FATIGUE PERFORMANCE OF NITINOL TUBING WITH A_f OF 25°C

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ABSTRACT

The purpose of this study is to assess the fatigue behavior of Nitinol tubing aged at different temperatures (350–500° C) and times to achieve a 25° C ±2° C A_f. Fatigue properties were determined in tension at a mean strain of 1.5% and alternating strain magnitudes of 0.2%, 0.3%, and 0.4%. Although there are insufficient data to draw statistical conclusions from this preliminary investigation, it is important to point out that there did not appear to be any major differences in the samples tested despite the differences in microstructure and therefore in plateau strength. Still, among the heat-treatment temperatures evaluated, 500° C produces the smallest amount of R-phase with the most fatigue-stable microstructure, and therefore is the recommended choice for good reproducible device performance.

KEYWORDS

Tension, Fatigue, Mechanical Properties, A_f, Aging

INTRODUCTION

Like many metallic biomaterials, Nitinol's mechanical properties are determined by the thermomechanical processing of the alloy. In addition, Nitinol's mechanical behavior at body temperature is strongly dependent upon its transformation temperatures [1,2]. Several implant devices, such as self-expanding Nitinol stents, rely on the superelastic properties of the material, which are affected by the A_f transformation temperature of the alloy [3]. As a result, optimization of the mechanical properties of Nitinol for medical device applications is achieved by using various aging treatments to target a specific A_f transformation temperature. Aging treatments in the 350 to 500° C range are routinely used to adjust the transformation temperatures of Nitinol medical devices [1]. Small changes in time and temperature have shown to have little effect on the performance of the device.

EXPERIMENTAL METHODS

- As-received $Ni_{50.8}Ti_{49.2}$ Nitinol tubing of 0.072 in. Outer Diameter (OD) by 0.051 in. Inner Diameter (ID) was measured using the bend-free recovery method [5] to determine the initial A_f temperature prior to any heat treatments.
- 6-in. pieces of the tubing were heat treated to the times and temperatures listed in Table 1.
- One sample of each group was then measured using the bend-free recovery method to determine the final A_f temperature (see Table 1). Another sample from each group was further characterized by Differential Scanning Calorimetry (DSC) [6].
- To avoid sample breaking near or inside the grip during the fatigue test, the outside diameter of the tubes was electropolished such that the gauge section had a slightly smaller outside diameter of 0.071 inches [7].
- The mechanical testing was performed on a MTS 858 Mini Bionix test system. A 2000N pneumatic grip system with MTS serrated grip surfaces of dimension 38mm by 58mm were used in the test. The test consists of a loading and unloading sequence from 0 to 6% strain and then a fatigue test at 1.5% mean strain with cut-off cycle counts of one million, i.e., if the samples do not break at one million cycle counts, the test is stopped. The 1.5% mean strain is consistent with the *in vivo* mean strain that SMART® stents experience [8]. Alternating strain magnitude for fatigue tests were chosen to be 0.2%, 0.3% and 0.4%. All mechanical testing was performed at 37° C using circulated warm air.

Table 1 Temperature, Time and Average Final A_f of the Samples Used in the Study

Heat Treat Temperature (° C)	Time (min.)	Final A_f (° C)
350	35	23
400	8	27
450	5	26
500	90	25

We evaluated the following properties: (1) A_f temperature after each heat treatment using the bend-free recovery method, (2) transformation temperatures using DSC testing, and (3) fatigue life and loading/unloading plateaus after aging.

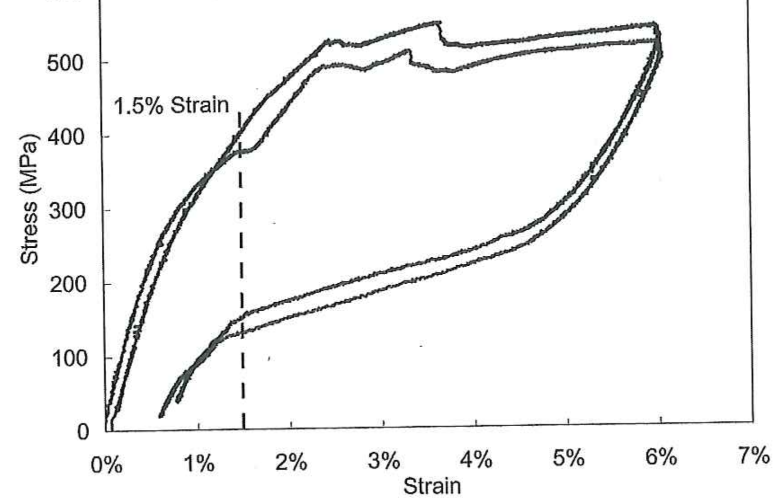

Figure 1 Stress-strain graph of Nitinol tubing heat treated at 450° C tested at 37° C.

The typical stress-strain responses of the tubing after the 450° C heat treatment process at 37° C is presented in Figure 1. The 1.5% mean strain used in the fatigue experiments is indicated on the figure. With this lower heat-treatment temperature, the tangential stiffness decreases at lower strain, which indicates the presence of a significant fraction of the R-phase. This is further confirmed with the DSC tests performed on the same heat-treated sample (Figure 2). There is a known variation between the bend-free recovery method and the DSC method when determining the A_f temperature due to the influence of strain. It is important to note that the DSC results were used as reference only and that all A_f temperatures were set using the bend-free recovery method.

Samples heat treated at 500° C (Figure 3), have a noticeable increase in the tangential stiffness over those heat treated at 450° C. This indicates that the R-phase is less of a factor at this temperature as indicated in Figure 4. The 1.5% mean strain for this heat treatment corresponds to the approximate onset of the plateau stress.

Figures 5 and 6 show the stress-strain responses of samples heat treated at 350° C and 400° C before and after the 10^6 cycle fatigue tests. As can be seen in these figures, it appears that the displacement cycling modified the tangential stiffness by eliminating the R-phase inflection. This indicates that the R-phase interacted with the fatigue-induced dislocations thereby stabilizing the structure. Additional mechanical and DSC testing is required to verify these observations.

The plateau stress as a function of the hea- treat temperature is plotted in Figure 7. Clearly there is a nonlinear relationship between plateau stress and aging temperature used to obtain an A_f of 25° C. It is interesting to note that these thermal treatments led to a plateau stress range from 300 MPa to over 500 MPa.

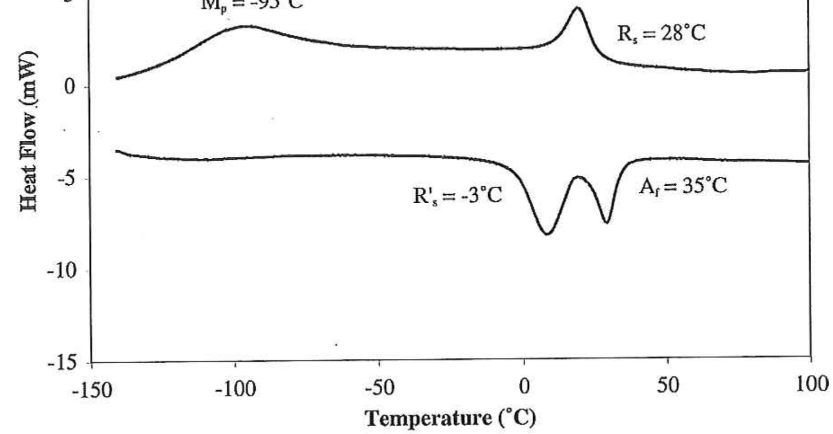

Figure 2 DSC results of Nitinol tubing heat treated at 450° C.

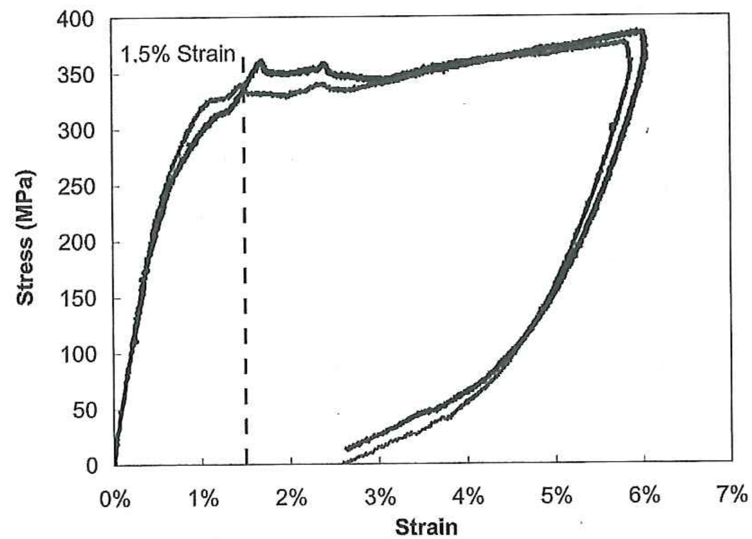

Figure 3 Stress-strain graph of Nitinol tubing heat treated at 500° C tested at 37° C.

Fatigue Properties

Fatigue testing on thin-walled tubes is nontrivial as pointed out by Tabanli *et al.* [9]. Sample failures inside or very close to the grips are still possible even with the proper gauge section reduction. Grip failures are normally attributed to excessive load from the grip; therefore these fatigue data

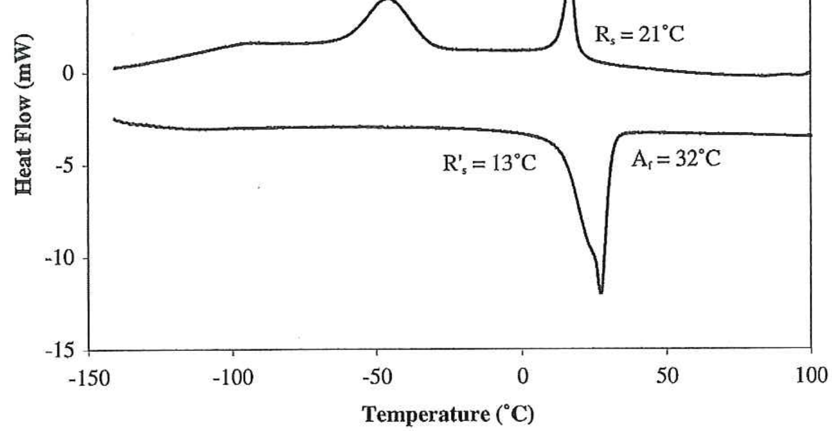

Figure 4 DSC results Nitinol tubing heat treated at 500° C.

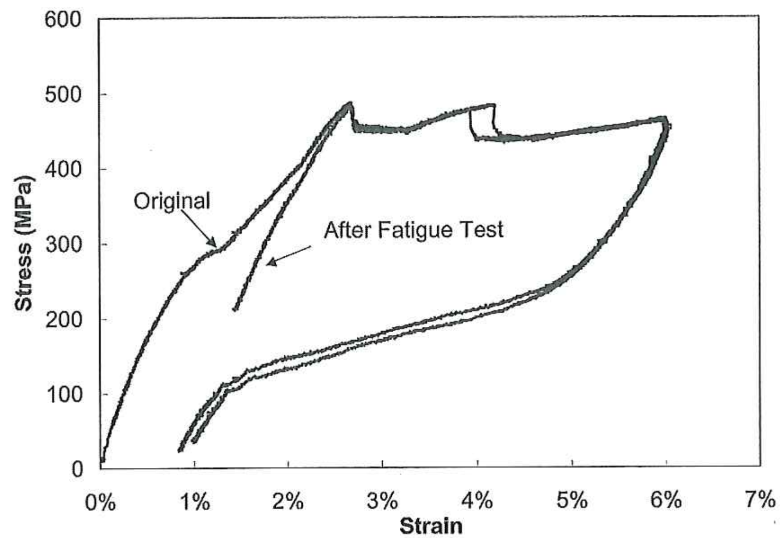

Figure 5 Stress/strain graph of Nitinol tubing heat treated at 350° C before and after fatigue test.

were not included in the analysis. Figure 8 summarizes the fatigue-tests results for samples that either broke at the test section or had run out at 10^6 cycle counts. There was no tubing failure with 0.2% half-alternating strain for any of the aging temperatures. At 350° C and 400° C, there were two samples with run out at 0.4% and 0.2% half-alternating strain magnitudes, yet there was a

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