A STUDY OF THE EFFECTS OF SURFACE MODIFICATIONS AND PROCESSING ON THE FATIGUE PROPERTIES OF NITI WIRE

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ABSTRACT

Several studies on the processing effects on NiTi fatigue were published in the recent past. However, there were disagreements and debates with respect to the interpretation on these results. In hope to provide a more comprehensive understanding of this issue, a study on the fatigue properties of NiTi wires, Ti-55.8/55.9 wt %Ni in composition, comparing the various effects of surface finishes and heat treatments was conducted using a rotating beam test method in a 37^{o}C water bath. This investigation covers the effects of various chemical, electrochemical as well as mechanical finishing methods on the fatigue properties of heat-treated NiTi wire. The effects of heat treatment parameters and the resulting transformation temperatures on NiTi fatigue were also studied. Where enough data was gathered S-N curves were generated for the various processing conditions. Surface, microstructure as well as fracture analyses were conducted to support the cyclic fatigue data.

KEY WORDS

Fatigue, S-N curves, rotating beam fatigue test, surface finishing, thermal mechanical processing.

INTRODUCTION

As the unique properties of Nitinol have become better understood the number of applications that take advantage of these properties whether as an implant, a component in delivery systems, or instrument has increased. A number of surface modification processes have been commonly used not only to improve the materials appearance but also to improve the fatigue life as well as the corrosion resistance. It's no mystery that surface finish can have an effect on fatigue life; smoother more uniform surfaces free of defects will typically endure more fatigue cycles than surfaces with imperfections.

Practically speaking materials that are drawn, processed, and handled for forming, assembly, and inspection will have some level of surface imperfections. These imperfections can be artifacts of the raw material production processes, carbides, inclusions [1], or segregations. They can be contamination, surface marks, laps, seams, or texture from the intermediate processes used to bring material to its final size or from the shape and property setting processes used to provide the desired functionality.

Knowing these imperfections exist, three common surface modification processes; Electro Polish, Chemical Etch, and Mechanical Polish, can be used to mitigate the effects of the various surface conditions on fatigue life. Each process has specific requirements in terms of fixturing and appropriateness depending on product shape and size and particular benefits based on the desired results.

Electro Polish typically requires a clean surface to promote current flow and prevent preferential polishing with the part connected to a current source submerged in an acid solution. Chemical Etching also requires a clean surface and uses an acid solution to remove material. The Mechanical Polish process is typically a batch process where parts are placed in a drum or barrel with various types of media and then cycled promoting the contact of the media and parts until the parts cycle is finished or the desired appearance is achieved.

Surface modifications post shape setting are not the only means to improve fatigue life. Surface modifications also cannot overcome or compensate for all of known or unknown fatigue limiting defects or irregularities present in the materials. Earlier publications have discussed and future publications will continue to discuss many of these factors; Material composition [1], upstream processing and heat treats [2] can also be developed to provide improved fatigue resistance for a given strain or stress condition.

MATERIAL AND EXPERIMENTAL PROCEDURES

All of the samples for this portion of the study were produced from cold-worked Ti-55.8/55.9 wt% Ni and shaped as straight wires using the same heat treatment - tooling, time, temperature, and heat source. The resulting A_f was ≈12°C. A baseline data set was created from the same wire shape set with the same heat treatment with no post processing steps. Three post processing surface finish treatments were used; Electro Polish, Chemical Etch, and Mechanical Polish, each of these was followed by a standard passivation step.

Fatigue testing was conducted using a rotating beam tester with a zero mean .8% max alternating strain, as depicted in Figure 1 and Figure 2. This tester rotates a wire sample at a constant speed guided in a test block that is machined with a radius to provide the level of strain desired. The tester tests up to 10 samples at a time and counts revolutions on each sample individually using a laser counter.

The tester is designed to provide comparative results for materials processed with different parameters at the same strain level or varying strain levels. Testing was performed in a 37^{o}C water bath at 1000 RPM.

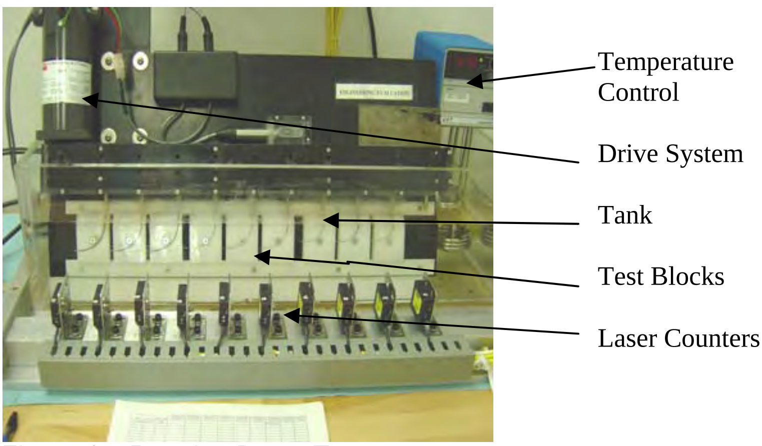


Figure 1 – Rotating Beam Tester

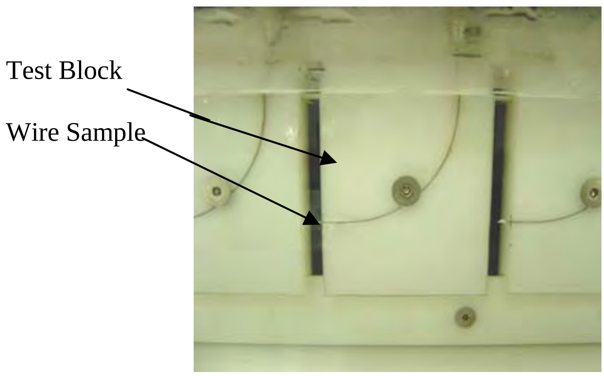


Figure 2 – Rotating Beam Test Block

RESULTS AND DISCUSSION

Figure 3 shows the sample population survival rates for the baseline population as well as the three finishing option populations. The fatigue data is presented as an average number that represents the survival percentage past a set number of cycles, in this case 90,000.

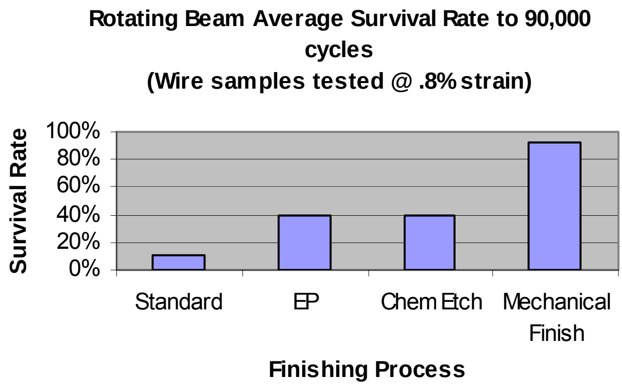


Figure 3 – Average Population Survival Rates

All parts were formed from wire that had a light oxide, straw colored finish. The survival rate for Standard parts was 10%, Electro Polished and Chemical Etched was 39% and Mechanically Polished was 92%.

The typical surface of as drawn and heat set wire is shown in Figure 4 and Figure 5. The surface contains small imperfections visible at this magnification from the drawing, handling and forming processes.

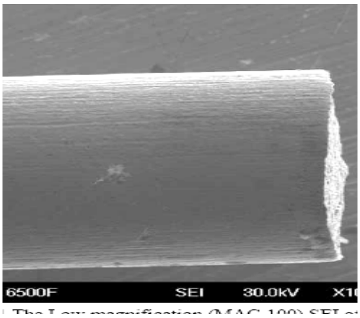


Figure 4 – SEM Image of As Drawn and Heat-treat Surface

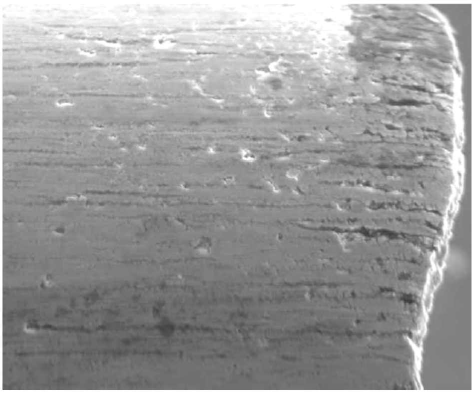


Figure 5 – SEM Image of As Drawn and Heat-treat Surface

The Electro Polish used for this experiment removed approximately .001" on diameter from the wire. The process smoothed the surface, attacking any thin or rough edges first. As can be seen in the photographs, Figure 6 and Figure 7, the resulting surface is smooth and under normal microscopy examination would appear reflective.

The surface appears very smooth with very little evidence of the as drawn or surface texture. Defects that were less than .0005" in depth are removed and the edges of surface initiated defects deeper than .0005" have been smoothed.

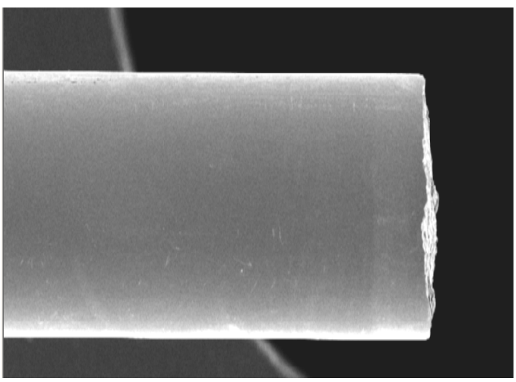


Figure 6 – SEM Image of Heat-treat and Electro Polished Wire Surface

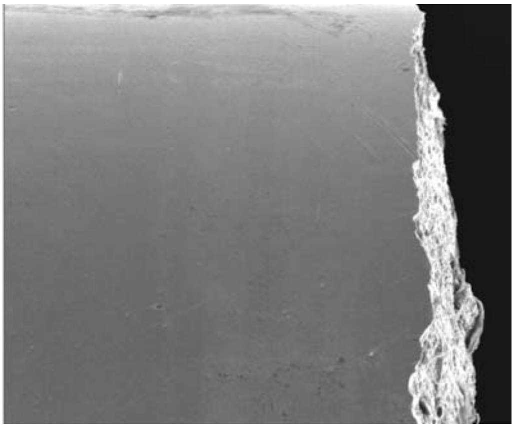


Figure 7 – SEM Image of Heat-treat and Electro Polished Wire Surface

The smoothing of the surface and removal of shallow defects reduces stress risers and increases the fatigue life. The effect on fatigue can be observed in Figure 3 where the population average survival rate to 90,000 cycles increases from the non post processed value of 10% to approximately 39%.

The Chemical Etching used for this experiment typically removed approximately .001” on diameter from the wire. This process smoothed the surface, attacking the surface in a more even manner removing material equally around the part. As can be seen in the photographs, Figure 8 and Figure 9, the resulting surface is smooth and reflective. As the material was removed more uniformly the surface still shows a pattern or texture based on the original as drawn surface but muted or smoothed from the chemical process.

As with the Electro Polish process surface defects that were less than .0005” in depth are removed and the edges of surface initiated defects deeper than .0005” have been rounded.

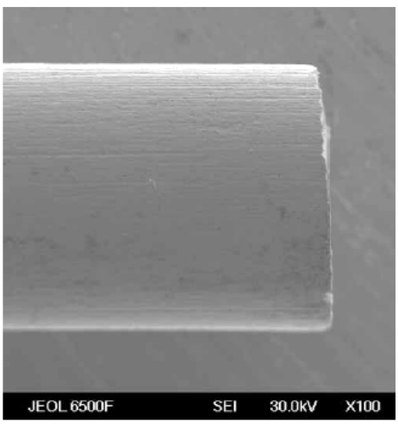


Fig. a-5-1 Low magnification (MAG 100) of the surface 1

Figure 8 – SEM Image of Heat-treat and Chemically Etched Wire Surface

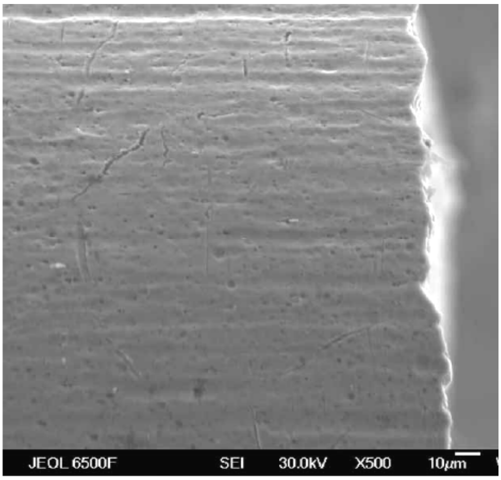


Fig. a-5-2 SEM image (MAG 500) of the surface finish.

Figure 9 – SEM Image of Heat-treat and Chemically Etched Wire Surface

Although the surface appears different the effect on fatigue is almost identical to that of Electro Polish and can be observed in Figure 3. The population average survival rate to 90,000 cycles increases from the non-post processed value of 10% to approximately 39%.

The Mechanical Polish used for this experiment typically removed approximately .0002" on diameter from the wire. This process removed the oxide layer and produced a visually reflective surface to the unaided eye. At magnification, Figure 10 and Figure 11, it can be seen that the surface is still similar in appearance to the as drawn surface, Figure 4 and Figure 5. The process appears to have added some texture by uniformly scratching the surface as the media impacted the parts during the finishing cycle.

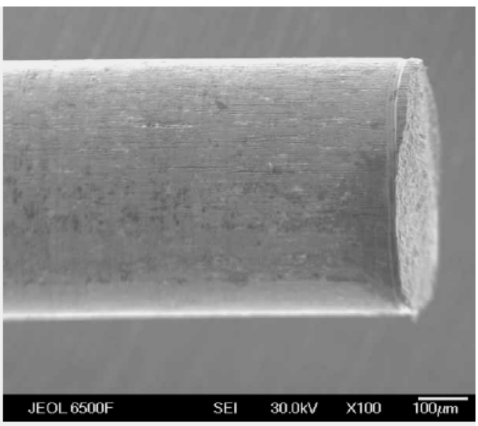


Fig. b-4-1 Low magnification (MAG 100) of the surface finish.

Figure 10 – SEM Image of Heat-treat and Mechanically Polished Wire Surface

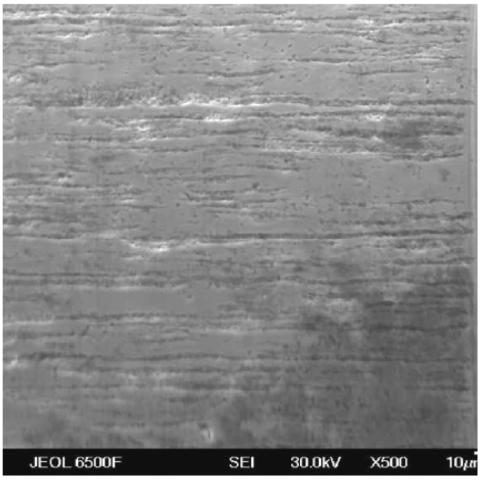


Fig. b-4-2 SEM image (MAG 500) of the surface finish.

Figure 11 – SEM Image of Heat-treat and Mechanically Polished Wire Surface

The population average survival rate to 90,000 cycles increases from the non-post processed value of 10% to approximately 92%. Since the material removal is minimal and the surface does not appear as smooth as the chemically processed parts, the mechanism responsible for the increased fatigue life is not the same as the Electro Polish and Chemical Etch processes.

A few theories exist for this mechanism; One is that the impacts associated with the process cause residual surface compressive stresses that must be overcome before a crack can initiate and spread, a second is that the uniform scratching creates non uniform stress fields that interrupt the propagation of cracks, and a third, that is related to the surface impacts, theorizes that the deformation of the surface creates stress induced martensite at the surface which is inherently more crack resistant delaying or impeding the initiation and propagation of cracks. Data supporting surface martensite or martensite acting as a crack inhibitor or interruption has been published [3] and the surface texture theory is similar to the crack blunting theory proposed by Morgan, Painter and Moffat. [4]

The graph in Figure 12 is a subsurface stress analysis of standard finished and mechanically finished parts. X ray diffraction was used to measure a group of wires in the axial or longitudinal direction and the graph reports the average of the stress readings at each depth.

This shows an increase in the subsurface compressive stresses for the mechanically finished part. The peak occurs at approximately .0004” depth and is - 9,000 psi.

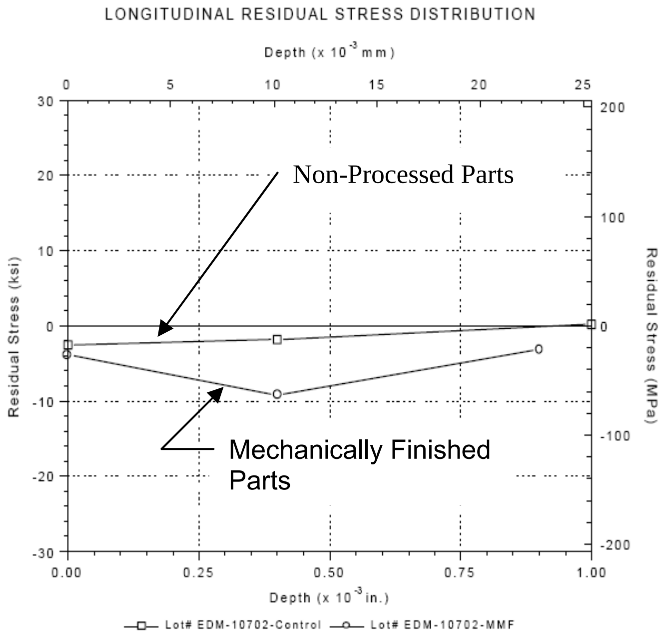


Figure 12 – Subsurface Stress Analysis

To examine the possibility of surface texture acting as non uniform stress fields or crack interruption zones and this contributing to increased fatigue life of the Mechanically Polished parts, untested wires were examined to characterize the finish pattern and texture. Figure 13 is an SEM image of a non-tested mechanically finished part. The two images of the same wire show the surface texture and pattern at the magnifications indicated.

The texture and pattern can be described as:
- Predominantly axial in direction.
- Each texture line is non-continuous.
- The texture lines vary in width, length, and depth.
- The texture lines are wavy.
- The space between the lines is random.
- The texture is relatively uniform in distribution around the circumference of the wire.

While this theory is possible there is no conclusive evidence to support or refute its validity at this time.

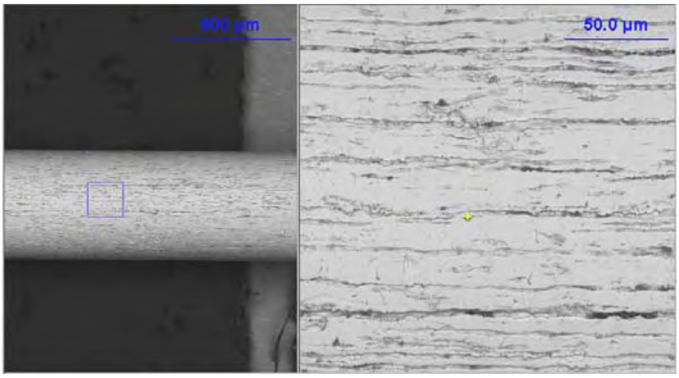


Figure 13 – SEM Image of Non-Tested Heat-treat and Mechanically Polished Part

SUBSURFACE MICROSTRUCTURE

The subsurface microstructure shows that in addition to removal of the oxide and the subsurface compressive stresses the B2 structure has been deformed by the Mechanical Polishing process. The deformation can be seen at varying depths near the surface of the test parts with higher dislocation density near the surface, Figure 14 and Figure 15.

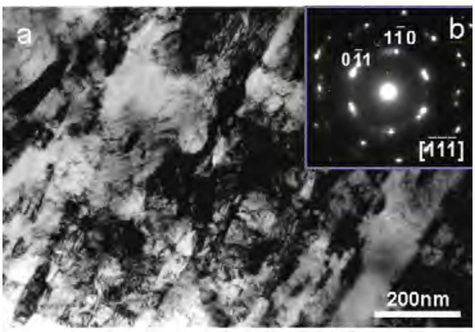


Fig. 2. The bright field image and the associated SAEDP at about 125 µm under the surface. The SAEDP shows the [-1-1-1] zone axis diffraction of the B2.

Figure 14 – Electron Diffraction and Transmission Electron Image of Mechanically Polished Microstructure 125 µm below the Surface

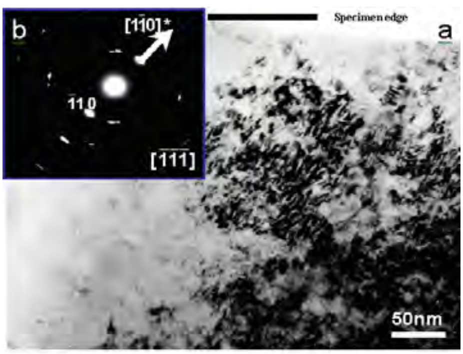


Fig. 1. The bright field image and the associated SAEDP at 200 nm under the surface. The SAEDI shows the [-1-1-1] zone axis diffraction of the B2.

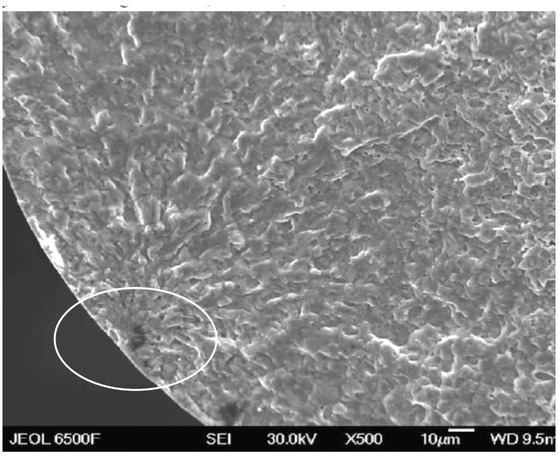
Figure 15 – Electron Diffraction and Transmission Electron Image of Mechanically Polished Microstructure 200 μm below the Surface

The combination of deformation and the subsurface compressive stress is responsible for the increased fatigue life compared to the Electro Polished and Chemical Etch processes.

SEM FRACTURE SURFACE ANALYSIS

The following fracture surface pictures, Figures 16 – 23, provide some documentation of the samples from the various fatigue tests.

Low Cycle Fatigue ~ 4,000 – 52,000 cycles
Failures typically initiated at very distinct points located at the surface or minimally subsurface. These points, Figures 16 – 19, were irregularities or surface interruptions from a variety of sources including handling dents or dings, pits or holes, carbides, and/or inconsistencies in the material matrix. Origination points of low cycle fatigue fractures are very distinct with typical radiating pattern [5].



g. d-1-2 SEM image (MAG 500) of the crack initiation site.

Figure 16 –SEM Image of Low Cycle Fatigue Fracture Surface 4,900 Cycles

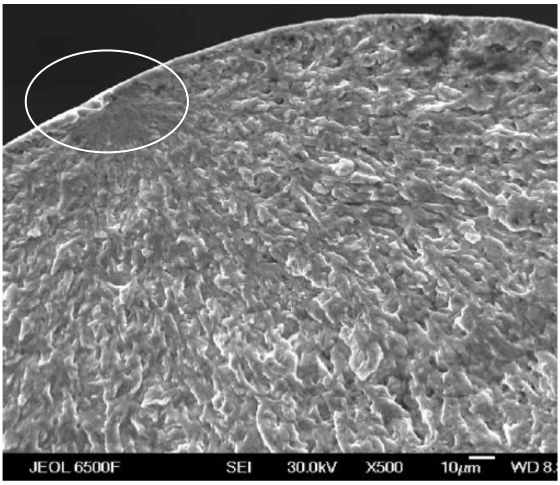
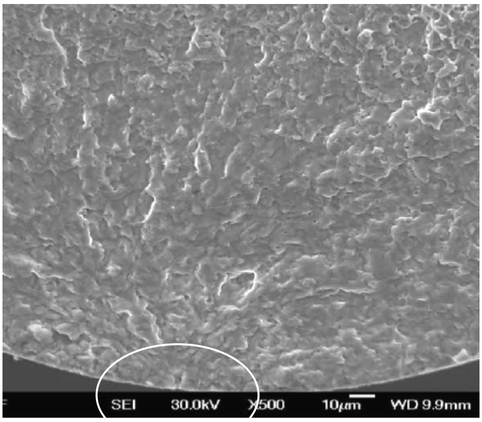


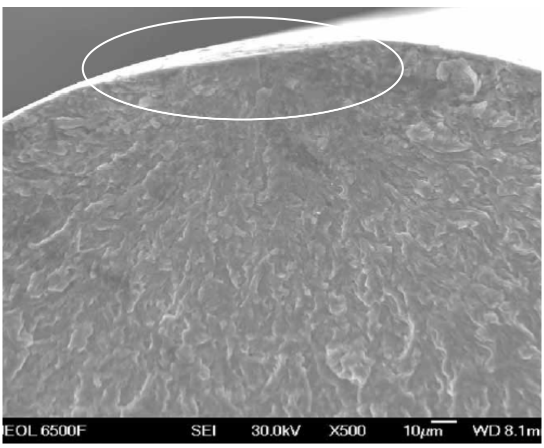
Fig. e-2-2 SEM image (MAG 500) of the crack initiation site.

Figure 17 – SEM Image of Low Cycle Fatigue Fracture Surface 51,140 Cycles



I image (MAG 500) of the crack initiation site.

Figure 18 – SEM Image of Low Cycle Fatigue Fracture Surface, 4,950 Cycles



e-3-2 SEM image (MAG 500) of the crack initiation site

Figure 19 – SEM Image of Low Cycle Fatigue Fracture, 37,416 Cycles

Medium Cycle Fatigue ~ 100,000 cycles
Origination point of fracture, Figures 20 – 21, is difficult to pinpoint. Fracture pattern originates from a wider arc and is not as distinct as the low cycle fatigue failures.

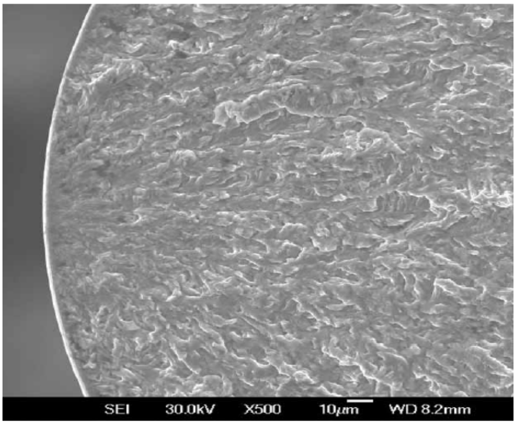
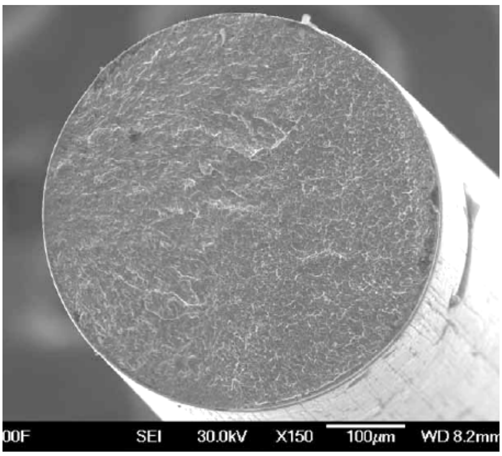


image (MAG 500) of the crack initiation site.

Figure 20 – SEM Image of Medium Cycle Fatigue Fracture



ow magnification (MAG 150) of the fracture surface.

Figure 21 – SEM Image of Medium Cycle Fatigue Fracture

High Cycle Fatigue ~ 530,000 cycles
The initiation point of the fracture is increasingly difficult to identify with the typical pattern that radiates from the fracture wide and spread out. Figure 22 and Figure 23.

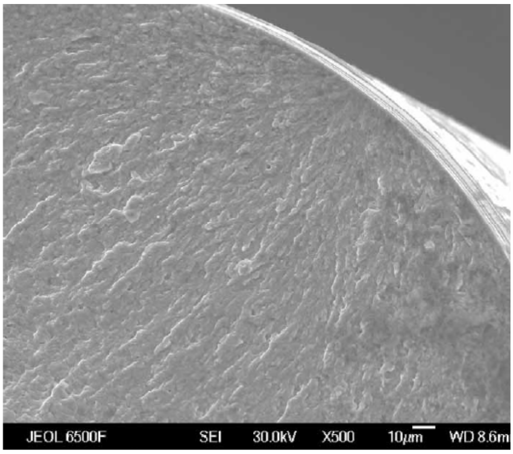


Fig. b-5-2 SEM image (MAG 500) of the crack initiation site.

Figure 22 - SEM Image of High Cycle Fatigue Fracture

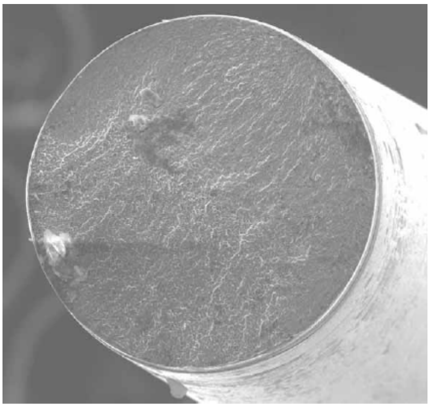

.ow magnification (MAG 150) of the fracture surface.

Figure 23 – SEM Image of High Cycle Fatigue Fracture

S-N CURVES FOR SURFACE FINISH

The following two S-N curves, Figure 24 and Figure 25 were generated to document the effect of surface finish on fatigue life. The first curve, Figure 24, compares parts produced with no post processing surface finish to parts mechanically finished and passivated.

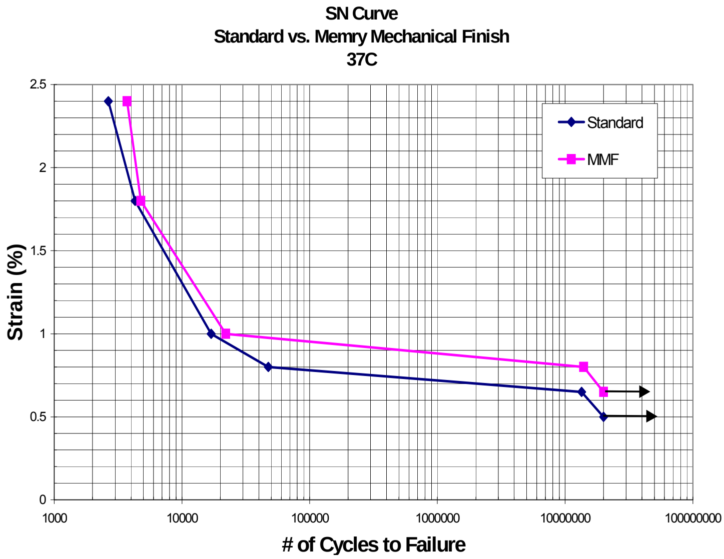


Figure 24 – S-N Curve for Standard & Mechanically Polished Wire

The Mechanical Polishing process shifts the S-N curve up and to the right increasing the fatigue lives of parts produced with this process. Both sets of parts were produced from the same material and heat-treated with the same process.

Figure 25 is an additional S-N curve that also includes an additional data set for parts processed using Chemical Etching. The data for this process falls in between the points for no post processing and for the mechanical polish post processing. The control group is the Standard parts, the CE group is Chemically Etched, and the Tumble Polish is the Mechanically Polished.

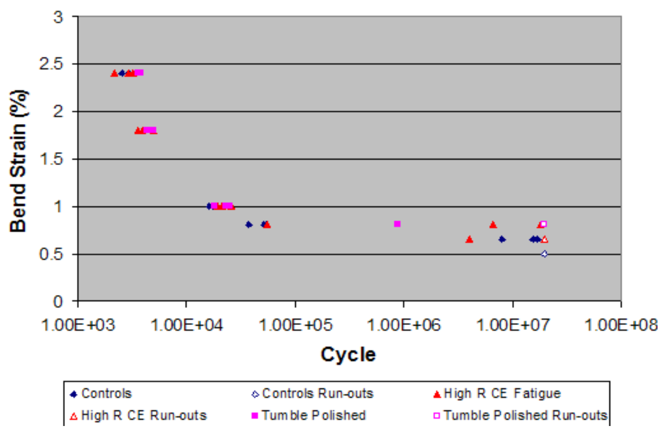


Figure 25 – S-N Curve for Standard, Chemically Etched and Mechanically Polished Parts

OTHER FACTORS THAT INFLUENCE FATIGUE LIFE

There are many other factors in addition to surface finishes that affect fatigue. While this study focused on the influence of surface finish on fatigue, the other factors that could influence the fatigue life of the material were noted and kept as consistent as possible.

Some of these factors include the base material's composition, uniformity, and cleanliness, the heat treat and the resulting stress/strain characteristics; specifically the shape of the curve and upper and lower plateau stresses levels [6].

Figures 26 represents a base line 6% stress-strain curve for tested material. Figure 27 is a 6% stress-strain curve for the same material heat treat with a slight temperature change. This introduces an R-phase transformation on the first part of the stress-strain curve lowering the slope and lowering the stress at the .8% test strain from 80,000 psi to 65,000 psi.

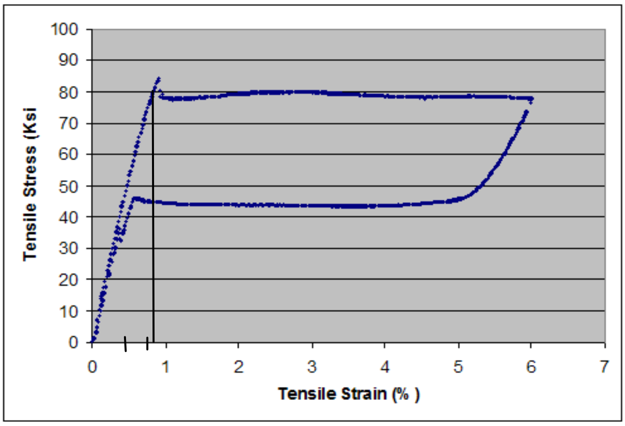


Figure 26–6% Stress Strain Curve for Heat- treat A Base Line

Heat-treat A - Heat-treat results
R_s = 7°C, R_f = -9°C
A_s = -6°C, A_f = 10°C
M_s < -70°C

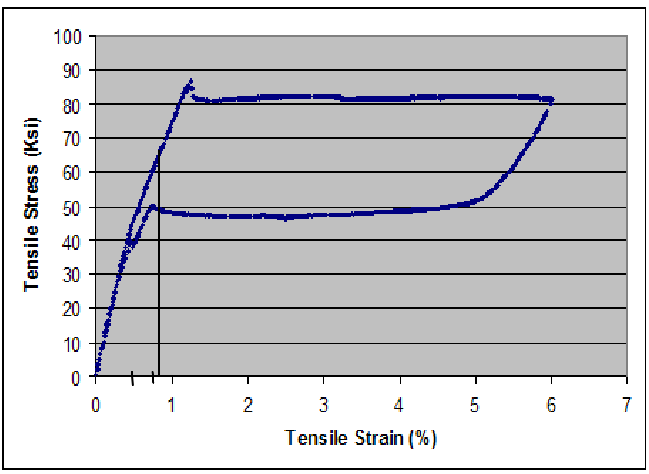


Figure 27 – 6% Stress Strain Curve for Heat-treat B

Heat-treat B – Heat-treat results
R_s = 27°C, R_f = 7°C
A_s = 8°C, A_f = 29°C
M_s < -70°C

A 15,000 psi change in stress level was tested and resulted in a 300% increase in fatigue survival in the initial testing.

CONCLUSIONS

Surface finish processes can have a significant effect on fatigue. Electro Polish and Chemical Etching improve the fatigue performance by 290% when compared to non-processed wire. This improvement is from the elimination of surface defects within the layer of material that is removed during these processes and the lowering of stresses around partially removed or deeper defects by rounding or smoothing the edges or initiation sites.

The improvement in fatigue life associated with Mechanical Polishing; 139% compared to Electro Polish or Chemical Etching, 820% compared to non-processed wire, is the result of small surface deformations that create residual compressive stresses. These stresses provide a crack resistant layer on the surface of the part that improves the fatigue life. The resulting surface texture may also contribute by stopping crack propagation.

Surface finish processes can improve the material as it is presented to these processes however it cannot completely overcome the condition of the starting material. Material chemistry, uniformity, thermal history has a large influence on fatigue. In the one example noted regarding a slight change in the heat-treat temperature the test stress level was lowered 15,000 psi and fatigue performance improved significantly

Through the testing program it was discovered that there is no generic S-N curve for Nitinol. Since there are so many factors that influence its fatigue performance, each life curve is only representative of the specific process steps used to prepare the test samples. The nature of the material allows it to be tailored to the applications stress and strain conditions. Testing at these conditions within the range of processing parameters provide the only true indication of part performance.

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