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A Study of the Effect of Diameter on the Fatigue Properties of NiTi Wire

Dennis W. Norwich and Audrey Fasching

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It is a generally held belief that controlled strain fatigue life is independent of wire diameter. This has led to the widespread use of general S-N or ε-N curves for engineering metals. Independence of wire diameter on fatigue life may not hold true for Nitinol. If so, care must be used when applying generic Nitinol S-N or ε-N curves to product design. To provide a more comprehensive understanding of this issue, a comparison study was conducted on the fatigue properties of various diameters of NiTi (Ti-55.8/55.9wt.%Ni) wire drawn from the same starting material. ε-N curves were generated for various diameters. The wire diameter reduction from 0.030″ to 0.010″ increased the 0.8% strain 80k cycle survival rate from 35 to 96%. For the scope of this study, all factors that may have an effect on fatigue life except for wire diameter were held constant.

Keywords controlled strain, fatigue, Nitinol, rotating beam fatigue test, S-N curve, ε-N curve

1. Introduction

The use of Nitinol components in the medical device industry has increased as engineers and designers have become more familiar with the unique pseudoelastic (commonly called superelastic) and shape memory properties of the binary alloy (Ref 1). Other industries such as the automotive, military, and commercial sectors are also taking advantage of the benefits of Nitinol that outweigh the high relative cost of the material (Ref 2).

As more engineers and designers use Nitinol in their products, the need for published data on the properties and performance of the material becomes more important. For stainless steel alloys there are countless volumes of credible published data to assist in the design process. This is especially true of fatigue data, which has proven to be predictable and consistent for the many available steel alloys. For Nitinol, there is comparatively little fatigue or S-N or ε-N data that has been published and proven out through the design and test process (Ref 3). Engineers and designers are left with little credible data to use on which to base their designs.

For a specific stainless steel alloy, the published fatigue properties are generally independent of material diameter or condition. Thus, published data can be used to accurately predict the life cycle of a product. This paper will research the fatigue properties of different diameters of Nitinol wire drawn from the same starting material to determine if the same consistency of fatigue life holds true for Nitinol as it does for steel. Every effort was made to keep all factors other than wire diameter constant throughout this experiment. This reduces the chance of any property other than wire diameter affecting the fatigue life of the wire.

2. Materials and Experimental Procedures

The fabrication process detailed below was designed to simulate that of a typical medical device that exhibits superelastic properties at room temperature. All of the samples for this study were produced from one lot of 0.052″ diameter wire (Ti-55.8/55.9wt.%Ni) which was cold worked and drawn to the following final diameters: 0.030″, 0.025″, 0.020″, 0.015″, and 0.010″. Each wire size was drawn with a similar drawing schedule to yield approximately 45% cold work on the final material. The test samples were shape set using the same heat treatment process: tooling, time, temperature, and heat source (Ref 4). After heat treatment, the parts were chemically etched, electropolished, and passivated. This surface treatment process reduced the diameter of each wire by 0.001″.

Fatigue testing was conducted using a rotating beam fatigue tester (RBT) with a zero mean 0.8% maximum alternating strain, as depicted in Fig. 1 and 2. Strain was determined by the diameter ratio methodology where ε = radius of wire/radius of curvature to neutral fiber. The samples were rotated at a constant speed guided in a test block machined with a radius to provide the level of strain desired. The fixture can test up to 10 samples at a time and counts revolutions on each sample individually using a laser counter. One hundred samples of each wire size were tested. The test was run to breakage or was suspended at 80,000 cycles. The results of this testing are shown in Fig. 3.

The RBT test is designed to provide comparative results for materials processed with different parameters at the same or varying strain levels. Tests were performed in a circulating 37 °C water bath at 1000 rpm to eliminate any heating effect

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Dennis W. Norwich and **Audrey Fasching**, Memry Corporation, Bethel, CT 06801. Contact e-mail: dennis_norwich@memry.com.

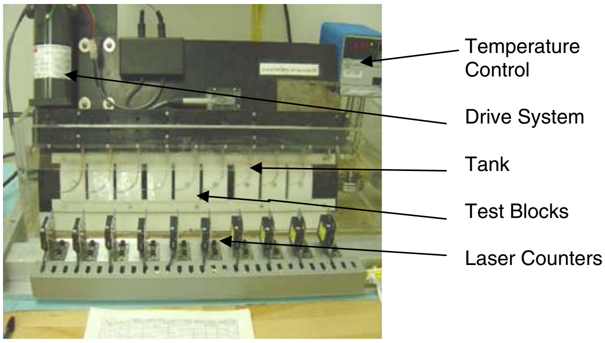

Fig. 1 Rotating beam fatigue tester

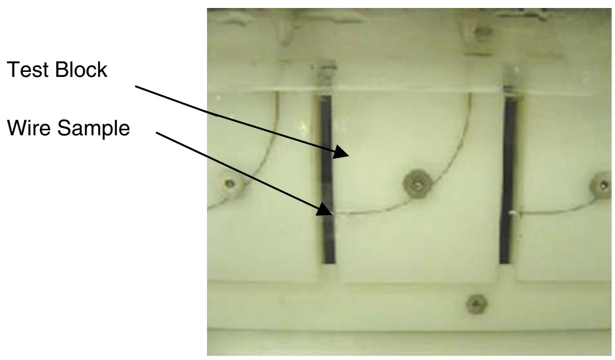

Fig. 2 Rotating beam test block

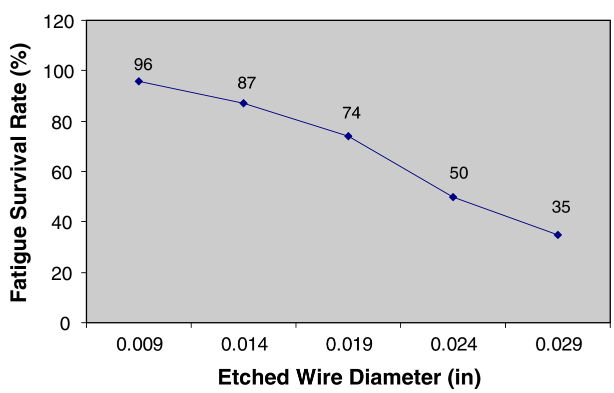

Fig. 3 Wire diameter vs. fatigue survival rate beyond 80k cycles at 0.8% strain

created in the specimen due to dissipative processes during cyclic loading (cyclic stress induced formation of martensite). The heat transfer from the specimen to the test environment helps to counteract this process (Ref 5).

Strain versus number of cycles to failure (ε-N) curves were generated for the 0.025″ and 0.010″ diameter wires. These wires were etched and electropolished to 0.024″ and 0.009″ final diameter and tested with the same rotating beam tester described above. Test blocks were made for each wire size to provide strains of 2.4, 1.8, 1.0, 0.8, and 0.65%. Three samples of each wire size were tested at each strain level. The test was

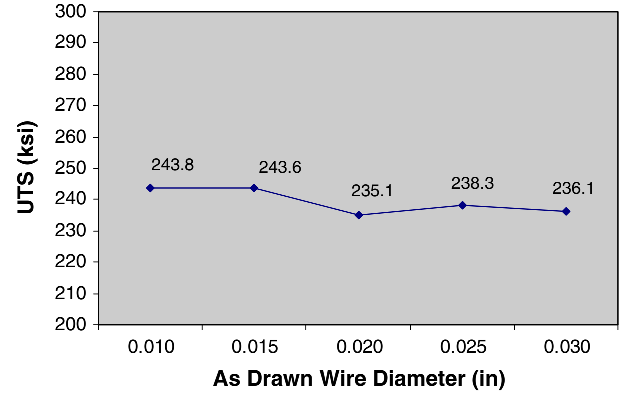

Fig. 4 Wire diameter vs. UTS as drawn

run to failure or suspended at 10 million cycles. The results of this testing are shown in Fig. 9.

Tensile tests were conducted on using an electromechanical tensile testing load frame with a 5 kN load cell as per ASTM F 2516-05. Tensile tests to failure (TTF) was performed on cold worked and shape set wire samples, while 6% strain tests were performed on only the shape set samples. Tensile testing was performed at 22 °C in air.

Differential scanning calorimeter (DSC) tests were conducted on cold worked and shape set parts per ASTM F 2004-3 to determine transformation temperature. Austenite finish temperature (A_f) was determined to be ~11 °C.

An outside laboratory was used to perform metallographic inclusion analysis on samples of each cold worked wire size. Metallographic mounts were created and examined using an SEM and an analytical computer program to measure and count inclusions. Five samples of each wire size were analyzed in both the longitudinal and the transverse directions for inclusion size, number of inclusions, and area fraction of inclusions.

3. Results and Discussion

The purpose of this study was to investigate the effect of diameter on the rotational bending fatigue life properties of Nitinol wire. Memry conducts an 80,000 cycle test at 0.8% strain as part of a wire fatigue qualification protocol. In this test, samples are fatigued at 0.8% strain until failure or 80,000 cycles. The results are reported as the percentage of parts exceeding the 80,000 cycle limit. Over years of data gathering, it has been noted that the smaller diameter wires survive this test at a much higher rate than the larger diameter wires. The results of this study corroborate and quantify this observation (see Fig. 3).

Although every measure was taken to ensure consistency in processing, basic tensile data were collected to confirm uniformity. Ultimate tensile strength (UTS) for the as-drawn wire for each wire size was determined and plotted in Fig. 4. The upper and lower plateaus (6% curves) for each heat-treated wire size are shown in Fig. 5 (Ref 6). A typical 6% stress-strain curve for a heat-treated test sample is shown in Fig. 6. Figure 7 depicts the stress-strain curves for each wire size to 2.4% strain, the maximum strain used for the fatigue study. Figure 8 is a

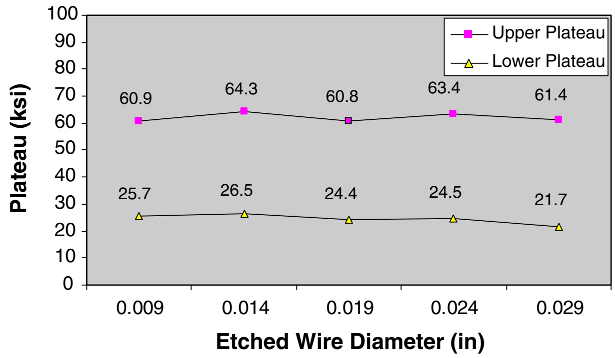

Fig. 5 Wire diameter vs. upper and lower plateau

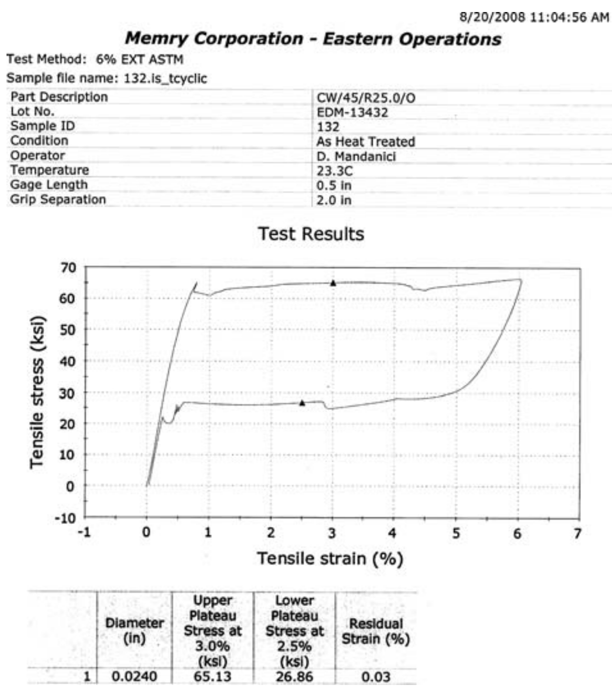

Fig. 6 The 6% curve of as heat-treated wire

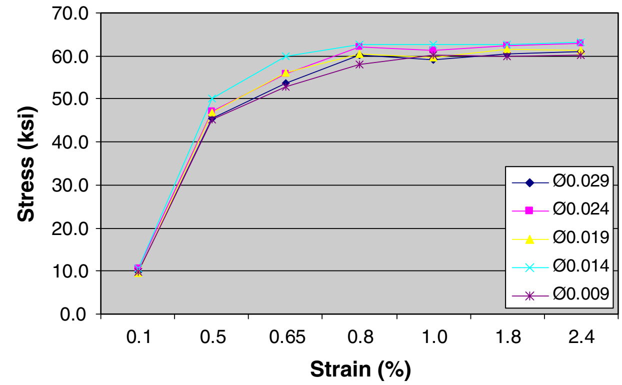

Fig. 7 Stress-strain curve for each wire size

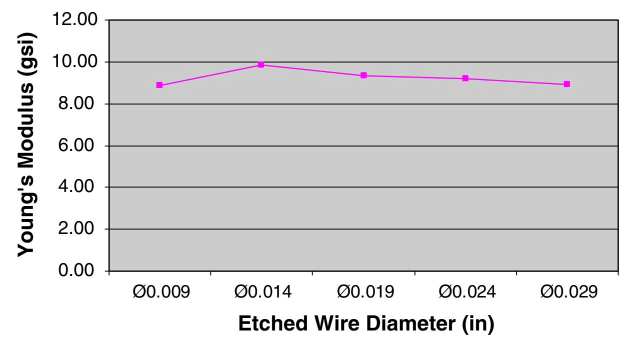

Fig. 8 Young's modulus vs. wire diameter

plot of Young's modulus for each wire size. The tensile data exhibit only small variations across the wire sizes. This validates the consistency of preparation of the test samples for each wire size.

ε-N curves were generated for the 0.024″ and the 0.009″ diameter wires used for this investigation. The results, shown in Fig. 9, were generated using three samples per strain level. The data indicate an increased fatigue life of the 0.009″ over the 0.024″ diameter wire. More data over a larger range of wire sizes will need to be collected to determine the extent of this effect.

An SEM image of a typical fatigue failure generated during this study is shown in Fig. 10. On the image, the fracture initiation site, likely an inclusion, is indicated by an arrow. This fracture surface is typical of fatigue failures, where the crack radiates out from the initiation point and is followed by a region of microvoid coalescence which indicates that final failure occurred by overload (Ref 7).

It is generally accepted that inclusions, especially those located at or near the surface of the wire, act as stress risers and initiation sites for fatigue cracks. Metallographic examination provides quantitative analysis of the inclusion size and content for the wire samples. The primary inclusions found in Nitinol alloys are titanium carbides (TiC) and intermetallic oxides ($Ti_4Ni_2O_x$). The titanium carbides tend to be small, uniform, and evenly distributed while the intermetallic oxides ($Ti_4Ni_2O_x$) are blocky and unevenly distributed throughout the matrix.

Inclusion analysis requires the examination of mounted and polished metallographic samples. Because inclusions can be unevenly distributed and analysis is conducted on a small area, the samples may not be representative of the entire matrix. The most useful data come from examination of multiple samples from the same material, but this is costly and time consuming.

Area fraction of inclusions and the largest inclusion size in both transverse and longitudinal directions are the most meaningful data gathered in the analysis when relating to fatigue properties of a material. A plot of the inclusion area fraction for each wire size in both the longitudinal and transverse directions is shown in Fig. 11. Since inclusions are fatigue initiation sites, a lower area fraction of inclusions should correlate to better fatigue performance. This plot did not correlate well with the cyclic fatigue data. The five largest inclusions for each wire size in both the longitudinal and transverse directions are plotted in Fig. 12. The trend lines show that transverse inclusions tend to get smaller as wire diameter decreases, while longitudinal inclusions tend to form stringers and elongate in the drawing direction. This phenomenon

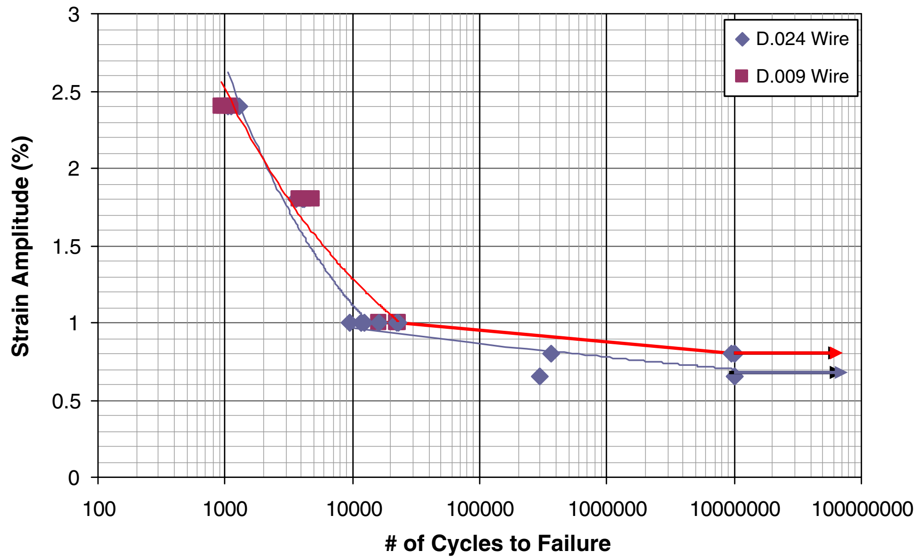

Fig. 9 ε-N curve 0.009″ vs. 0.024″ diameter heat-treated wire

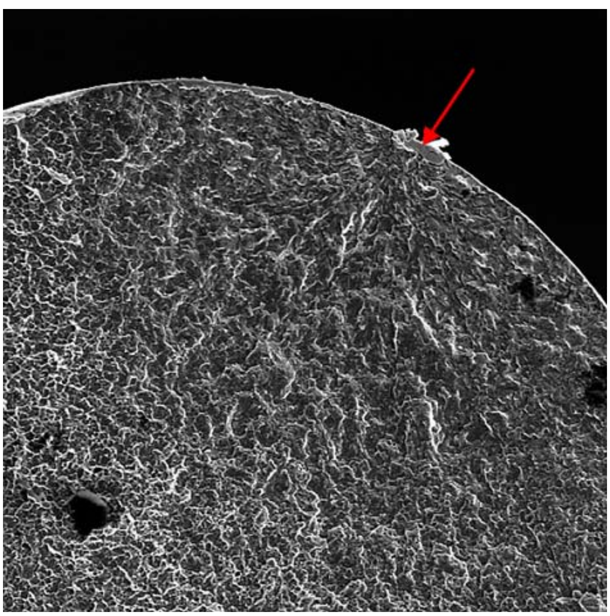
Fig. 10 SEM image of fatigue fracture surface 0.024″ diameter wire

can be explained by the drawing process; each reduction breaks up and strings out individual inclusions. The smaller diameter wires have more reduction steps than the larger diameter wires and therefore the inclusions are more broken up and spread out (Ref 3). Larger, blocky inclusions are more likely to initiate fatigue cracks than small, symmetrical inclusions. This may explain the increase in fatigue life for the smaller diameter wires.

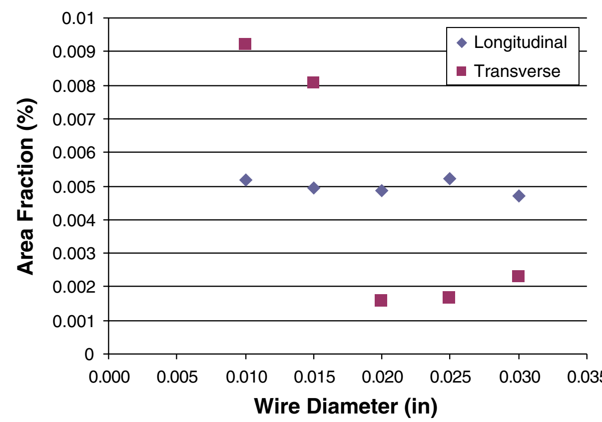

Fig. 11 Inclusion area fraction vs. wire diameter

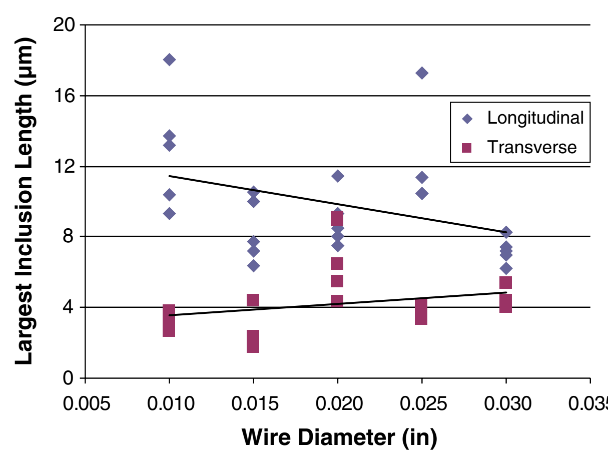

Fig. 12 Five largest inclusions vs. wire diameter

4. Summary

Final wire diameter has been shown to have an influence on the fatigue properties of Nitinol. The wire diameter reduction from 0.030″ to 0.010″ increased the 0.8% strain 80k cycle survival rate from 35 to 96% as shown in Fig. 3. For the scope of this study, all factors that may have an effect on fatigue life except for wire diameter were held constant. These factors include starting ingot material, wire drawing process, sample heat treatment and fixturing, post-processing surface treatment, and fatigue testing procedures. The ε-N data (Fig. 9) show slightly increased fatigue life for 0.009″ over the 0.024″ diameter wire.

Inclusion size and dispersion are generally accepted as factors that can negatively impact fatigue life. While the inclusion analysis performed for this experiment is not entirely conclusive, the trend lines in Fig. 12 do show the inclusions becoming smaller in the transverse direction and stringing out in the longitudinal direction. This makes logical sense and may contribute to greater fatigue life in the smaller diameter wires with all other factors held constant.

The results of this study along with other published fatigue data on Nitinol wire demonstrate that the fatigue performance of Nitinol is very product specific. Unlike stainless steel, there is no generic S-N or ε-N curve for Nitinol. There are many factors that can affect fatigue life that were held constant for the scope of this experiment. With a difference in wire diameter, there was a difference in fatigue performance. More data need to be collected to determine the significance of the findings in this paper. However, engineers and designers must recognize that no generic S-N or ε-N curve will provide an accurate prediction of the fatigue life of Nitinol. Each product, material, and set of process parameters must be individually tested and analyzed to provide accurate fatigue life data.

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