The Effects of Varying Active A_f Temperatures on the Fatigue Properties of Nitinol Wire

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Abstract

The influence of deformation temperature on mechanical properties of Shape Memory and Superelastic Nickel-Titanium (Nitinol) alloys has been studied and is well documented. In determining the effectiveness of a device when it is deployed and maintained under strain at ambient or body temperature, both the material properties and the environments encountered by the final device must be taken into account. In device design, it is important to understand the thermomechanical history of the wire supplied. However, data is lacking in terms of deriving a relationship between transition temperatures to a breadth of mechanical properties. Engineers of medical devices tailor the final material properties of wires used in a medical device application typically through a shape-setting process. Heat treating Nitinol wires in this type or a similar process aids in obtaining a desired shape as well as in reaching a target Active A_f prior to final surface preparation. These processing steps can affect fatigue life. By applying alternating tension and compression states through rotary beam fatigue testing, one may generate data to predict the life expectancy of Nitinol wires. Specimens with varying Active Austenitic Finish temperatures have been subjected to multiple strain levels. The relevancy of this type of study, which involves the generation of fatigue data, supplements thermal and mechanical data to provide design engineers additional information for the development of Nitinol wire implants.

Introduction

Through thermomechanical processing, including cold drawing, inter-pass annealing, and training heat treatments, superelastic medical grade Nitinol wire is produced. After the shape-setting or straight annealing processes, final Active Austenitic Finish values are instilled in Nitinol materials. This process directly affects Nitinol material structure, properties, and final performance. The implication of this thermal property and its influence on fatigue life for a particular environment will be explored through rotary beam fatigue testing (RBT). In a previous study, the fatigue behavior of Nitinol tubing exposed to various aging heat treatments produce materials with an Active A_f of 25°C +/- 2°C [1]. Using a known mean strain, with differing alternating strains, no major trends were discovered on the fatigue of these materials. The current study focuses on 0.323 mm Nitinol (Ni 55.8wt%-Ti; Ti49.2at%, Ni50.8at%) wires in the zero mean strain condition. As the wires rotate, the outer surfaces are at the apex are exposed to a cyclic stress reversal of tension and compression states. The aim is to decipher whether a trend of fatigue life is achievable through manipulating the Active Austenitic Finish of a product, through similar processing, while offering results for different testing temperatures. Moreover, as engineers encounter material selection issues, importance must be placed on the relationship between mechanical and the thermal properties. The employment of rotary beam fatigue testing as a tool for life prediction has proven useful in drawing conclusions on the tour of the wire item being implanted and in exploiting the association of temperature dependency of Nitinol wire.

As studied by Pelton et al, changes in mechanical properties of Nitinol are closely linked to changes thermal properties by the Clausius-Clapeyron relationship:

$$\frac{d\sigma}{dT} = -\frac{\Delta H}{\varepsilon T} \qquad (1)$$

In this expression, σ is the plateau stress, T signifies test temperature, ε is the transformational strain, ΔH represents the enthalpy of the transformation per unit volume. As shown in Figure 1, a superelastic Nitinol wire with an Active A_f of 11°C exhibits an increase in upper plateau stress as the difference between test temperature and Active A_f increases [2].

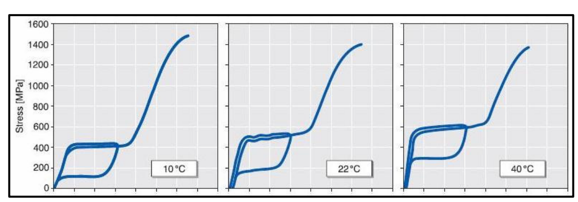

Figure 1: The effect of test temperature on Nitinol wire with an Active A_f of 11°C [2].

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The goal of this testing approach is to offer those engineers, working with medical devices, additional information on the temperature sensitivity of Nitinol superelastic wires. Tensile testing completed on wires for the current study followed a similar trend as found in previously published works. It has been found that temperatures for tensile testing as well as for fatigue testing are of the utmost importance. Fine-tuning the proper Active A_f is equally critical in the assessment of Nitinol properties. In addition to tensile testing of superelastic NiTi wires, the Clausius-Clapeyron relationship is applied to RBT. By utilizing six different ΔT of test temperature and Active A_f, a correlation may be drawn to tensile test data.

Figure 2 shows an inclusion found in supplied material at Ø=2.16 mm. These non-homogeneous microscopic discontinuities are found as either single particles or long stringers, well dispersed within the NiTi matrix. Scanning Electron Microscopy-Backscattered Electron Imaging (SEM-BEI) shows the longest dark feature found. Within the testing volume, features are a combination of inclusions and voids for a multitude of sizes. The particular inclusion in Figure 2 had more titanium than bulk of the surrounding NiTi magnified at 5000X based on Energy Dispersive X-ray Spectroscopy (EDS).

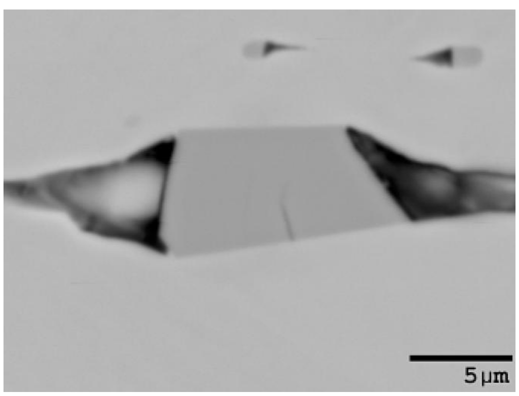

Figure 2: Typical feature found on 2.16 mm raw material at 5000X.

Materials and Methods

Nitinol round wires, from the same section of ingot, were cold worked from 2.16 mm to 0.323 mm with nominally 45% reduction in area on the final drawing die sequence. The material was then straight annealed to exhibit the following Active A_f values: 3.20°C, 12.0°C, and 20.8°C, as tested by bend and free recovery. The straight annealing process for these three material conditions was completed with the same inert atmosphere, time at temperature, and tension. Heat treating temperature was the only variable adjusted in processing of the Niti wires. As well documented, the dependence of stress and strain with temperature change in Nitinol materials follows the Clausius-Clapeyron relationship [3]. The relationship suggests that the difference in testing temperature to Active A_f must be taken into account when heat-treating samples and subsequently during testing.

Tensile testing and RBT testing were conducted at nominally 22.0°C and 37.0°C; these temperatures were chosen as being common testing and operation temperatures of final medical-grade wire devices. A Positool Rotary Beam U-Bend Wire Spin Fatigue Tester (10-040) was employed to evaluate the fatigue performance of the Niti round wires. Testing was completed in a temperature-controlled water bath. The following strain levels (%) were tested: 0.80, 0.90, 1.00, 1.50, 2.00, 2.50. At the highest strain level of 2.50%, ten specimens were tested; seven samples were tested on the remaining strain levels. Test completion was based on either wire fracture or by reaching a run out criterion for each strain level. Test protocols allowed the wire to be rotated up to 100 million alternating cycles. The material is cycled at a constant frequency of 3,600 revolutions per minute (RPM) [4].

Experimental Results

Six ΔT values were evaluated in this experiment. The ΔT was calculated as shown in Equation 2 below:

$$\Delta T = (| T_{test} - Active\ A_f\ |) \qquad (2)$$

The temperature differences ranged as follows: 1.20°C, 10.0°C, 16.2°C, 18.8°C, 25.0°C, and 33.8°C. Results of room temperature mechanical testing are found in Table 1. In Table 2, the mean mechanical properties of the same materials were tested at body temperature. During tensile testing, the material was cycled to 8% strain, and then returned to 0% strain, then pulled to failure. The upper plateau stresses were measured at 4% offset. The 22°C tests were completed in ambient air while the 37°C tests were conducted in an environmental chamber.

Table 1: Mean Mechanical Properties at Room Temperature

Active A_f (°C)	Upper Plateau Strength (MPa)	Lower Plateau Strength (MPa)	Permanent Set (%)
3.20	575	179	0.14
12.0	561	178	0.16
20.8	552	188	0.24

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Table 2: Mean Mechanical Properties at Body Temperature

	Upper Plateau Strength (MPa)	Lower Plateau Strength (MPa)	Permanent Set (%)
3.20	619	275	0.26
12.0	607	298	0.18
20.8	603	305	0.17

The following graph in Figure 3 plots the data from the table to better represent the effect of test temperature on plateau stresses. There is an approximate 7% - 8% increase in upper plateau stress with a 15°C temperature change. One can deduce that the material stresses decrease with increasing Active A_f, or in other words, upper plateau stresses are directly proportional to the ΔT.

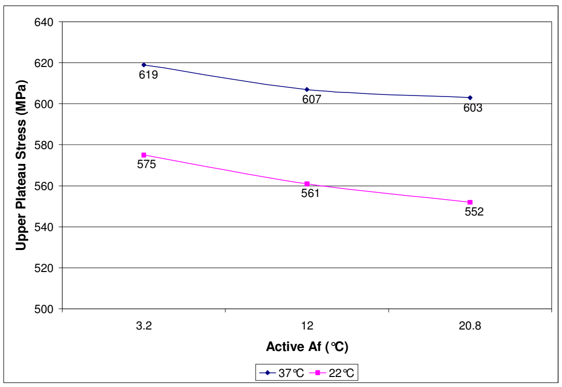

Figure 3: Effect of test temperature on upper plateau stress.

Nitinol fatigue data is typically plotted on an ε-N curve as shown in Figures 4 and 5. At body temperature, the mean fatigue life is inversely proportional to the ΔT between Active A_f and test temperature at selected strain levels. As expected, a similar trend was discovered when the material was cycled at body temperature. These trends coincide with the results of tensile testing. From the data, one can deduce that if a wire were to have a greater stress state, then it would be more prone to failure at a lower number of alternating cycles.

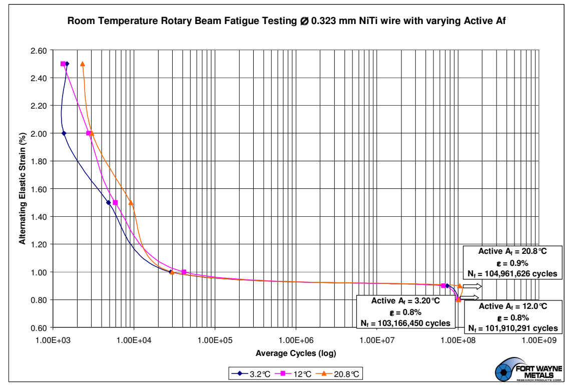

Figure 4: Room temperature RBT testing of 0.323 mm NiTi wire.

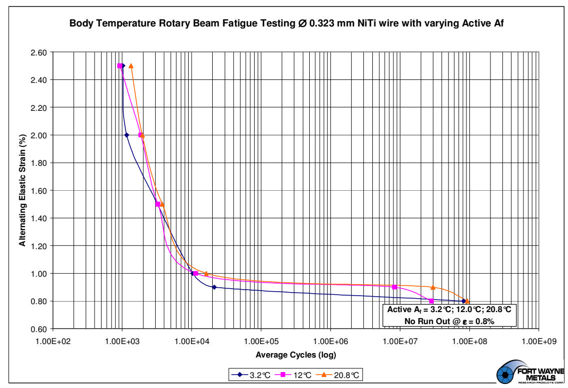

Figure 5: Body temperature RBT testing of 0.323 mm NiTi wire.

At room temperature, the 12.0°C Active A_f materials reached run out at 0.80% strain, as indicated by the arrow. The 20.8°C Active A_f materials experienced run out during 0.90% strain testing. Trends drawn from the tensile and RBT testing at both room temperature (RT) and body temperature (BT) are listed in Table 3.

Table 3: Data Trends

Active A_f	3.20°C	12.0°C	20.8°C
Loading Plateau Stress	RT < BT	RT < BT	RT < BT
ΔT(|22°C-Active A_f|)	18.8°C	10°C	1.2°C
ΔT(|37°C-Active A_f|)	33.8°C	25°C	16.2°C
Mean Fatigue Life*	RT >> BT	RT >> BT	RT >> BT

*Observed at selected strain levels.

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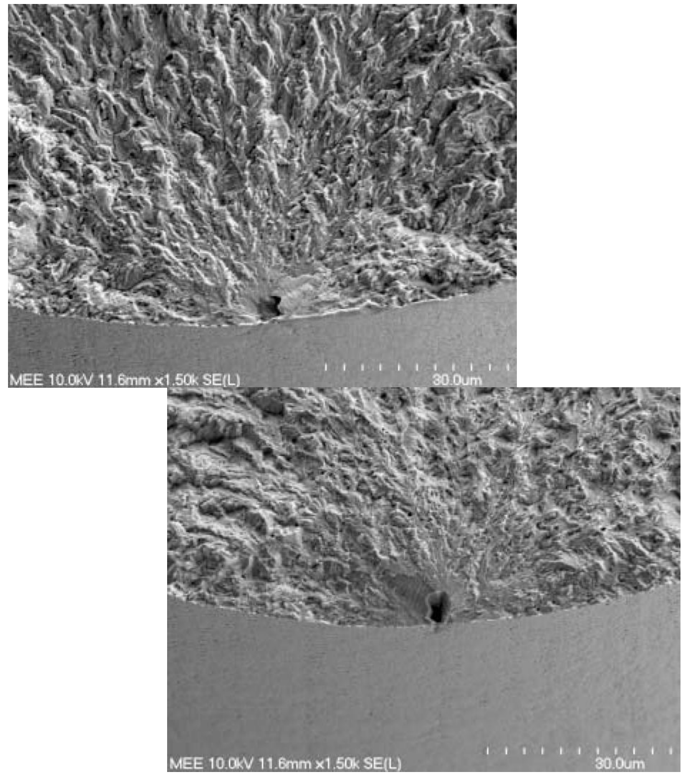
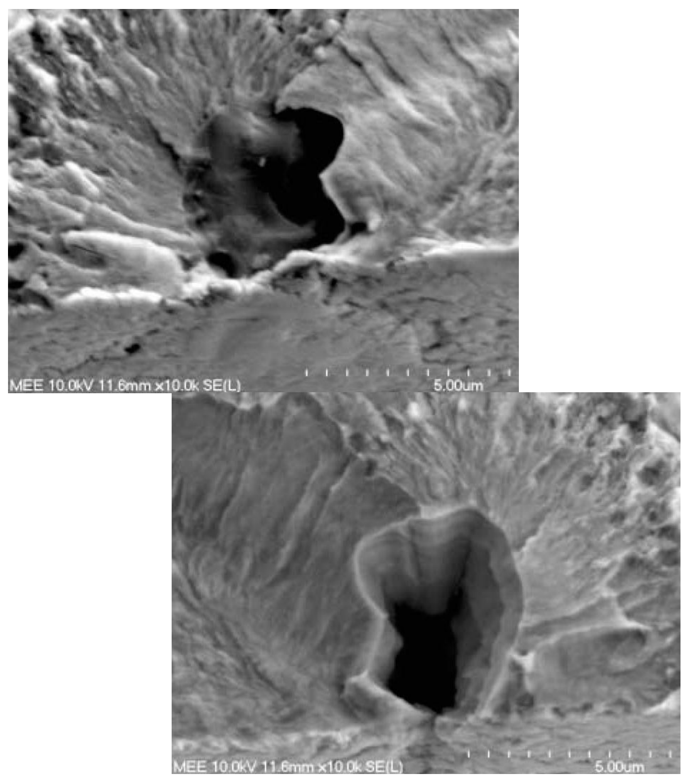
By utilizing SEM-BEI and employing the EDS function, fracture surfaces were thoroughly evaluated for morphology and chemical composition and compared. Fracture surfaces are generally on a flat and transverse plane where the stress is concentrated, with striations evident in the material. No significant gross plastic deformation such as necking or bending was observed. In addition, the fracture surfaces were typical of fatigue fracture mechanisms and partly due to ductile fracture mechanism for the remainder of the cross section. The extrinsic structures found exhibited varying levels of titanium, carbon, oxygen, and lesser amounts of nickel of varying size. Radial markings on the fracture surfaces indicate a single initiation site with a nonmetallic inclusion at the fracture origin; other surfaces had radial markings on the fracture surfaces indicated with multiple crack initiation sites within a small area on the surface of each sample.

The following images (Figures 6 & 7) show mating sides (A & B) of wire segments broken through fatigue fracture. Figures 6a and 7a present an inclusion embedded in the surface while Figures 6b and 7b show a cavity from the missing defect. The sample was heat treated to have an Active A_f of 20.8°C, and was RBT evaluated at 0.80% alternating strain at body temperature. EDS analysis in Figure 8 displays the spectrum of a predominately Ti and C compound.



Figures 6a and 6b: Mating fracture sides A & B at 1500X.



Figures 7a and 7b: Mating fracture sides A & B at 10000X.

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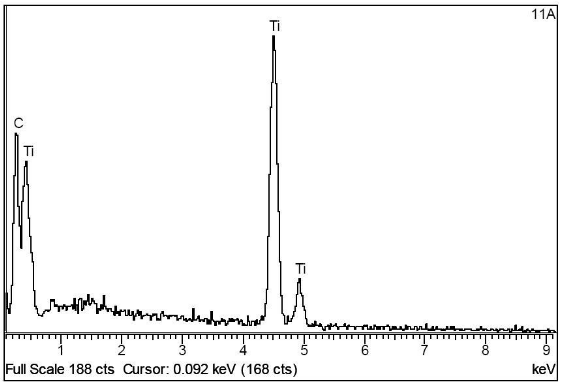
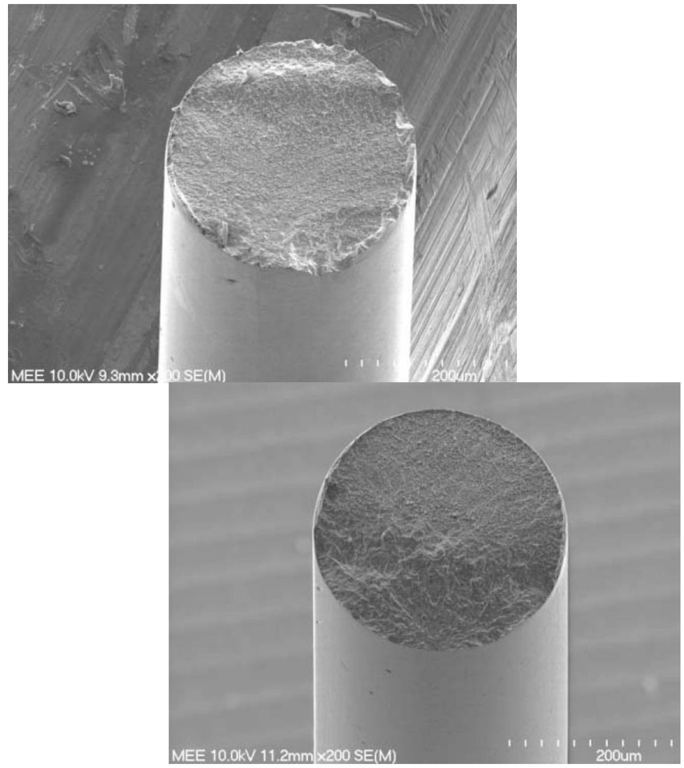


Figure 8: EDS spectra of inclusion matter.

For most wire samples, regardless of alternating strain level, testing temperature, and Active A_f, the fracture surfaces exhibited inclusions and radial markings. At high strain levels, the outer surface of the wire was rough and had jagged edges, as in Figure 9a, while at low strain levels, the outer roundness of the wire was intact, as indicated in Figure 9b. Some brittle, ceramic inclusions (Figures 10a & 10b) divided and could be located on both sides of the mating surfaces. In the rare instances where inclusions or pre-existing discontinuities were absent in observed origin area, small secondary cracks (Figure 11) were identified on the wire fracture surface. When an angular pit was observed, but no inclusions were found, an inclusion may have once resided in that location, but had fallen out after fracture. Previous studies have found similar defects [5, 6].



Figures 9a & 9b: Rough outer wire surface at high strain level 2.50% and smooth outer wire surface at low strain level 0.80% at 200X.

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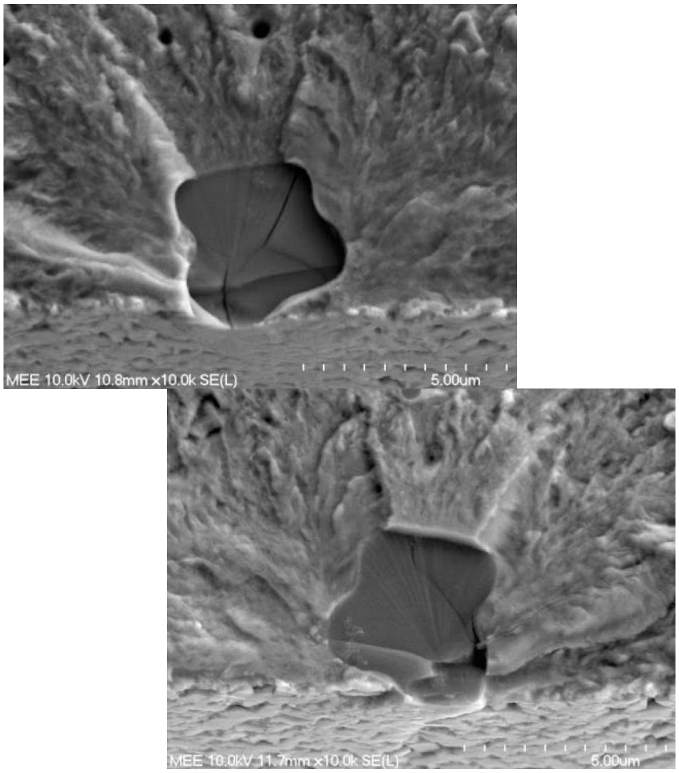

Figures 10a & 10b: Side A and B of 12.0 BT 0.80% brittle inclusion split and is located on both fracture surfaces at 10000X.

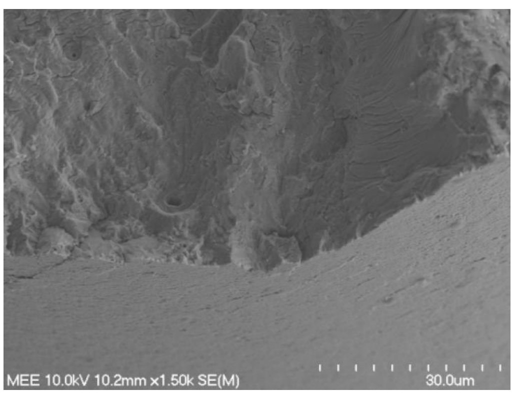

Figure 11: Small secondary cracks found on fracture surfaces at 1500X.

Discussion

As general trends indicate, the fatigue life at room temperature was much greater than those samples tested at body temperature. In addition, the plateau stresses of the room temperature tested specimens were less than those tested at body temperature, as derived from tensile testing. Both observances are concurrent with the implications of the Clausius-Clapeyron relationship. The stress-induced Martensite (SIM) strain level is traditionally approximated by the onset of the loading plateau from the σ-ε curve. At this strain level, the material will undergo a phase transformation in which localized Martensitic structures appear in the parent volume of material being tested. The dissimilar heat treating temperatures should shift this value among samples. The material which exhibited the best RBT wear longevity was found in material with the smallest Active A_f to testing temperature difference ($\Delta T=|1.2°C|$). Material with largest temperature spread ($\Delta T=|33.8°C|$) had the lowest number of alternating cycles completed for selected strain levels.

Summary and Conclusion

In an aforementioned study, Nitinol tubing was used in an attempt to draw conclusions of material properties with the same Active A_f [1]. In the current study, Niti round wires were under investigation, thus avoiding testing issues encountered by Lopes et al. The premature failure of the tube was attributed to flaws on the tube ID; conversely, through testing round wire specimens, these errors were eliminated. Moreover, in deriving sheer property relationships, the solid round wire provided consistent results due to the internal stresses being across a constant cross section.

Continuing studies should centralize testing with greater sample set near the SIM strain levels. Data sets accumulated from this type of testing would be more representative of the critical phase transformation of the matrix structure, which produce dramatic changes to the material properties and fatigue performance. Run out was not achieved for all samples in this preliminary work; however, verification of this introductory work is underway. Based on ε-N curves generated, testing at high alternating strain levels yielded that increasing strain levels incurred in the sample are inversely proportional to the number of completed alternating cycles as expected. When testing in intermediate strain levels near the SIM strain level, the values strayed due to partial localized transformation of Austenite to Martensite structures, but mostly followed a similar trend as other strain levels. When specimens are prepared properly, DSC testing may be used as an indicator of phase transformations of Austenite to Martensite and aid in determining the possibility of R-phase appearance. Finally, phases and precipitates should be determined at fracture surfaces.

When used in conjunction with tensile testing results, the values obtained through flexural endurance testing are critical to the final device design. Through preliminary results, it has been noted the fatigue life of materials tested at ambient temperature and body temperature have shown dependency to Active A_f. With respect to RBT, fatigue life and ΔT are inversely proportional. A wire with the smallest difference in

Active Austenitic Finish temperature and testing temperature has shown to withstand more alternating cycles at selected strain levels when compared. Additionally, the wire with an Active A_f furthest from the chosen testing temperature exhibited higher loading plateau stresses through tensile testing, and subsequently lower fatigue life. Optimizing the balance of plateaus, test temperature, Active A_f, and fatigue performance will guide the medical device engineer in choosing the most suitable Nitinol wire.

Acknowledgements

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