower in both orientations.

The effect of water was the greatest on the 7.5 % carbide material. Whereas the hydrated C-R orientation remained relatively similar to the dry, the R-L orientation exhibited a high degree of relationship between test environment, with the hydrated samples roughly 50 % that of the dry.

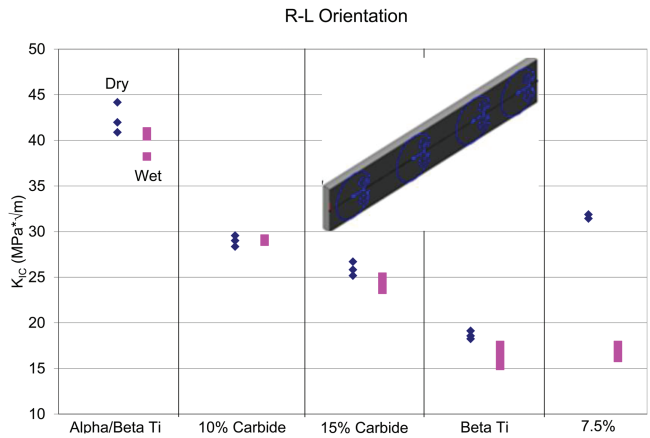


FIG. 10—*R-L fracture toughness, dry and wet.*

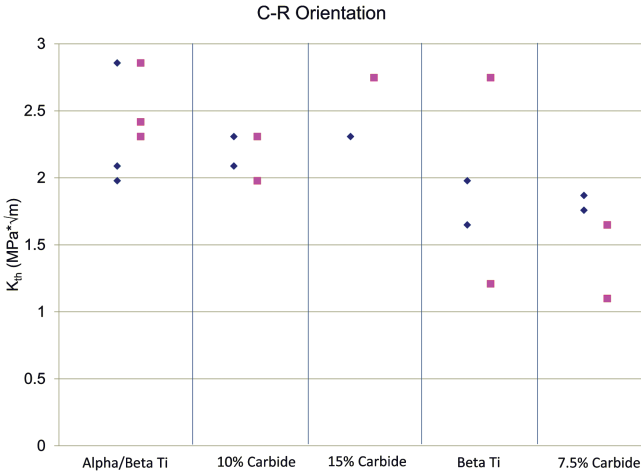


FIG. 11—*C-R stress intensity threshold, dry and wet.*

Fatigue Crack Propagation Results

Fatigue crack propagation experiments yielded results that trended with the previous toughness data. Stress intensity threshold parameters determined are provided in Figs. 11 and 12.

The alpha/beta titanium matrix materials had the highest fatigue crack propagation thresholds among the materials evaluated. Because of the small number of samples available for testing, it is difficult to assess the true variability within the data; however, the difference between the matrix materials appears most evident in the R-L orientation.

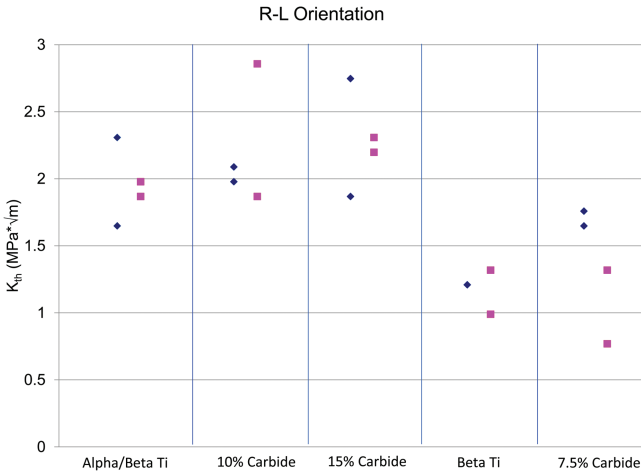


FIG. 12—*R-L stress intensity threshold, dry and wet.*

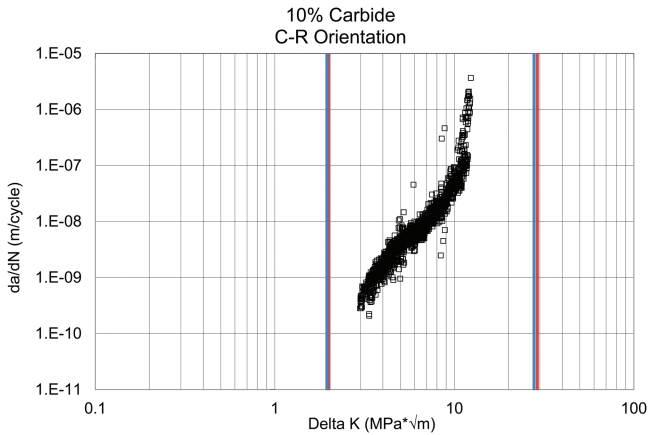


FIG. 13—10 % Carbide in α/β matrix, C-R orientation, dry.

When tested in the hydrated condition, the α/β matrix materials appeared to have minimal shifts of stress intensity thresholds and crack propagation rates, highlighted in Fig. 13. Results across all α/β matrix materials were consistent. The β matrix materials, however, had a much greater dependence upon hydration, highlighted by Fig. 14. As was observed in the previous fracture toughness results, the threshold stress intensity decreased and the crack propagation rates increased.

Fractography

Post-fracture specimens were evaluated in a SEM to determine fracture mode, matrix ductility, and relative appearance of the carbides. A representative

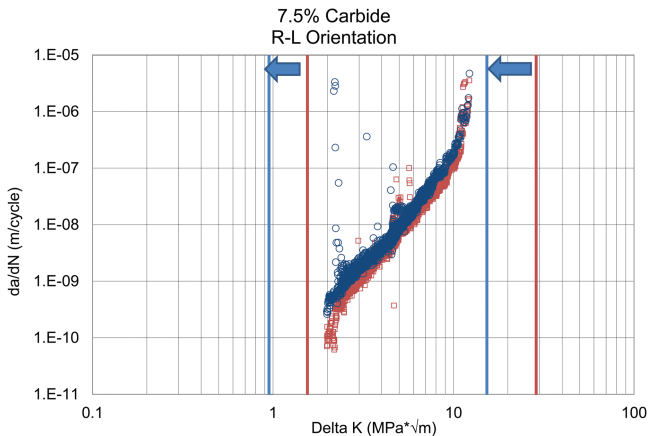


FIG. 14—7.5 % Carbide in β matrix, R-L orientation, dry and wet.

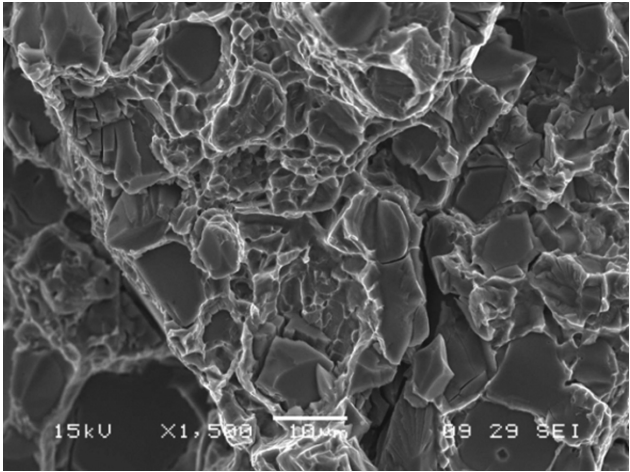


FIG. 15—10 % Carbide in alpha/beta matrix, C-R orientation, dry fracture surface.

micrograph of the 10 % carbide material is provided in Fig. 15. The ductile nature of the alpha/beta matrix material, typical of these alloys, can be observed by the presence of ductile microvoid coalescence. It appears that the carbides exhibit some degree of brittle fracture. This is important as it demonstrates a degree of load sharing and bonding between the matrix material and the carbides. Similar observations were made with the 15 % (Fig. 16) as well as the 7.5 % carbide material. The beta matrix material with no carbides, however, yielded a primarily brittle fracture surface, containing both transgranular and

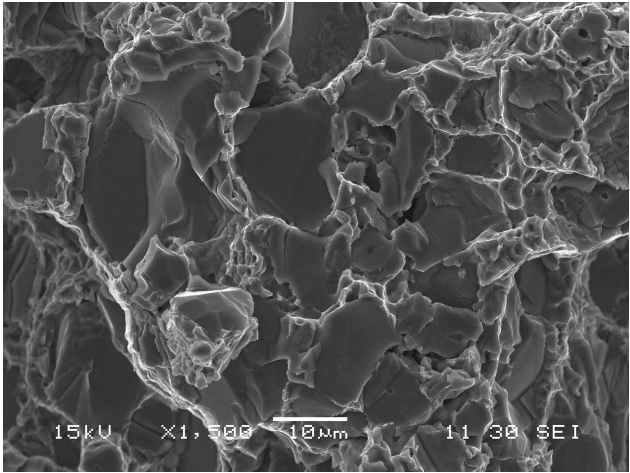


FIG. 16—15 % Carbide in alpha/beta matrix, C-R orientation, dry fracture surface.

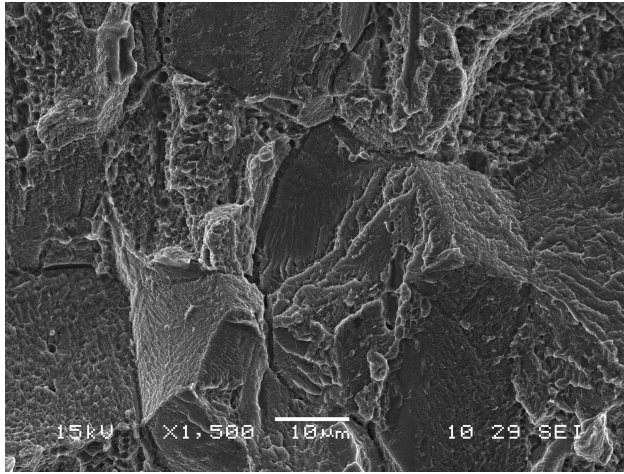


FIG. 17—Beta Ti matrix material, No carbides, C-R orientation, dry fracture surface.

intergranular fracture, shown in Fig. 17. This is consistent with the high tensile strength and low toughness observed. As highlighted by Fig. 18, no discernible difference was observed in the fracture surface of those samples evaluated in the R-L orientation compared to the C-R orientation.

Although the fracture toughness of the 7.5 % carbide material had the greatest change because of the presence of water, no significant change in fracture morphology could be observed, as shown in Figs. 18 and 19. Evaluation of the beta matrix material tested in hydrated conditions indicated a predominantly intragranular fracture morphology, as shown in Fig. 20. The change in

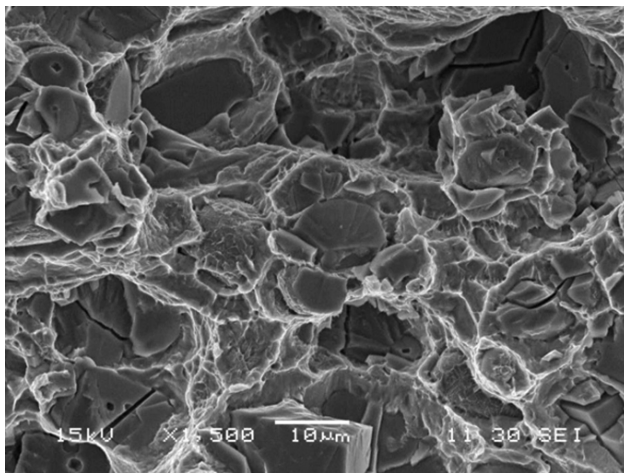


FIG. 18—7.5 % Carbide in beta matrix, R-L orientation, dry fracture surface.

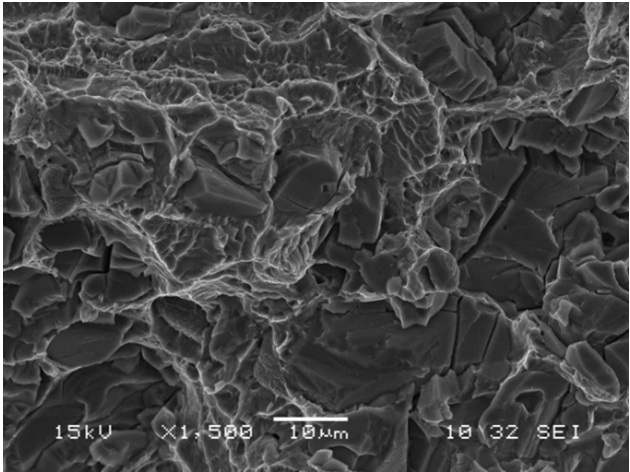


FIG. 19—7.5 % Carbide in beta matrix, R-L orientation, wet fracture surface.

fracture morphology would indicate a dependence of the composite fracture toughness on the matrix material, with beta titanium phases exhibiting reduced ductility in the presence of water.

In all materials evaluated dry, the fatigue crack propagation surface appeared to be transgranular cleavage, as highlighted in Figs. 21 and 22. No crack arrest lines could be observed and there appeared to be evidence of carbide brittle cleavage as well. This further reinforces the conclusion that there

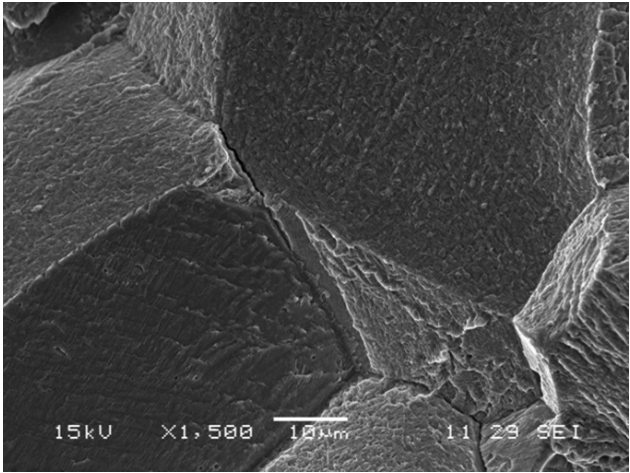


FIG. 20—Beta Ti matrix material, no carbides, R-L orientation, wet fracture surface.

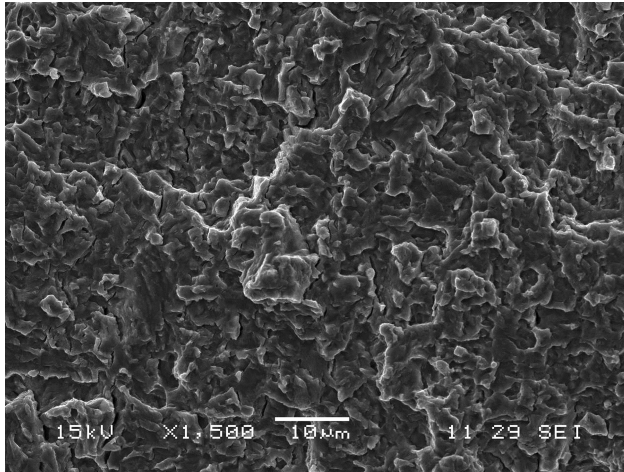


FIG. 21—Alpha/beta Ti matrix, no carbides, C-R orientation, dry fatigue surface.

exists some degree of load sharing and bonding between the carbides and the surrounding matrix material.

For all materials, with the exception of the beta matrix with no carbides, the morphology of the fatigue surface in hydrated conditions remained similar. For the beta matrix material, however, the fatigue crack propagation mode changed from a predominantly transgranular fracture to a predominantly intergranular fracture mode (Figs. 23 and 24).

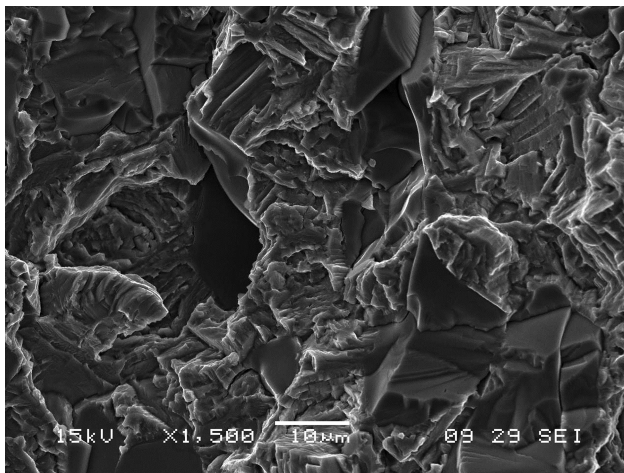


FIG. 22—10% Carbide in alpha/beta Ti matrix, C-R orientation, dry fatigue surface.

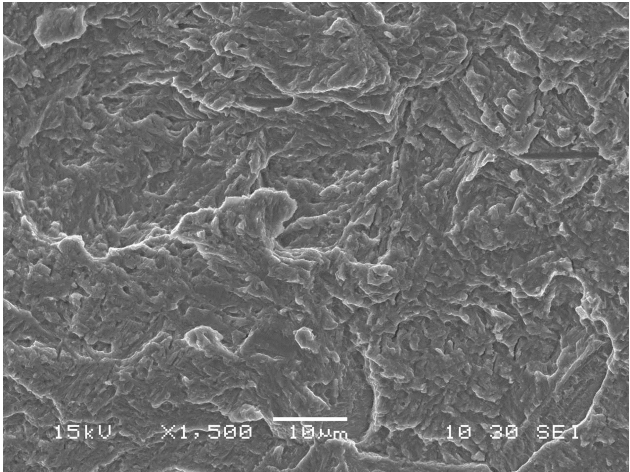


FIG. 23—*Beta Ti matrix, no carbides, R-L orientation, dry fatigue surface.*

Discussion

In accordance with ASTM E399 [13], post-test analysis is required to establish whether the fracture toughness values obtained are truly plane strain results, or if they operate in a mixed-mode condition. Analysis of the load/clip opening displacement (COD) results indicated that the alpha/beta material with no carbides exhibited significantly more ductility during fracture than the test metal matrix composite groups, as shown in Fig. 25. Because the maximum load sustained during the fracture toughness test was more than 10 % larger than the load used to calculate the fracture toughness, this analysis method was unable

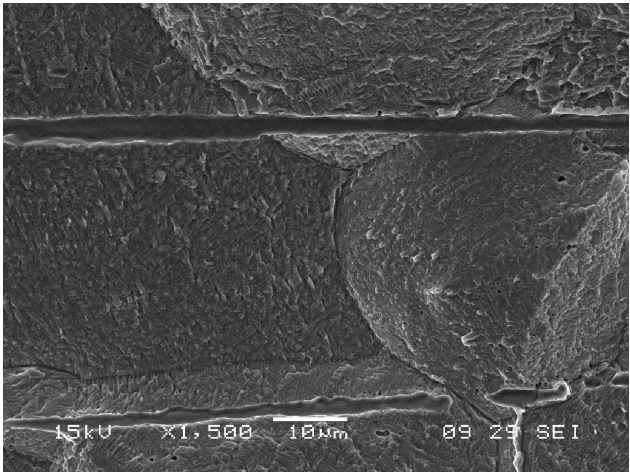


FIG. 24—*Beta Ti matrix, no carbides, R-L orientation, wet fatigue surface.*

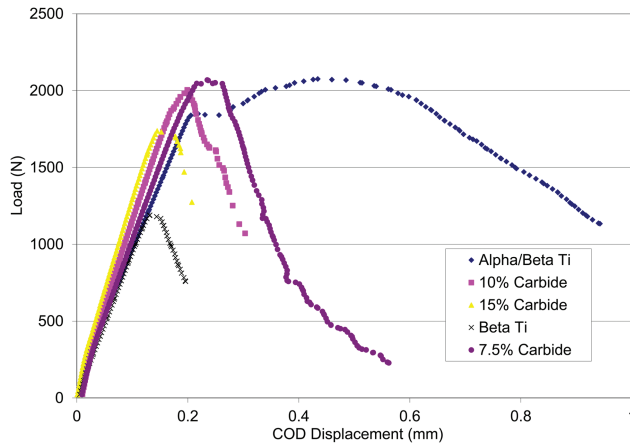


FIG. 25—Fracture toughness COD results.

to determine true K_{IC} values. This could be overcome by either increasing the sample width (moving to a plane strain condition) or by using ASTM E1820 [15] for this sample configuration. All values calculated for the remaining materials passed this analysis and were deemed valid K_{IC} results.

The powder metallurgy manufacturing process appeared to reduce the sample to sample variability in fracture toughness results compared to standard titanium extrusion processes. The loosely aligned matrix microstructure is visible in the metallurgical cross-section Figs. 1–5. The lone exception is the alpha/beta titanium with no carbides. This material had a highly elongated microstructure along the extrusion direction and the greatest variability within a single material combination, regardless of orientation.

In general, the sample to sample fracture toughness variability appeared to be greater within the C-R orientation than in the R-L. As the matrix microstructure (in the powder processed material) is generally equiaxed, the variability is believed to be tied to the carbides themselves. Cross-section of Figs. 1–5 highlight their variable size and orientation. Especially evident in the 10% carbide material, these carbides appeared to be aligned in the extrusion direction with greater areas of matrix material separating generally aligned carbides. By testing in the C-R orientation, this variability may have been highlighted, compared to the R-L orientation.

Initial data scatter was observed during crack propagation testing. When a sample was cycled, the first 4–6 data points were generally scattered to a higher crack propagation rate than that observed during the bulk of the testing. Whereas the R factor and final pre-crack stress intensities were chosen to minimize residual plastic zone effects at the root of the crack, it is possible that some plasticity was observed which influenced the initial measurements. This data scatter was consistent across all materials and orientations, indicating that

it may have been a function of test equipment, rather than material or carbide concentration. These data points were not excluded from analysis, nor did they significantly affect it because of the robustness of the data set.

The tests conducted here provide significant amounts of data that could be used in the design of a wear-resistant, dynamic medical device. However, to down-select a material for development, a way to properly visualize and convey the material properties was desired. Therefore, the fracture toughness and stress-intensity threshold parameters were plotted together, as shown in Fig. 26. In general, it is desired to utilize material which has increased fracture toughness, as well as stress intensity thresholds. Although these two parameters may trend with each other, it is also desired to utilize a material with consistent performance, regardless of orientation and environment. In general, the alpha/beta matrix materials had the highest fracture toughness and stress intensity threshold. Both the 10 % and 15 % carbide material had comparatively consistent results in both orientations and environmental conditions, indicating that they would be best suited for biomedical applications. Whereas there was one outlier of the 15 % carbide results, it was higher in toughness and fatigue propagation resistance compared to the others.

The beta matrix materials, however, exhibited a higher degree of dependence on orientation and environment. Although the beta material with no carbides present exhibited the highest tensile properties, it also yielded the lowest ductility, highlighted by the fracture surface in Fig. 17. As the primary variable controlled between metal matrix composite materials was chemistry, it is believed that by increasing the beta stabilizing content, the ductility of the material was significantly impeded by the change in chemistry. Because toughness

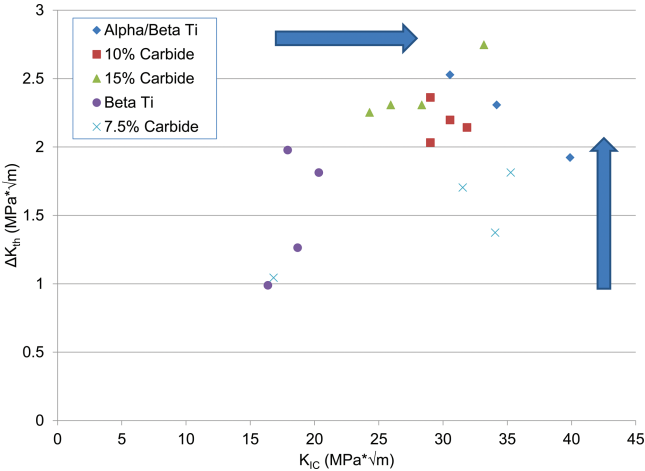


FIG. 26—Fracture toughness and stress intensity threshold results, all materials, all conditions.

generally trends with ductility, the increase in beta stabilizer may have resulted in a reduction in toughness. No precipitates were observed in the metallurgical cross-section (Fig. 4), however local areas of chemistry variations were found, highlighted by the light and dark striations in the SEM image. It is possible that some local concentrations that are higher in beta stabilizing content may contribute to the reduced fracture toughness and fatigue crack resistance.

The beta stabilized matrix with 7.5 % carbide, in general, appeared to have improved properties compared to that with no carbide. As noted previously, small amounts of alpha titanium can be observed in the metallurgical cross-section (Fig. 5). The increase in alpha phase, because of a reduction in beta-stabilizing content, appeared to increase ductility, highlighted in Fig. 18. This did not, however, translate to an increase in crack propagation resistance in wet conditions, as there was a large reduction in the R-L orientation wet fracture toughness.

In general, titanium alloys are corrosion resistant to immersion in water. However, crack propagation caused by notches or sharp cracks in seawater has been noted [16]. Published literature regarding beta titanium alloys suggests that the chemistry, processing, and microstructure all play a role in the crack propagation and sensitivity to their environment; however, performance is generally positive [17,18]. Therefore, the sensitivity noted in the beta stabilized matrix material with 7.5 % carbides is somewhat surprising. The generally equiaxed matrix microstructure would indicate the variation caused by orientations in the wet condition is primarily a result of manufacturing process or carbides. As seen in Fig. 5, these appeared to be some of the larger and more "blocky" carbides with respect to the other composites. This indicates that the carbide size and shape may play a role in the fatigue and fracture properties of these materials.

These tests were only a method to provide an indication of risk associated with material selection and biological implant, as all hydrated tests conducted were in deionized water. The human body is considered to be a much more corrosive environment. To make more detailed calculations regarding crack-propagation rates during in vivo use, similar testing would be required in an appropriate media.

Conclusion

Using the referenced ASTM standards, the fracture toughness and fatigue crack-propagation rates of several metal matrix materials were evaluated for biomedical applications. It appears that the increased ductility of an alpha/beta matrix contributes to increased fracture toughness. The carbides present tend to reduce the toughness. Although hydration did not significantly affect the alpha/beta matrix composites, it did affect the higher beta matrix content alloys, reducing fracture toughness and increasing crack propagation rates. Overall,

the 10 % or 15 % carbide in alpha/beta matrix material appeared to have the most consistent results, regardless of orientation and environment. Of course, the final material choice must also take other properties such as wear resistance into account.

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