

**CHANGES IN THE MECHANICAL BEHAVIOR OF NITINOL  
FOLLOWING VARIATIONS OF HEAT TREATMENT  
DURATION AND TEMPERATURE**

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**CHANGES IN THE MECHANICAL BEHAVIOR OF NITINOL**  
**FOLLOWING VARIATIONS OF HEAT TREATMENT**  
**DURATION AND TEMPERATURE**

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# TABLE OF CONTENTS

<b>ACKNOWLEDGEMENTS.....</b>	<b>iii</b>
<b>LIST OF TABLES.....</b>	<b>v</b>
<b>LIST OF FIGURES.....</b>	<b>vi</b>
<b>LIST OF ABBREVIATIONS.....</b>	<b>ix</b>
<b>SUMMARY .....</b>	<b>x</b>
<b>CHAPTER 1: INTRODUCTION .....</b>	<b>1</b>
Nitinol.....	1
<i>Discovery</i> .....	1
<i>Transformation Mechanisms</i> .....	1
<i>Effects of Heat Treatment</i> .....	6
<i>Effects of Cold-Working</i> .....	9
<b>CHAPTER 2: EXPERIMENTAL METHODS.....</b>	<b>12</b>
<b>CHAPTER 3: EXPERIMENTAL RESULTS .....</b>	<b>15</b>
<i>Hot-Rolled Nitinol</i> .....	15
<i>Cold-Drawn Nitinol</i> .....	22
<b>CHAPTER 4: DISCUSSION .....</b>	<b>28</b>
<b>CHAPTER 5: CONCLUSIONS.....</b>	<b>45</b>
<b>REFERENCES.....</b>	<b>48</b>

## LIST OF TABLES

Table 1: Material properties as reported by Frick et al. <sup>5</sup> with the exception of (*) indicating temperatures of the material used in this study.....	9
Table 2: Definitions of Properties extracted from stress-strain results, as defined by ASTM-F2516.	13

## LIST OF FIGURES

Figure 1: Schematic representation of shape-memory transformation.....	3
Figure 2: Simplified schematic of stress-induced martensite transformation. ....	4
Figure 3: Schematic of the stress-strain curve during shape-memory, pseudoelastic and plastic deformation material responses due to an increase in testing temperature.....	5
Figure 4: R-phase effect on Nitinol stress-strain response <sup>3</sup> .....	6
Figure 5: Sketch representation of a typical stress-strain response. ....	14
Figure 6: Upper Plateau Strength (UPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hour durations. ....	15
Figure 7: Lower Plateau Strength (LPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	16
Figure 8: Shifted LPS curves of hot-rolled Nitinol. The original 8 hr duration curve is shown with the 90 min curve shifted to the left by one temperature group and the 10 min curve shifted to the left by six temperature groups. Both 10 min and 90 min curves have been truncated on the left side.....	17
Figure 9: Start of Lower Plateau Strength (SLP) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	18
Figure 10: Ultimate Tensile Strength (UTS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.....	19
Figure 11: Residual Elongation (RE) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	20
Figure 12: Strain to failure of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	21
Figure 13: Simple schematics representing the overall trends seen in the hot-rolled data;.....	21
Figure 14: Upper Plateau Strength (UPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	22
Figure 15: Lower Plateau Strength (LPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations .....	23

Figure 16: Start of Lower Plateau Strength (SLP) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	24
Figure 17: Ultimate Tensile Strength (UTS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	25
Figure 18: Residual Elongation (RE) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	26
Figure 19: Strain to failure of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	27
Figure 20: Simple schematics representing the overall trends seen in the cold-drawn data; .....	27
Figure 21: Upper Plateau Strength (UPS) of hot-rolled (H) and cold-drawn (C) Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	31
Figure 22: Lower Plateau Strength (LPS) of hot-rolled (H) and cold-drawn (C) Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	31
Figure 23: Hysteresis, Upper Plateau Strength (UPS) minus Lower Plateau Strength (LPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	33
Figure 24: Hysteresis, Upper Plateau Strength (UPS) minus Lower Plateau Strength (LPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	34
Figure 25: Upper Plateau Strength (UPS) vs. Lower Plateau Strength (LPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	36
Figure 26: Representative curves from regions 1-H, 2-H, and 3-H demonstrating the effects of high, moderate, and low power heat treatments respectively. ....	38
Figure 27: Upper Plateau Strength (UPS) vs. Lower Plateau Strength (LPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	39
Figure 28: Representative curves from regions 1-C and 2-C demonstrating the effects of moderate and low power heat treatments respectively. ....	40
Figure 29: Upper Plateau Strength (UPS) vs. Lower Plateau Strength (LPS) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	41

Figure 30: Upper Plateau Strength (UPS) vs. Residual Elongation (RE) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.....	42
Figure 31: Upper Plateau Strength (UPS) vs. Start of Lower Plateau strength (SLP) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.....	43
Figure 32: Ultimate Tensile Strength (UTS) vs. Residual Elongation (RE) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations. ....	44



## LIST OF ABBREVIATIONS

A	austenite
A <sub>s</sub>	austenite start temperature
A <sub>f</sub>	austenite finish temperature
EF	strain to failure
LPS	lower plateau strength
M	martensite
M <sub>d</sub>	martensite deformation limit temperature
M <sub>f</sub>	martensite finish temperature
M <sub>s</sub>	martensite start temperature
Ni	Nickel
Nitinol	Nickel-Titanium
R-phase	rhobohedral-phase
RE	residual elongation
SLP	start of lower plateau strength
T	temperature
Ti	Titanium
UPS	upper plateau strength
UTS	ultimate tensile strength

## SUMMARY

The successful use of Nickel-Titanium (Nitinol) in biomedical applications requires an accurate control of its unique mechanical properties. The purpose of this study is to analyze the effects of a wide range of heat treatments on the mechanical behavior of hot-rolled and cold-drawn Nitinol. Results comprise an understanding of the effect of heat treatment temperature and time variation on final material response which is imperative for optimization of material properties. Thirty-three heat treatment variations are tested by combining three durations, 10 minutes, 90 minutes, and 8 hours, with eleven different heat treatment temperatures between 200°C and 440°C. Following heat treatment, the Nitinol samples undergo tensile testing with upper plateau strength, lower plateau strength, ultimate tensile strength, strain to failure, and residual elongation compared for all test groups.

Heat treatment “power” is used to describe the efficacy of different combinations of heat treatment temperature and duration. When using hot-rolled Nitinol, results show a low heat treatment power does not create significant precipitation hardening or a significant decrease in martensite transformation stress, resulting in a high upper plateau strength, high residual strain values, and evidence of plastic deformation upon unloading. Moderate power treatments lead to sufficient hardening of the material and a decrease in martensite transformation stress resulting in a pseudoelastic response. Increasing to a high treatment power further decreases the transformation stress and increases the martensite transformation temperature leading to a shape-memory response in hot rolled Nitinol. When using cold-drawn Nitinol, low and moderate heat treatment power levels result in the material exhibiting a pseudoelastic response. Increasing heat treatment power shows the same effects on martensite transformation stress and temperature as seen with the hot-rolled material resulting in a material response transition from pseudoelastic to shape memory.

# CHAPTER 1

## INTRODUCTION

### **Nitinol**

#### *Discovery*

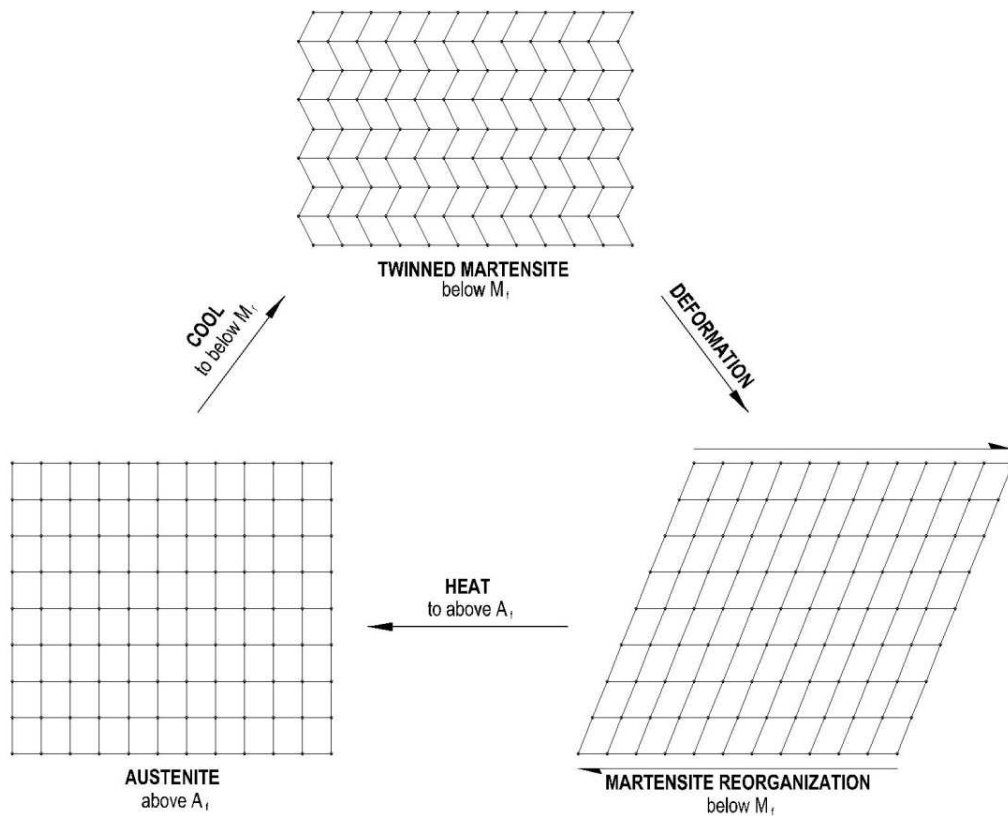
Nitinol is a near-equiatomic nickel titanium alloy that exhibits the unique properties of shape-memory and pseudoelasticity. In 1958, William J. Buehler, a metallurgist at the U.S. Naval Ordnance Laboratory (NOL), discovered this alloy while working on a project to develop metallic materials for the nose cone of the U.S. Navy Polaris reentry vehicle. He named the material NITINOL (Nickel Titanium Naval Ordnance Laboratory) but it wasn't until 1961 that the exceptional shape memory property was uncovered<sup>1</sup>. During a laboratory management meeting a thin strip of Nitinol was used to demonstrate the material's unique fatigue-resistant properties. The strip was bent into an accordion shape by short longitudinal folds and passed around the conference table to be repeatedly compressed and stretched at room temperature without breaking. Dr. David S. Muzzely, one of the Associate Technical Directors, used his pipe lighter to apply heat to the strip and the compressed Nitinol stretched out longitudinally. This marked the discovery of the alloy's shape-memory effect, a characteristic by which the alloy can change its shape reversibly and repeatedly with heating and cooling<sup>1,2\</sup>

#### *Transformation Mechanisms*

The shape-memory and pseudo-elastic capabilities of Nitinol are attributed to a reversible phase change from an austenitic to a martensitic microstructure<sup>3</sup>. Each atom of nickel is surrounded by four atoms of titanium, creating a three-dimensional symmetric grid. The unique crystal structure formed by the atomic forces binding these atoms has the ability to exhibit a solid-state transition between the two phases<sup>4</sup>. The first requisite is the parent phase, an

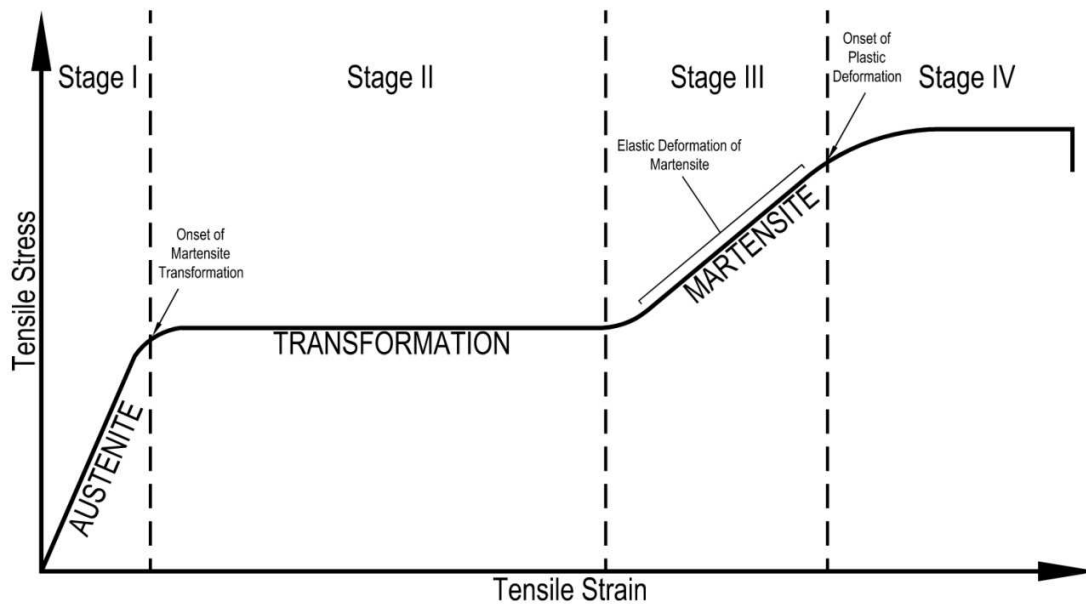
atomically ordered, cubic B2 austenite phase. Second, restructuring occurs into a complex, monoclinic B19' martensite phase. This transformation allows for the recovery of large strains (8-10% compared to 1% in traditional metals) either through an increase in material temperature or a decrease in applied mechanical stress<sup>5</sup>.

A schematic of the shape-memory transformation is shown in Figure 1. Cooling Nitinol below the martensite start and finish temperatures,  $M_s$  and  $M_f$  respectively, restructures the material into the low-temperature, unstable, martensitic phase, which enables easy deformation. At this phase the material is composed entirely of a twinned lattice structure characterized by needle-like crystals arranged in a herringbone fashion. With the application of stress at this state, reorientation and detwinning occurs as twin boundaries move to produce a shape that better accommodates the applied load. Reheating the material through the austenite start and finish temperatures,  $A_s$  and  $A_f$  respectively, results in a return to the original form in the stronger, high-temperature, austenitic phase. This reversible process describes the shape-memory effect of Nitinol<sup>1,3,4</sup>.



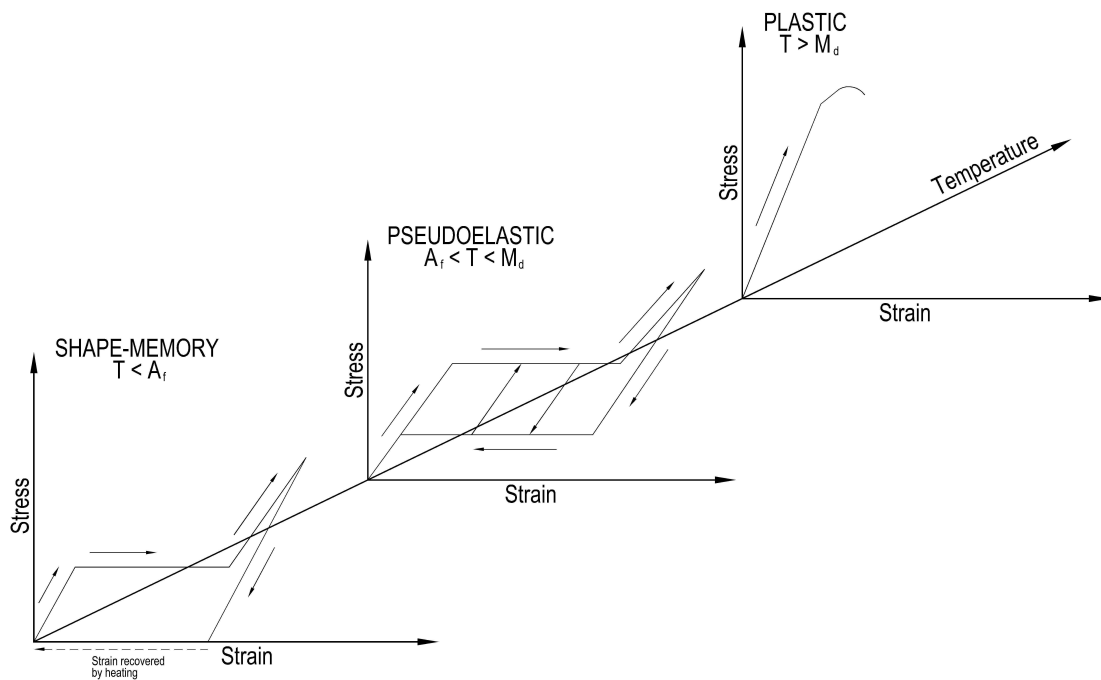
**Figure 1:** Schematic representation of shape-memory transformation.

Formation of martensite can also be stress-induced by loading the austenitic phase above the  $A_f$  temperature (Figure 2). With initial loading, the material behaves in a linear elastic manner (Stage I) until the initiation of martensite formation at a strain of  $\sim 1\%$ . A plateau region (Stage II) characterizes the transformation process and usually continues to a strain of  $\sim 6\%$  at which point the material is nearly fully martensite. The lattice structure responds elastically with continued deformation (Stage III) until a critical stress is reached where yielding occurs and plastic deformation continues until failure (Stage IV) <sup>4,7</sup>.



**Figure 2:** Simplified schematic of stress-induced martensite transformation.

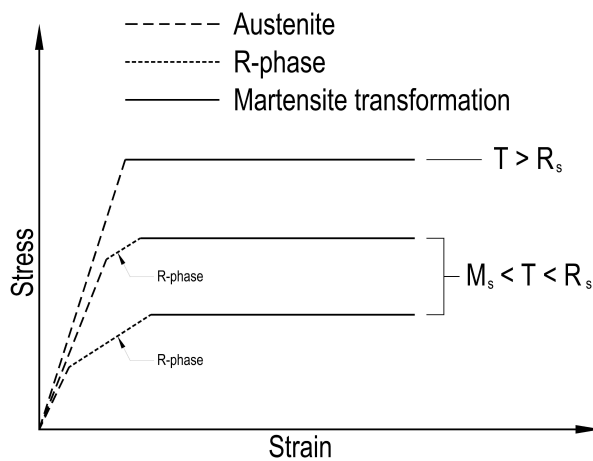
Stress-induced phase transformation that occurs between the  $A_f$  temperature and the martensite deformation limit temperature,  $M_d$ , is reversible as the local interatomic bonds remain intact. Strain induced before the onset of plastic deformation is recovered during unloading, a phenomenon known as ‘pseudoelasticity’. The pseudoelastic response, obtained in the temperature range  $A_f < T < M_d$ , follows a hysteresis, with the upper plateau corresponding to the forward transformation from austenite to martensite and the lower plateau corresponding to the reverse transformation from martensite back to the parent austenite phase. Above the  $M_d$  temperature, which is greater than the  $A_f$  temperature, stress-induced martensite will not form. Instead, plastic deformation occurs as the critical stress required to form martensite is greater than that required to drive dislocations, resulting in a loss of pseudoelasticity and a behavior similar to a traditional metal<sup>5</sup>. Figure 3 is a simple schematic representation of the stress-strain response during shape-memory, pseudoelastic, and plastic deformation material behavior due to different testing temperatures<sup>1,3,4</sup>.



**Figure 3:** Schematic of the stress-strain curve during shape-memory, pseudoelastic and plastic deformation material responses due to an increase in testing temperature.

In near-equiatomic Nitinol the austenite to martensite transformation may occur directly, ( $A \rightarrow M$ ) or proceed through an intermediate phase known as the R-phase (rhombohedral-phase).<sup>6,7</sup> During transformation the cubic lattice of the austenite phase elongates along one of its diagonals reducing the cube angle and producing a rhombohedral structure. When the R-phase reorientation has accommodated its maximum strain, the elastic energy increases until the R-phase transforms to martensite; capable of accommodating more strain and further reducing strain energy<sup>8</sup>. This intermediate phase is characterized mechanically by a slope change in the stress-strain curve prior to the martensite transformation plateau. As seen in Figure 4, the material is composed of both austenite and R-phase constituents when testing occurs within the range  $M_s < T < R_s$ . As described earlier, the initial slope of the curve is the elastic deformation of the austenite phase. A sufficient increase in stress leads to a slight change in slope, marking

the onset of the R-phase component. The R-phase region of the curve links the austenite component to the transformation plateau where martensite formation begins. Increasing the testing temperature increases the austenite region of the curve until it is increased above  $R_s$  which typically removes the R-phase component resulting in a direct transformation from austenite to martensite<sup>3</sup>.



**Figure 4:** R-phase effect on Nitinol stress-strain response<sup>3</sup>.

#### *Effects of Heat Treatment*

Heat treatment or aging is one of the simplest and most economical methods for manipulating the transformation properties of shape-memory alloys<sup>9</sup>. Heat treatment effect is dependent on time, temperature, processing history, and the amount of prior cold work<sup>10</sup>. Thus, variations in heat treatment temperature and duration can be used to identify optimal aging treatments to produce desired shape-memory or pseudoelastic effects for specific applications. It has been established that aging treatments performed on slightly nickel-rich Nitinol lead to the formation of  $Ti_3Ni_4$  precipitates<sup>11</sup>. The formation of coherent or semi-coherent precipitates leads to the development of strong local stress fields in the Nitinol matrix. These local stress fields result in local resolved shear stresses creating preferential nucleation sites for martensitic



transformation<sup>11</sup>. The critical stress level required for martensite transformation is thus lowered and  $M_s$  increases as predicted by a shift in the Clausius-Clapeyron line in stress-temperature space<sup>11,18</sup>.

Effect of aging on transformation temperature is linked to precipitate size and coherency. As previous studies report precipitates are perfectly coherent at about 10 nm and lose coherency completely at about 300 nm<sup>11</sup>. As this incoherent critical particle size is approached, dislocations are generated around the precipitates and local shear stress magnitudes decrease. In contrast, more coherent precipitates increase local shear stress and consequently increase transformation temperatures, decreasing the critical transformation stress<sup>13,9,12</sup>. It has also been noted that larger but still coherent precipitates create a larger strain field when compared to smaller coherent precipitates, and thus have a more dramatic effect on transformation temperatures<sup>5</sup>. Frick et al. reports the effects of 1.5 hour heat treatments on precipitate size and transformation temperatures for similar compositions of Nitinol to the ones used in this study<sup>5</sup>. Select results are provided in Table 1 along with the transformation temperatures of the as received material used in this study. Yan et al. reports the effect of precipitate size on fatigue properties<sup>14</sup>. With precipitate size of ~10 nm following heat treatment of 400°C and ~300 nm following a treatment at 500°C, the smaller coherent precipitates improved fatigue resistance. Supporting literature describes a decrease in fatigue resistance as precipitates of ~100 nm begin to lose coherency<sup>14</sup>.

Aging also results in a decrease of Ni concentration as precipitates change the Ni content of the surrounding matrix. This depletion of Ni has also been shown to increase transformation temperatures<sup>11,18</sup>. More complex, multi-stage transformation behaviors, including the existence of an R-phase, are also the result of aging. It is important to note that as increasing heat treatment temperature results in the formation and growth of  $Ti_3Ni_4$  precipitates, high

temperatures of  $\sim 600^\circ\text{C}$  solutionizes nearly equiatomic Nitinol<sup>5</sup>. Mahesh et al. reports increasing transformation temperatures with increasing heat treatment temperatures up to  $500^\circ\text{C}$ <sup>9</sup>. Similarly, Frick et al. presents a minimum transformation stress with increasing heat treatment temperature up to  $450^\circ\text{C}$  followed by a progressive increase following  $550^\circ\text{C}$  and  $600^\circ\text{C}$  treatments.

Along with increasing  $M_s$ , studies show that the formation of small coherent  $\text{Ti}_3\text{Ni}_4$  precipitates also increases the stress needed for plastic flow<sup>5</sup>. This precipitate strengthening or hardening effect is attributed to suppression of dislocation motion and the resulting preference for the stress-induced martensitic transformation. Gall et al. provides evidence for this effect by reporting a higher value of  $M_d$  for materials with increased  $\text{Ti}_3\text{Ni}_4$  precipitates due to a higher Ni concentration<sup>7</sup>. Precipitation hardening is also evident in the shape of the stress-strain curve. It is important here to note that when the material resides in the self-accommodated martensitic state, stage II and stage III deformation continues via martensite reorientation and martensite deformation<sup>7,18</sup>. It is believed that precipitates suppress martensite deformation resulting in a shortened stage II plateau as martensite reorientation is favored. Similarly, the effect on the slopes of stage I and stage III shows evidence for the activation of inelastic deformation mechanisms in addition to elastic deformation of the austenite and martensite phases<sup>7</sup>.

**Table 1:** Material properties as reported by Frick et al.<sup>5</sup> with the exception of (\*) indicating temperatures of the material used in this study.

Material	Precipitate size	Transformation temperatures
Hot-rolled (as received)	< 1 nm	*A <sub>s</sub> = -15°C to -8°C
Hot-rolled (aged at 300°C for 1.5 h)	not measured	R-to-M = -50°C
Hot-rolled (aged at 350°C for 1.5 h)	~10 nm	R-to-A = above 25°C A-to-R = below 25°C Estimated M <sub>s</sub> = -20°C to -35°C
Hot-rolled (aged at 400°C for 1.5 h)	not measured	M-to-R = 0°C R-to-M = -15°C Estimated M <sub>s</sub> = -35°C
Hot-rolled (aged at 450°C for 1.5 h)	~50 nm	M-to-R = 14°C R-to-A = above 25°C Estimated M <sub>s</sub> = 5°C to -10°C
Hot-rolled (aged at 550°C for 1.5 h)	~300 nm	R-to-M = -35°C
Hot-rolled (aged at 600°C for 1.5 h)	~1 nm	not confirmed
Cold-drawn (as received)	not confirmed	*A <sub>s</sub> = -16°C
Cold-drawn (aged at 350°C for 1.5 h)	not confirmed	not confirmed
Cold-drawn (aged at 450°C for 1.5 h)	not confirmed	M-to-R = 15°C Estimated M <sub>s</sub> = 0°C
Cold-drawn (aged at 550°C for 1.5 h)	not confirmed	not confirmed
Cold-drawn (aged at 600°C for 1.5 h)	~ 1 nm	M-to-A = -5°C A-to-M = 20°C

### *Effects of Cold-Working*

Cold-working (drawing or rolling) is a typical process utilized to generate the appropriate material shape for specific applications as well as produce shape-memory or pseudoelastic properties<sup>16</sup>. This type of deformation processing imparts large plastic deformations creating a high density of dislocations within the alloy<sup>16,17,18</sup>. These dislocations generate an internal stress state that inhibits interface mobility, suppressing the martensite phase, and resulting in an overall decrease of the transformation temperatures. This leads to an increase in the critical stress needed to induce martensite transformation. This ‘pinned’ residual martensite remains until the dislocations are removed. An increase in transformation hardening in the stress-strain curve is also found. With increasing percentages of cold-working, M<sub>s</sub> decreases and the stress level for the onset of plastic strain increases<sup>11,17</sup>. Cold-working also results in the appearance of the R-

phase during transformation, attributed to the increased dislocation density creating locations for the R-phase to nucleate<sup>17</sup>.

Following a fixed percentage of cold-working, the effect of heat treatment is dependent on heat treatment temperature. With increasing heat treatment temperature the cold worked material undergoes an increase in dislocation annihilation as well as precipitate growth. It is important to note that the large dislocation density of cold-worked Nitinol affects precipitate growth. Precipitates tend to form on isolated dislocations, hindering the dislocation movement and thus annihilation. Also, dense dislocation populations inhibit precipitate formation, causing precipitates to be smaller and lose coherency faster when compared to non-deformed material<sup>5,13</sup>. This leads to an “interaction” effect of precipitate growth and dislocations that combine to create the microstructure result of heat treatment. Despite these combined effects, Frick et al. demonstrates that heat treatment of cold-drawn Nitinol has a similar influence on the stress-strain properties when compared to the effects of heat treatment on hot-rolled material<sup>5</sup>. Therefore, the authors conclude that precipitates have a strong influence even in light of the presence of residual martensite and a high dislocation density<sup>5</sup>.

Nitinol of near-equiatomic composition is one of the most technologically significant shape-memory alloys currently available with increasing importance in the medical device industry<sup>9</sup>. Along with the capability to exhibit shape-memory and pseudoelastic effects, Nitinol also shows excellent biocompatibility<sup>9</sup>. The successful use of Nitinol for biomedical and other industry applications is linked to the ability to control its unique transformational and mechanical properties. The thermomechanical response is dependent on alloy composition, material processing, heat treatment, and prior thermomechanical cycling<sup>13,17</sup>. Although prior work has studied heat treatment effects in both cold drawn and hot rolled Nitinol, this previous work has

focused on isolated heat treatment parameters and not on the range of temperatures and times that could be possibly used in practice. This study compares the effects of various heat treatment temperatures and durations on the mechanical behavior of both cold-drawn and hot-rolled Nitinol. The objective of the present work is to uncover the trends in material responses due to changes in heat treatment temperature and duration to allow for proper optimization of material properties for specific applications.

## CHAPTER 2

### EXPERIMENTAL METHOD

Two forms of commercial polycrystalline Nitinol were used in this study; referred to here as “hot-rolled” material (55.9 wt.% Ni) and “cold-drawn” material (56.0 wt.% Ni). Hot-rolled samples were cut from 12.7 mm diameter cylindrical bars available from Saes Smart Metals Inc. The bars were hot-rolled, straightened, and centerless ground. Electro-discharge machining was used to cut dog bone tensile samples with a 10.6 mm gauge length and a 1.5 x 0.5 mm gauge cross section. A light sand-blasting was performed on all samples to remove surface oxidation produced by machining. Cold-drawn test samples were cut from cold-drawn wire with a nominal diameter of 0.508 mm available from Fort Wayne Metals Inc.

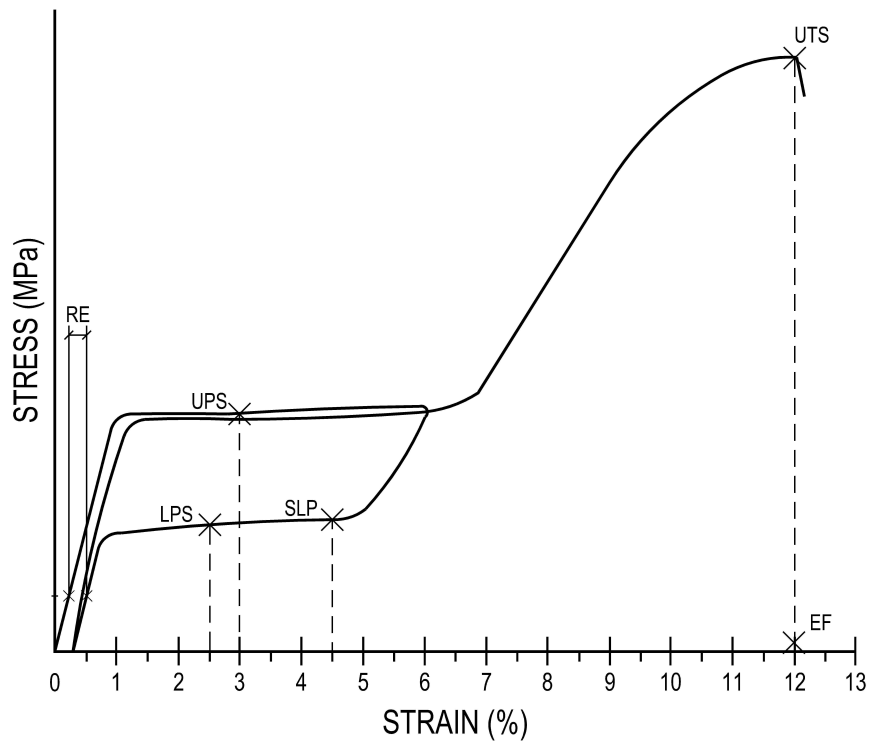
Samples were heat treated at the specified temperature in an Isotemp Muffle Furnace, Fisher Scientific Model 550-126. A Fluke digital multimeter was used simultaneously to monitor heat treatment temperature. At the conclusion of the specified heat treatment duration all samples were immediately water quenched. Following heat treatment, testing was performed at 37°C on a universal testing machine (MTS Insight 2) using a 2kN load cell, a laser extensometer to record strain, and an air thermal chamber to maintain testing temperature. Tensile testing was executed per ASTM-F2516, *Standard Test Method for Tension Testing of Nickel-Titanium Superelastic Materials*. Prior to testing, critical dimensions were measured for each sample using digital calipers. Samples were loaded into the thermal chamber and heated to 37°C. A thermocouple reading was used to ensure testing temperature was reached by the chamber as well as the individual sample. As specified by ASTM-F2516, testing protocol involved pulling the specimen to 6% strain and reversing the motion to unload the specimen to

less than 7 MPa (Cycle 1), followed by pulling the specimen to failure (Cycle 2). Cycle 1 was completed at a rate of 0.42 mm/min and Cycle 2 completed at a rate of 4.2 mm/min.

Thirty-three heat treatment variations were tested on both the hot-rolled and cold-drawn Nitinol. Sample groups were formed by combining three durations, 10 minutes, 90 minutes, and 8 hours, with eleven different heat treatment temperatures ranging from 200°C to 440°C in 30 degree increments with additional groups at 385°C and 395°C. Tensile testing was performed on a minimum of n=3 from each sample group. Stress-strain behavior was analyzed along with the following properties from each stress-strain curve: upper plateau strength, lower plateau strength, start of lower plateau strength, ultimate tensile strength, strain to failure, and residual elongation. Definitions of all properties extracted can be found in Table 2. A sketch representation of a typical stress-strain response is provided in Figure 5.

**Table 2:** Definitions of Properties extracted from stress-strain results, as defined by ASTM-F2516.

Property	Definition
Upper Plateau Strength (UPS)	Stress at 3% strain during the initial loading of the sample.
Lower Plateau Strength (LPS)	Stress at 2.5% strain during unloading of the sample after loading to 6% strain.
Start of Lower Plateau Strength (SLP)	Stress at 4.5% strain during unloading of the sample after loading to 6% strain.
Ultimate Tensile Strength (UTS)	Maximum resistance to fracture.
Strain to Failure (EF)	Maximum strain reached.
Residual Elongation (RE)	Difference between the strain at a stress of 7.0 MPa during unloading of the sample and the strain at a stress of 7.0 MPa during initial loading of the sample.



**Figure 5:** Sketch representation of a typical stress-strain response.

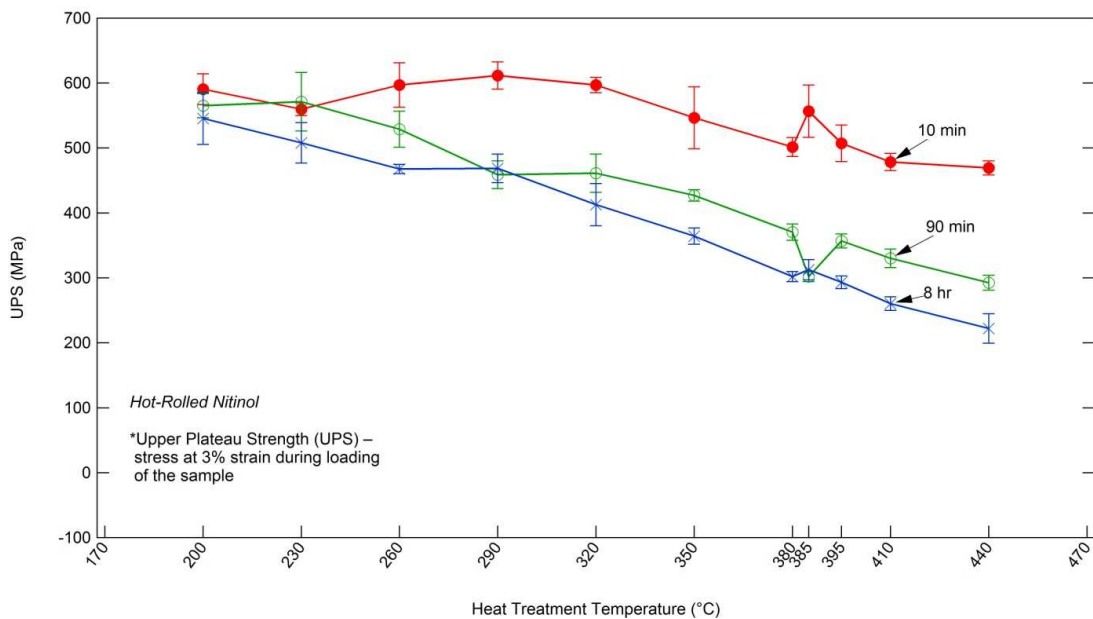


## CHAPTER 3

### EXPERIMENTAL RESULTS

#### Hot-Rolled Nitinol

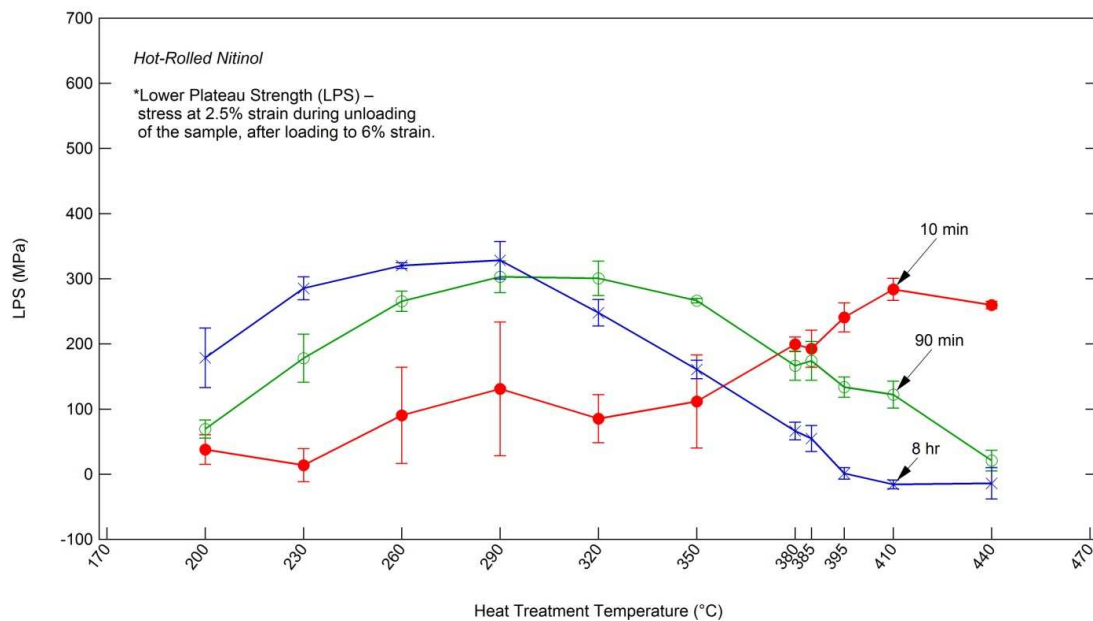
Figure 6 shows the Upper Plateau Strength (UPS) of the hot-rolled Nitinol for each heat treatment temperature, grouped by heat treatment duration. Results for the 90 minute and 8 hour durations show an approximately linear decrease in UPS with increasing heat treatment temperature. The 10 minute duration curve exhibits this same decrease following heat treatment at 290°C. A schematic showing the overall trend for hot-rolled UPS is shown in Figure 13(a).



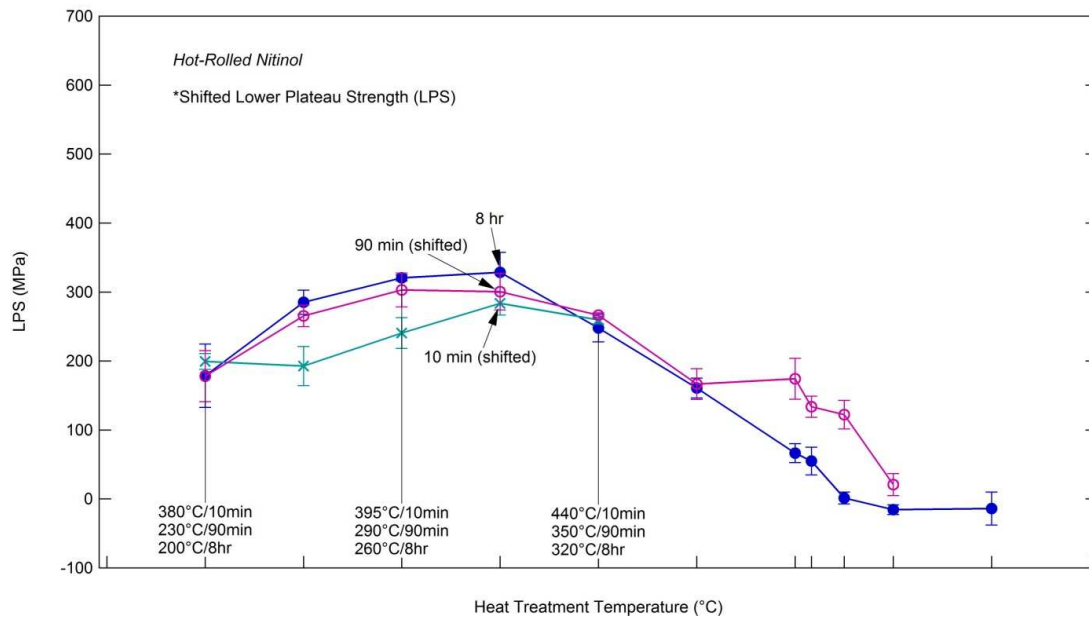
**Figure 6:** Upper Plateau Strength (UPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hour durations.

Figure 7 shows the Lower Plateau Strength (LPS) of the hot-rolled Nitinol for each heat treatment temperature, grouped by heat treatment duration. The 90 minute and 8 hour heat treatment durations exhibit a bell-curve response with increasing heat treatment temperature.

Both durations exhibit an increase in LPS to a maximum value found at 290°C followed by a continued decrease for the remaining temperature groups. The 10 minute duration sample groups exhibit low LPS values for the 220-350°C heat treatment temperatures due to a partially pseudoelastic response. The 10min/380°C sample group exhibits an average LPS value (199.3 MPa) similar to the 90min/230°C group and 8hr/200°C group (178.1 and 178.6 MPa respectively). The remaining 10 minute duration groups from 385-440°C demonstrate a very similar bell-curve response to those seen in the 200-320°C temperature range for the 90 minute and 8 hour groups. This trend can be seen in Figure 8 which displays the 8 hour LPS data plotted with the 90 minute curve (shifted to the left by one temperature group) and the 10 minute curve (shifted to the left by six temperature groups). A schematic showing the overall trend for hot-rolled LPS is shown in Figure 13(b).

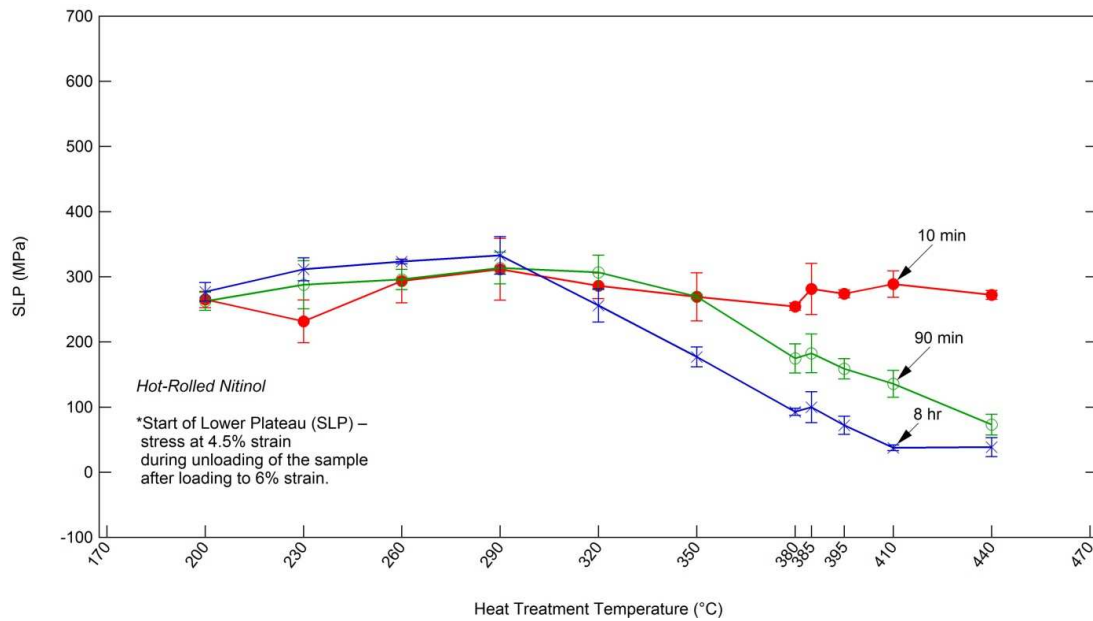


**Figure 7:** Lower Plateau Strength (LPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.



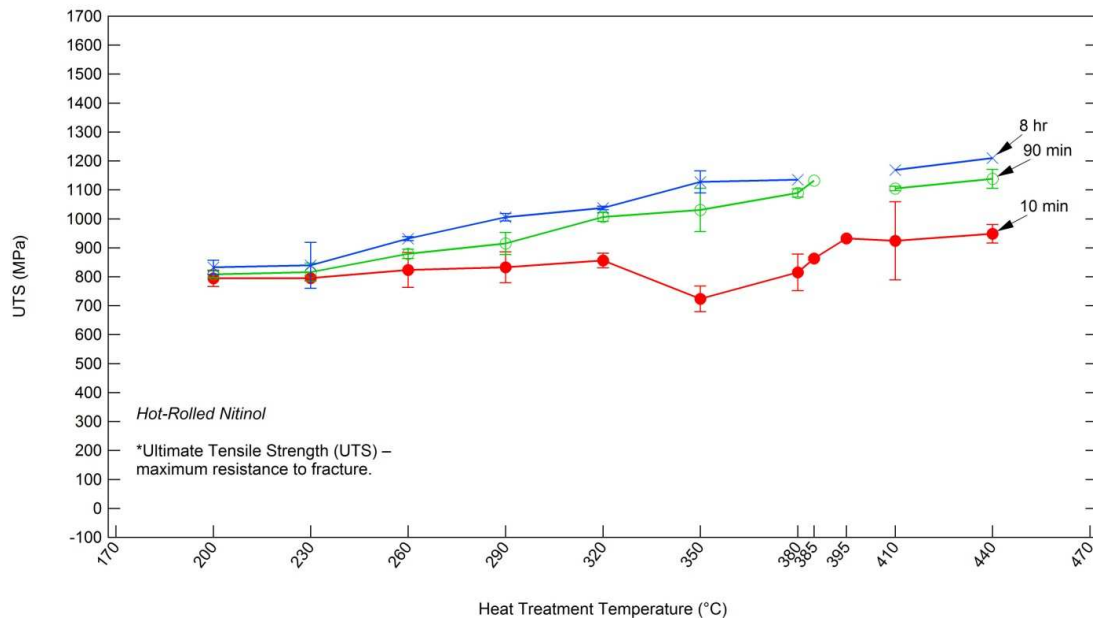
**Figure 8:** Shifted LPS curves of hot-rolled Nitinol. The original 8 hr duration curve is shown with the 90 min curve shifted to the left by one temperature group and the 10 min curve shifted to the left by six temperature groups. Both 10 min and 90 min curves have been truncated on the left side.

Figure 9 shows the Start of Lower Plateau Strength (SLP) of the hot-rolled Nitinol for each heat treatment temperature, grouped by heat treatment duration. All durations show a slight increase in SLP value as heat treatment temperature increases from 200°C to approximately 290°C. Increasing heat treatment temperature beyond 290°C causes a decrease in SLP value for the 90 minute and 8 hour duration groups. A schematic showing the overall trend for these groups is shown in Figure 13(c). The 10 minute duration groups do not show a significant decrease in SLP values, presenting a similar stress value at 4.5% strain during unloading for all heat treatment temperatures.



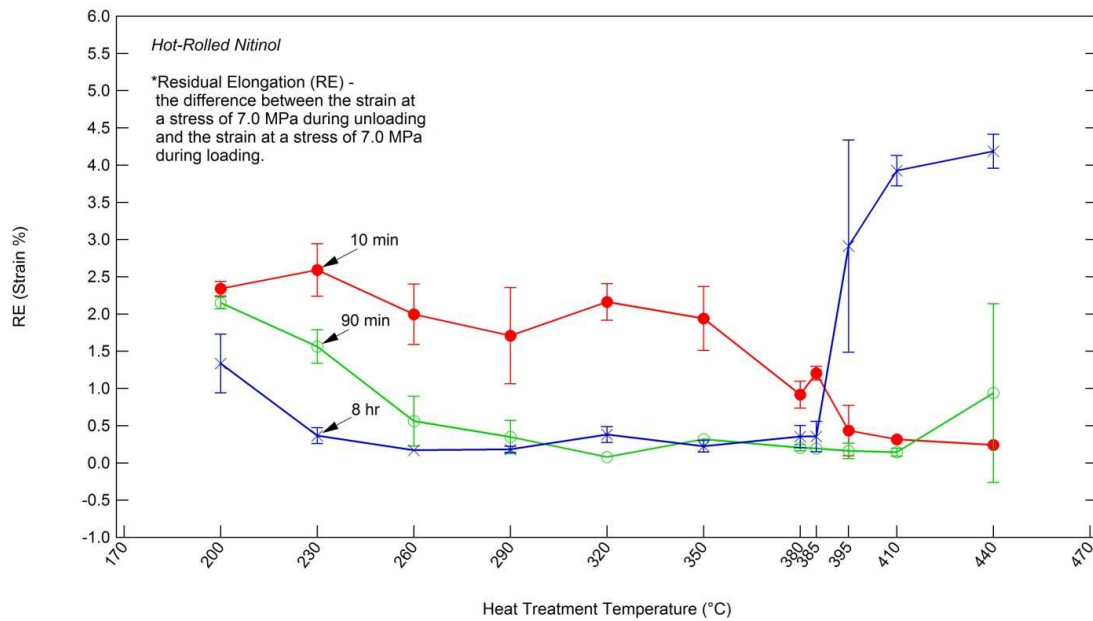
**Figure 9:** Start of Lower Plateau Strength (SLP) of hot-rolled Nitinol following heat treatment at 200–440°C for 10 min, 90 min, and 8 hr durations.

Figure 10 shows the Ultimate Tensile Strength (UTS) of the hot-rolled Nitinol for each heat treatment temperature, grouped by heat treatment duration. Values were not recorded for the 385°C/8hr or 395°C/8hr and 90 min groups due to samples slipping from the tensile grips near the end of testing. A nearly linear increase of UTS with increasing heat treatment temperature is seen for all three heat treatment durations. UTS values are similar for all three durations at 200°C and 230°C. After 230°C UTS values increase; the 8 hour and 90 minute curves exhibit a slightly steeper slope in comparison to the 10 minute curve. Also, at all temperatures from 260°C to 440°C with recorded UTS values, the 8 hour group exhibits the highest UTS followed by the 90 minute group, which is followed by the 10 minute group. A schematic showing the overall trend of the UTS for the hot-rolled Nitinol is shown in Figure 13(d).



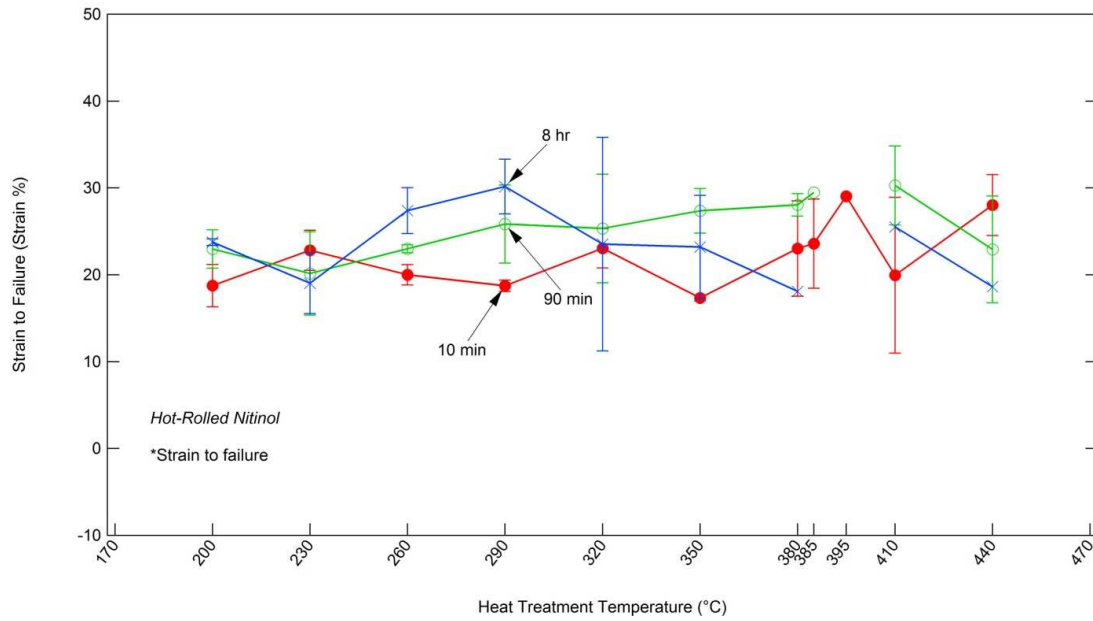
**Figure 10:** Ultimate Tensile Strength (UTS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

Figure 11 shows the Residual Elongation (RE) of the hot-rolled Nitinol for each heat treatment temperature, grouped by heat treatment duration. The 90 minute and 8 hour curves show a decrease in residual elongation with increasing heat treatment temperature from 200-260°C. The 90 minute curve exhibits a nearly steady RE result for the 290-410°C temperature range followed by an increase at 440°C. The 8 hour curve exhibits a similar steady RE response ending with a significant increase from 385°C to 395°C followed by a continued rise to a max at 440°C. In contrast, the 10 minute curve shows similar RE at heat treatment temperatures from 200-350°C followed by a decrease at 380°C. Residual elongation values for the 10 minute curve at temperatures 395-440°C closely match the those seen by the 90 minute and 8 hour curves at the 290-385°C temperatures. A schematic showing the overall trend of RE for the hot-rolled Nitinol is shown in Figure 13(e).

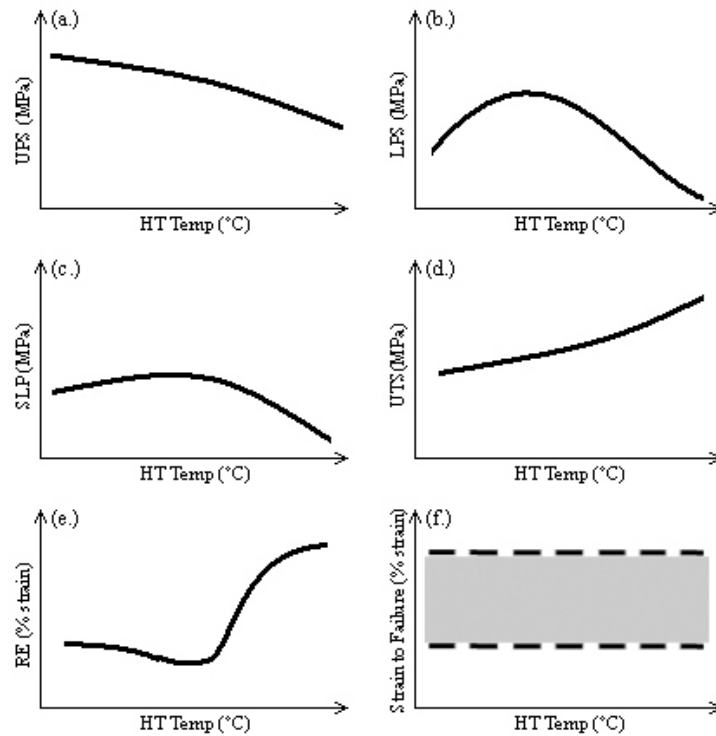


**Figure 11:** Residual Elongation (RE) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

Figure 12 shows the strain to failure results of the hot-rolled Nitinol for each heat treatment temperature, grouped by heat treatment duration. Heat treatment temperature and duration do not show a significant influence on strain to failure. Strain to failure varies between 10 and 40% for all heat treatment durations at all heat treatment temperatures tested. A schematic showing the overall trend of strain to failure for the hot-rolled Nitinol is shown in Figure 13(f).



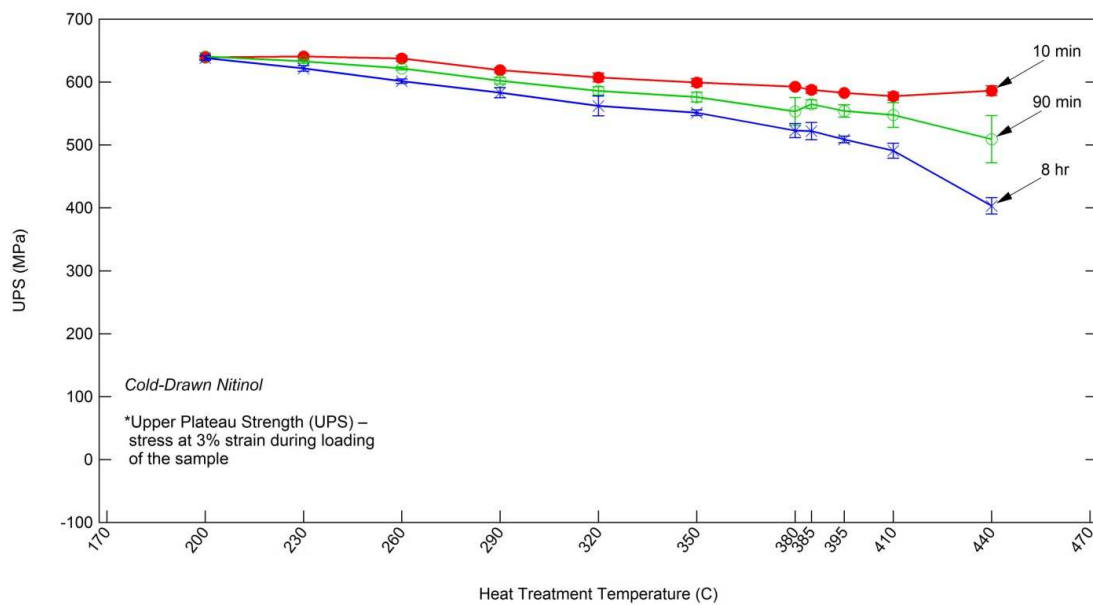
**Figure 12:** Strain to failure of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.



**Figure 13:** Simple schematics representing the overall trends seen in the hot-rolled data; a) UPS, b) LPS, c) SLP, d) UTS, e) RE, f) Strain to failure.

## Cold-Drawn Nitinol

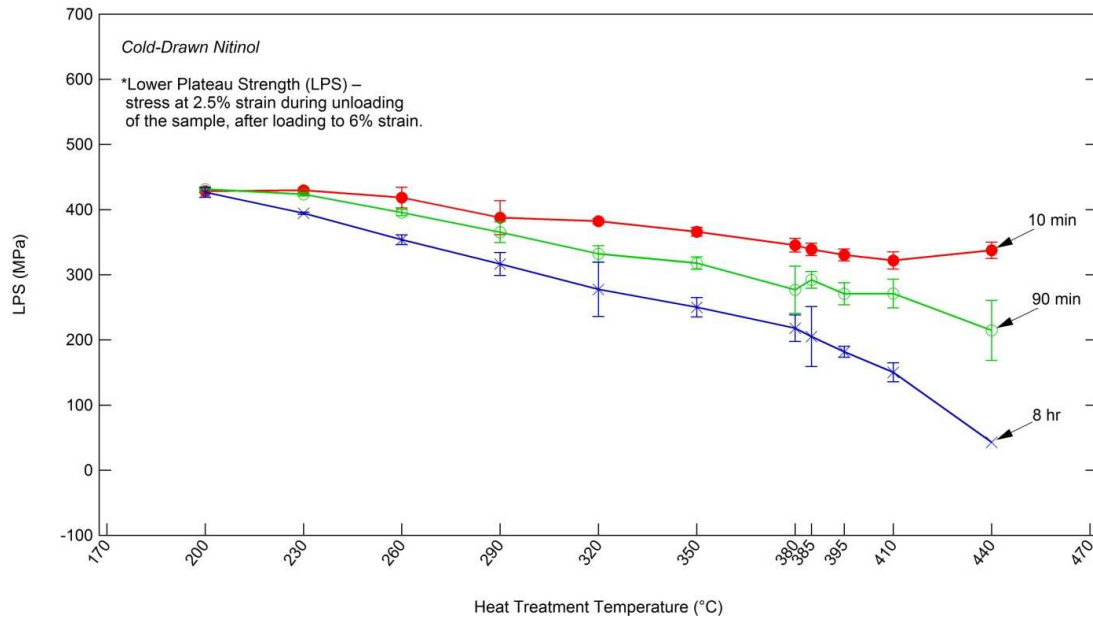
Figure 14 shows the Upper Plateau Strength (UPS) of the cold-drawn Nitinol for each heat treatment temperature, grouped by heat treatment duration. Results for all three durations (10 minutes, 90 minutes, and 8 hours) show an approximately linear decrease in UPS with increasing heat treatment temperature. A schematic showing the overall trend for cold-drawn UPS is shown in Figure 20(a).



**Figure 14:** Upper Plateau Strength (UPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

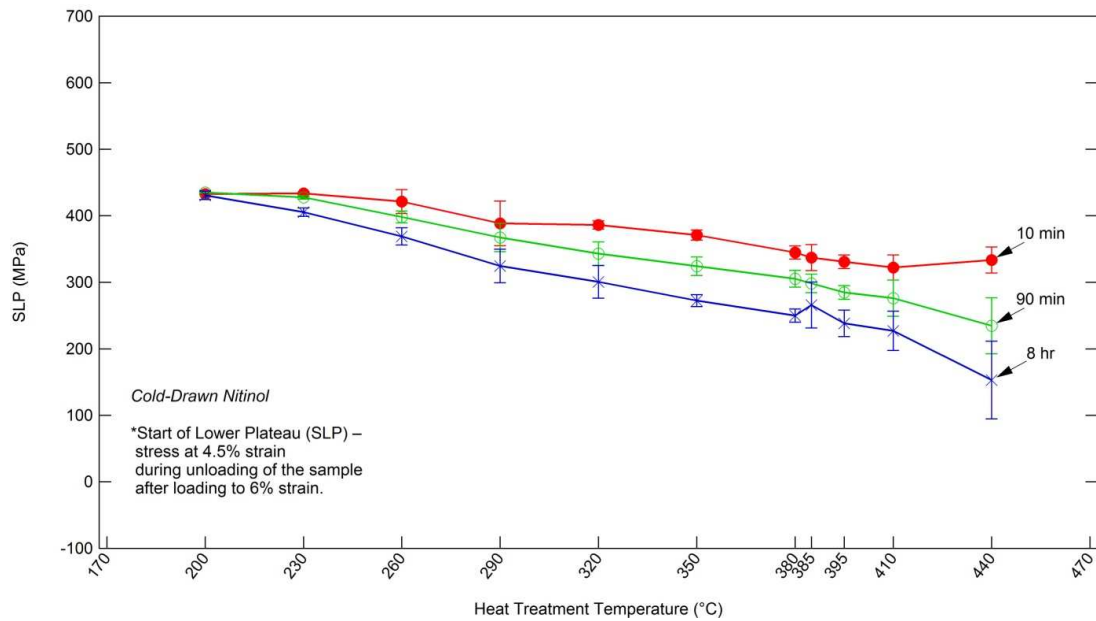
Figure 15 shows the Lower Plateau Strength of the cold-drawn Nitinol for each heat treatment temperature, grouped by heat treatment duration. Results for all three durations show an approximately linear decrease in LPS with increasing heat treatment temperature. The 8 hour curve exhibits a steeper decrease of LPS with increasing temperature, followed by the 90 minute curve and 10 minute curve respectively. A schematic showing the overall trend for cold-drawn LPS is shown in Figure 20(b).





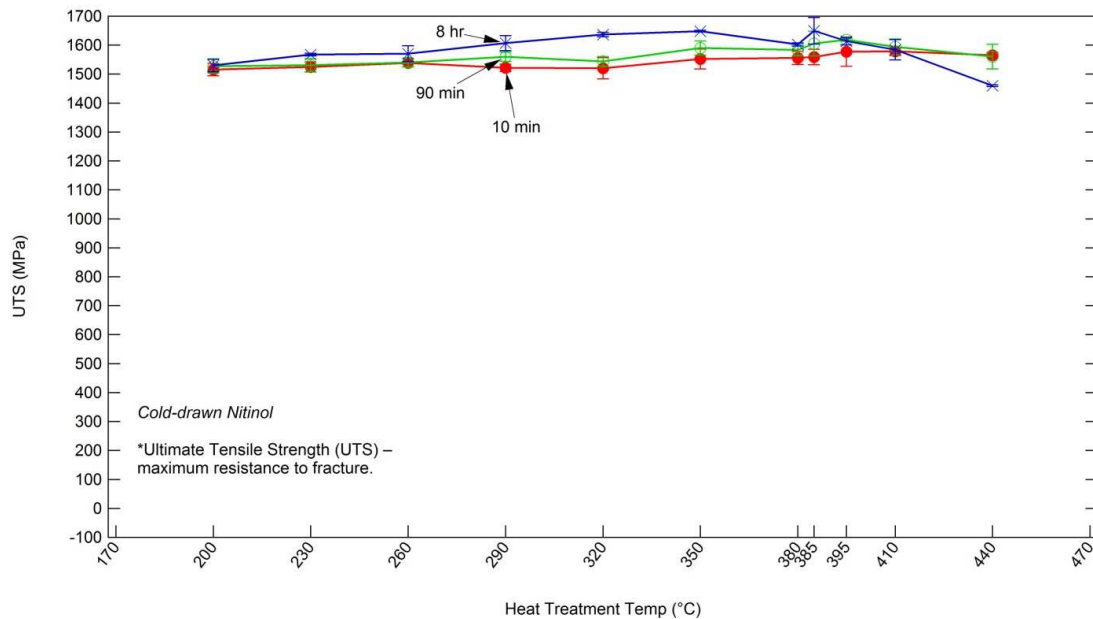
**Figure 15:** Lower Plateau Strength (LPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations

Figure 16 shows the Start of Lower Plateau Strength (SLP) of the cold-drawn Nitinol for each heat treatment temperature, grouped by heat treatment duration. The cold-drawn SLP results demonstrate the same trend as that seen for the cold-drawn UPS and LPS. All three durations show an approximately linear decrease in SLP with increasing heat treatment temperature. Again, the 8 hour curve exhibits a steeper decrease of SLP with increasing temperature, followed by the 90 minute curve and 10 minute curve respectively. A schematic showing the overall trend for cold-drawn SLP is shown in Figure 20(c).



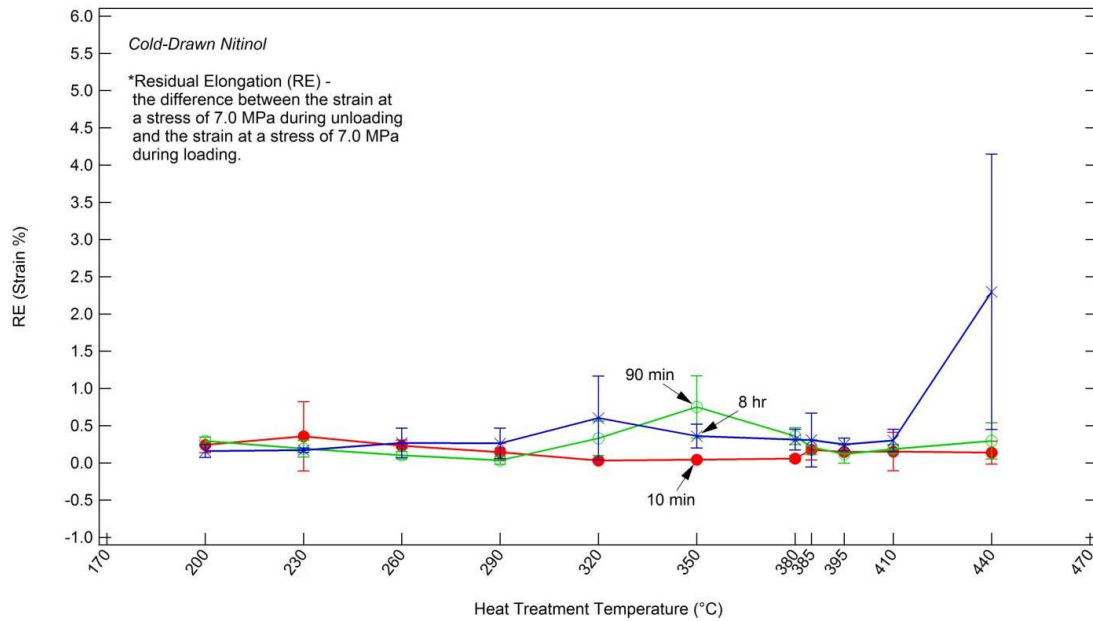
**Figure 16:** Start of Lower Plateau Strength (SLP) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

Figure 17 shows the Ultimate Tensile Strength (UTS) of the cold-drawn Nitinol for each heat treatment temperature, grouped by heat treatment duration. The 90 minute and 8 hour curves show a slight increase in UTS with increasing heat treatment temperatures from 200-350°C followed by a dip at the 380°C heat treatment. UTS values for the 90 minute duration group increase at the 385 and 395°C temperatures followed by a decrease in UTS with increasing temperatures from 410-440°C. The 8 hour curve also shows an increase of UTS at the 385°C temperature. This is followed by a significant decrease of UTS with increasing temperature from 395-440°C. The 10 minute duration groups exhibit a steady to slight increase of UTS with increasing heat treatment temperatures. A schematic showing the overall trend of the UTS for the cold-drawn Nitinol is shown in Figure 20(d).



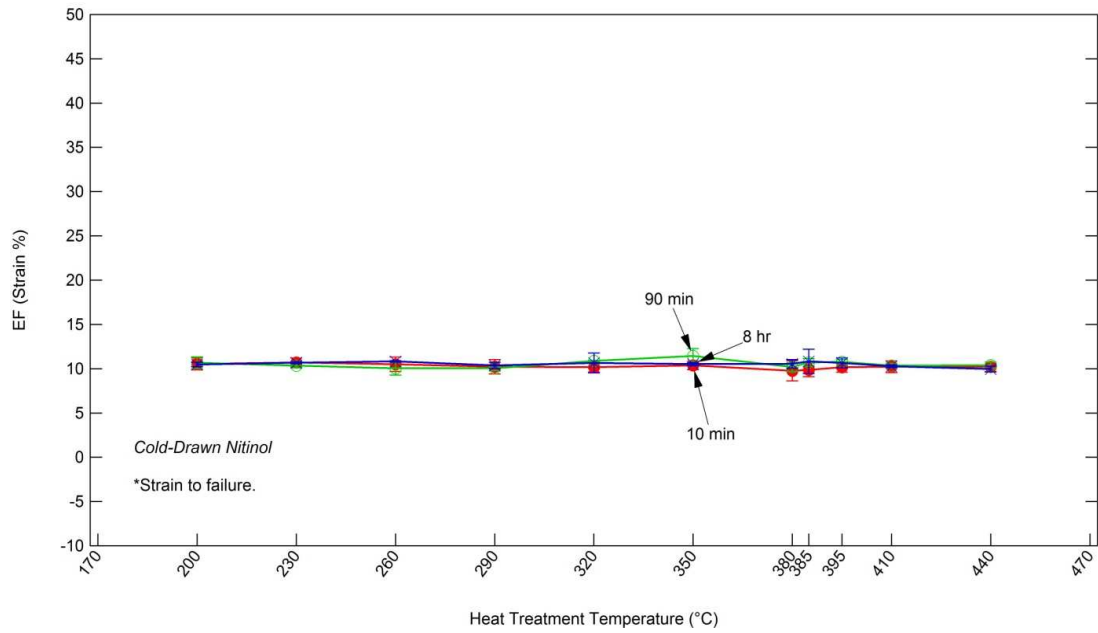
**Figure 17:** Ultimate Tensile Strength (UTS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

Figure 18 shows the Residual Elongation (RE) results of the cold-drawn Nitinol for each heat treatment temperature, grouped by heat treatment duration. All three durations show steady, similar results for the 200-290°C heat treatments. The 8 hour duration group shows an increase in RE at 320°C followed by a slight decrease in RE with increasing temperatures from 350-410°C. At 440°C the 8 hour duration shows a large jump in RE from ~0.3% at 410°C to ~2.3% at 440°C. The 90 minute curve shows an increase of RE with increasing temperature from 290-350°C followed by a decrease of RE at 380°C. A steady RE (~0.15%) is then seen for the remaining heat treatment temperatures. The 10 minute duration groups show a similar RE result (~0.16%) for all temperature groups. A schematic showing the overall trend of RE for the cold-drawn Nitinol is shown in Figure 20(e).

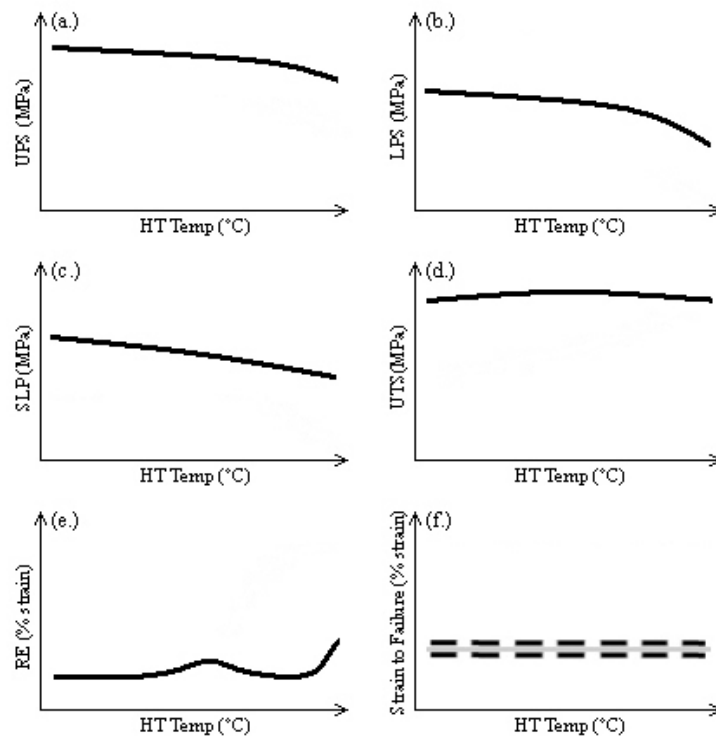


**Figure 18:** Residual Elongation (RE) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

Figure 19 shows the strain to failure results of the cold-drawn Nitinol for each heat treatment temperature, grouped by heat treatment duration. Heat treatment temperature and duration do not show an influence on strain to failure. Results show a steady strain to failure, between 9% and 11%, for all samples tested. A schematic showing the overall trend of strain to failure for the cold-drawn Nitinol is shown in Figure 20(f).



**Figure 19:** Strain to failure of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.



**Figure 20:** Simple schematics representing the overall trends seen in the cold-drawn data; a) UPS, b) LPS, c) SLP, d) UTS, e) RE, f) Strain to failure.

## CHAPTER 4

### DISCUSSION

This study investigates the effects of various heat treatments on the mechanical behavior of both hot-rolled and cold-drawn Nitinol. While a number of heat treatment temperatures have been investigated by previous authors, variations on heat treatment duration along with the eleven temperature groups investigated here have not previously been reported. Tensile testing was performed to compare the stress-strain behavior of each sample group with the following data extracted from each stress-strain curve: upper plateau strength, lower plateau strength, start of lower plateau strength, ultimate tensile strength, strain to failure, and residual elongation. The results provide insight into the effect of heat treatment on processing-structure-property relationships in Nitinol and also reveal some interesting trends in various transformation properties.

It has previously been established that increasing heat treatment temperature results in an increase in precipitates in hot-rolled Nitinol<sup>5</sup>. The size and coherency of  $Ti_3Ni_4$  precipitates influences the transformation temperatures and subsequently the stress-strain response of the material. Increasing coherent precipitates increases internal stress which leads to a decrease of the isothermal martensite transformation stress. It has also been reported that the yield strength and recoverable strain increase following coherent precipitate formation<sup>14</sup>. Thus, heat treatment increases  $M_s$  and introduces precipitation hardening. As reported by Frick et al. heat treatment temperatures above 450°C lead to incoherent precipitates in hot-rolled Nitinol with the eventual result of a solutionized material following a heat treatment of 600°C or higher. Previous studies performed on the same composition and processing of hot-rolled material used here have shown

that the as-received hot-rolled material contains almost no precipitates. Heat treated material exhibited a low dislocation density, indicating precipitation formation as the dominant factor controlling aging effects in the hot-rolled material<sup>15</sup>.

When working with cold-drawn material, previous studies have shown increasing heat treatment temperature results in a decrease in dislocation density and an increase in precipitate growth on the dislocations. Therefore, heat treatment effect becomes a function of these two interacting processes. The decrease in dislocation density decreases the martensite transformation stress while the precipitate growth blocks dislocation annihilation, decreases martensite transformation stress, and helps block plastic flow. This interaction continues until both processes are overcome by recrystallization of the material at high temperatures. Previous studies report a relative minimum martensite transformation stress in cold-drawn material following a heat treatment of 550°C<sup>5</sup>.

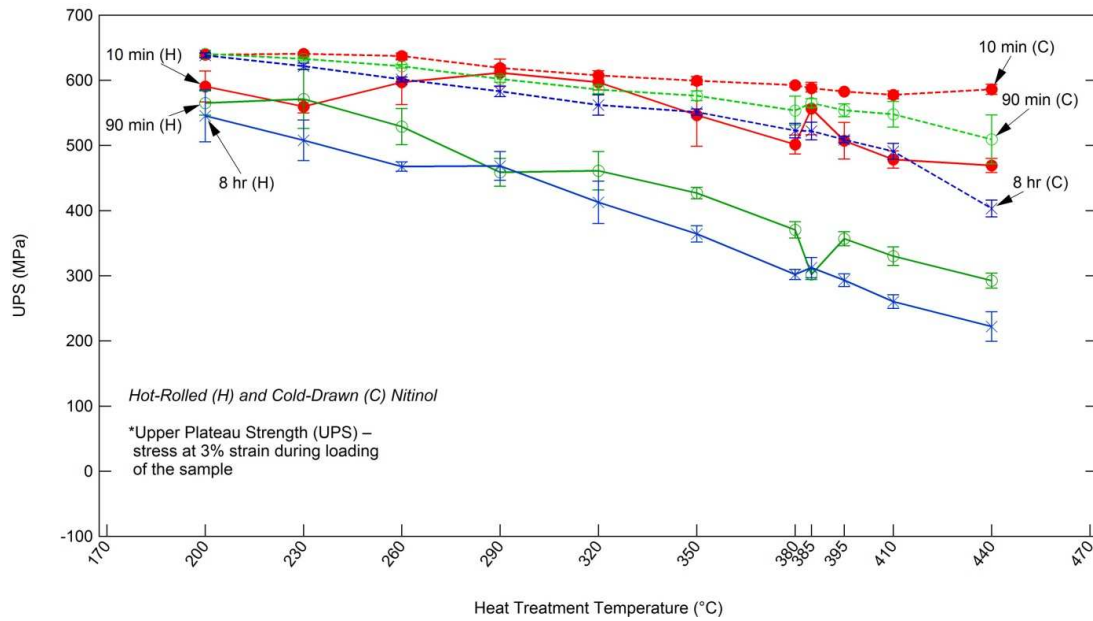
When considering the effect of heat treatment temperature on martensite transformation stress, hot-rolled and cold-drawn results shown here correspond well with the expected outcome. As seen in Figure 6, upper plateau strength (UPS) of the hot-rolled Nitinol is shown to decrease with increasing heat treatment temperature. This is attributed to the increase in internal stress fields as the size of the coherent precipitates increases in the hot-rolled material, leading to an increase in  $M_s$  and a lower martensite transformation stress, correlated here to the UPS result. The maximum heat treatment temperature tested here (440°C) does not exhibit signs of solutionizing the material which would be indicated by a rise in UPS at this temperature. Cold-drawn results (Figure 14) also demonstrate a decrease in UPS with increasing heat treatment temperature for all durations tested. This can be explained by the interaction effect of both

precipitate growth and decrease in dislocation density leading to an increase in  $M_s$  and decrease in the stress needed to induce martensite transformation.

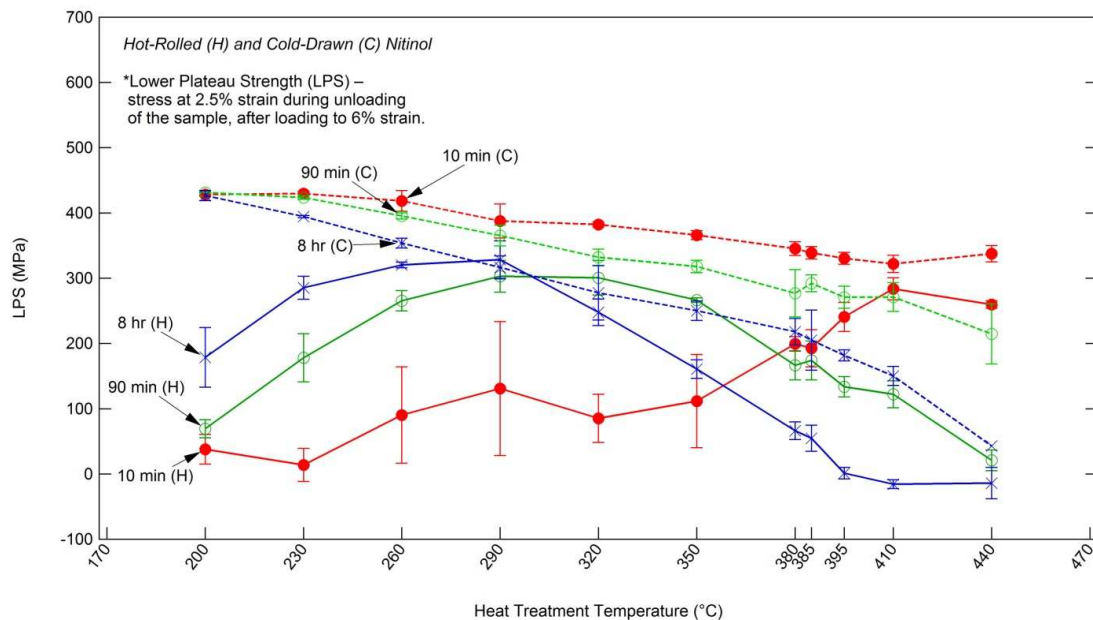
Figure 21 shows the combined results of UPS for the hot-rolled and cold-drawn samples. Cold-drawn UPS values at all heat treatment temperatures are higher for all heat treatment durations when compared to those of the hot-rolled material treated for the same duration. It is known that deformation processing of the cold-drawn material induces a high density of dislocations<sup>16</sup>. Previous studies show that this high dislocation density inhibits interface mobility during the reorientation process, subsequently inhibiting martensite transformation<sup>17</sup>. Therefore, martensite and austenite transformation temperatures are decreased resulting in an increase in the stress needed to induce martensite transformation<sup>5</sup>. Deformation of the material during cold-drawing also increases the stress level required for the onset of plastic strain<sup>11</sup>.

In contrast, the hot-rolling process occurs at high temperatures, between 845°C and 955°C, which is above the solutionizing temperature<sup>16</sup>. Heat treatment of either material decreases the martensite transformation stress by precipitate formation and decreasing the dislocation density. Based on these observations one would expect, as seen here, cold-drawn UPS (corresponding to the martensite transformation stress) would be higher in comparison to the hot-rolled material UPS treated at the same temperature for the same duration. In other words, the cold-drawn material begins as a stronger material requiring a higher stress to induce martensite transformation and plastic deformation. While increasing heat treatment temperature has the same effect as it does on hot-rolled material, the UPS of cold-drawn material remains higher for each temperature and duration tested. Comparison of LPS for the hot-rolled and cold-drawn samples (Figure 22) shows cold-drawn values higher than those for the hot-rolled material treated at the same temperature for the same duration.





**Figure 21:** Upper Plateau Strength (UPS) of hot-rolled (H) and cold-drawn (C) Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

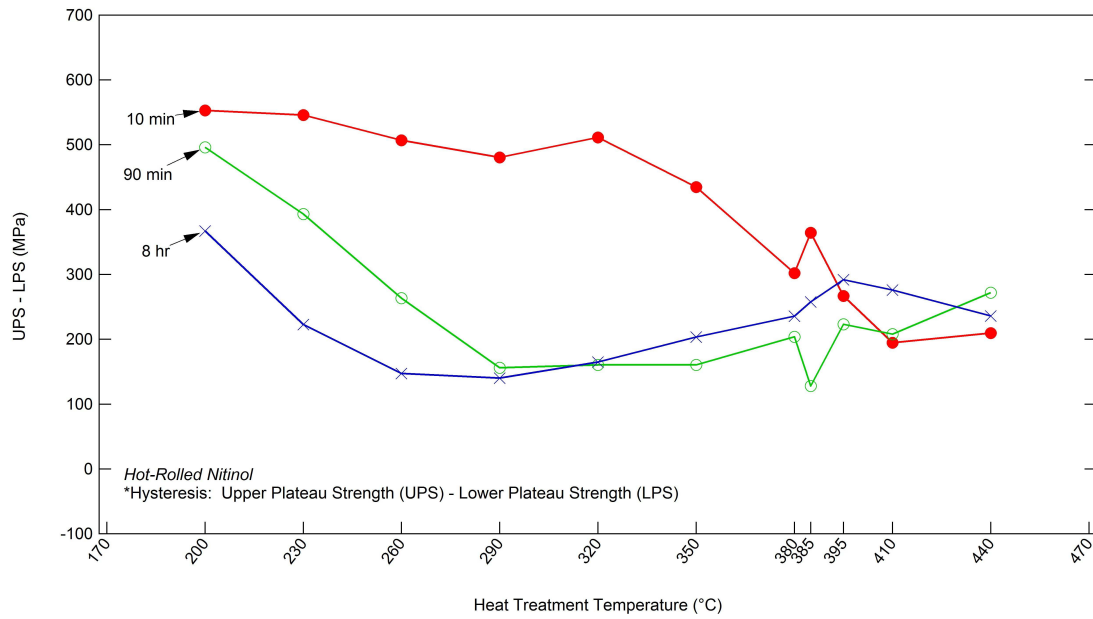


**Figure 22:** Lower Plateau Strength (LPS) of hot-rolled (H) and cold-drawn (C) Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

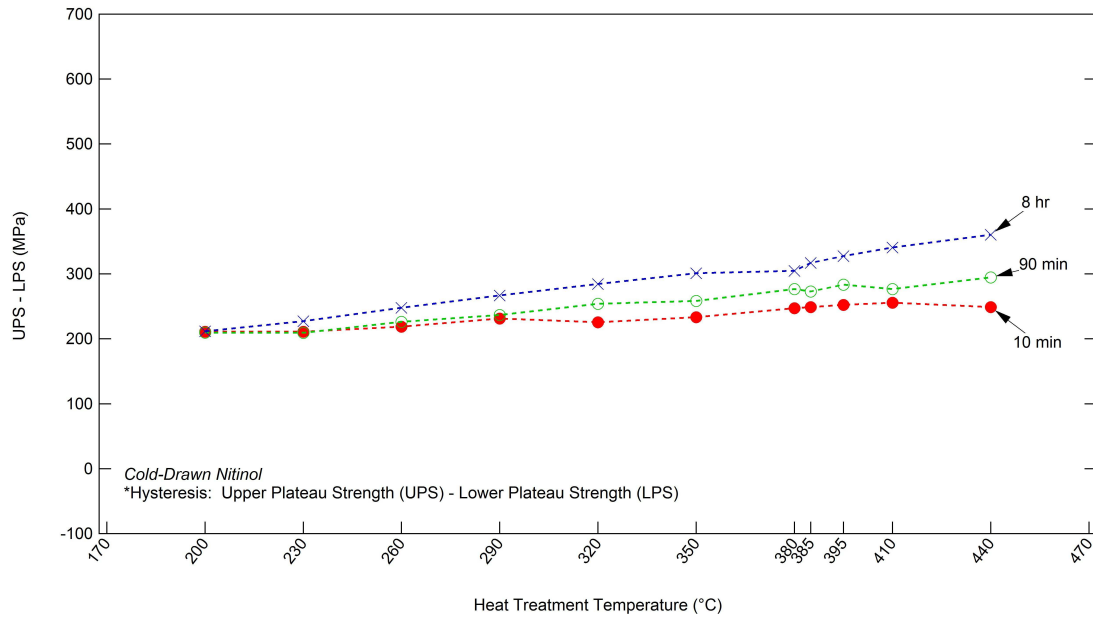
Examining the difference between UPS and LPS allows for a hysteresis trend to be explored. As seen in Figure 23, the hot-rolled 90 minute and 8 hour duration curves show a wide hysteresis at the 200°C heat treatment, (496MPa and 367MPa respectively). This hysteresis trend demonstrates a narrowing with increasing heat treatment temperature up to ~320°C followed by a slow widening in hysteresis as heat treatment temperature continues to increase. This tendency is attributed to the overall hot-rolled trend of UPS decreasing with increasing heat treatment temperature combined with the bell-curve trend of the LPS. This hysteresis pattern predicts plastic deformation of the material at wide hysteresis points corresponding with high stress values, a partially pseudoelastic to more fully pseudoelastic effect for the narrow hysteresis regions, and a shape-memory effect for the wider regions corresponding to low stress values. For the hot-rolled 10 minute duration the trend shows a wide hysteresis for heat treatment temperatures from 200-320°C (~520MPa) followed by a narrowing of the hysteresis with continued increase of heat treatment temperature. This again predicts plasticity of the material at high stress values, followed by a partial to fully pseudoelastic effect with narrowing of the hysteresis at a lower stress values. A second indicator for the prediction of hot-rolled material response can also be found by examining trends in residual elongation (Figure 11). A larger residual strain indicates a plastic or shape-memory response while low values indicate pseudoelasticity.

The cold-drawn material exhibits a hysteresis between UPS and LPS of approximately 200-400MPa. The difference between UPS and LPS (Figure 24) shows a moderately narrow hysteresis that widens slightly with increasing heat treatment temperature for all durations tested. Examining Figure 18 shows a similar low residual elongation for all samples with the exception of the group heat treated for 8 hours at 440°C. This predicts a pseudoelastic response for all

groups with the exception of a plastic or shape-memory response predicted for the outlier. Reexamining Figure 24, we see that group 8hr/440°C group presents a moderate UPS value which would indicate a shape-memory response as opposed to the plastic response high stress values commonly produce.



**Figure 23:** Hysteresis, Upper Plateau Strength (UPS) minus Lower Plateau Strength (LPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

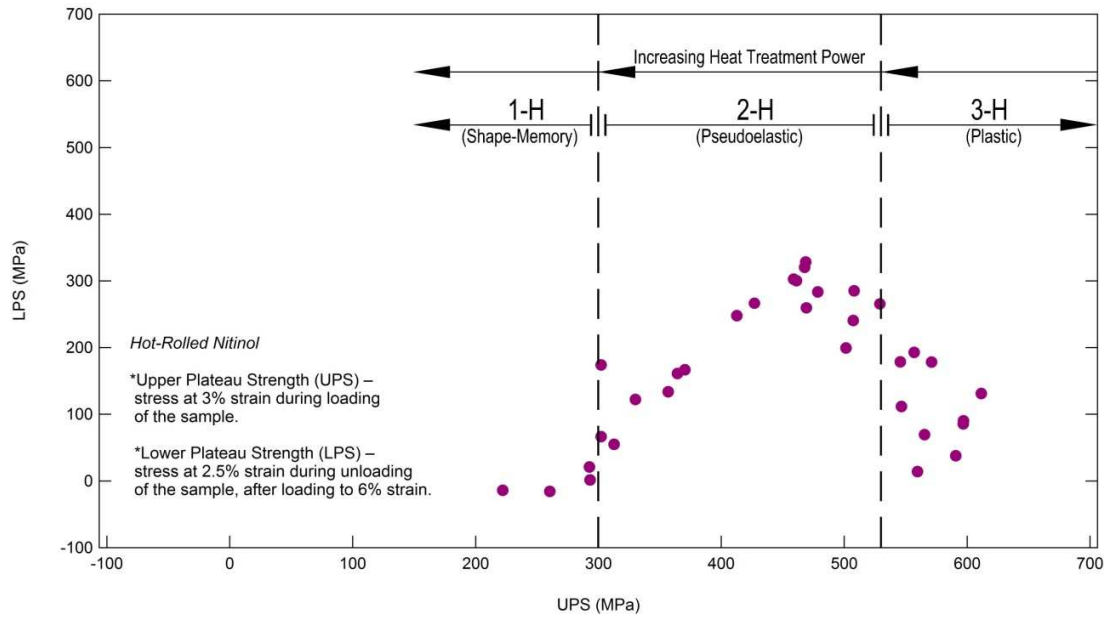


**Figure 24:** Hysteresis, Upper Plateau Strength (UPS) minus Lower Plateau Strength (LPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

As described above, heat treatment effects on hot-rolled and cold-drawn material increase coherent precipitates leading to an increase in internal stress and subsequently a decrease in the stress needed to induce martensite transformation. This occurs until solutionizing of the material at approximately 600°C. Increasing heat treatment temperature also shows an increase in martensite transformation temperatures until solutionizing temperatures are reached, followed by a decrease in the transformation temperatures<sup>5, 18</sup>. Therefore, if testing temperature is held constant, it is expected that the material will exhibit pseudoelasticity when the  $A_f$  temperature is below testing temperature, and exhibit increasing partial pseudoelasticity to full shape-memory as the martensite transition temperatures approach the testing temperature with increasing heat treatment. The critical stress value for slip or plastic deformation must also be considered when predicting material behavior. Cold-drawn material exhibits an initial high critical stress value

due to deformation processing, and increasing heat treatment temperature is expected to increase the critical flow stress in hot-rolled material.

For discussion purposes here, the term ‘heat treatment power’ will be used to describe the efficacy of different heat treatment temperature and duration combinations – high power indicates high temperature and longer time while low power indicates low temperature and short time. Low heat treatment power corresponds to a material response exhibiting plastic deformation, moderate heat treatment power corresponds to a fully pseudoelastic response, and high heat treatment power corresponds to a material exhibiting shape-memory. When considering the hot-rolled material, as heat treatment power increases the effects include an increase in  $M_s$ , a decrease in martensite transformation stress, and an increase in critical flow stress, all attributed to the increase of coherent precipitates. When considering the cold-drawn material, as power increases the expected effects include an increase in  $M_s$  and a decrease in martensite transformation stress, both attributed to precipitation and a decrease in dislocation density. Figure 25 shows the UPS values versus LPS values for the hot-rolled material following all heat treatment variations tested. The hot-rolled data shows a triangular shaped trend. LPS increases with increasing UPS from ~200-400MPa, steady UPS and LPS values are seen within the UPS range of ~400–530 MPa, followed by decreasing LPS values at the higher UPS range of ~530-610MPa. For discussion, three stress regions for the hot-rolled material are defined as follows: region 1-H (UPS < 300MPa), region 2-H (UPS in the range of 300-530MPa) and region 3-H (UPS > 530MPa).



**Figure 25:** Upper Plateau Strength (UPS) vs. Lower Plateau Strength (LPS) of hot-rolled Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

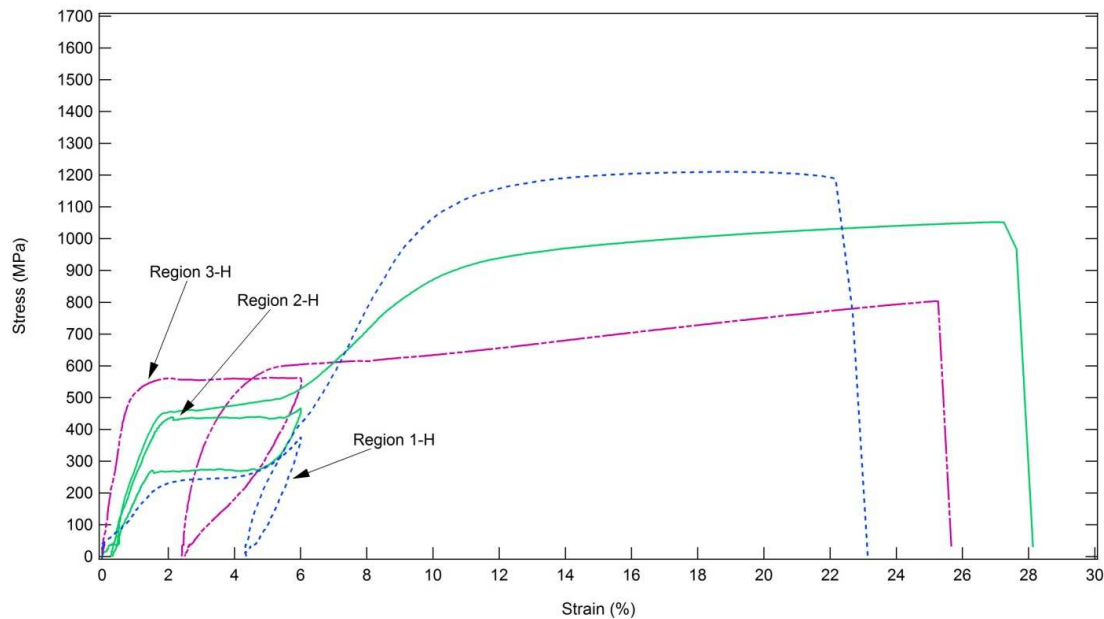
For the hot-rolled material, low power heat treatments reside in region 3-H, with the material exhibiting plastic deformation to partially-pseudoelastic responses. Low power heat treatments of the hot-rolled material do not appear to create significant precipitation hardening or a significant decrease in the martensite transformation stress. Thus, a higher stress is needed to induce transformation resulting in the combined processes of martensite reorientation and martensite deformation.

A shift from plastic deformation to more of a pseudoelastic response is seen by an increase of heat treatment power and its expected effects. Increasing to a moderate heat treatment power, hot-rolled material resides in region 2-H, as partial to fully-pseudoelastic responses are created. Based on these observations, moderate power heat treatments appear to result in a sufficient hardening of the material to block plastic flow, combined with a decrease in transformation stress and an increase of  $M_s$  as evident by a decrease in UPS. While  $M_s$  increases,

it remains far enough below the testing temperature to prevent a shape-memory effect. Thus, plastic deformation is prevented and the material recovers with a pseudoelastic response.

A progression from pseudoelastic to shape-memory response is seen by a further increase in heat treatment power. Following a high power heat treatment, the hot-rolled material resides in region 1-H, exhibiting a partially-pseudoelastic to full shape-memory effect. High power heat treatments appear to cause the same effects (decrease in transformation stress and sufficient hardening) as moderate power treatments with the difference of the transformation temperature's proximity to the testing temperature. As transformation temperatures are increased and approach the testing temperature the deformed martensite becomes more stable at this temperature. This stability causes the martensite to remain deformed following the release of the external stress, exhibiting the shape-memory effect.

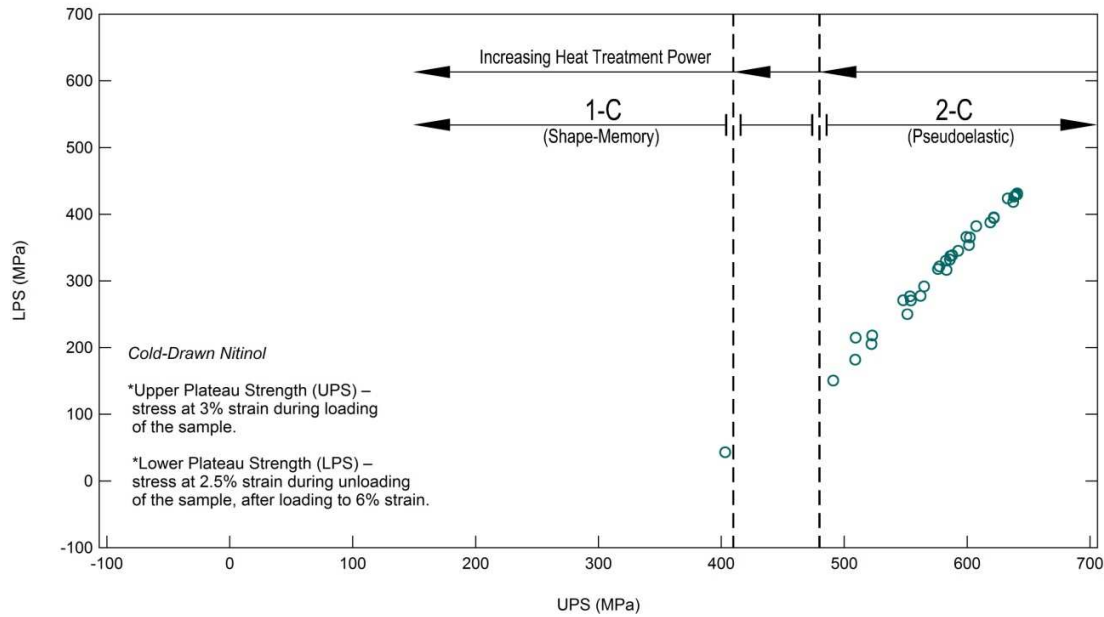
Representative hot-rolled curves from each stress region (1H-3H) are shown in Figure 26. The shape of the stress-strain curves provides evidence for the effects of different heat treatment powers. Pseudoelasticity is evident by the extent of the return of the lower plateau corresponding to the reverse transformation from martensite back to the parent austenite phase. With increasing heat treatment power we see a gradual transition towards a shorter stage II transformation plateau and a higher stage III slope. Similarly, it is known that precipitates suppress martensite deformation and martensite reorientation is favored, resulting in a shortened stage II plateau and a higher stage III slope. Therefore, this provides evidence for the increase in precipitation hardening with increasing heat treatment power. In summary, for the hot-rolled material the transition from low to moderate to high heat treatment powers corresponds to a gradual shift to shorter stage II plateaus, higher stage III slopes and an evolution from plasticity to pseudoelastic to shape-memory responses as seen in regions 3, 2, and 1 respectively.



**Figure 26:** Representative curves from regions 1-H, 2-H, and 3-H demonstrating the effects of high, moderate, and low power heat treatments respectively.

Figure 27 shows the UPS versus LPS for the cold-drawn material following all heat treatment variations tested. The cold-drawn data shows a linear progression of increasing LPS with increasing UPS. As predicted, one point indicates a different material response as seen by a much lower UPS/LPS result. For discussion, two stress regions are defined for the cold-drawn material as follows: region 1-C (UPS < 410 MPa) and region 2-C (UPS > 480MPa).



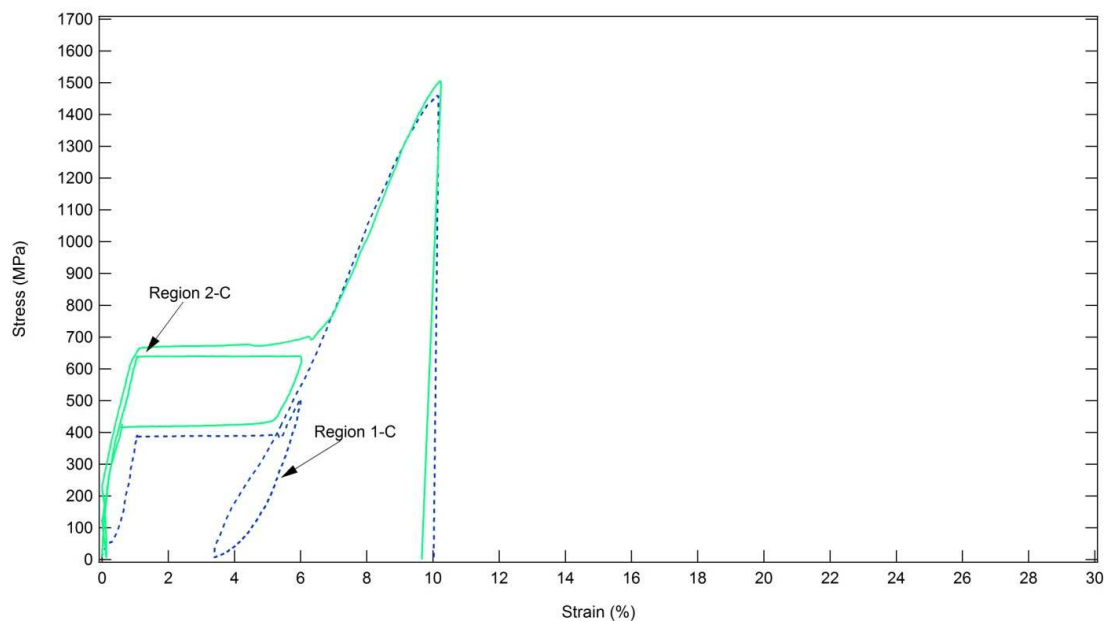


**Figure 27:** Upper Plateau Strength (UPS) vs. Lower Plateau Strength (LPS) of cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

While high stress values seen by the cold-drawn UPS/LPS results may predict plastic deformation, low residual elongation values and inspection of the stress-strain curves indicate good pseudoelastic responses for all samples within region 2-C. Due to the high initial critical flow stress values of the cold-drawn material and the precipitation formation blocking plastic flow, all heat treatment variations tested exhibit the expected results of the moderate or high power designation as defined earlier. All test groups found within region 2-C exhibit a pseudoelastic response corresponding to a cold-drawn moderate heat treatment power. The linear trend within this region confirms that as heat treatment power is increased, precipitate formation and dislocation annihilation lead to a decrease in martensite transformation stress. Also, for all groups within region 2-C,  $M_s$  remains far enough away from the testing temperature to produce a pseudoelastic response upon unloading.

As heat treatment power is increased, a cold-drawn high power treatment (region 1-C) results in a continued decrease of the transformation stress and approximation of transformation

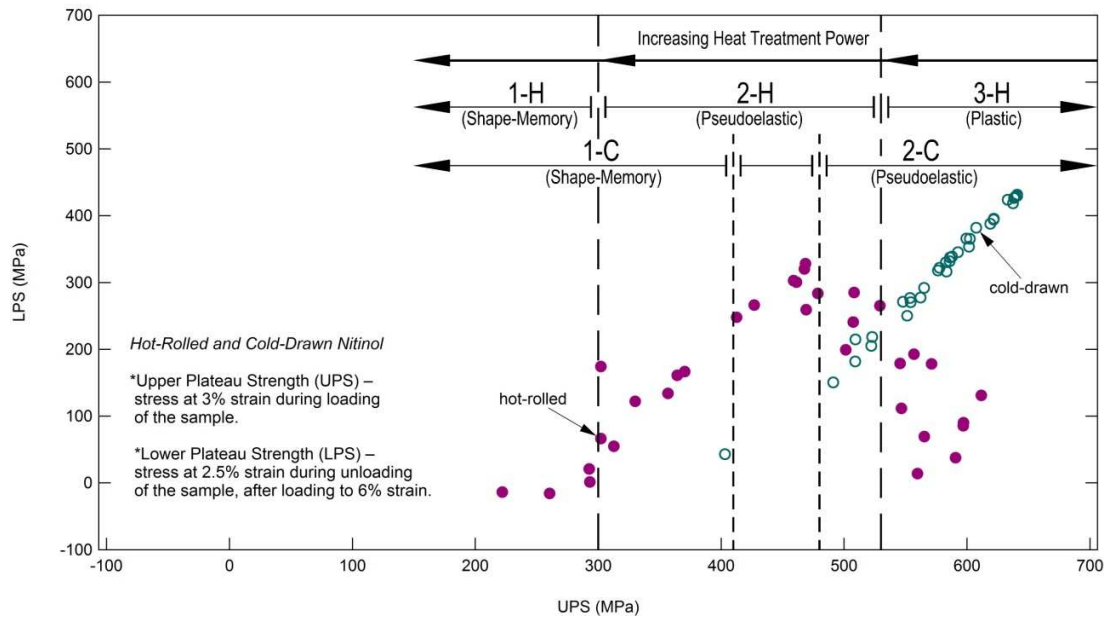
temperatures to the testing temperature. This predicts a shape-memory response which is confirmed by inspecting the stress-strain curves for this sample group (33W). It is important to note that no heat treatments tested resulted in data within the stress range between region 1-C and 2-C. Therefore it is difficult to predict precisely where a pseudoelastic response would end and a shape-memory response would begin. It is reasonable to assume that with other heat treatment variations a transition from pseudoelastic to shape-memory can be produced within the two regions defined here. In summary, for the cold-drawn material, moderate to high heat treatment powers exhibit pseudoelastic and shape-memory responses as seen in regions 2-C and 1-C respectively. Representative cold-drawn curves from each stress region (1-C and 2-C) are shown in Figure 28.



**Figure 28:** Representative curves from regions 1-C and 2-C demonstrating the effects of high and moderate power heat treatments respectively.

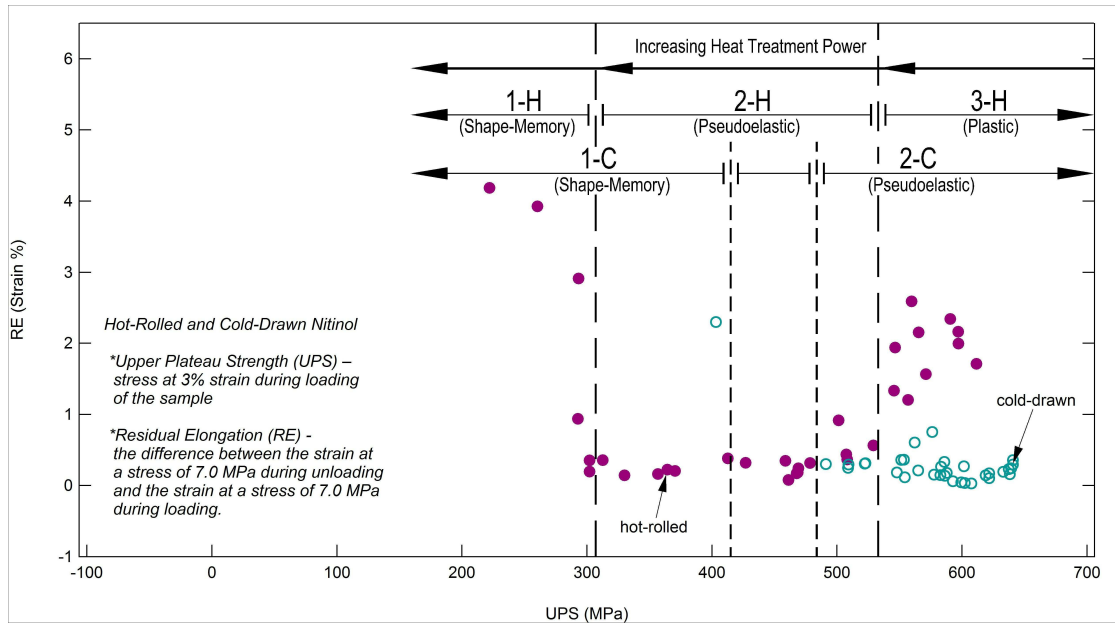
Figure 29 shows the UPS vs. LPS values for the hot-rolled and cold-drawn material plotted together for reference. It is important to note that heat treatment powers (low, moderate,

and high) do not span the same stress ranges for hot-rolled and cold-drawn material. Therefore, it is imperative to consider this difference when utilizing these two processes.



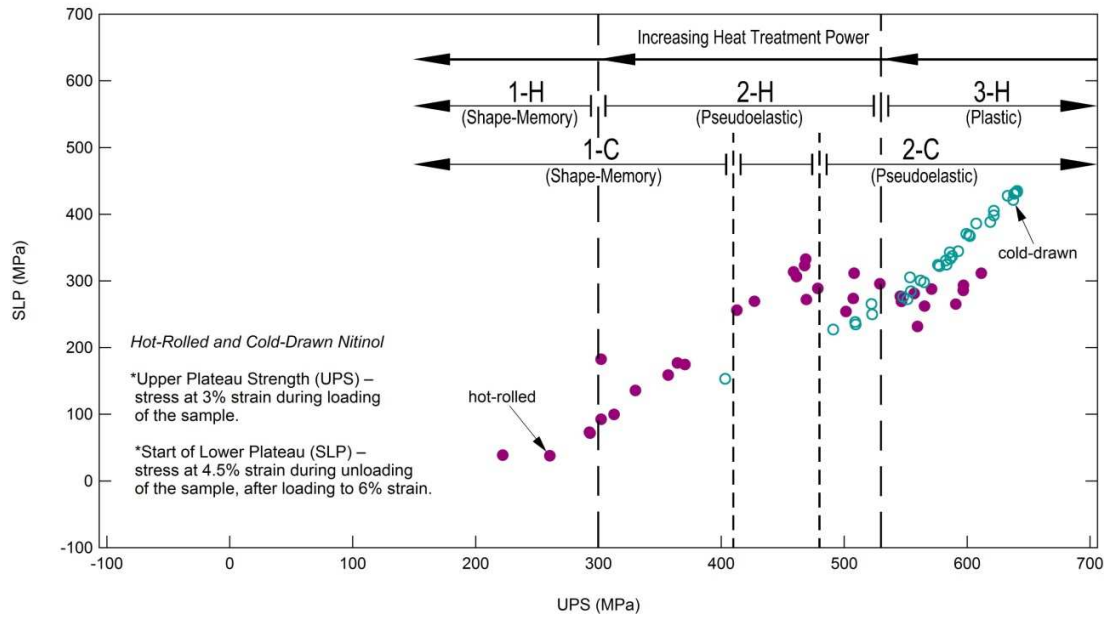
**Figure 29:** Upper Plateau Strength (UPS) vs. Lower Plateau Strength (LPS) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

UPS vs. RE for all samples tested from the hot-rolled and cold-drawn material are shown in Figure 30. As evident by the change in residual elongation, regions 3-H, 2-H, and 1-H display the transition from plasticity to pseudoelasticity to shape-memory for the hot-rolled material and regions 2-C and 1-C display the transition from pseudoelasticity to shape memory for the cold-drawn material. As described previously, this change in material response is attributed to the variation in heat treatment power.



**Figure 30:** Upper Plateau Strength (UPS) vs. Residual Elongation (RE) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

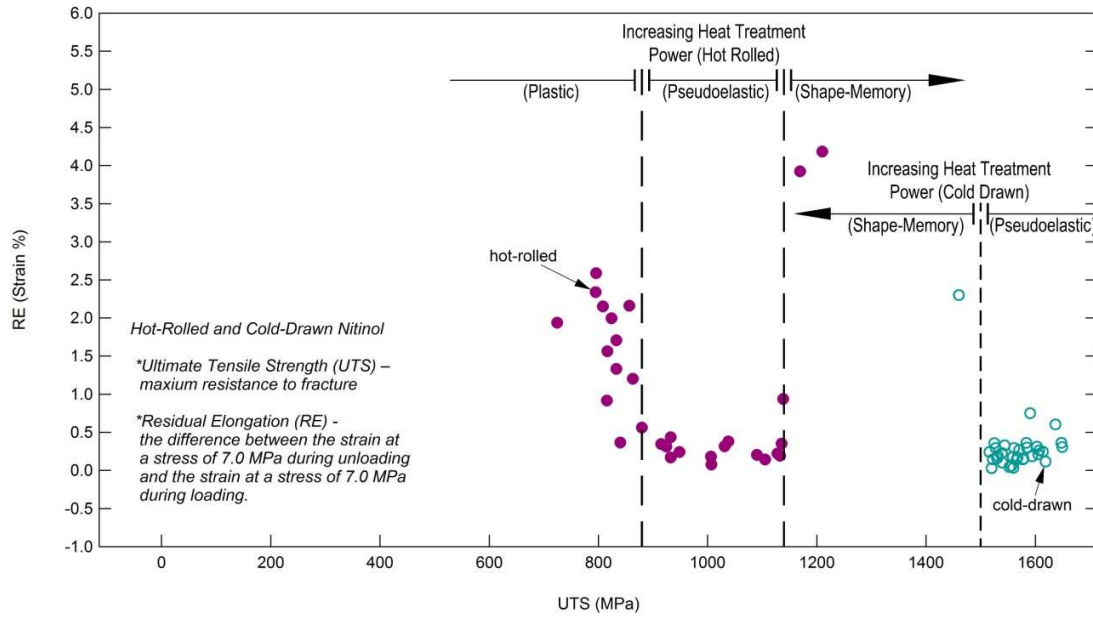
By examining UPS vs. SLP for all hot-rolled samples (Figure 31) there is a horizontal linear trend within regions 1-H and 3-H and an increasing sloped linear trend within region 2-H. This is indicative of the widening hysteresis (as UPS increases SLP remains fairly constant) due to a shape-memory and plastic response in contrast to a similar hysteresis (SLP increases with increasing UPS) for samples within the pseudoelastic region, 2-H. Similarly, Figure 31 also shows a linear increase of SLP with UPS within region 2-C, as expected by the good pseudoelastic response.



**Figure 31:** Upper Plateau Strength (UPS) vs. Start of Lower Plateau strength (SLP) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

For the hot-rolled material, UTS increases with increasing heat temperature for all heat treatment durations (Figure 10). This correlates to an increase in heat treatment effect, or power, as precipitation hardening raises the critical stress required for plastic deformation. As described previously, increasing heat treatment power results in a transition of the hot-rolled material response from plastic, to pseudoelastic, to shape-memory. Figure 32 shows the UTS vs. RE for the hot-rolled and cold-drawn material. For the hot-rolled material, as UTS increases, corresponding to an increase in heat treatment power (from left to right), residual elongation follows a high to low to high trend, indicative of the material response shift from plastic, to pseudoelastic, to shape-memory. In contrast, increasing heat treatment power in the cold-drawn material does not have a great effect on UTS (Figure 17). Deformation processing of the cold-drawn material increases the critical stress for plastic deformation resulting in high UTS values for all samples tested. The effect of increasing heat treatment power is only seen as

transformation temperatures approach testing temperature resulting in a shape-memory response. Thus for the cold-drawn material, Figure 32 represents an increase of heat treatment power from right to left, characterized by a shift from low residual elongation values to a high residual elongation for the sample group exhibiting shape-memory.



**Figure 32:** Ultimate Tensile Strength (UTS) vs. Residual Elongation (RE) of hot-rolled and cold-drawn Nitinol following heat treatment at 200-440°C for 10 min, 90 min, and 8 hr durations.

## **CHAPTER 5**

### **CONCLUSION**

*“All heat treatments are not created equal”*

The successful use of Nitinol for biomedical and other industry applications depends on an accurate control of its unique transformational and mechanical properties. Heat treatment or aging is often used as an economical method for manipulating these properties for specific engineering purposes. Heat treatment effect is dependent on time, temperature, processing history, and the amount of prior cold work. This study compares the effects of various heat treatment temperatures and durations on the mechanical behavior of both cold-drawn and hot-rolled Nitinol. Different heat treatment combinations can be used to identify optimal aging treatments that produce a desired plastic, shape-memory or pseudoelastic effect. By altering heat treatment temperature and duration it is also possible to create heat treatment combinations that produce similar effects on the material properties.

For discussion purposes here, different combinations of heat treatment temperature and duration lead to a change in heat treatment “power”. When using hot-rolled Nitinol, low heat treatment powers do not create significant precipitation hardening, or a significant decrease in martensite transformation stress. This results in a high upper plateau strength, high residual strain values, and evidence of plastic deformation upon unloading.

Increasing heat treatment power to moderate power levels results in a decrease in upper plateau strength and a decrease in residual elongation, as seen by the transition from a partial to fully pseudoelastic response. Moderate power heat treatments result in sufficient hardening of the material by increasing the critical stress required for plastic deformation, a decrease in

martensite transformation stress and an increase in martensite transformation temperature. Although transformation temperatures are increased, moderate power heat treatments result in  $M_s$  remaining far enough below the testing temperature to allow the material to recover with a pseudoelastic response.

Increasing heat treatment power beyond the moderate level results in a progression from a pseudoelastic to a shape-memory response characterized by a further decrease in upper plateau strength and an increase residual elongation. Reaching a high heat treatment power continues to increase the transformation temperature, decrease the transformation stress, and provides sufficient precipitation hardening. The deformed martensite becomes stable as  $M_s$  approaches the testing temperature, resulting in the shape-memory response.

When considering Nitinol in the cold-drawn form, the same heat treatment variations tested here on the hot-rolled material fall into only two designations, moderate and high power. Critical stress values for plastic deformation are initially high in the cold-drawn material as a result of the deformation process. Even the lowest power heat treatments tested here result in the material exhibiting a pseudoelastic response, and thus eliminating the “low power” category for the cold-drawn material. Within the moderate heat treatment power level, the cold-drawn material exhibits a decrease in upper plateau strength and a fairly constant small residual elongation resulting in a good pseudoelastic response. This is attributed to precipitate formation and dislocation annihilation leading to a decrease in martensite transformation stress and an increase in  $M_s$  that remains far enough away from the testing temperature to produce a pseudoelastic response upon unloading.

Further increasing heat treatment power results in the cold-drawn high power treatment. This power level is characterized by the continued decrease in martensite transformation stress,



and the approach of  $M_s$  to the testing temperature. Consistent with the hot-rolled high power treatment, this results in a decrease in upper plateau strength, an increase in residual elongation, and the shape-memory material response.

By examining the data presented here it is possible to select an appropriate heat treatment combination to produce the desired transformational and mechanical properties for both hot-rolled and cold-drawn Nitinol.

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