MEAN STRAIN EFFECTS AND MICROSTRUCTURAL OBSERVATIONS DURING IN-VITRO FATIGUE TESTING OF NITI

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ABSTRACT

The non-linear nature of the superelastic phase transformation in NiTi means that conventional fatigue life theory is difficult to apply. The volume fraction of martensite/parent phase and its role in the fatigue mechanism is not understood. This paper considers the fatigue life of superelastic NiTi alloys when tested at 2Hz in Ringers solution. In agreement with other researchers [1] [2] it was found that low alternating strains but high mean strain values resulted in the longest fatigue lives. To support the mechanical testing data, optical and electron microscopy was used to investigate the mechanism of fatigue and the role of the NiTi microstructure.

KEYWORDS

NiTi, Fatigue mechanisms, Martensite, Fracture surfaces

INTRODUCTION

Fatigue life remains one the most discussed yet least understood aspects of NiTi alloys. The FDA requirement of a fatigue life exceeding 400 million cycles for intravascular stents means that a better understanding of the factors affecting fatigue life and the mechanism of crack initiation and growth is essential [3]. Although cyclic deformation by bending is by far the most likely fatigue mode of in-vivo NiTi stents, fundamental mechanical properties are often best obtained from tensile studies. In an attempt to better understand the fundamentals of NiTi fatigue effects this paper we study uniaxial tensile fatigue data and consider how imposed mean strains and testing temperatures affect the fatigue life. In addition, optical micrography and fractography will be employed to study the crack initiation and growth mechanism.

EXPERIMENTAL

Fatigue testing was carried out with specimens immersed in a body fluid simulant known as Ringers solution that was maintained at a 38ºC using a digital water heater. All testing was performed under strain control using an extensometer on wires of Ti-55.9wt%Ni, straight annealed. The Af temperature of the alloy as measured by DSC was 12ºC. The wire surface was in the as-pickled condition.

Fatigue data was collected at a frequency of 2Hz for three different means strains: 2%, 4%, 6% and seven different alternating strain amplitudes: 0.5%, 0.75%, 1.0%, 1.5%, 2.0%, 2.5% and 3.0%. In addition to the 38ºC tests the experiment was repeated at a temperature of 20ºC for a mean strain of 6% and alternating strain amplitudes of: 0.5%, 0.75%, 1.0%, 1.5%, 2.0%, 2.5% and 3.0%.

RESULTS

Although the data is sparse and only preliminary Figure 1 is consistent with the following observations: At high strain amplitudes, the mean strain has little effect upon fatigue life. However, at low strain amplitudes (1.0% and less) increased mean strain is consistent with a significant increase of fatigue life.

Figure 2 plots the number of cycles to failure for 6% mean strain specimens cycled at two different temperatures, 38ºC and 20ºC; the data is consistent with the following observations: At high strain amplitudes the mean strain has little effect upon fatigue life. However, at 1.0% alternating strain the samples cycled at 20ºC yield the greatest fatigue life whilst at 0.5% alternating strain the samples cycled at 38ºC result in the highest fatigue life. It should be noted that these results are preliminary and that a more statistically significant series of tests is required to reach firm conclusions.

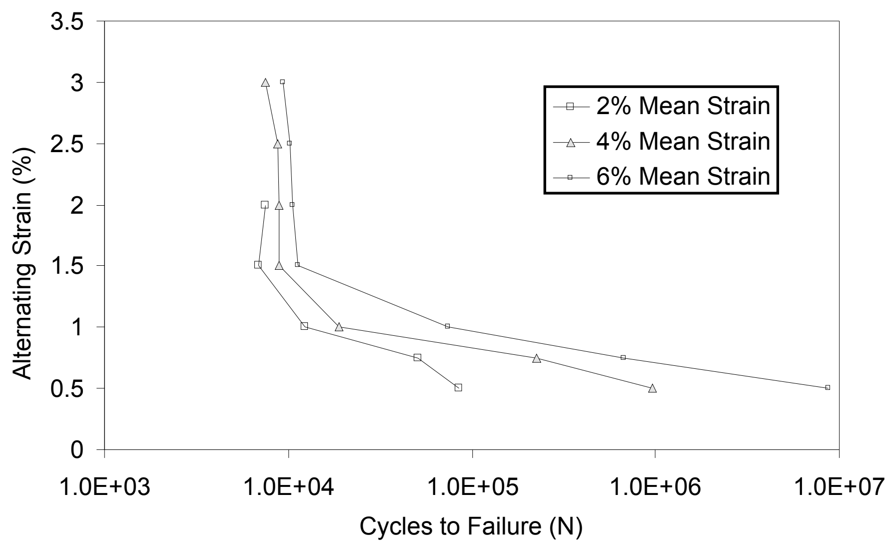


Figure 1: Cycles to failure of NiTi samples cycled in tension at 38ºC. All data points are fractures, i.e. none of the samples exceeded the cycles to failure shown above

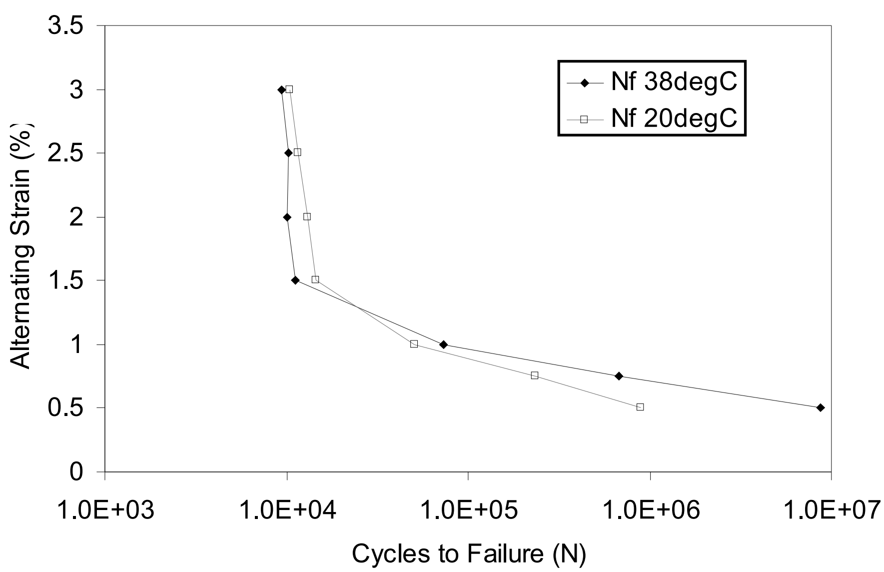


Figure 2: Cycles to failure of NiTi samples cycled in tension at a mean strain of 6.0%. All data points are fractures, i.e. none of the samples exceeded the cycles to failure shown above

When discussing the mechanisms behind these fatigue effects it is worth considering the stress/strain relationship of the mean strains and alternating strains. Figure 3 shows an idealized diagram of how the stress/strain hysteresis profiles of the samples cycled at an alternating strain of 1.5% and the samples cycled at 0.5% mean strain fit into a full superelastic transformation cycle.

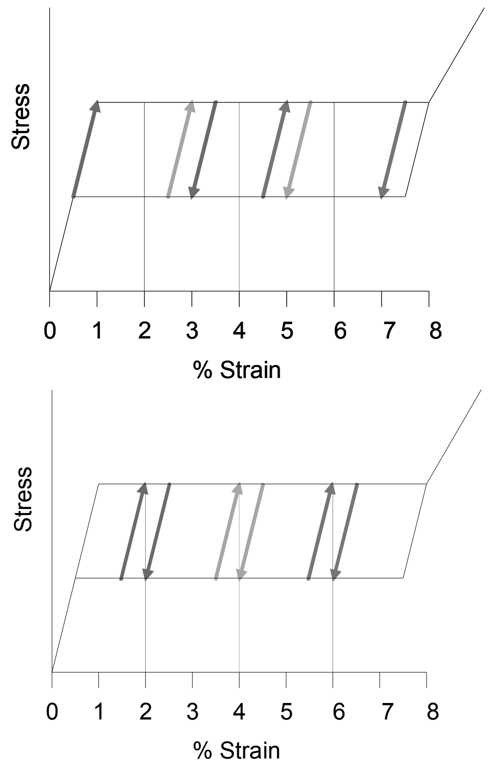
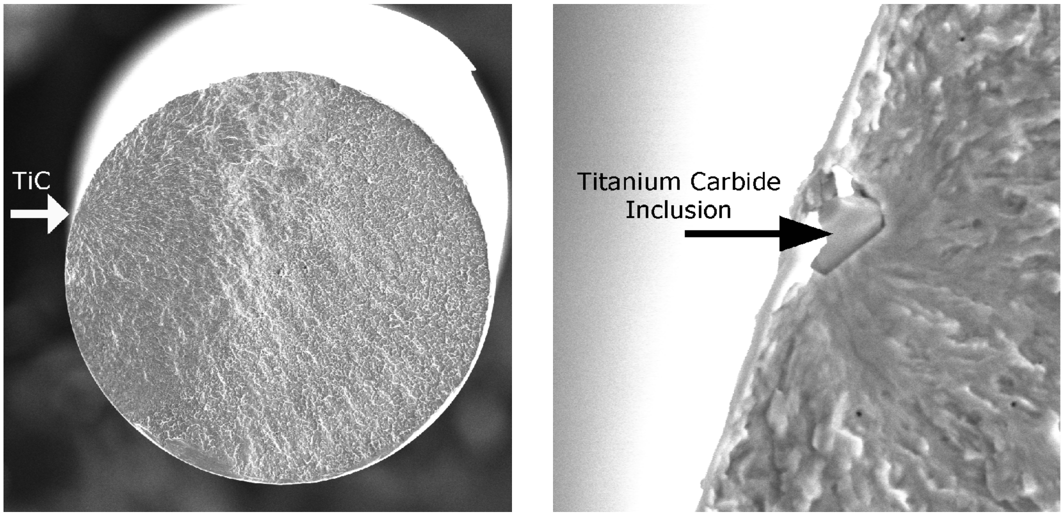


Figure 3: An idealized diagram of how the stress/strain hysteresis profiles of the samples cycled at an alternating strain of 1.5% and the samples cycled at 0.5% mean strain fit into a full superelastic transformation cycle.

At higher alternating strains the fatigue cycles rely upon considerable volumes of material transforming between the martensite and parent phase and will necessarily create dislocations within the structure which may ultimately lead to shorter fatigue lives.

In addition to dislocations, the relatively high alternating strains may create defects due to discontinuities and inclusions within the microstructure. Figure 4 is an example of the type of fracture surfaces observed at high alternating strains. Figure 4a is typical of a conventional fatigue failure involving high nominal stress and low to moderate stress concentration. However, in this case the wires were actually experiencing high nominal strains rather than high nominal stresses. The fatigue crack has initiated at a TiC inclusion at the surface of the wire (Figure 4b) and the area of final fast fracture (ductile) is comparatively large.



(a) *(b)*

Figure 4: An example of the fracture surfaces observed at high alternating strains. The wire diameter is 0.65mm and the inclusion size approximately2µm.

At low alternating strains the volume of transforming material is much less and the mismatch between the strain in the NiTi and inclusions is much lower. Figure 5 is typical of the fracture surface observed at low alternating strains. In comparison to Figure 4 the area of crack growth is much larger and the area of ductile fast fracture much smaller. This is typical of fatigue failure where the nominal stress is low but the stress concentration is high. However, in this case the wires were actually experiencing low nominal strains rather than stresses. Unlike those samples cycled at high alternating strains the point of initiation at low alternating strains was diffuse and could not be traced back to inclusions. The shear lips present on the fractured wire imply a crack front that is partially in tensile mode and partially in shear mode.

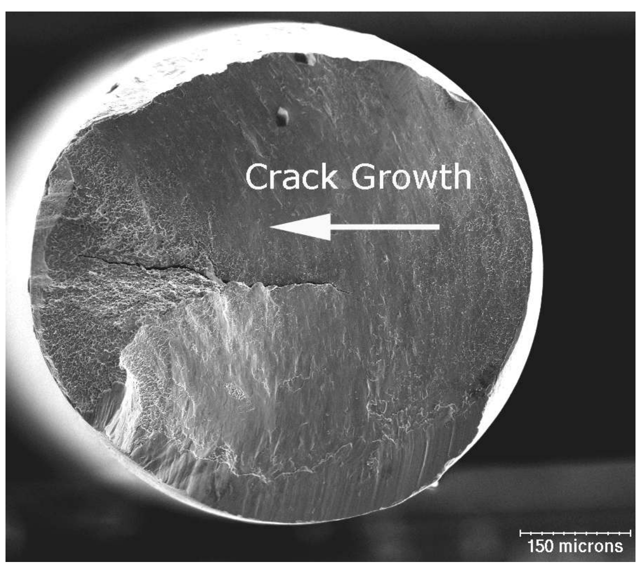
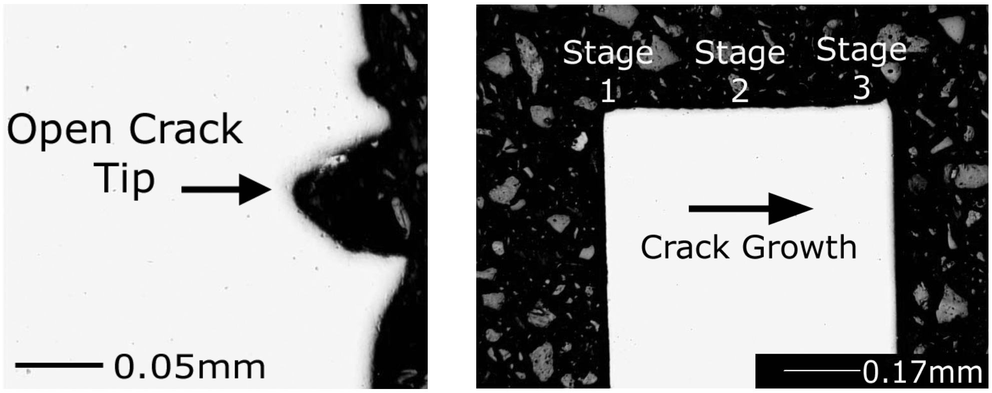


Figure 5: An example of the fracture surfaces observed at low alternating strains.

Figure 6 is a longitudinal view of a wire cross-section that was cycled at low alternating strain but did not break. The open crack tip is typical of the early stages of fatigue crack growth resulting from a highly dislocated structure.

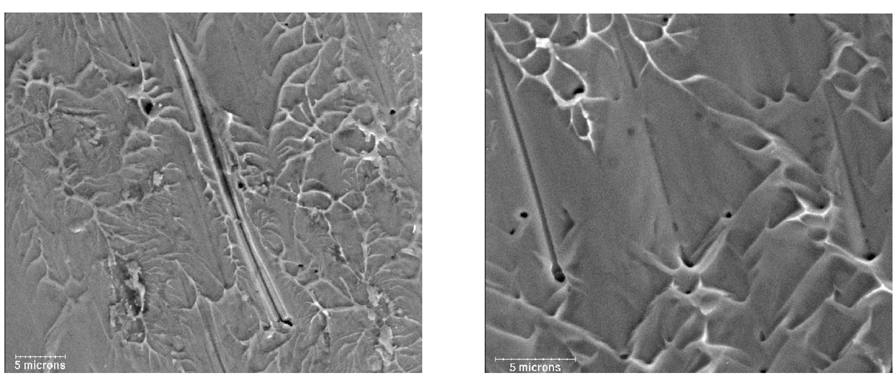


(a) (b)

Figure 6: (a) Longitudinal view of a wire cross-section that was cycled at low alternating strain but did not break. The open crack tip is typical of the early stages of fatigue crack growth resulting from a highly dislocated structure. (b) The three stages of fatigue crack initiation, growth and failure.

A cross-sectioned view of a fracture surface from the low alternating strain failures shows the classic characteristics of fatigue crack propagation. Stage I represents initial crack growth resulting from a highly dislocated structure. Stage II is the crack propagation approximately normal to the tensile axis which extends over a much greater distance than Stage 1. Stage II corresponds to the crack growth area in Figure 5 and is characterized by fatigue striations on the fracture surface. Stage III is final fast fracture. Stage III corresponds to the area of final fracture in Figure 5 and is characterized by micro-void coalescence leading to ductile overload.

An interesting feature of the low alternating strain fracture surfaces is microcracking. Figure 7a is typical of this cracking. Many of these cracks appear to be associated with a void resulting in a 'comet' type appearance, Figure 7b. In addition, the fracture surface morphology at the crack tips is consistent with martensite. Specifically, the morphology of the martensite is that sometimes described as a 'herring-bone' type. Herringbone martensite has been shown to consist of four correspondence variants.



(a) (b)

Figure 7: (a) Microcracking in the fracture surface of a NiTi sample that failed in tension fatigue. (b) Stabilzed martensite at microcrack tips.

It was noted that the frequency of microcracking and occurrence of martensite at the crack tips actually increased with mean strain. In other words the higher the mean strain, the greater the amount of stabilized martensite in the fracture surface.

Figure 8 is a surface response plot constructed from the same preliminary data shown in Figure 1. It is plotted for alternating strain versus mean strain. For alternating strains above 1.5% mean strain appears to have little effect. The fracture surfaces indicate that failure is controlled by surface inclusions and high nominal alternating strains.

Below 1.5% alternating strain, the level of mean strain has a much greater effect. The increase in fatigue life with mean strain is consistent with lower nominal alternating strains and 'classic' crack initiation and growth.

The mechanism behind the apparent increase of fatigue life (at low alternating strains) with mean strain level appears to be associated with microcracking and stress induced martensite. The samples tested at higher mean strains result in larger amounts of stress induced martensite. In addition, the microcracks in the samples cycled at higher mean strains appear to be blunted by localized martensite.

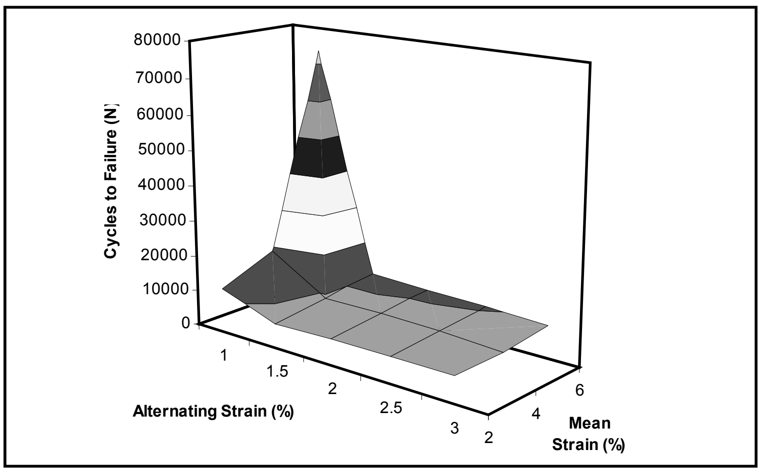


Figure 8: Surface response plot for alternating strain Vs. mean strain.

It is known that certain materials exhibit increased fatigue life due to phase changes ahead of the crack tip. The phase change absorbs energy and effectively toughens the material. Figure 9 describes a possible mechanism for the longer fatigue lives and crack blunting. The advancing crack tip increases the stress intensity to a level where martensite is induced ahead of the crack tip and stabilized by residual stresses and plastic deformation. The results confirm both the importance of transformation strain and phase state within the material during fatigue loading.

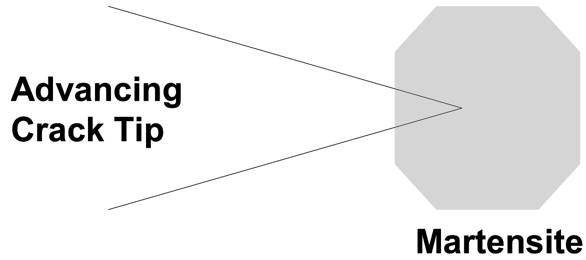


Figure 9: The stabilization of martensite ahead of the crack tip

The effect of testing temperature on the fatigue life of the alloys is counterintuitive. As the testing temperature is increased so the stress required for transformation increases. With this in mind it is reasonable to assume that the fatigue life would decrease with testing temperature. Figure 2 shows that at high alternating strains this is possibly true. However, below 1.0% alternating strain the specimens cycled at the higher temperature display the longer fatigue lives. It should be noted that this data is preliminary and as such no firm statistical conclusions should be drawn, however the results are consistent with such a theory.

Consistent with the increased fatigue life due to higher mean strains, it is possible that the higher transformation stress at the higher temperature results in larger amounts of stabilized martensite and/or an increased rate of stabilization. Figure 10 supports this theory. Martensite variants can be seen on the unetched surface of the wire after fatigue testing. The variants appear to be of the same type, i.e. they are all stabilized in the same orientation.

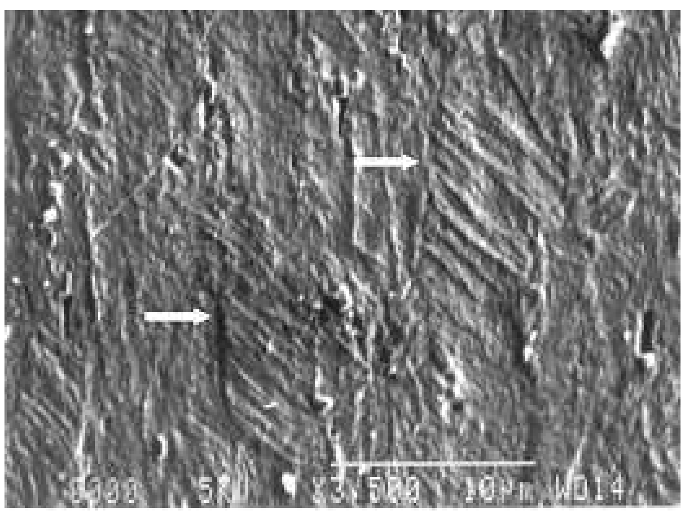


Figure 10: Stabilized martensite variants on the surface of NiTi wire after fatigue testing

CONCLUSIONS

- The results are consistent with high mean strains and low alternating strains resulting in the highest fatigue lives
- High alternating strain failures tend to be associated with inclusions within the alloy
- Crack initiation points for low alternating strain failures tend to be more diffuse and are not usually associated with inclusions
- Higher mean strains resulted in higher amounts of martensite in the fracture surface
- Extended fatigue lives appear to correspond to increasing amounts of stabilized martensite variants
- Microcracking within the fracture surface appeared to be associated with stabilized martensite implying a possible blunting/energy absorption mechanism prolonged fatigue lives

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