A Study of the Properties of a High Temperature Binary Nitinol Alloy Above and Below its Martensite to Austenite Transformation Temperature

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Abstract

High temperature Nitinol alloys provide a challenge to end users of the material because they are martensitic and soft at room temperature. These are commonly referred to as Shape Memory alloys as they revert to their superelastic (pseudoelastic) form and austenitic structure at a temperature above ambient. For this study, a NiTi wire, Ti-55.3 wt %Ni in composition (Alloy-B) and heat treated to an Af ≈ 60°C was used. Tensile testing was performed to fully characterize the performance of the material at a series of temperatures above and below its transformation temperature. This paper will summarize the properties of the material along with the affects of multiple strains on key material performance characteristics.

Keywords

Nitinol, Transformation Temperature, Martensitic, Austenitic, Tensile Testing, Shape Memory, Strain, Superelastic, Pseudoelastic

Introduction

As shown in previous studies, Nitinol alloys when fully recrystallized after high temperature annealing exhibit a single stage martensitic transformation from the parent B2 to B19' monoclinic martensite. For functional use in a superelastic or shape memory application, the material is optimized by cold working and heat treating at lower temperatures so that nano sized subgrains, a high density of dislocations, and very fine Ni rich precipitates are present in the material (Ref 1, 2). This microstructure leads to a two stage transformation of B2→R Phase→B19' martensite (Ref 1).

This study was developed to determine the effects of different heat treat temperatures and multiple strains on a NiTi wire, Ti-55.3 wt %Ni in composition and heat treated to an Af ≈ 60°C. The first heat treat temperature used was 525°C for 4 minutes. This is a typical heat treat temperature and time that would be used for a Ti-55.8 wt %Ni Nitinol to provide superelasticity at room temperature. The second lower heat treat temperature of 430°C for 4 minutes was used to determine if the lower temperature would provide superior superelastic properties in the much warmer Ti-55.3 wt %Ni alloy. The constant Af over a wide range of heat treatment parameters may be explained by the balancing affect between annealing and the precipitation of Ni rich precipitates (Ref 1).

Methods

<u>Sample Preparation:</u>

Test samples were fabricated from a 40% cold worked ø0.0428 wire (CW40-B-42.8). Samples for test were constrained on a fixture and shape set straight in a salt pot for the prescribed times and temperatures. This was followed by an immediate room temperature water quench.

<u>Determination of Transformation Temperature:</u>

Transformation temperature was determined through both DSC (differential scanning calorimetry) per ASTM F2004 and BFR (bend and free recovery) per ASTM-F2082. Results from each method agreed within one degree. This is excellent correlation for alloys showing a clear R phase transformation (Ref 3).

<u>Tensile Testing:</u>

Tensile testing was performed in a controlled temperature chamber using extensometer strain control per ASTM F2516.

Results

The data shown below summarize the tensile testing performed at various temperatures on the straight heat treated wire samples. Each sample was pulled to 6% strain and then the load was reduced to less than 1ksi. The cycle was repeated 3 times per sample at each temperature noted. The strain was balanced after each cycle.

Table 1: Typical Room Temperature Martensitic Heat Treatment, Ti-55.3 wt %Ni

Heat Treatment 525˚C/4min Af=57˚C									
	Test Temperature								
	50C	55C	60C	65C	70C	75C	80	85	90
Stress at 3% Strain (ksi) 1st Cycle	28	34	46	54	58	72	72	82	85
Residual Strain (%) 1st Cycle	5.2	5.1	4.9	4.7	4.4	1.8	0.4	1.0	1.6
Stress at 3% Strain (ksi) 2nd Cycle	106	103	100	89	77	62	69	69	73
Residual Strain (%) 2nd Cycle	3.0	3.1	3.0	3.1	3.2	0.8	1.0	1.1	1.5
Stress at 3% Strain (ksi) 3rd Cycle	167	155	165	161	155	54	66	60	76
Residual Strain (%) 3rd Cycle	Break	Break	Break	Break	Break	1.4	1.0	1.6	1.2

Table 2: Modified Room Temperature Martensitic Heat Treatment, Ti-55.3 wt %Ni

Heat Treatment 430˚C/4min Af=58˚C						
	Test Temperature					
	50C	55C	60C	65C	70C	75C
Stress at 3% Strain (ksi) 1st Cycle	55	59	68	71	78	81
Residual Strain (%) 1st Cycle	4.0	0.3	0.1	0.1	0.1	0.1
Stress at 3% Strain (ksi) 2nd Cycle	60	53	62	66	74	75
Residual Strain (%) 2nd Cycle	3.2	3.7	0.0	0.0	0.1	0.1
Stress at 3% Strain (ksi) 3rd Cycle	173	85	59	65	71	72
Residual Strain (%) 3rd Cycle	Break	3.0	0.0	0.0	0.0	0.0

For comparison purposes, a room temperature superelastic alloy (Alloy-BB) was shape set using the high temperature (525˚C for 4 minutes) heat treatment. The same tensile testing was performed at temperatures both above and below its martensite to austenite transformation temperature. The results are tabulated below.

Table 3: Typical Room Temperature Austenitic Heat Treatment, Ti-55.8 wt %Ni

Heat Treatment 525˚C/4min Af=10˚C							
	Test Temperature						
	-20C	-10C	0C	10C	20C	30C	40C
Stress at 3% Strain (ksi) 1st Cycle	36	41	48	54	64	73	81
Residual Strain (%) 1st Cycle	4.7	4.2	0.3	0.1	0.0	0.0	0.0
Stress at 3% Strain (ksi) 2nd Cycle	40	39	46	52	61	69	77
Residual Strain (%) 2nd Cycle	4.5	4.6	0.1	0.1	0.0	0.1	0.1
Stress at 3% Strain (ksi) 3rd Cycle	33	36	45	51	61	70	75
Residual Strain (%) 3rd Cycle	4.8	4.7	0.1	0.0	0.0	0.0	0.1

When subjected to a high temperature heat treatment typical of that used for a room temperature superelastic alloy, the warmer Alloy-B material did not develop its full superelastic properties until approximately 25˚C above its Af temperature. In contrast, the same alloy when heat treated at a lower temperature developed its full superelastic properties at its Af temperature. The room temperature superelastic material (Alloy-BB) actually developed its full superelastic properties 10˚C below its Af temperature. The above data and results are depicted in the following six Figures.

Figure 1: Upper Plateau Stress vs. Temperature – First Cycle

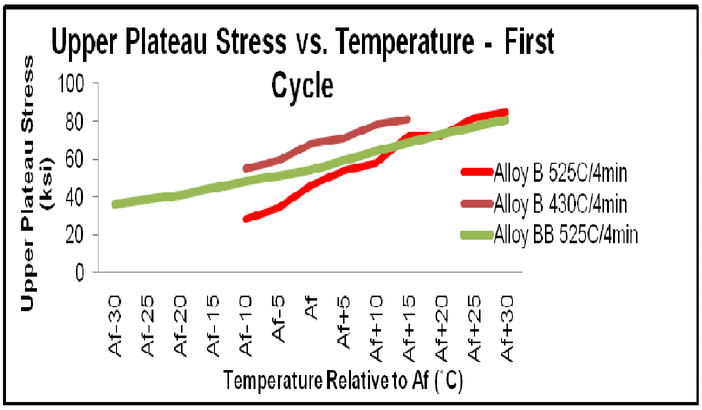


Figure 2: Residual Strain vs. Temperature – First Cycle

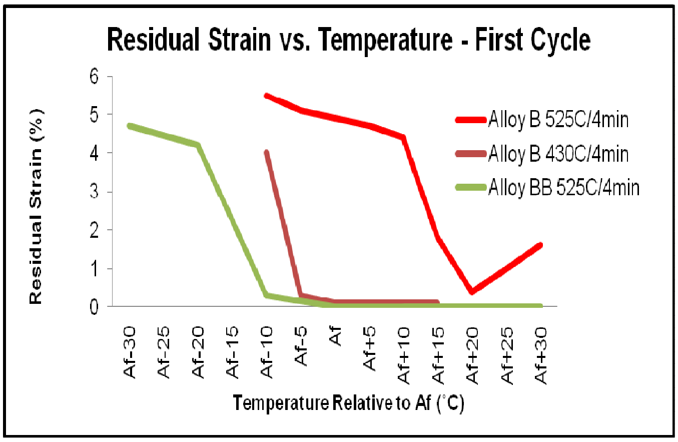


Figure 3: Upper Plateau Stress vs. Temperature – Second Cycle

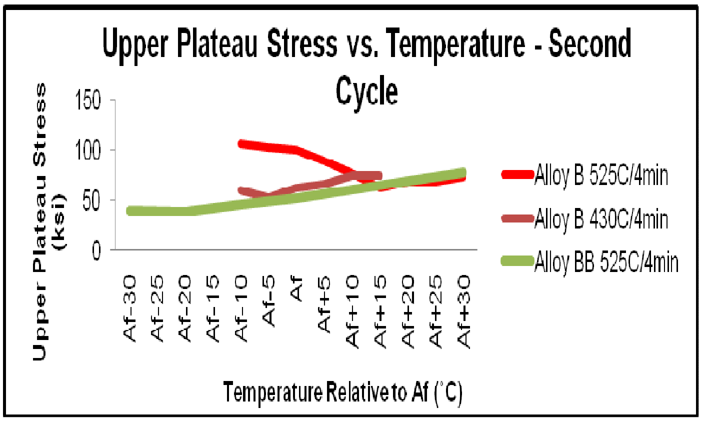


Figure 4: Residual Strain vs. Temperature – Second Cycle

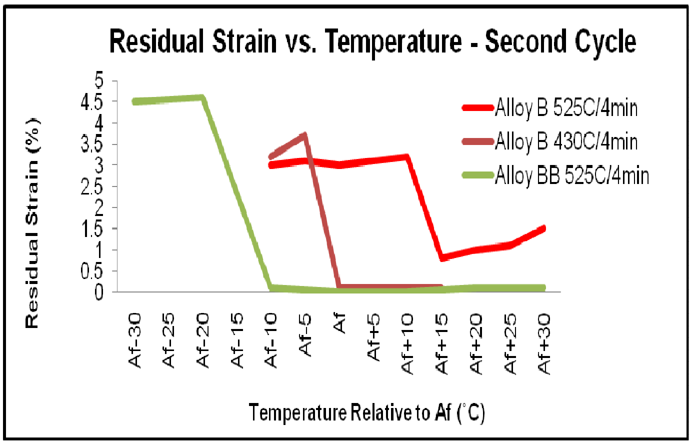


Figure 5: Upper Plateau Stress vs. Temperature – Third Cycle

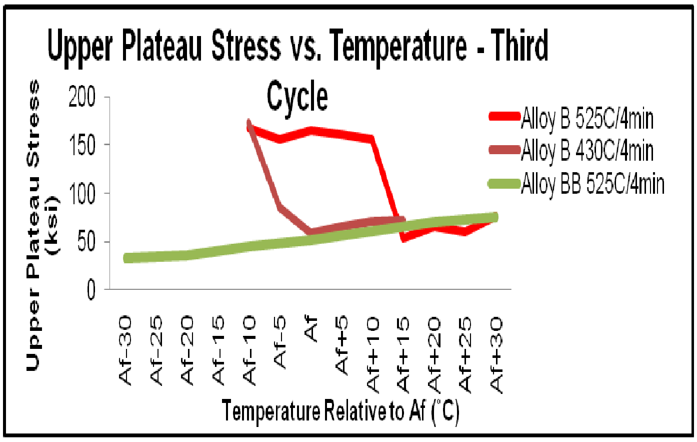
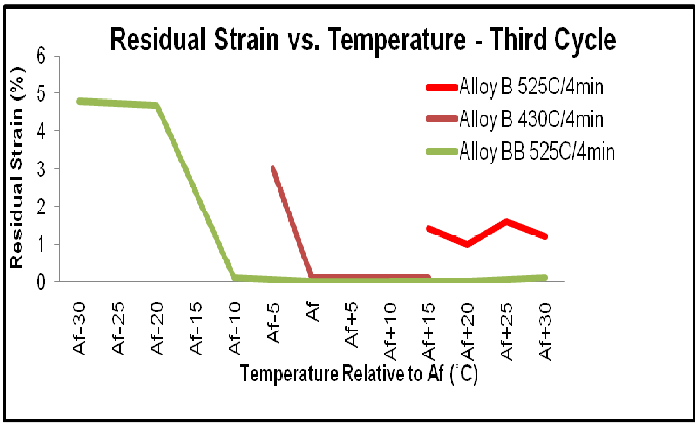


Figure 6: Residual Strain vs. Temperature – Third Cycle



Tensile Test Graphs

The following Figures are graphical representations of the data presented above. Note the differences in superelastic plateaus depending on heat treatment and ambient temperature. The colder Alloy BB material shows much less dependence on ambient temperature than the warmer Alloy B material.

Figure 7: Alloy B - Heat Treatment 525˚C/4min Tested at Af Temperature

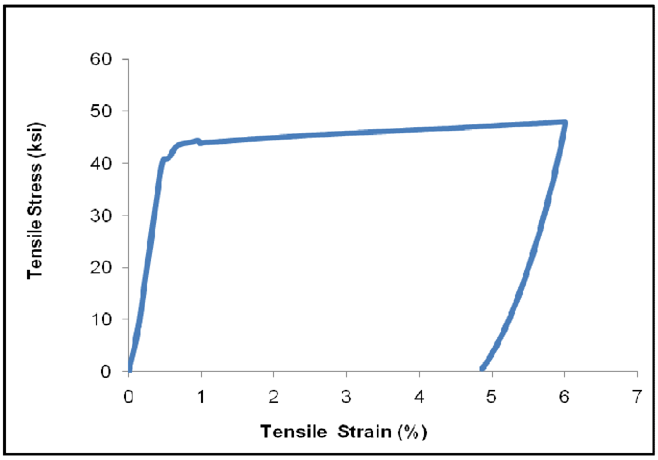


Figure 8: Alloy B - Heat Treatment 525˚C/4min Tested at 10˚C Above Af Temperature

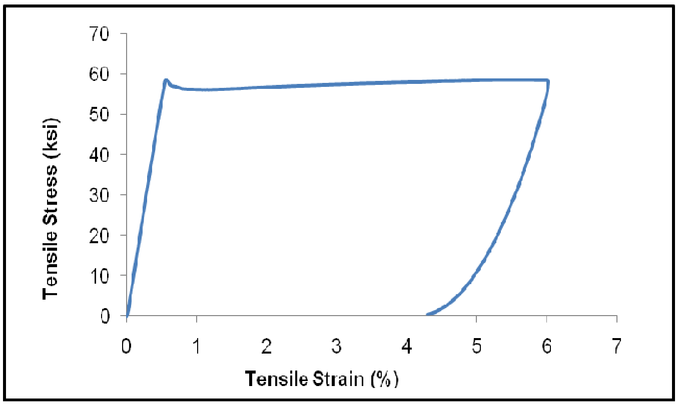


Figure 9: Alloy B - Heat Treatment 430˚C/4min Tested at Af Temperature

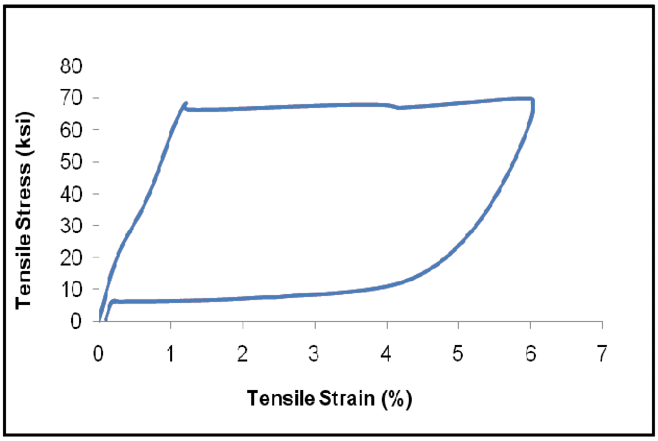


Figure 10: Alloy B - Heat Treatment 430˚C/4min Tested at 10˚C Above Af Temperature

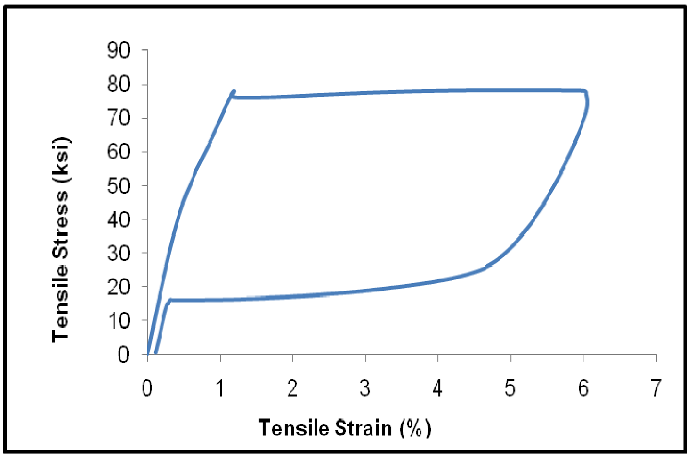


Figure 11: Alloy BB - Heat Treatment 525˚C/4min Tested at Af Temperature

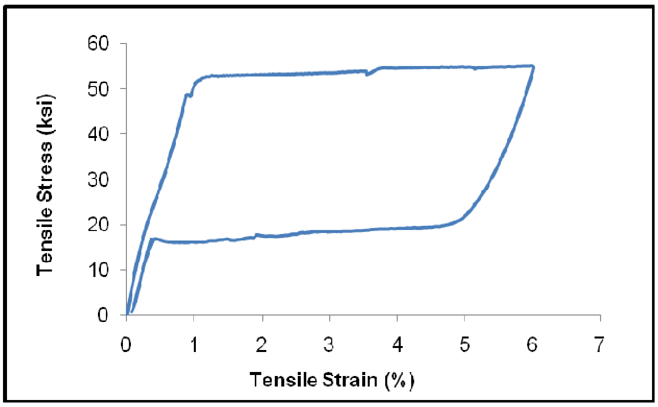
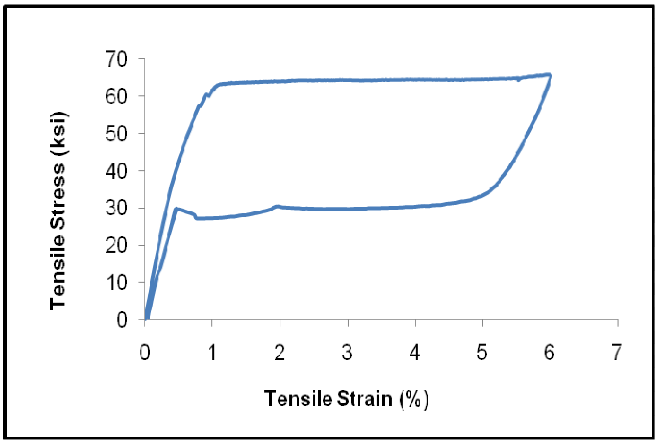


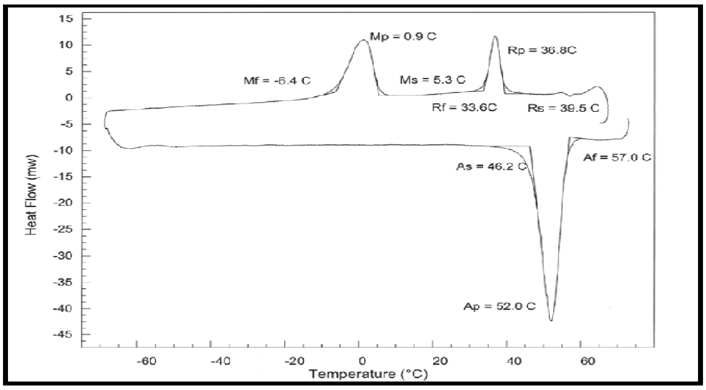
Figure 12: Alloy BB - Heat Treatment 525˚C/4min Tested at 10˚C Above Af Temperature



Transformation Temperature Analysis

The following figure is a DSC scan representative of the typical results for a heat treated sample used for this study. The graph clearly shows the 2 stage transformation of B2→R Phase→B19' martensite (Ref 1).

Figure 13: DSC Curve Alloy B - Heat Treatment 525˚C/4min



Fracture Surface Analysis

Scanning Electron Microscope (SEM) analysis was used to analyze the fracture surface on tensile test samples tested both above and below the sample martensite to austenite transformation temperature. The following images depict ductile yielding followed by overload fracture. The fracture surfaces exhibit microvoid coalescence morphology independent of temperature or phase (Ref 4). This is consistent with previous research and literature.

Figure 14: SEM Image of Alloy B Fracture Surface at 20˚C

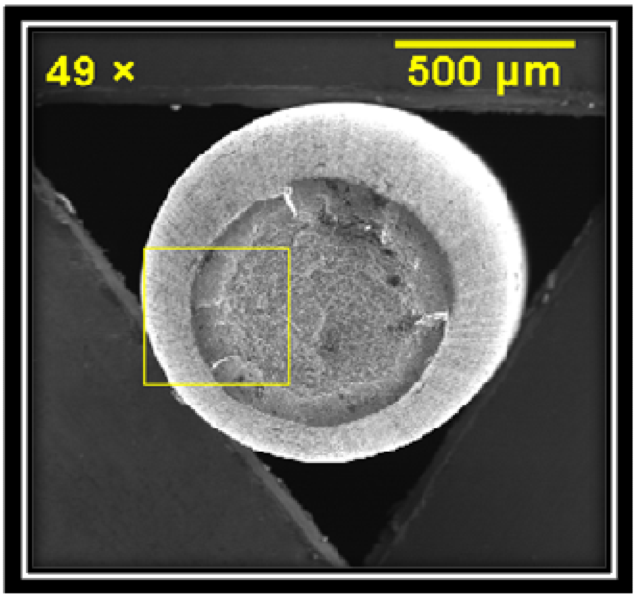


Figure 15: SEM Image of Alloy B Fracture Surface at 20˚C

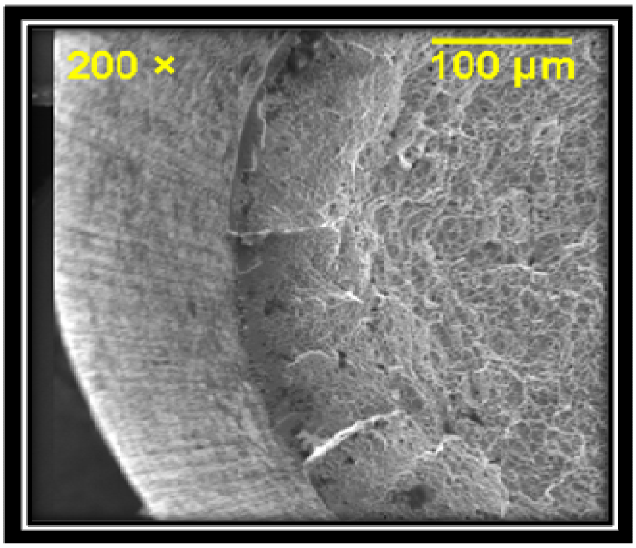


Figure 16 SEM Image of Alloy B Fracture Surface at 75˚C

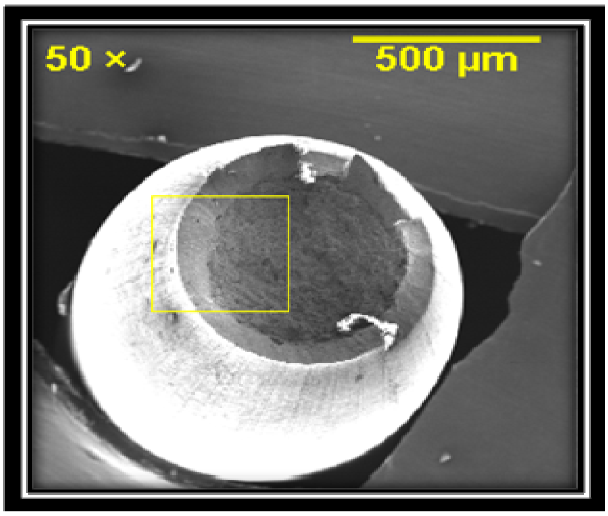
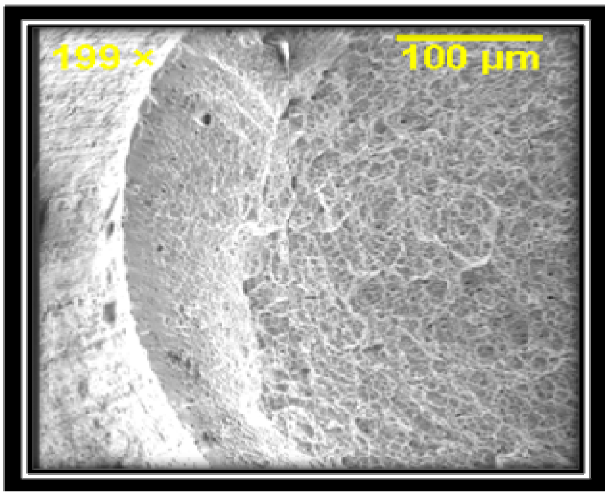


Figure 17: SEM Image of Alloy B Fracture Surface at 75˚C



Discussion

- The superelastic properties of a warm Ti-55.3 wt %Ni alloy are much more dependent on heat treat temperature and ambient test temperature than a room temperature superelastic Ti-55.8 wt %Ni alloy. This is due to the lower frequency of precipitates and dislocations in the lower Ni content alloy (Ref 2, 5).
- The higher heat treat temperature for the warmer Nitinol alloy also retards the nucleation and growth of the Ni rich precipitates that act as barriers to dislocation motion and strengthen the alloy. This prevents the NiTi structure from providing full superelastic properties (Ref 2, 5).
- The lower temperature heat treat for the Ti-55.3 wt %Ni alloy provides superior superelastic properties to the higher temperature heat treat.
- The effect of the optimum heat treatment while evident on the initial strain cycle is exaggerated upon multiple strain cycles as seen in the accompanying data tables.

Conclusions

- As alloys are developed with different transformation temperatures, sufficient studies must be performed to determine the appropriate individual heat treatment. The times and temperatures needed to develop optimum properties are alloy dependent and cannot be carried over from prior experience.
- With the further development of ternary (NiTiCo for example) and other more complex alloys, this attention to alloy individuality will become more important.

References

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