Characterizing Fatigue and Fracture Response of Medical Grade Nickel-Titanium Alloys by Rotary Beam Testing

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ABSTRACT: With respect to a dynamic test protocol, fatigue information has been reliable in predicting wire wear longevity as well as critical structural integrity. It has been noted that the efficacy of modern medical devices are confined by the life cycle of the material of which they are composed. The aim of this study is to expound on the means of how rotary beam fatigue test methods merit attention as a viable characterization technique. By simulating alternating tension and compression states through rotary beam fatigue testing, it is possible to predict the life expectancy of Nitinol wires. Fatigue testing has been employed to characterize the influence of subtle changes in ingot inclusion content, chemistry variations of raw material, ingot transformation temperatures of Nitinol, and surface finish conditions. Arrays of specimens have been subjected to multiple strain levels, yielding calculations of the approximate stress/strain values of ultimate failure at the outer surface of monofilament wires. Test protocols were administered up to 100 million alternating cycles where desired. If preferred, fatigue testing may be conducted in temperature-controlled ambient air or liquid environments. Characterization of fracture surfaces has proven useful in evaluating the factors influencing failures. The utilization of fatigue data and fracture mechanics surpasses tensile testing in providing information to the design engineer. Results from studying flexural endurance, statistical Weibull life prediction analysis, fracture analysis, and a determination of stress/strain levels at the site of failure may prove useful in determining desired material properties for next generation medical devices.

KEYWORDS: Nitinol, wire, rotary beam, fatigue, NiTi, zero mean, strain

Introduction

Some common fatigue test methods are axial, bending, and torsional testing. In 2000, a study which used a wire diameter of 0.267-mm and controlled conditions for investigating a possible threshold at which inclusion morphology would impact fatigue performance was conducted. This previous work compared the melting practices, the last metal workings, and last heat treatment in the material; however, chemical composition, and surface conditions may also be explored. Figure 1 shows the comparison of inclusions found in various supplied material at $\Omega=2.032$-mm [1]. 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 non-homogeneous microscopic discontinuities which may inhibit slip and act as stress raisers. Another study utilized a tabletop testing device which uses oscillating loads to create a mean strain in fatigue Nitinol materials [2]. It was found that increased fatigue life was obtained for mean strains above 1.5%. In a rotary beam strain-controlled study, varying heat treatments and various test temperatures were applied. It was proven that in an isothermal strain-controlled environment, superelastic Nitinol was superior to stainless steels [3]. In this study, there is also mention of how fatigue-crack propagation analysis may be used complementary to the results from fatigue testing. A cyclic frequency of 1 000 revolutions per minute (RPM) 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.

![Backscattered Electron Image](image1.png)

![Backscattered Electron Image](image2.png)

FIG. 1--Examples of inclusions found in Nitinol with diameter 2.032-mm material [1].

Through experimentation, one may survey various alloys, suppliers, and processes utilized in the production of medical grade fine wire. When used in conjunction with standard tensile testing of medical grade Nitinol wires, rotary beam fatigue analysis allows testing parameters and environments to be adjusted to represent *in vivo* usage. In a similar light, this useful tool may be used as a quality check prior to shipment to end device manufacturers during process validation. While testing, the number of alternate bends and the time for ultimate failure can be measured through this type of experimentation. In some cases, anomalies upon the NiTi wire surface seem to be compounded due to the sensitivity of the testing equipment and the nature of targeted volume of material being tested. The results from rotary beam fatigue testing (as will be abbreviated RBT for this venue), may aid in the facilitation of developing medical grade “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 rupture. [4]

The flow of Nitinol material goes through the following fabrication chain all prior to RBT: melting, hot working, cold working, and shape-setting heat treatment. Any segment change may affect final material properties and give rise to other complications. By using materials with equivalent thermomechanical processing of Ti49.2 at%, Ni50.8 at% as a baseline, an attempt to derive differences between Niti wire properties and performance is possible. As will be explained further in detail, the largest tensile and compressive forces are exerted at the outermost fibers of a round wire during RBT. This test protocol doesn’t focus solely on the end use of Nitinol products but also was designed to be used as standardization of material quality.

During 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 & M’) acting in the plane of symmetry at the ends of the wire. In the following few figures (FIG. 2 – FIG. 5b), it should be noted that a rectangular cross section is substituted for the RBT round wire specimen. As depicted in Figure 2, 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, a similar means of reaction flexure occurs when a wire is implanted in the cardiovascular system. The upper surface is in compression, while the lower surface is in tension. The x-axis, also known as the neutral axis ideally exhibits zero stress/strain, while the y-axis is based on the axis of symmetry in plane bending [5].

FIG. 2--Solid wire in pure bending [5].

If the wire were to be divided up into many cubic units with faces 90° to each other and parallel to the three coordinate planes, they would be depicted as in Figures 3a and 3b.

FIG. 3a & FIG 3b--Note axes of rotation. A moment balance can be applied at equilibrium by assuming that resisting moments must be equal and opposite when a uniaxial stress component \( \sigma_x \) is applied. [5]

From this view, at any point a finite distance from the neutral axis is under flexural loading, and there is a state of uniaxial stress from the \( \sigma_x \) component. In Figure 3a, segment AB shortens, while A’B’ lengthens as \( M > 0 \). The upper face of the wire is in compression with \( \varepsilon_x \) and \( \sigma_x \) being negative (-). The lower face, with \( \varepsilon_x \) and \( \sigma_x \) being positive (+), indicates a tension state. The neutral surface, the surface parallel to the upper and lower faces of the wire, has \( \varepsilon_x = \sigma_x = 0 \). This surface intersects the plane of symmetry through arc of circle DE in Figure 4, and intersects a transverse section along the neutral axis ‘z’ from Figure 3b. The distance from any point to the neutral surface is noted as ‘y’, \( \rho \) signifies the radius of arc DE, and the central angle to DE is \( \theta \), 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 5a shows a strain wave form indicating peak-to-peak variation during a controlled flexure mechanical test through time. At any inflection point on the curve, the material is at maximum strain amplitude loading, with a zero mean strain along the constant slopes. During testing, the stresses in the material remain below the superelastic limit, as referenced in Figure 5b, so there is no permanent set present.

FIG. 5a--*Rotary cycle wave form* [3]. FIG. 5b--*No permanent deformation in the wire taking place* [5].

**Materials and Methods**

The Valley Instrument Company, Rotary Beam U-Bend Wire Spin Fatigue Tester (10-040) as shown in Figure 6 may be utilized to test Nitinol wire in the superelastic condition above the Active Austenitic Finish temperature (Active A<sub>f</sub>). RBT may be conducted in a temperature-controlled ambient air or in a liquid environment. A water flow system may also be used to control multiple testing systems. Solvent baths used in experimentation include Reverse-Osmosis water (RO H<sub>2</sub>O), 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 to 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 Figure 7. The specimen, with a known length, is mounted into the drive chuck system while the “non-driven” 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.
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 (exploratory research and development tests) research demands a minimum number of specimens to be from six to twelve [6]. Typical strain values may range from 0.6% to 2.1%; a strain level is considered low or high if it lies below or above the SIM (Stress-Induced Martensite) strain level, respectively. At high strain levels ten samples are typically chosen, due to the expected shortness of the fatigue life; in contrast, seven samples are tested at low strain levels. It is worth mentioning that at extremely low levels of strain, the time to failure is exorbitantly long. A test protocol may include using multiple strains which are calculated by using the starting bend radius and increasing the center distance.

The reversible load which 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. At fracture, the instrument electrically grounds the test sample and thus terminates the test automatically. Some material or processes may show significant differences regarding cycles to rupture, which is desirable in comparison of materials. In regards to lower strain levels, a desired run-out time expectancy determination becomes of concern. Reinoehl, et al set 20,000,000 cycles, which is approximately 3.9 days, as run-out [1]. In today’s testing scheme, 100 million cycles (19.3 days) seems to be more representative of long term testing. As follows in Equation 1, dimension analysis yields the number of cycles a specimen is subjected:

\[
\frac{3600 \text{revs}}{1 \text{min}} \times \frac{60 \text{min}}{1 \text{hour}} \times \frac{24 \text{hours}}{1 \text{day}} \times \text{#days} = \text{cycles}
\]

Keep in mind that the aim of this type of testing is to represent the fatigue a wire endures when implanted in the cardiovascular system. The average resting heart rate is approximately 60 beats per minute, which is 86,400 beats per day, and over 31.5 million times in a year. During an average lifetime, the human heart will beat more than 2.5 billion times [7]. Also, to simulate in vivo conditions, body temperature testing (~37°C) may be performed.

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 studied using SEM analysis. S. Miyazaki found that fractures occur at both inclusions and grain boundaries. Those which nucleated at grain boundaries were found through samples of Electron Beam Melting. Fractured surfaces which revealed TiC inclusions were found in material of Induction Melting using a carbon crucible. Large stress concentrations are expected to occur at both grain boundaries as well as near inclusions [8]. EDS analysis is used to determine the chemical composition of the interstitial particles.

**Experimental Results**

Nitinol fatigue test data is traditionally presented in the form of an $\varepsilon$-$N$ (strain-life) diagram as shown in Figure 8. The data shows a truncation at 20,000,000 cycles as the run-out parameter.
The dependent variable, fatigue life $N$, in cycles is plotted on the abscissa, using an arithmetic or logarithmic scale, as desired. The independently controlled variable, the maximum strain amplitude $\varepsilon_A$, is plotted on the ordinate typically with an arithmetic scale [6]. A regression line, or similar techniques, may also be fitted to the fatigue data. As the fatigue curve achieves a slope of zero and the endurance limit has been reached, the line now represents the fatigue strength at run-out. Replicate tests are designed to ensure good distribution in data acquisition. This type of frequency distribution curve of fatigue lives, taken at a given strain, for an array of samples, allows the variation of fatigue characteristics throughout a volume of material to be extensively studied [6].

![Nitinol Rotary Beam Fatigue Data](image)

**FIG. 8--RBT data presented with $\varepsilon$-$N$ curve with 20 000 000 cycles run-out.**

By creating survival probability plots, one is able to predict life for a specific parameter. Figure 9 is an example of high strain level testing. Weibull analysis is a method for modeling data sets containing values greater than zero, such as failure data. In this case, being applied to Nitinol fatigue data, Weibull analysis can predict the wire longevity and endurance in simulated use. The line graph suggests the survival probability of each wire manufacturer at various cycles. A reliability goal must be defined properly in order to choose the ideal wire for a particular application.
For RBT testing, fracture surfaces may be analyzed using SEM and EDS analysis, or other comparable microscopy techniques. Fatigue-crack propagation has also been studied using TEM [9]. Figure 10 shows typical fracture surfaces at inclusion sites. Fracture surfaces are generally on a flat and transverse plane while the stress is concentrated with striations evident in the material. The fracture surface is characteristic of a ductile fracture mechanism; in addition, severe necking and plastic flow is not present. Radial markings indicate a single initiation site where a nonmetallic inclusion resides in these specimens. These brittle, ceramic inclusions may either split, or remain on one side of a fractured specimen. For comparison, the size and composition of the inclusion are also measured. Typical extrinsic defects have been found to be a variant of a Titanium, Carbon, and Oxygen compound. Figure 11 shows a grain boundary as the suspect stress raiser at failure.

FIG. 9--Survival plot at 1.7% strain amplitude.
Discussion

By understanding the cornerstones of material science relationships, this method of testing characterizes the interwoven properties of an array of Nitinol alloys. Creating a designed experiment allows the examination and subsequent characterization of material in the functional condition. As described, appearances of defects near the surface are easily exposed and their effects become magnified through RBT. In traditional tensile testing, one measures the variability in stress at a given number of cycles (typically one, i.e. monotonic loading); in contrast, RBT measures the variability in cycles at a given strain. Furthermore, tensile testing generates vertical scatter between data, and fatigue testing produces horizontal scatter bars [10].
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:

\[
\frac{d\sigma}{dT} = -\frac{\Delta S}{\varepsilon} = -\frac{\Delta H}{\varepsilon T},
\]

(2)

In this expression, \(\sigma\) is the uniaxial stress, \(\varepsilon\) is the transformational strain, \(\Delta S\) represents the entropy of transformation per unit volume, and \(\Delta H\) signifies the enthalpy of the transformation per unit volume. The difference in testing temperature to Active Af must be taken into account when heat treating samples as well as during testing [11]. 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 Af should be completed be as accurately as possible. In doing so, a variation of the Bend and Free Recovery test should be employed to provide the final Active Af value, instead of Differential Scanning Calorimetry. Essentially, one should aim to test with same ‘\(\Delta T\)’ in test temperature and Active Af. Test termination criterion based on testing temperature limits, through achievement of a desired cycle of the failure criterion, and through wire breakage, is needed to be clearly defined prior to testing.

**Conclusions & Recommendations**

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

Continuing studies should not be limited to solid NiTi wire but should encompass DFT®1 on the single-chuck design [12]. 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 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. The purpose of this type of study would be to analyze differences in how some raw material suppliers prepare cross-sectional mounts longitudinally, and others transversely, during inclusion testing. 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

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1 DFT (Drawn Filled Tube) is a Registered Trademark of Fort Wayne Metals Research Products Corporation, Fort Wayne, Indiana
conclusions in relation to homogeneity and possible anisotropy effects. It must be mentioned that metallurgical limitations on sample preparation by possibly disturbing the local phase of material are possibly encountered. Using FEA, one may also predict 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 set-up includes the equipment set-up, 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 $\varepsilon$-N diagrams, survival plots, and include highlights of the notable features, anomalies, and trends.

References


