Final Report DOE Project, "Origins of Asymmetric Stress-Strain Response in Phase Transformations, DEFG02-93ER14393

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Two graduate students who received full or partial funding from the DOE grant received their PhDs during 1998. Dr. Ken Gall who received his PhD in May 1998 was fully supported by the DOE project. He accepted a faculty position at University of Colorado, Boulder starting in June 1999. Dr. Mark Balzer who developed the high pressure rig with Professor Sehitoglu was partially supported by the project and started as a Research Scientist at Jet Propulsion Laboratory, Pasadena, California.

The following PhD theses received support from the DOE grant.

(i) The Effect of Stress State and Precipitation on Stress-Induced Martensitic Transformations in Polycrystalline and Single Crystalline Shape Memory Alloys: Experiments and Micro-Mechanical Modeling", PhD Thesis, Ken Gall

(ii) Mechanical Behavior of Metals under Triaxial Stress: Apparatus and Experiments, PhD Thesis, Mark Balzer

Papers published based on the research conducted with the DOE grant during 1996-1999 is given below.

1.Jacobus, K., H. Sehitoglu, and M. Balzer, "Effect of Stress State on the Stress-Induced Martensitic Transformation in Polycrystalline Ni-TI Alloy," *Metallurgical Transactions*, 27A, 3066-3073, 1996

2.Balzer, M. and H. Sehitoglu, *Experimental Mechanics*, 37:1, 87-95, 1997.

3.Gall, K., H. Sehitoglu, and H. J. Maier, 1998a"Stress Induced Martensitic Phase Transformation in Polycrystalline CuZnAl Shape Memory Alloys in Different Stress States," *Meta. Trans.*, 29A, 765-773, 1998

4.Gall, K. and H. Sehitoglu, 1999a "The Role of Texture in Tension/Compression Asymmetry in Polycrystalline NiTi, *International Journal of Plasticity*, 15,69-92,

5. Gall, K., H. Sehitoglu, Y. I. Chumlyakov, Y. L. Zuev, and I. Karaman, 1998b "The Role of Coherent Precipitates in Martensitic Transformations in Single Crystal and Polycrystalline Ti-%0.8% Ni," *Scripta Materialia*, 39:6, 699-705, 1998

6. Gall, K., H. Sehitoglu, Y. I. Chumlyakov, I. V. Kireeva, and H. J. Maier, 1999b "The Influence on Critical Transformation Stress Levels and Martensite Start Temperatures in NiTi: Part I Discussion of Experimental Results,"ASME, *Journal of Engineering Materials and Technology*, , 121, 19-27

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7.Gall, K., H. Sehitoglu, Y. I. Chumlyakov, I. V. Kireeva, and H. J. Maier, 1999c "The Influence of Aging on Critical Transformation Stress Levels and Martensite Start Temperatures in NiTi: Part II Aged Microstructure and Modeling, ASME, *Journal of Engineering Materials and Technology*, 121, 28-37

8. Gall, K., H. Sehitoglu, Y. Chumlyakov, and I. Kireeva, 1998c, Pseudoelastic Cyclic Stress-Strain Response of Overaged Single Crystal Ti-50.8%Ni, *Scripta Materialia*, 40,1,7-12

9. Gall, K., H. Sehitoglu, Y. Chumlyakov, and I. Kireeva, 1999d, "Tension-Compression Asymmetry of the Stress-Strain Response of Aged Single Crystal and Polycrystalline NiTi," *Acta Materialia.*, 47,4,1203-1217

10. Gall, K., Huseyin Sehitoglu, and Yuriy I. Chumlyakov, 1999e, The Effect of Precipitates on Martensitic Transformations in Single Crystal NiTi Shape Memory Alloys, Constitutive and Damage Modeling of Inelastic Deformation and Phase Transformation, Proceedings of Plasticity, 99, Ed. A. Khan, 207-210

11. Gall, K. H. Sehitoglu, and H. Maier, Asymmetric Stress-Strain Response in Shape Memory Alloys, Physics and Mechanics of Finite Plastic and Viscoplastic Deformation, Proceedings of Plasticity 1997, A. Khan editor, 153-154

12. Sehitoglu, H. and Chumlyakov, Y. , 1999, Guest Editors, Special Issue on Shape Memory Alloys, ASME J. Engineering Materials and Technology

13. Gall, K. , Sehitoglu, H., R. Anderson, I. Karaman, Y. Chumlyakov, I. V. Kireeva, On the Mechanical Behavior of Single Crystal NiTi Shape Memory Alloys and Related Polycrystalline Phenomenon, presented at the TMS meeting honoring Ali Argon, accepted to Materials Science and Engineering, 1999

14. Gall, K., T. J. Lim, D. McDowell, H. Sehitoglu, and Y. I. Chumlyakov, "The Role of Intergranular Constraint on the Stress Induced Martensitic Transformation in Textured Polycrystalline NiTi," *International Journal of Plasticity*, 16 (2000) 1189-1214

15. Sehitoglu, H., I. Karaman, R. Anderson, X. Zhang, K. Gall, H. J. Maier, and Y. Chumlyakov, "Compressive Response of NiTi Single Crystals," *Acta Materialia*, 48, 2000.

16. Sehitoglu, H., I. Karaman, X. Zhang, H. Kim, Y. Chumlyakov, H.J.Maier, I. Kireeva, " Deformation of NiTiCu Shape Memory Single Crystals in Compression, Metallurgical and Materials Transactions, 2001, 32A,3, 477-489

Significant progress was made in the last three years of research. Many of the original findings are described in Appendix 1.

For the first time, experiments on NiTi under pressure loadings were conducted in Ref. (1). This work showed that the stress-strain response of NiTi is highly pressure sensitive and there was an asymmetry of tension and compression results. The results were obtained based on the special rig developed in (Ref. 2) by Sehitoglu and his students. Several experiments under pressure were also conducted on CuZnAl alloys with also pressure dependent response.

The stress-strain results for NiTi were predicted with a micro-mechanical model, which accounted for variant-variant interaction and texture effects in the case of NiTi alloys (Ref. 3). It was found that the polycrystalline version of these materials has a strong texture due to the cold rolling process (Figure 4). Consequently, they almost behave as single crystals oriented in the [111] direction (Figure 3). We showed that if the texture effects are not accounted for the models give the incorrect trends when compared with experiments (Figure 5). Our work also showed that the evolution of the variants in tension is much more rapid compared to the compression case (Ref. 3).

In the second year of the work, our attention focussed exclusively on the deformation behavior of single crystals. Several key results were achieved with single crystals. Initially, we studied the role of aging treatment on tension compression asymmetry and crystal orientation dependence. It was shown that the orientation dependence of critical resolved shear stress is significant in the case of peak aged crystals while the orientation dependence decreases with overaging. A micro-mechanical model was developed to explain these trends based on the determination of the local shear stresses due to the precipitate on the 24 possible martensite variants (Figure 6). It was found that those variants that have high resolved shear stress due to external loading experience low local stresses due to the precipitate weakening the orientation dependence (Refs. 4-6). Overall the results and the model showed that the introduction of precipitates reduce the critical transformation stress in these materials and reduce the orientation dependence (Figure 7). It was also noted that overaging results in loss of Ni in the matrix resulting in decrease in strength levels.

In the third year of the research, our attention focussed on cyclic loading and recoverable strains as a function of orientation and stress direction (Refs. 8-9). We made preliminary attempts at explaining the orientation dependence of the recoverable strains based on the CVP formation and detwinning models (Table 2) but pointed out that further work is necessary in this area. The precipitates curtail the detwinning phenomenon and reduce the overall transformation strain levels in these alloys (Table 2). Finally, we note the unusual amount of hardening under cyclic loading (Ref.9) with combined effects of twinning and slip. The strengthening in compression was found to be remarkably high (Figure 10). Further study of cyclic loading was put aside for the proposed research .

Appendix 1

Shape memory alloys have unique properties such as recoverability of deformation upon heating and pseudoelastic stress-strain behavior for large strains. These are promising alloys finding applications ranging from bioengineering and medicine to mechanical and civil engineering.

The materials science background has been documented for these alloys over the years in a variety of materials journals dating back from the work of Chang and Read but a number of key issues remain relevant to orientation dependence and detwinning effects.

This proposal is aimed at disclosing some of the most pertinent needs in this area and providing a systematic approach to address these needs with experiments on single and polycrystals. These experiments will reveal understanding of microstructure to facilitate description at both the micro- and continuum levels in the quest for bridging length scales.

1.1. Tension-Compression Asymmetry in NiTi, and Texture effects

NiTi (Nitinol) is the most promising of the shape memory alloys. It possesses moderate strength as a structural alloy, pseudo-elastic or shape memory behavior depending on composition, aging treatment, processing, and test temperature. It exhibits high recoverable strains (nearly 8% under tension in polycrystals). Previous work has identified that the stress-strain behavior of NiTi exhibits tension-compression asymmetry and pressure dependence

To gain insight into tension-compression asymmetry we consider Figure 1. Both crystals are oriented in the [100] direction. The crystal on the right is subjected to compressive stress, while the one on the left is subjected to tension. The uniaxial stress is denoted as Σ_{11}. In Figure 1, n and m denote the normal to habit plane (the undistorted plane) and transformation direction respectively.

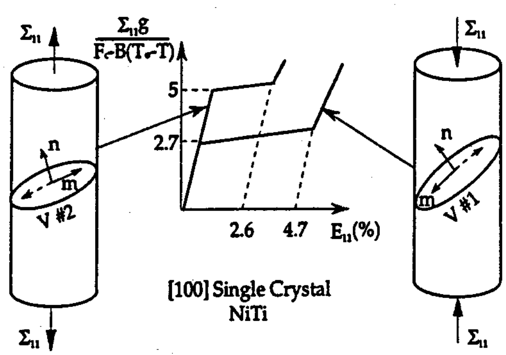

Figure 1 Schematic demonstrating the origin of tension-compression asymmetry

Unlike slip, the transformation is unidirectional, and CVP#2 (or V#2) will be activated in tension but not in compression. When the load is reversed a different CVP (CVP#1) becomes active. The CVP refers to a correspondent variant pair of martensite, which is martensite in the form of two twin related crystals, that transforms from the austenite crystal.

The CVP #1 (correspondent variant pair #1) in the case of compression is more favorably oriented with respect to the loading axis , thus this [100] crystal requires a lower critical stress Σ_{11} for yielding. In Figure 1, the middle schematic shows the yielding stresses for the tension and compression. The yield strength in tension is nearly a factor of two higher than in compression case. The normalized stress in vertical axis includes the transformation shear strain, g, the critical value of the driving force for the transformation F_c and the chemical free energy term $B(T_0 - T)$ where T_0 is equilibrium temperature.

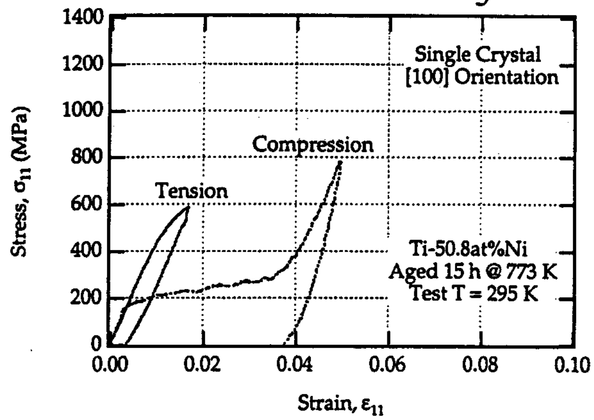

Figure 2 Experimental demonstration of the tension compression asymmetry for the case [001] NiTi

Experimental results shown in Figure 2 confirm the predictions in Figure 1. These results were obtained from single crystals in [001] direction. Note that the tensile stress level exceeds the compressive stress levels by a factor of two. Since the ductility in this orientation is low, the straining in tension was limited to less than 0.02. The papers by provide further details on these experiments and models. If we examine [111] crystals the critical compressive stress exceeds the critical tensile stress. Similar experiments have not been reported in the literature because of the difficulty of growing single crystals.

The treatment above considered the behavior of single crystals. To transition to polycrystalline material behavior treatments considered both CuZnAl and NiTi alloys. These treatments considered the transformation from an austenite to an internally twinned martensite structure. For textured polycrystalline NiTi deformed under tension it was demonstrated that the martensite evolution is very abrupt, consistent with the Luders type deformation experimentally observed. This behavior occurs because majority of the grains are oriented along the [111] crystallographic direction, which is soft under tensile loading. The drawn polycrystalline NiTi has a strong texture of the <111>{110} type, thus it deformed in a manner consistent with the [111] single crystals. This is demonstrated in Figure 3.

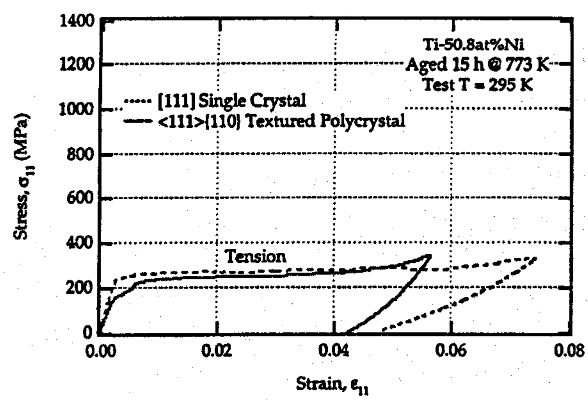

Figure 3 Comparison of a [111]single crystal and the textured polycrystal,

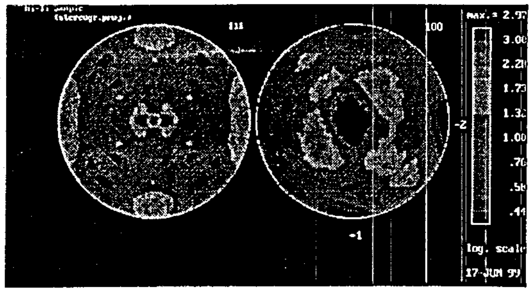

Figure 4 Demonstration that NiTi polycrystals have strong texture

To predict the experimental stress-strain behavior of polycrystalline NiTi, crystallographic texture of the material was measured. Figure 4 shows the pole figures resulting from a polycrystalline sample. From the Figure 4, a strong crystallographic texture of the <111>{110} type was established. By using the crystallographic theory of CVP formation and the self consistent scheme for micro- to macro- transition the stress-strain curves were obtained. The predicted stress-strain curves for both the textured (solid lines) and untexured (dashed lines) polycrystalline NiTi are given in Figure 5 using the texture results in Figure 4. The material modeled in Figure 5 has a lower Ni concentration than the material in Figure 3, thus the critical transformation stress levels are higher at an equivalent test temperature. When the texture effects are included, the results reproduce the experimental trends (not shown here) extremely accurately. In the untextured case, the predicted stresses under tension are slightly higher than compression. This trend is clearly not observed in experiments.

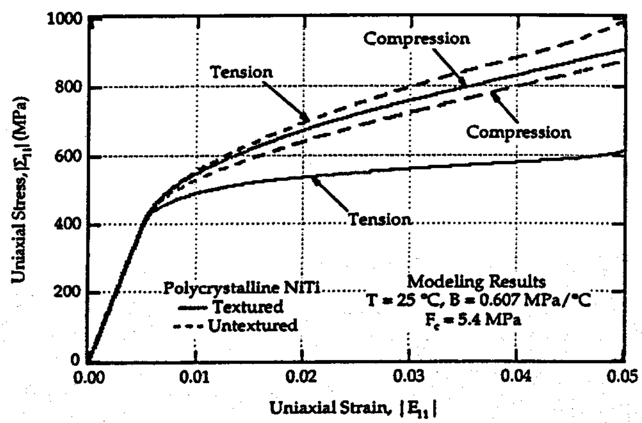

Figure 5 Modeling Results of Polycrystalline NiTi showing the role of texture as responsible for tension-compression asymmetry

We note that this is the first time the texture information has been incorporated into the determination of stress-strain response for NiTi. These models correctly account for the self accommodating nature of the martensite variants, ie. the interaction among the CVPs. However, microstructural observations reveal that, within a CVP, martensite detwins (one twin grows and widens at the expense of the other) and an additional transformation strain is produced. Detwinning induced strains are stress direction and orientation dependent. Admittedly, none of the previous developments account for the detwinning effect. Also, there is no discussion of the detwinning induced strains in any of the textbooks on the topic. Therefore, this topic is a major emphasis in the proposed work (see Sections 1.3 and 4).

1.2 Modeling and Experiments on Orientation Dependence and The Role of Precipitates on Deformation Behavior

It is well known that heat treatments that produce coherent or semi-coherent precipitates lead to large changes in the macroscopic mechanical properties of NiTi alloys. Consequently, precipitation has been used to tailor pseudo-elasticity effects the critical stress for transformation the martensite start temperatures, and fatigue properties. A TEM photograph of the precipitate morphology is shown in Figure 6(a). The strong contrast surrounding the precipitates indicates that internal strains are present.

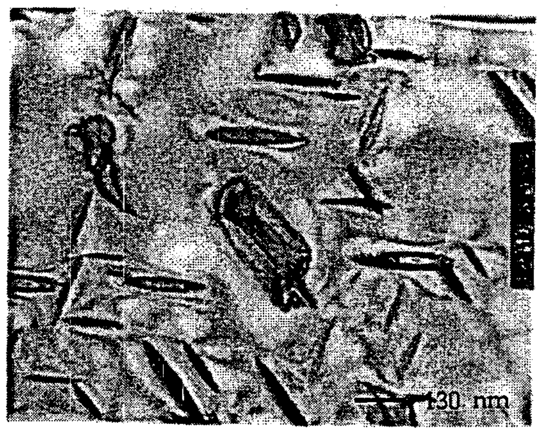

Figure 6 (a) A TEM photograph showing the precipitate structure in single crystal NiTi

The precipitates have a considerable influence on the tension-compression asymmetry in NiTi. Using unique experimental results and micro-mechanical modeling it was possible to rationalize the change in orientation dependence when the precipitates are coherent, semi-coherent or incoherent . Tensile and compressive stress-strain behaviors were established on aged single crystals ([100], [110], and [111] orientations). The peak aged NiTi single crystals showed an orientation dependence (max. 55 %) and tension-compression asymmetry (max. 54 %) of the critical resolved shear stress,CRSS. Using a micro-mechanical model, this deviation from Schmid's law was attributed to the strong local stress fields surrounding the coherent Ti_3Ni_4 precipitates. The unique orientation relationship that exists between the precipitate variants and the martensite correspondent variant pairs (CVP's) allows the stress fields to alter the orientation dependence of (Critical Resolved Shear Stress, CRSS). In the overaged samples, the coherency stresses are lowered, and CRSS was relatively independent of the crystal orientation and stress state, thus Schmid's law was obeyed.

To understand the role of local stress fields on martensite nucleation, we consider Figure 6(b). In addition to the resolved shear stress due to external loading , τ^{n}_{rss-e}, there now exists a resolved shear stress due to the local stress fields around the precipitates, a so-called internal resolved shear stress, τ^n_{rss-i}. This average internal resolved shear stress, $\bar{\tau}^n_{rss-i}$, is necessary to correctly estimate the effect of the precipitates on the macroscopic critical stress, σ_{cr}, and temperature, M_s, required to trigger the transformation. Given the eigenstrains it is possible to calculate the local stress and strain fields outside of the disk shaped precipitates. With the additional local coherency fields, the transformation criteria is given as,

$$\tau^n_{rss-e} + \bar{\tau}^n_{rss-i} \geq \tau_{crss}(T, c_{Ni}) \qquad (1)$$

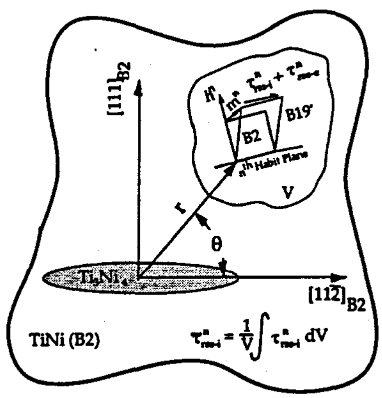

Figure 6(b) Schematic on the Proposed Effect of local mismatch strains(stresses) on the overall resolved shear stresses

The above equation states that the transformation will proceed when the sum of the internal and external resolved shear stresses is greater than a critical value. To estimate the effect of local stresses on the martensitic transformation, it is appropriate to calculate the local resolved shear stresses acting on the 24 martensite CVP's near the precipitate, τ^n_{rss-i}.

The results of the simulations are summarized in Table 1. The resolved shear stress factor (Schmid factor), α^n_{11}, is given for CVP #2, 4, 6, 9, 11, 13,and 20. When this factor is multiplied by the critical uniaxial stress, σ_{cr}, CRSS is obtained. In the [111] orientation CVP#2, 9 and 11 have the largest resolved stress due to external loading but CVP# 4 and 6 experience the largest internal resolved shear stresses. The CVP that is activated has the highest value of internal and external summation. Consequently, the transformation will be biased differently, depending upon the orientation of the loading axis. Also, when the Ni rich NiTi is over-aged, it is found that the precipitates coarsen to approximately 1000 nm, they become incoherent, and the local stress fields disappear.

	RSS Factor, α_{11}^{n}			Intern (RSS) (MPa) $\bar{\tau}_{rss-}^{n}$	Predicted CRSS τ_{crss} (MPa)		
CVP #	[111]	[100]	[110]		111	100	110
2	0.39	0.18	-0.20	60	174	-	-
4	-0.27	0.18	-0.20	148	-	223	-
6	-0.1	0.18	-0.01	148	-	223	-
9	0.39	0.21	-0.37	60	174	-	-
11	0.39	0.21	-0.02	60	174	-	-
13	-0.1	0.21	0.18	130	-	218	-
20	-0.27	0.18	0.41	90	-	-	219

Table 1 Prediction of Internal Stress Fields on Martensite CVPs showing that the CVP with high RSS factor experiences small internal stress

A summary of CRSS results as a function of precipitate size is given in Figure 7. Several observations are noteworthy. Upon aging the CRSS decreases significantly (compare solutionized CRSS and 50nm precipitate size CRSS results) and this decrease is consistent with the increase in local stress fields due to the presence of precipitates(Table 1, Column 5). In the peak aged condition (50 nm precipitate size), the strength level has decreased by as much as a factor of three and orientation dependence is highest (compare [111] and [001] CRSS levels for peak aged case). This decrease in CRSS with aging and orientation dependence is accurately predicted with the model. The results clearly show that the stress fields outside the coherent or semi-coherent precipitates effectively decrease the orientation dependence in aged alloys. With further aging, in the overaged case, the strength increases compared to the peak aged case but it is still well below the solutionized material. The reason why the strength level for the overaged case is lower than expected is due to the loss of Ni in solution. After over-aging the average composition of the matrix drops from 50.8 at% Ni to approximately 50.4 at% Ni.

It is noted that the above experiments focussed on the effect of aging on transformation stress levels at the onset of the transformation. As the martensite grows the lattice mismatch strains and local stress fields will change. Near the completion of the transformation the local stress fields will be governed by the lattice mismatch between the precipitates and the B19' martensite. The newly formed stress fields are responsible for the inhibition of detwinning, and they also will control the reverse transformation upon unloading. This is a topic that will be covered in the proposed work (see Section 4). In summary, previous work has established that coherent or semi-coherent precipitates reduce the critical transformation stress levels by a combined effect of increasing the internal stresses and reduction of %Ni. There has been no systematic study on the role of precipitates on detwinning strains and overall recoverable strains. It is expected that the detwinning effect will have a considerable influence at high strain levels (>0.02) on the unloading-reloading response. These will be discussed in Sections 2.3 and 4 of this proposal.

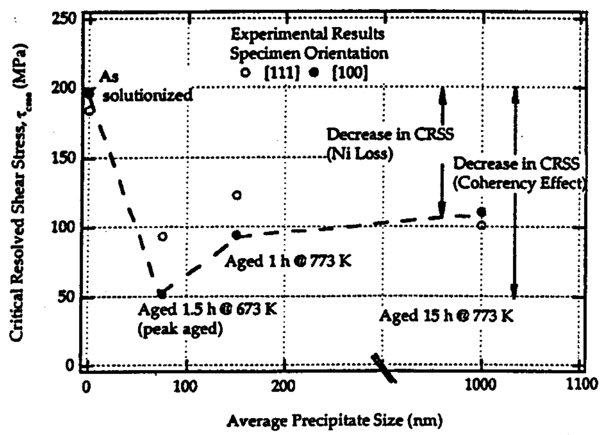

Figure 7 Critical Resolved Shear Stress under Tension as a function of the precipitate size, $T > M_s$ where M_s is martensite start temperature

1.3. Recoverable Strain and Detwinning

The ability of the shape memory alloy to recover the transformation strain upon heating is potentially useful in many applications. The orientation dependence of recoverable strain levels, ε_0, as a function of heat treatment needs to be fully understood for effective utilization of the shape memory alloys. Two models have been forwarded to predict the maximum recoverable strain, the lattice deformation models proposed by Saburi et al.and the CVP growth and CVP detwinning proposed by Buchheit and Wert. Both models handle the end state (single crystal martensite) and the initial state (single crystal of austenite. There is very limited experimental data to check these models. The evolution of the detwinning with stress in both single and polycrystalline case in addition to the recoverable strain in single and polycrystalline materials are all of considerable interest and will be considered in this research.

The phenomenon of detwinning for a single crystal can be best described with the aid of Figure 8(a). Initially, the material is in the austenitic B2 phase. Upon application of external stress, the transformation initiates when a critical stress is reached. The martensite that is formed is internally twinned. Upon further loading, the martensite detwins (one twin variant grows in expense of the other) finally resulting in a single crystal of martensite.

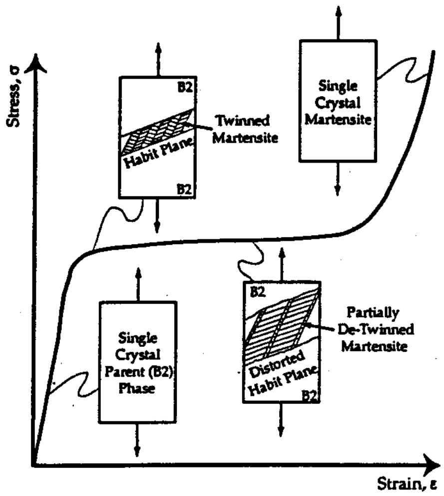

Figure 8(a) The process of CVP formation and detwinning, $T > M_s$

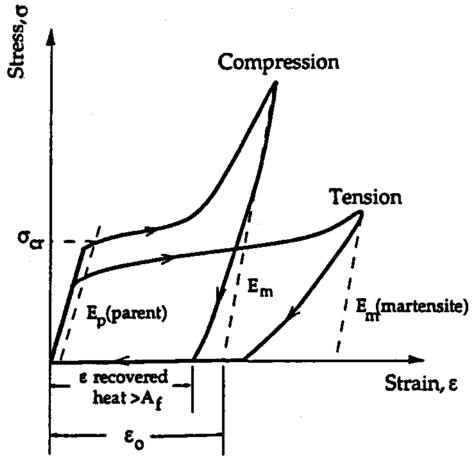

Figure 8(b) Schematic demonstrating the determination of the maximum recoverable strain.

Now we illustrate the determination of the recoverable strain. A schematic of the stress-strain curves in tension and compression is shown in Figure 8(b). In compression upon transformation to martensite the material remains elastic even at high stresses. Then, the recoverable strain can be measured upon heating at zero stress. In tensile loadings plasticity of austenite occurs making the recoverable strain determination more difficult. Note that upon unloading considerable non-linearity has been observed in tension case. During the unloading, spontaneous reverse detwinning of the martensite is believed to occur.

To precisely estimate the ε_0 magnitude, the initial linear unloading path has been extrapolated to zero stress (Figure 8(b)). The point where this extrapolated line intersects the total strain axis is defined as ε_0. This technique accounts for the non-linearity (spontaneous reverse transformation or reverse detwinning) observed in the unloading paths. Since the non-linearity was considerably larger in tension versus compression, the ε_0 magnitudes were measured using unloading modulus under compression for all tests.

In Table 2, the recoverable strains are summarized as a function of crystal orientation and aging treatment, as determined by experimental results on single crystals. All of the tensile ε_0

magnitudes are preceded by a ≥ sign. This is because the measured recoverable strain levels under tension may actually be slightly larger since the transformation may not have been 100 % complete upon unloading. Miyazaki *et al.*, have demonstrated that under large tensile stresses, plastic strains make it more difficult to precisely determine $\_{o}$. However, under compression, the measured recoverable strain levels are proposed to be close to the exact ones. This is because the compression curves begin to show an upward slope which indicates that the transformation is nearly complete. For all of the tensile and compressive stress-strain tests presented here, the strains were perfectly recovered upon heating above the austenite finish temperature, A_f (Figure 8(b) and Table 2).

In Table 2, the predictions are only given for the solutionized material (no precipitates). In each grid two predictions are given, the first represents the maximum recoverable strain for a single twinned CVP, ε_o^{cv}. The numbers in parenthesis represent the total maximum recoverable strains obtained by superposition of CVP formation strain, ε_o^{cv} and detwinning strain, ε_o^{dt}. The difference between the two numbers is the detwinning strain. The detwinning strain is small in [100] orientation but it is rather large in [111] and [110] orientations. We also note that the amount of detwinning strain in compression is a very small fraction of the overall transformation strain.

	Experimentally Measured				Predicted (Solutionized Case)	
	ε_o (1.5 h 673 K)		ε_o (15 h 773 K)		ε_o^{cv} (ε_o^{cv} + ε_o^{dt})	
	Ten.	Comp.	Ten.	Comp.	Ten.	Comp.
[100]	≥ 1.2 %	3.5 %	≥0.5 %	4.3 %	2.7 (2.9)%	5.2 (5.3)%
[110]	≥5.2 %	3.2 %	≥6.5 %	3.6 %	5.3 (8.8)%	4.8 (5.0)%
[111]	≥6.6 %	2.3 %	≥7.3 %	3.0 %	5.1 (8.6)%	3.5 (3.8)%
poly	≥5.3 %	2.6 %	≥5.5 %	2.7 %	–	–

Table 2 Summary of recoverable strains obtained under tension and compression

Three observations are noteworthy: (1) The recoverable strains $(\varepsilon_o^{cv} + \varepsilon_o^{dt})$ in compression are approximately 50% of tension results for [110] and [111] while they are higher for the [100] case, (2) The predictions for recoverable strains are provided for the solutionized case (if a theory existed for the precipitate case the numbers would be lower), and (3) there is considerable orientation dependence of the recoverable strain results.

Peak aging more effectively inhibits the detwinning of martensite CVP's compared to over aging. Consequently, the ε_o levels in peak aged NiTi are on the order of the strains predicted by exclusively considering the formation of a martensite CVP. Since detwinning is more favored in tension versus compression, peak aging has a more dramatic effect on tensile ε_o levels. In the over aged samples, the ε_o levels were qualitatively consistent with the predictions of the CVP formation and detwinning model. However, for all orientations, the strain levels fell short of the predicted values. This is partially attributed to the fact that the precipitates are unable to undergo the transformation and therefore cannot contribute to the experimentally measured transformation strain. Predictions in the presence of precipitates have not been reported and will be considered in the current work (Section 4).

In the peak-aged state, the measured ϵ_0 values under tension are on the order of the strains predicted after removing detwinning from the model (Table 2).

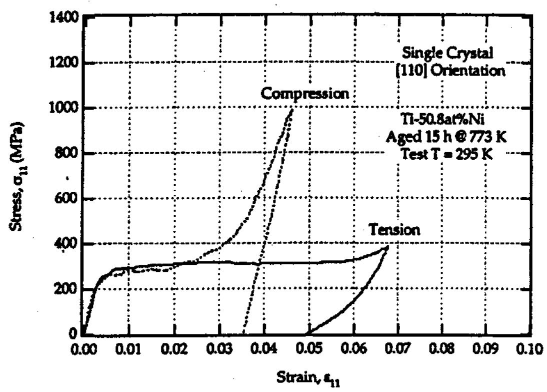

Figure 9 Deformation under tension and compression demonstrating similar CRSS but different energy dissipation

As the transformation proceeds, the internal stresses built up in the peak-aged NiTi are larger than the built up internal stresses in the over aged NiTi. These new internal stresses are a direct consequence of the mismatch strains between the martensite plates, the precipitates, and the parent phase. It is insightful to take a closer look at the tensile deformation of the [111] crystal, since it is most favorably oriented for the detwinning process (Table 2). Even in the peak-aged state, the [111] orientation demonstrates recoverable strain levels slightly larger than predicted without considering detwinning (Table 2).

To illustrate the results further, we consider the tension and compression experiments in [110] orientation in Figure 9. We note that in this orientation and heat treatment the critical transformation stresses are very similar yet the overall transformation strains are different (>6.5% in tension and 3.6% in compression). These results (Figure 9) demonstrate unequivocally that the proposed models in literature utilizing the product "critical stress x transformation strain = constant" have shortcomings. The area under the stress-strain curves is constant for different orientations only if the contribution from detwinning strains is removed. The proposed work will study this issue in detail with compression-tension experiments in two orientations that produce maximum recoverable strains respectively.

The only comprehensive study which provides evidence of twinned and partially detwinned martensite variants is that published by Van Humbeeck and colleagues (Liu et al, 1999). They show convincing proof that at strains of the order of 6% many CVPs are partially detwinned. Working with polycrystals, they showed that depending on the loading direction (rolling versus transverse) with respect to the Type II twinning (shear) direction different degrees of de-twinning develops during deformation. The stress levels in the case of loading in the transverse direction (where detwinning is not favored) result in a nearly factor of 4 higher stresses compared to the rolling direction stress levels where detwinning occur readily. Clearly, since the effects of detwinning are large a comprehensive understanding tuned with single crystal experiments is needed.

1.4. Cyclic Deformation of NiTi Materials

In metallic alloys it has been known that fatigue deformation produces microstructures at small strains (after many cycles) that are equivalent to unidirectional large deformations. During the low cycle fatigue of polycrystalline NiTi, the critical transformation stress level decreases, the transformation stress-strain slope increases, the dissipated hysteresis energy decreases, and residual transformation strains accumulate. Using some of the aforementioned experimental results as a guide, several phenomenological constitutive models for the fatigue of shape memory alloys have been developed. With a thermodynamic framework, these mathematical models have successfully reproduced the cyclic stress-strain response of polycrystalline NiTi. However, a link between modeling predictions and experimental results has only been attempted at large length scale phenomenological levels.

Moreover, many fatigue studies have not considered careful control of the precipitate size in NiTi shape memory alloys. The precipitates complicate the growth characteristics of martensite and strongly affect the monotonic mechanical properties of these alloys in both the single crystal and polycrystalline form. To help address the above issues, preliminary experimental results on the fatigue of precipitated (aged) Ni rich NiTi single crystals are presented here.

The strong advantage of single crystals is that the characteristics of the deformation mechanisms (martensite plate variants and slip systems) are a known function of the external loading axis of the crystal. Thus, the single crystals allow a much more direct comparison between the cyclic stress-strain response and the evolving microstructural features. This type of understanding will provide basis for future fatigue models that are more representative of the actual deformation mechanisms.

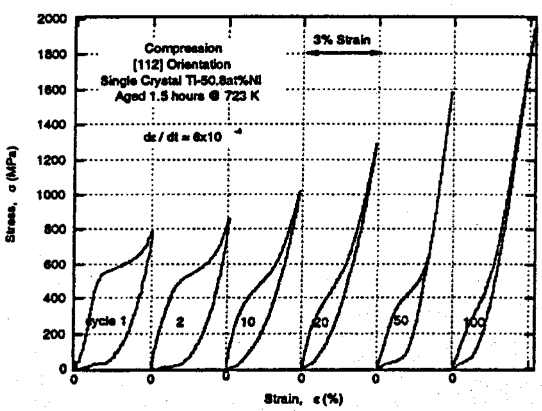

Figure 10 Cyclic Compression behavior of [112] NiTi illustrating the dramatic changes in hysteresis

Cyclic deformation under compression loadings is given in Figure 10. The strain range is 3% and Cycles 1 - 100 is shown in this figure. Two observations are noteworthy. Considerable strengthening occurs in compression with cycles, and the hysteresis loop narrows with cycling. Whether extraordinary hardening shown in Figure 10 holds for solutionized material (without precipitates) is not known. Any model that attempts to predict the behavior of polycrystals, has to note the compatibility problems at grain boundaries because the transformation strains are highly orientation dependent. Similar incompatibilities are expected in the martensitic phase upon transformation. Another consideration is the inclusion of slip processes and slip/twinning interactions that could produce the " S " shape stress-strain curves observed in Figure 10.