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Characterizing Fatigue Response of Nickel-Titanium Alloys by Rotary Beam Testing

ABSTRACT: Fatigue information has been reliable in predicting wire critical structural integrity. The aim of this study is to expound on the characterization technique of rotary beam fatigue testing (RBT). By alternating tension and compression stress states through RBT, it is possible to determine the life expectancy of Nitinol monofilament round wires. Fatigue testing has been employed to characterize the influence of subtle changes in inclusion content, chemistry variations of raw material, ingot transformation temperatures of Nitinol (NiTi), and surface finish conditions for implant grade wires. Currently, an ASTM standard does not exist that concentrates solely on fatigue testing shape memory alloys. By exploiting part geometry, this testing technique serves to compliment other characterization methods. Evaluation of fracture surfaces has proven useful in diagnosing the factors influencing failures. The utilization of fatigue data and fracture mechanics compliments tensile testing in providing information to the design engineer. Results from studying flexural endurance, statistical Weibull life assessment analysis, fracture analysis, and a determination of stress/strain levels at the site of failure have proven useful in determining desired material properties for next generation medical devices.

KEYWORDS: Nitinol, round wire, rotary beam, fatigue, fracture, $R=-1$, controlled strain

Introduction

Some common fatigue test methods include axial, bending, and torsion. In 2000, a study was conducted using a wire diameter of 0.267 mm to investigate the impact of melt origin on fatigue performance [1]. This research compared the melting practices, final workings, and final shape-setting heat treatment in the metal; however, fatigue lives did not differ for each supplier through bending strains of 0.72, 0.84, 1.0, 1.2, 1.7, and 2.5 %. Figures 1(*a*) and 2(*a*) show the comparison of inclusions found in Supplier A and Supplier B, two common Nitinol material vendors, utilizing scanning electron microscopy (SEM). Longitudinal mounts along the drawing direction were completed on nominal wire diameters of 2.03 and 2.16 mm, for Suppliers A and B, respectively. Energy dispersive X-ray spectroscopy (EDS) spectra shown in Figs. 1(*b*) and 2(*b*) suggest the inclusions are titanium-rich. While some defect particles reside in the bulk of the material, they have also been attributed to the use of contaminated feedstock. These melt-related defects often cause nonhomogeneous microscopic discontinuities that may inhibit slip and act as stress raisers.

Another study utilized a tabletop testing device that oscillates forces, creating a mean strain state of Nitinol materials [2]. It was found that fatigue life of Nitinol diamond-shaped specimens increased for mean strains above 1.50 % at the same alternating strain. In a rotary beam strain-controlled study, varying heat treatments and various test temperatures were imposed [3]. It was proven that in an isothermal strain-controlled environment, superelastic Nitinol was superior to stainless steels. Yang mentioned that fatigue-crack propagation analysis may be used complementary to the results from fatigue testing [3]. A cyclic frequency of 1000 revolutions per minute (r/min) was used in a system equipped with wire fracture detection, cycle counting, and over-temperature protection. The use of fracture surface analysis can be used in conjunction with these types of models.

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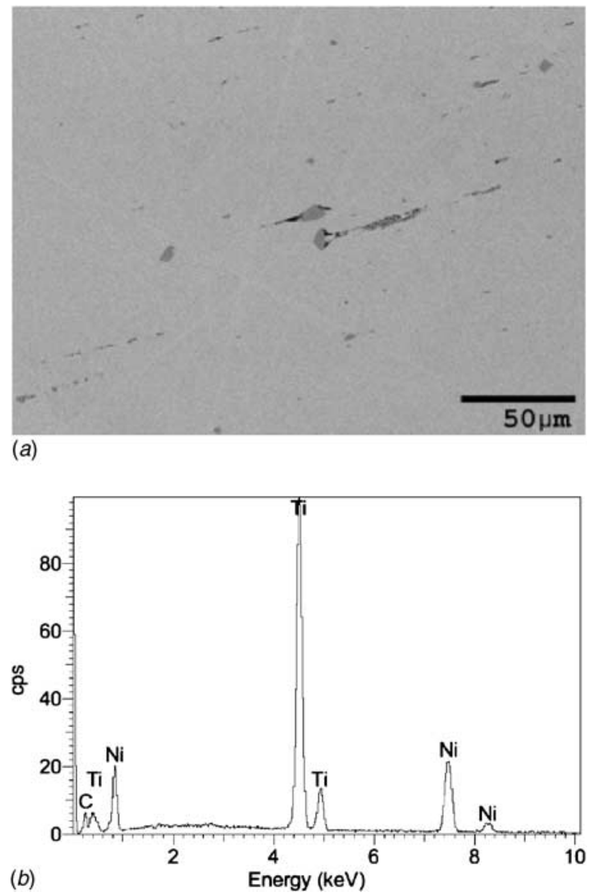

FIG. 1—*1(a) and 1(b)—Typical bulk material from Supplier A at 2.03 mm.*

Through experimentation, one may survey various alloys, suppliers, and processes utilized in the production of medical-grade fine wire. By controlling testing parameters and environments, tensile testing of medical grade Nitinol wires provides ancillary data to *in vitro* RBT analysis. In a similar light, this tool may be used as a quality check prior to shipment to device manufacturers during process validation. Due to the strain gradient in this type of test, any surface anomalies will be dominant in ultimate fatigue failures. The results from RBT may aid in the development of medical device focused safety factors, material development, and process evaluation. Intrinsic and extrinsic attributes, along with variation in processing routes, may all influence the comparison of cycles to failure [4].

The fabrication chain of Nitinol prior to RBT is: melting, hot working, cold working, and shape-setting heat treatment. Altering any chain segment affects final material properties. By using materials with equivalent thermomechanical processing with Ti49.2 at %, Ni50.8 at % as a baseline, derivations of the differences between in NiTi wire properties and performance are possible. As will be explained subsequently in detail, the greatest tensile and compressive normal forces are exerted at the outermost fibers of a round wire during RBT. The testing modus operandi does not solely focus on the end use of Nitinol products; it is regularly implemented as standardization of material quality.

Throughout RBT, the mechanical deformation that takes place in a solid wire may be studied as a member in pure bending. In this light, the wire contains a plane of symmetry and is exposed to equal and opposite couples (M and M′) acting in the plane of symmetry at the ends of the wire. As depicted in Fig. 3, the solid wire will bend concave upward and uniformly under the action of the couples, but will remain symmetric with respect to that plane. The wire is curved due to bending forces, analogous to the means of reaction flexure occurs when a wire experiencing forces in the cardiovascular system. The upper surface is in compression, while the lower surface is in tension. A

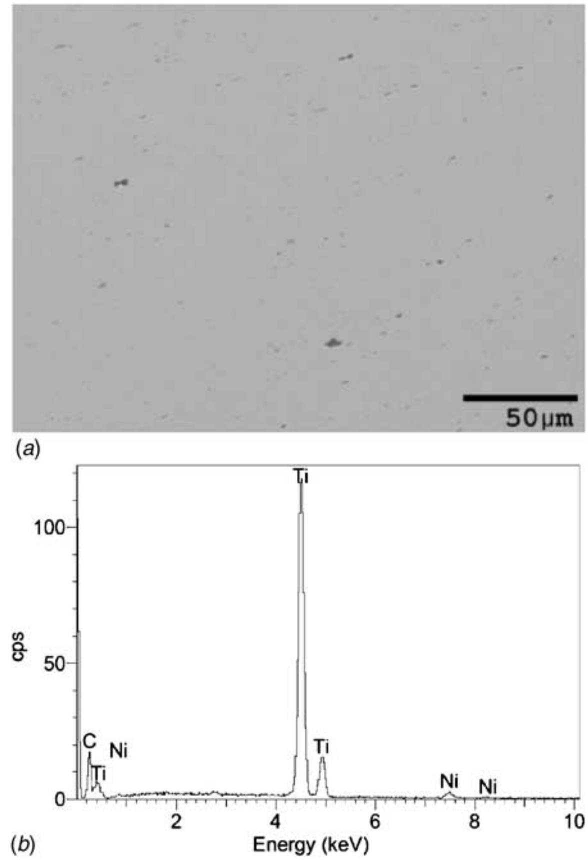

FIG. 2—*2(a) and 2(b)—Typical bulk material from Supplier B at 2.16 mm.*

dashed line through the core of the wire presents the neutral axis (NA), shifted to the compressive side.

At any point a finite distance from the neutral axis is under flexural loading, and there is a state of uniaxial stress from the σ_x component. In Fig. 4, segment AB shortens, while $A'B'$ lengthens as $M>0$. The upper face of the wire is in compression with ε_x and σ_x being negative (−). The lower face, with ε_x and σ_x being positive (+), indicates a tension state. The neutral surface, the surface parallel to the upper and lower faces of the wire, has $\varepsilon_x=\sigma_x=0$. The distance from any point to the neutral surface is noted as "y," ρ signifies the radius of arc *DE* (*NA*), and the central angle to *DE* is θ, knowing that the length $DE=L$ [5].

Rotating the wire samples causes a reversing cyclic stress, where the wire surface experiences an alternating tension and compression state. Figure 5(*a*) shows a strain waveform indicating peak-to-peak variation during a controlled flexure mechanical test through time. At any transition point on the curve, the material is at maximum strain amplitude with an equal amplitude about a zero mean strain ($R=-1$). During testing, the stresses in the material remain below the superelastic limit, as referenced in Fig. 5(*b*); permanent set is nonexistent as the round wire is not yielding.

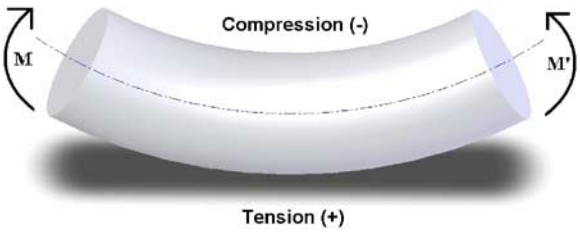

FIG. 3—*Solid wire in pure bending.*

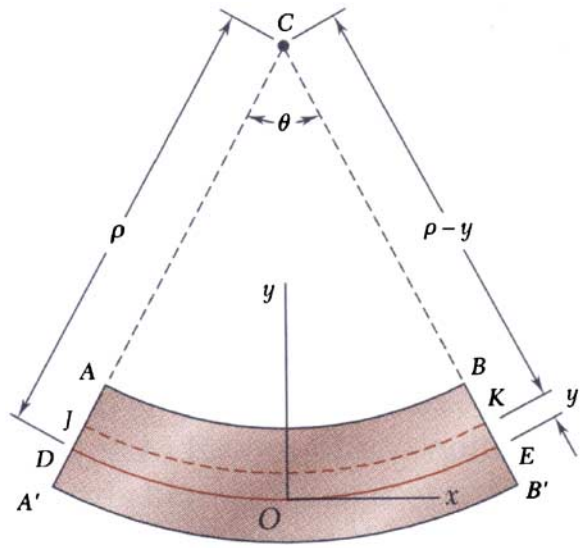

FIG. 4—*Neutral axis DE with radius of curvature ρ as noted [5].*

Materials and Methods

The Valley Instrument Company, Rotary Beam U-Bend Wire Spin Fatigue Tester (10-040) as shown in Fig. 6 may be utilized to test Nitinol wire in the superelastic condition above the Active Austenitic

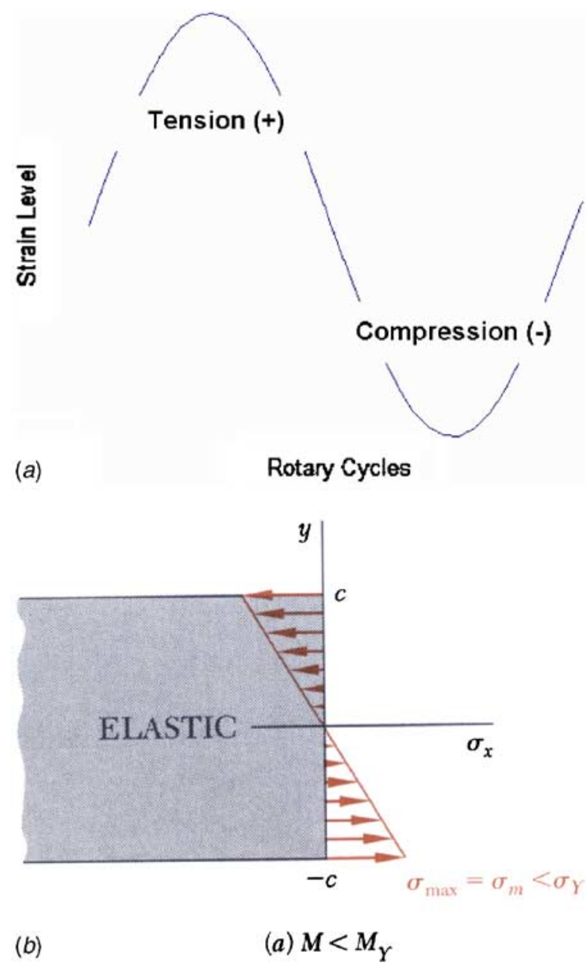

FIG. 5—*(a)—Rotary cycle waveform [3]; (b)—No permanent deformation in the wire taking place [5].*

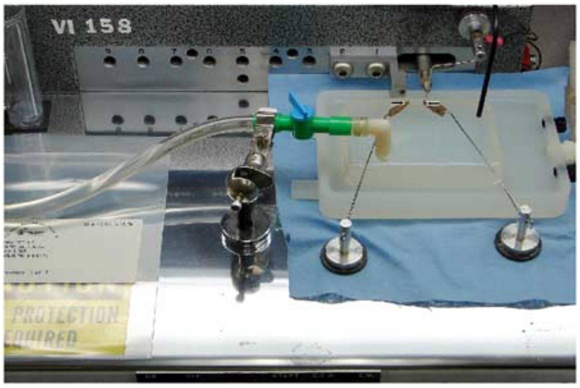

FIG. 6—*Valley instruments rotary beam spin fatigue tester.*

Finish temperature (Active A_f). Rotary beam fatique testing may be conducted in a temperature-controlled ambient air or in a liquid environment. Solvent baths used in experimentation include reverse-osmosis water (RO H_2O), saline, or Ringer's solution [4].

Testing instrument construction involves a motor-driven chuck and an adjustable bushing support that allows variable positioning of the free end of the specimens. The various holes in the bushing-bar provide strain adjustment for the specimen. Using a calculated distance from the chuck to form an arch, the design allows the axis of the chuck and the axis of the loose wire end in the bushing to be exactly parallel, as shown in Fig. 7. The specimen, with a known length, is mounted into the drive chuck system while the "nondriven" end is inserted into the free bushing. To prevent vibration, two support guides are positioned on the radius of the specimen, but outside of the apex, such that the guides do not affect the region of maximum strain.

The material is cycled in the chuck by a motor at a constant frequency of 3600 revolutions per minute, synchronous to an electronic clock with a resolution of 1/100th of a minute, thus giving the apparatus a resolution of 36 revolutions each minute [4]. Testing a planned grouping with a randomization of samples is an essential feature of a well-executed experiment. Attempt should be made to balance potentially detrimental effects of variables such as laboratory humidity, while accommodating for possible test equipment malfunction during the test program. The number of specimens, or sample size required, depends on the type of test conducted. Preliminary and exploratory research testing demands a minimum number of specimens to be from six to twelve [6]. Typical strain values may range from 0.60 % to 2.50 %; a strain level is considered low or high if it lies below or above the SIM (stress-induced martensite) strain level, respectively. The SIM appears where a volume of the parent phase of the material, austenite, transforms to martensite due to the application of a stress above that active A_f temperature. At high strain levels twelve samples are typically chosen, due to the

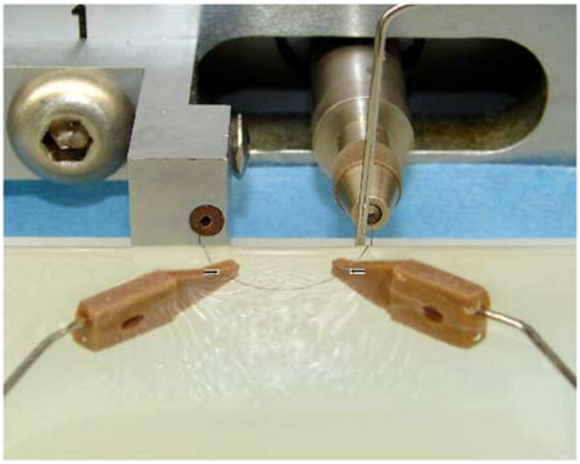

FIG. 7—*U-bend of Nitinol wire mounted within rotary chuck and bushing.*

expected shortness of the fatigue life; in contrast, six samples are tested at low strain levels. It is worth mentioning that at extremely low levels of strain, the time to failure is long ($N_f \sim > 10^7$ cycles) in relation to high strain levels ($N_f \sim 10^3$ cycles). In addition, it is recommended to set up testing to capture and evaluate data scatter experienced near the SIM strain level. A test protocol may include using multiple strains that are calculated by using the starting bend radius and increasing the center distance.

The reversible force that is incurred on the test sample is constant and stationary with the device. A cycle is counted as the number of turns the chuck completes during testing. The material is cycled to fracture, or continues to a predetermined number of cycles as a runout. At fracture, the instrument electrically grounds the test sample and thus terminates the test automatically. Through comparison of materials, some material or processes may show significant differences regarding cycles to rupture. In regards to lower strain levels, a desired runout time expectancy determination becomes of concern. Reinoehl et al. set 20 000 000 cycles, that is approximately 3.9 days, as runout [1]. In today's testing scheme, 400 million cycles (77.16 days at 3600 r/min) seems to be more representative of long-term testing. As follows in Eq 1, dimension analysis yields the number of cycles a specimen is subjected to:

$$\frac{3600\ \text{revs}}{1\ \text{min}} \cdot \frac{60\ \text{min}}{1\ \text{h}} \cdot \frac{24\ \text{h}}{1\ \text{day}} \cdot \ \#\,\text{days} = \ \#\,\text{cycles} \qquad (1)$$

Upon completion of rotary beam testing, fractured specimens are measured for length. If fracture has occurred, and the two pieces are of unequal length, the actual stress/strain state at the point of fracture can be extrapolated. The fracture ends are then studied using SEM analysis. Miyazaki found that fractures occur at both inclusions and grain boundaries [7]. Those which nucleated at grain boundaries were found in samples of electron beam melted material.

Fractured surfaces that revealed TiC inclusions were found in material that was melted in a carbon crucible. Large stress concentrations are expected to occur at both grain boundaries as well as inclusions [7]. EDS analysis is used to determine the composition of the foreign particles.

Experimental Results

Nitinol fatigue test data are traditionally presented in the form of an ε-N (strain-life) diagram as shown in Fig. 8 [1]. The data show 20 000 000 cycles as the runout parameter. From the data shown, it is evident that both suppliers' product yielded similar fatigue life at various strain levels for 0.267-mm round wire. The dependent variable, fatigue life N_f, in cycles is plotted on the logarithmic scale abscissa. The independently controlled variable, the strain amplitude (ε_A), is plotted on the ordinate with an arithmetic scale. A regression analysis line, or similar techniques, may be fitted to the data. As the fatigue life curve approaches a slope near zero, the line now represents the fatigue limit designated as a runout. Replicate tests are designed to ensure distribution in data acquisition. This type of frequency distribution curve of fatigue lives, taken at a given strain for an array of samples, permits the variation of fatigue characteristics throughout a volume of material to be extensively studied [6].

By creating survival probability plots, one is able to determine fatigue life for a specific parameter. Figure 9 is an example of specific strain level testing. In compliance with the proximity of data points in Fig. 8, the overall apparent trend is that both suppliers exhibited similar fatigue survival probability at various cycles for 1.7 % strain. Weibull analysis is a method for modeling datasets, such as fatigue failure data. In this case, being applied to Nitinol fatigue data, Weibull analysis can be employed to determine wire endurance in use. A reliability goal must be defined properly in order to choose the ideal wire for a particular application.

Postmortem investigations of RBT samples include metallographic examination of the failed specimens; moreover, fracture surfaces may be analyzed using SEM and EDS analysis, or other comparable microscopy techniques [8]. Figures 10(*a*)–10(*e*) represent two wire segments with mating fracture surfaces; Fig. 10(*f*) displays the resulting chemical analysis of the nonmetallic inclusion

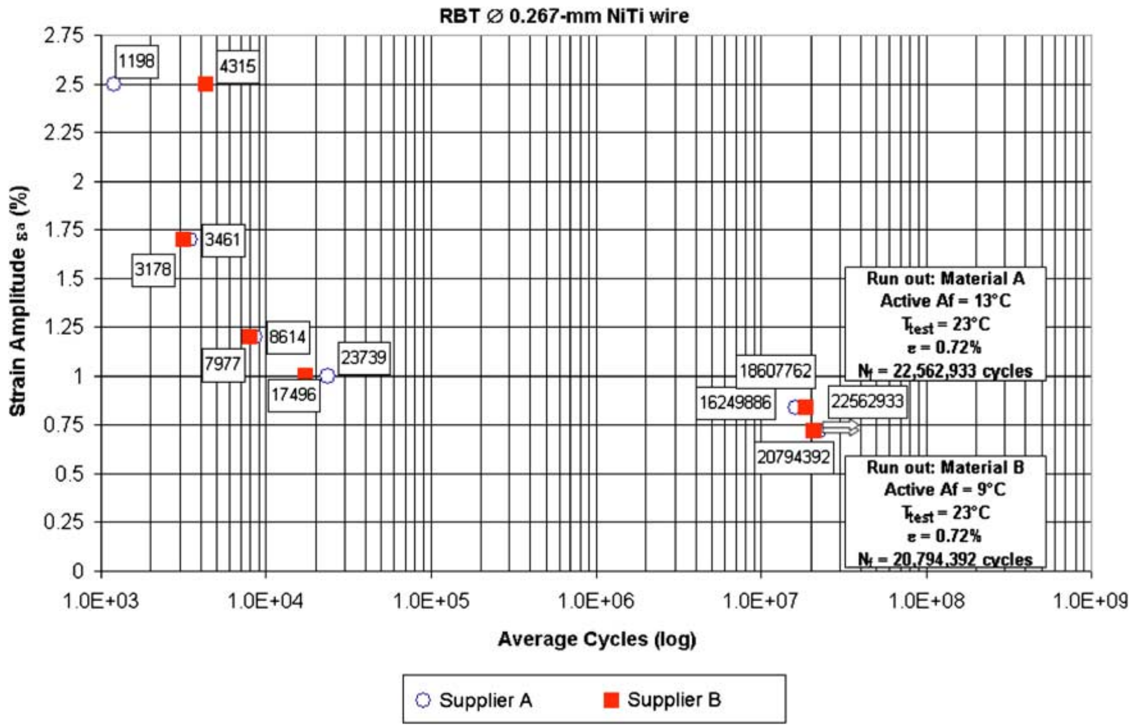

FIG. 8—*Rotary beam fatigue testing (RBT) data presented with ε-N curve with runout values noted.*

found at the initiation site (marked in side A) for Supplier A. The fatigue fracture area comprised about 55 % of the wire cross section. The inclusion had an angular shape and was of approximately 4.4 μm across. Figures 11(*a*)–11(*e*) characterize the mating initiation site of an RBT wire fracture found in material from Supplier B. Furthermore, Figure 11(*f*) shows the characteristic peaks of the defective region through EDS. In this case, the fatigue fracture area comprised of 42 % of the wire cross section. The nonmetallic inclusion is located at the wire surface of the initiation site (side A)

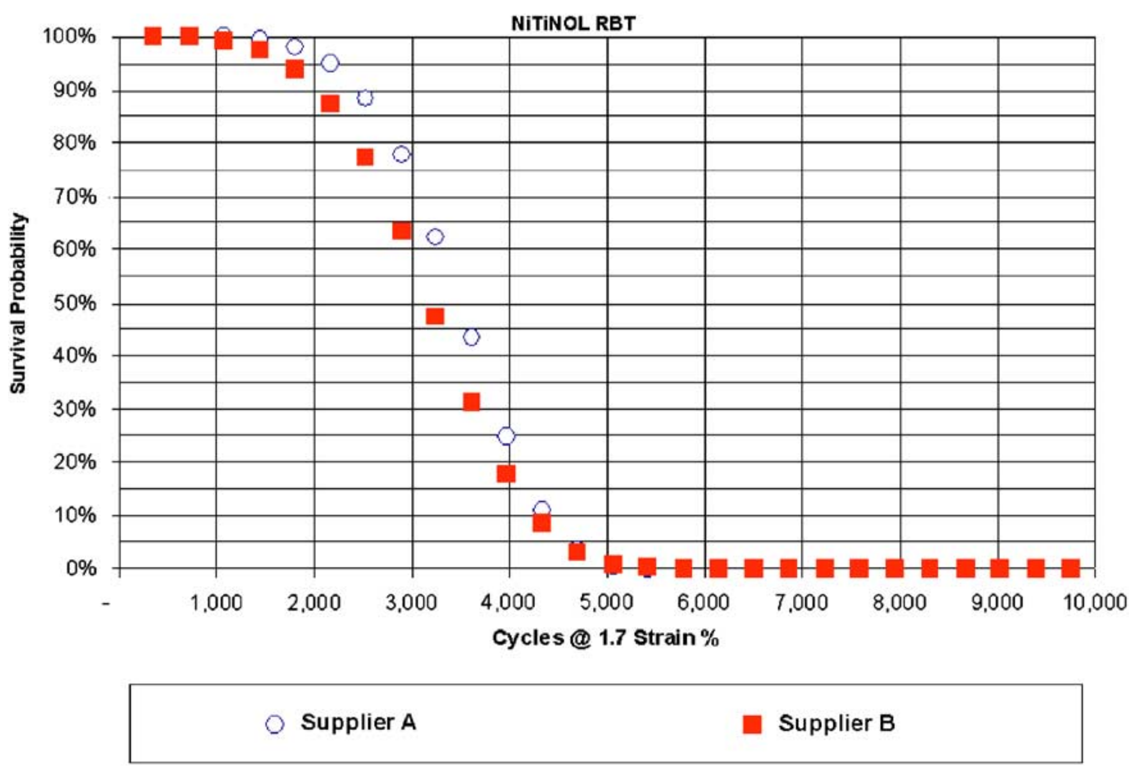

FIG. 9—*Survival plot studying 1.7 % strain amplitude.*

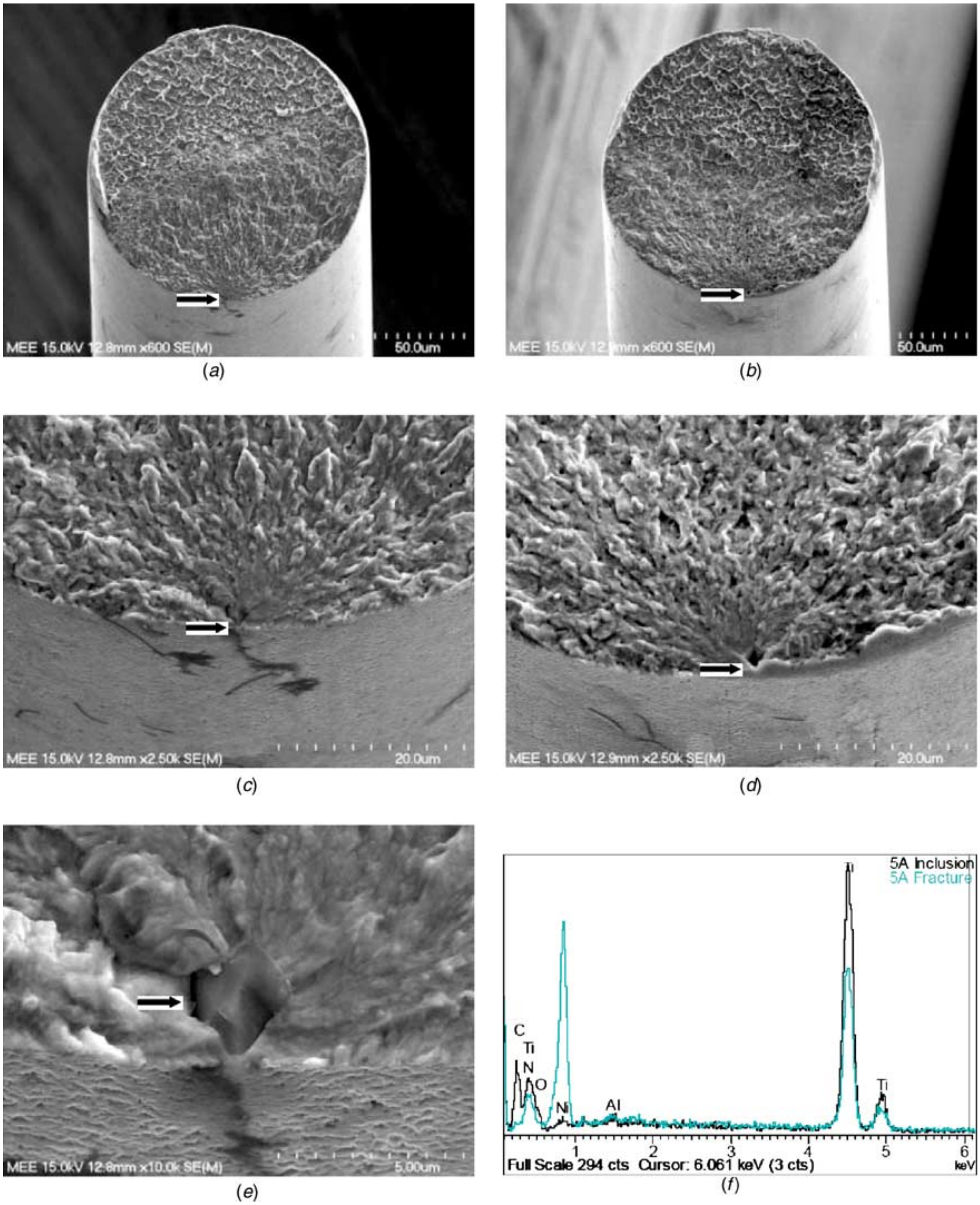

FIG. 10—*(a–e) Supplier A 0.127 mm fracture site.*

and has an angular shape of relatively 3.2 μm across. Fracture surfaces are generally on a flat and transverse plane while the stress is concentrated with striations evident in the material. For both suppliers, EDS analysis of the melt intrinsic defects indicate a variant of a titanium, carbon, nitrogen, and oxygen compound residing at the initiation zone; these results are in correlation with those found by Reinoehl. Figure 12 shows a grain boundary as the suspect stress raiser at failure found by Miyazaki [7].

Discussion

By understanding the cornerstones of material science relationships, this method of testing characterizes the interwoven properties of Nitinol alloys. Creating a designed experiment allows the ex-

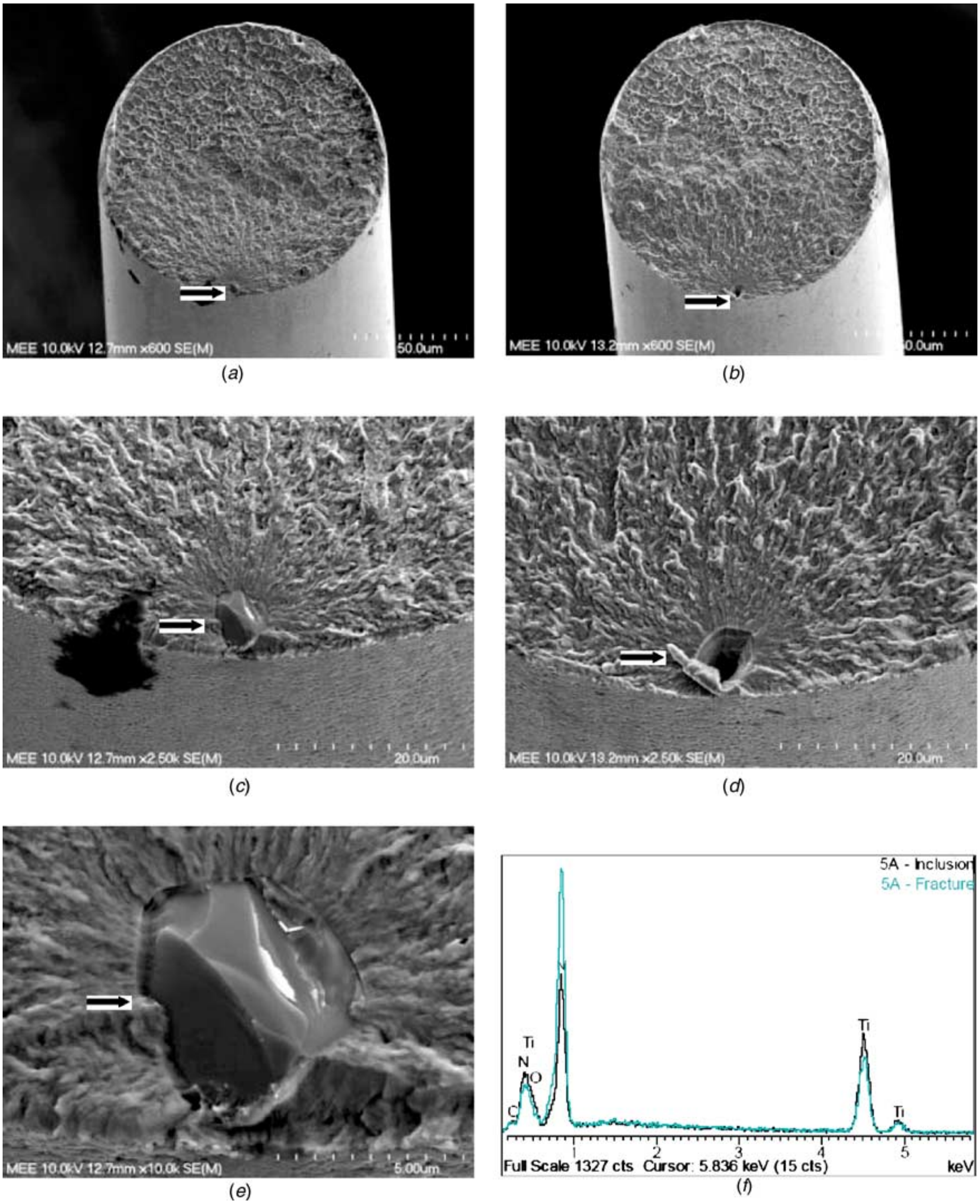

FIG. 11—*(a–f) Supplier B 0.127 mm fracture site.*

amination and subsequent characterization for an array of material in the functional condition. As described, appearances of defects near the surface are easily exposed and their effects become magnified through RBT.

There is a clear dependence of stress and strain with temperature change in Nitinol. This mechanical-thermal property link is well documented by the Clausius-Clapeyron relationship:

$$\frac{d\sigma}{dT} = -\frac{\Delta S}{\varepsilon} = -\frac{\Delta H}{\varepsilon T} \tag{2}$$

In this expression, σ is the uniaxial stress, ε is the transformational strain, ΔS represents the entropy of transformation per unit volume, and ΔH signifies the enthalpy of the transformation per unit volume [9]. The difference in testing temperature to Active A_f must be taken into account when heat

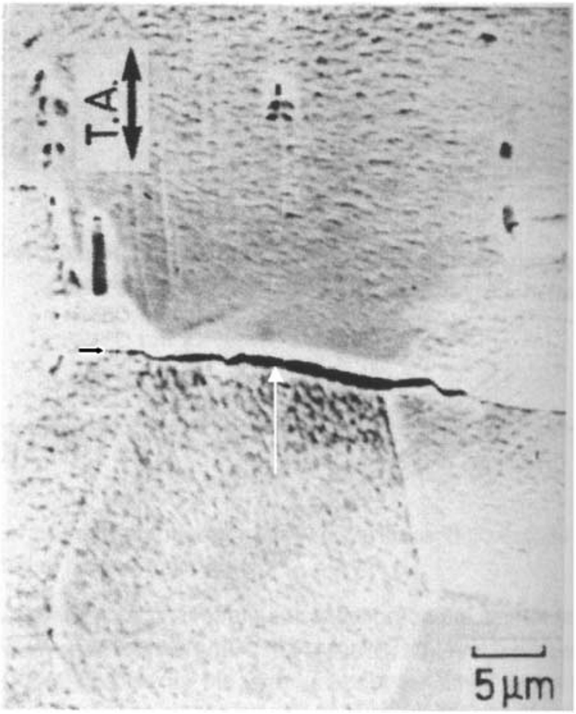

FIG. 12—*Grain boundary failure [7].*

treating samples as well as during testing. The tensile testing environment is critical; concern must be placed to ensure similar environments for RBT. After a shape-setting heat treatment, testing of the final Active A_f should be completed using a variation of the bend and free recovery test [10]. This method should be employed to provide the final Active A_f value, instead of differential scanning calorimetry. Essentially, one should aim to test with the difference in test temperature and Active A_f as shown in Eq 3 below:

$$\Delta T = (|T_{test} - Active\ A_f|) \tag{3}$$

Prior to experimentation, test termination criterion should outline (at a minimum) temperature limits, desired cycle runout, and wire breakage.

Conclusions and Recommendations

Fatigue information is considered a steadfast means for determining wire life in dynamic utility. To serve as a quality index, the fatigue behavior profiles and endurance limit values can be obtained through RBT. Of interest to both the wire manufacturer and the consumer, RBT offers a means for production quality control and makes possible the measurement of material performance on the basis of statistical models.

Continuing studies should not be limited to solid NiTi wire but should encompass DFT®[2] on the single-chuck design [11]. Strands and cables should be tested on a dual-chuck system and fracture surfaces should be studied for strain distribution through the cross section. If needed in future testing, a more advanced time meter display with an extended counter would aid in prolonged studies. There is also a limitation on strain imparted and center distance due to design of machine and having difficulty reaching small chuck-to-bushing distances for very high strain levels. Some testing protocols should be designed to study crack nucleation and propagation with partial testing conducted through a fraction of the predicted material lifetime. When testing, there should be a focus on subjecting samples to strain levels above and below the approximated SIM as hinted by the onset of

[2]DFT (Drawn Filled Tube) is a registered trademark of Fort Wayne Metals Research Products Corporation, Fort Wayne, IN.

the upper loading plateau from the tensile test data. This value is where an expected reversible martensitic phase transformation takes place.

Particular importance should be placed on micro-cleanliness and homogenizing melt practices as inclusions were found to be possible fracture initiation points. Particle morphology may affect crack propagation behavior and should be studied in detail. A correlation study should explore the orientation of inclusion particles and how they may have an effect on fatigue life. Since grain boundaries have also been known as suspect for stress concentrations, a relationship of grain sizes and length of grain boundaries to fatigue life may be studied to attempt to draw conclusions in relation to homogeneity and possible anisotropy effects. It must be mentioned that metallurgical limitations exist due to localized transformations of the material phase as a results of sample preparation. Using FEA, one may also determine the strain conditions evident in the material prior to and during fracture using RBT. By studying the various surface finishes of Nitinol wire, a window may be opened to the medical device engineer as far as material performance goes in a tailored product.

As aforementioned, rotary beam fatigue testing setup includes the equipment setup, initial strain calculations, sample preparation, monitoring of specimens during testing, and recording length of time for the duration of the test. The number of cycles the specimen experiences is subsequently calculated, thus providing invaluable insight on material response. The fractured specimens are further characterized and fracture surfaces are evaluated. The chemical composition of extrinsic defects may shed light on upstream processing avenues.

Comprehensive fatigue test reports encompass ε-N diagrams, survival plots, and include highlights of the notable features, anomalies, and trends.

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