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Title: Stress-Induced Martenistic Phase Transformations in NiTi Shape
Memory Alloys During Dynamic Loading

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STRESS-INDUCED MARTENSITIC PHASE TRANSFORMATIONS IN NiTi SHAPE MEMORY ALLOYS DURING DYNAMIC LOADING

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ABSTRACT

This research explores the near-adiabatic, high strain rate stress-induced martensitic phase transformation in NiTi shape memory alloys using both a compressive and tensile Split Hopkinson Pressure Bar (SHPB). The results of the dynamic loading tests are presented with emphasis on the loading rate, stress-strain response, specimen temperature and post-test microstructural evaluation. In addition to the large strain rates, tensile specimens of various geometries are tested to large strain levels such that void growth and failure mechanisms are identified. The dynamically loaded specimens failed in a mixed mode, ductile void growth followed by transgranular failure. The void growth in incipient failure specimens showed ductile void growth throughout the specimen cross-section.

INTRODUCTION

Over the last decade shape memory alloys (SMAs) have seen growing use in the mechanical, medical, and aerospace industries [1]. Most of the applications have been 1-D in nature where wires, strips, and rods were employed, i.e. as actuators in active wings [2], and robotic systems [3]. NiTi SMAs have also been investigated for their high damping capacity and material response under high loading rates [4-6].

Thermomechanical properties of SMAs undergo significant changes with differences in the chemical composition, cold work, heat treatment and thermomechanical cycling. It has been shown that the austenite to martensite phase transformation temperatures of NiTi SMAs can be

shifted by the presence of lattice defects introduced by cold working [7]. There are also many experimental results on the thermomechanical response of stress-induced martensite at temperatures above the austenite finish temperature, A^f , (the pseudoelastic response) showing the effects of strain level, stress level, cycle number, pre-straining and strain-rate [8-10].

The quasi-static mechanical response of the stress-induced martensitic transformation in fully annealed NiTi Shape Memory Alloys (SMAs) has been experimentally characterized for multiple variations of loading paths and material conditions. However, the stress-induced martensitic phase transformation in NiTi SMAs has not been experimentally explored under high strain rates. Even though the quasi-static transformation induced plastic strain development has been studied extensively, the effect of high strain rate, large plastic strain and the accompanying void growth leading to ultimate failure has not been studied. In this research, dynamic loading tests are performed on NiTi specimens using both a compressive and tensile Split Hopkinson Pressure Bar (SHPB) which allows for investigation of strain rates from $10^2 \leq \dot{\epsilon} \leq 10^4$. The tensile SHPB is also equipped with momentum trapping which enables testing of incipient specimen failure.

EXPERIMENTAL PROCEDURE

The NiTi SMA utilized in this research was purchased from Nitinol Devices and Components in the form of 2-in. diameter bar in the as-drawn condition, with the final draw imparting 20% cold work. Table 1 shows the elemental

material composition as stated by the manufacturer, and shows that a relatively high concentration of oxygen is present. For the compressive SHPB tests, cylindrical specimens with a 5.0 mm diameter and 5.0 mm long were electro-discharge machined from the bar. Seven specimen blanks approximately 2-in. long and .625-in. diameter were electro-discharge machined from one diameter of the bar for tensile SHPB specimens. The specimen blanks were machined into three different final specimen geometries. The specimens consisted of 4 uniaxial specimens and 6 notched specimens. The notched specimens consisted of four specimens with a 0.167-in. radius notch, "E" notch, and 2 specimens with a 0.100-in. radius notch, "A" notch, as described by Hancock and Mackenzie [11]. The uniaxial, "E" and "A" notch geometries result in stress states of

$$\left[\frac{-P}{2\tau} \right]_{\text{Bridgman}} = .167, \left[\frac{-P}{2\tau} \right]_{\text{Bridgman}} = .3, \text{ and } \left[\frac{-P}{2\tau} \right]_{\text{Bridgman}} = .37$$

respectively, where P is the level of hydrostatic tension and τ is the flow stress.

Differential Scanning Calorimetry (DSC) results of the as-drawn material confirmed that the high level of dislocations imparted by the drawing resulted in no thermally induced martensitic phase transformation. Therefore, before the specimens could be tested, the effect of the cold work in the specimen from both the drawing and the machining must be removed through an annealing process. The final machined specimens were wrapped in titanium foil and annealed in a flowing argon environment for 1 hour at 800°C and quenched in water. DSC results from the annealed material showed a large latent heat of transformation and the resulting transformation temperatures are given in Table 2, where M^s represents the martensitic start temperature, etc.

The initial microstructure of the specimen showed a large percentage of precipitants, as seen in Figure 1. Initial analysis of unetched specimens identified the precipitants as Ti_2Ni . Image analysis of the precipitants showed they comprised approximately 2% of the cross-sectional area, with a mean area of $20 \mu m^2$ and a mean aspect ratio of 2.1. As seen in the figure, the precipitants also appear as long stringers that align with the drawing direction of the bar, and exist both at grain boundaries and within the grain itself. The grain size of the specimens also was measured at approximately $112.5 \mu m$ perpendicular to the

drawing axis, determined using the 1.125 times the mean intercept length method.

Figure 1 Microphotograph of annealed NiTi showing precipitant microstructures.

The room temperature loading path on the NiTi SMA specimens, as identified by the DSC results in Table 2, is schematically shown in Figure 2 using the stress-temperature phase diagram for fully annealed NiTi SMAs described by Bo and Lagoudas [12]. Figure 2 shows an isothermal loading in which stress-induced martensite is formed upon loading and returns to the austenitic phase upon unloading. However, during the dynamic loading induced by the SHPB the specimen will not remain isothermal due to the release of latent heat of transformation from the stress induced phase transformation and the near adiabatic conditions associated with a dynamic loading. Therefore, a large temperature increase is expected which will increase the stress level required for the development of stress-induced martensite, shown as ΔT in Figure 2. To quantify the temperature rise in the specimen, K-type fast response thermocouples are held in physical contact with the specimen and a silicon paste is applied to ensure good thermal contact between the specimen and the thermocouple. This method of temperature measurement will not capture the complete temperature rise in the specimen due to the complex nature of the boundary conditions, however qualitative results can be measured.

Table 1 Elemental Composition of NiTi Alloy

Element	Ti	Ni	O	H	Co	Fe	Cu	C
Wt. %	55.5	Balance	0.034	0.0005	<0.01	<0.01	<0.005	0.0036

Table 2 Transformation Temperatures of NiTi annealed at 800°C/1 hr. quenched

Transformation	M^f	M^s	A^s	A^f
Temperature, °C	-40	-13	-12	9

Figure 2 Stress-Temperature phase diagram for fully annealed NiTi.

To study the response of the NiTi SMA under dynamic compressive loading, specimens were loaded in the compressive SHPB with a 10-inch striker bar while varying the striker velocity. Striker velocity was varied between 12 m/s to 19 m/s in the attempt to maintain similar strain rates for each loading cycle; however, the severe hardening that occurred in the specimen led to difficulties in maintaining a constant strain rate. The SHPB results for two cyclically loaded specimens are presented, with each specimen cycled at a different mean strain rate. The results of specimens loaded at a mean rate 500 ϵ/s , a mean rate of 1500 ϵ/s and quasi-statically are all compared.

To study the growth of voids that lead to ultimate specimen failure, tests were performed in the tensile SHPB that unload just before failure of the specimen occurs, i.e. incipient failure tests. The test procedure therefore first involves testing a specimen to failure, from which calculations involving the total incident pulse time can be used to identify the velocity and pulse length for further incipient failure tests. Additionally, specimen geometries including a uniaxial bar and Hancock & Mackenzie "E" and "A" notch geometry were tested to identify the effects of hydrostatic stresses on the void, or damage, nucleation and growth within the specimen.

RESULTS AND DISCUSSION

Cyclic Compressive Tests

The incident, reflected and transmitted pulses from a SHPB test apparatus on an annealed NiTi specimen with a 10-in. striker bar with velocity of 12.5 m/s are shown in Figure 3. The incident pulse shows a typical SHPB input pulse with a short rise time and constant amplitude. The reflected tensile pulse also shows a short rise time but quickly decays to nearly zero before a second compressive pulse is seen. This second

compressive pulse is half the amplitude of the incident pulse and is a result of the large recovery during the reverse phase transformation. The true strain rate vs. true strain curves for compressive SHPB tests on annealed NiTi specimens with striker bar velocities of 12.5 m/s and 18.5 m/s are shown in Figure 4. These results show that a constant strain rate was not achieved throughout the test. The inability to maintain a constant strain rate is due to the large amount of energy absorbed by the specimen during the phase transformation.

The true stress-true strain results for annealed NiTi specimens loaded at three different strain rates are shown in Figure 5. The SHPB tests with mean strain rates of 1500 ϵ/s and 500 ϵ/s are shown along with quasi-static (10^{-3} ϵ/s) results. The obvious difference in the results lies in the stress-induced martensitic plateau seen in the quasi-static tests, but not seen in the high strain rate tests. Delineated on the quasi-static curve is a close approximation of the detwinned martensitic strain accumulated during the phase transformation, approximately 4% strain. The arrows, approximately 4% strain in length, between the high strain rate and quasi-static curves show the significance of this value. These results indicate that high strain rate tests do not develop a detwinned martensitic microstructure, but rather a self-accommodated martensitic microstructure with very little detwinned martensite. The apparent lack of detwinned strain is also seen in the unloading portion of the curve by estimating the unloading of the quasi-static test at the maximum stress attained in the high strain rate test. This is delineated in Figure 5 as the dotted line labeled as "estimated unloading" and matches the approximate 4% strain difference seen in the loading. Assurance that a martensitic transformation actually occurred during the loading is shown in the 500 ϵ/s test, where a substantial portion of the strain is recovered upon unloading through the reverse phase transformation. Large plastic strains, as developed in the 1500 ϵ/s test, have been shown to inhibit the reverse transformation in quasi-static tensile tests [13]. However, dynamic tensile tests with large-scale plastic deformation, ~45% plastic strain, did not inhibit the reverse transformations [14].

Additionally, a comparison of the 1-wave and 3-wave analysis methods was performed to assess the stress equilibrium achieved in the samples. In every test performed the comparison showed that the 3-wave analysis oscillated around the 1-wave analysis for the duration of the test, indicating that stress equilibrium was maintained in the specimen during the entire test.

Figure 3 Strain gage output signals for NiTi specimen with striker bar velocity of 12.5 m/s.

Figure 4 True Strain Rate-True Strain results for two striker velocities on compressive NiTi specimens.

The quasi-static loading seen in Figure 5 shows the stress-induced martensitic phase transformation initiating at 575 MPa and completing at 750 MPa. The martensitic phase then loads elastically and reaches an elastic limit at 1050 MPa where it begins to linearly harden. At the same stress level, 1050 MPa, the high strain rate test begins to harden at the same linear rate seen in the quasi-static test, although the elastic limit still occurs at 575 MPa. The deformation between 575 MPa and 1050 MPa is therefore assumed to be the stress-induced martensitic phase transformation, albeit without the stress plateau or the detwinned strain associated with the stress-induced phase transformation. There are several possible explanations for why these phenomena might not occur in the high strain tests. The release of latent heat, as described in the previous section, may strongly influence the material response. In the high strain rate tests shown in Figure 5, the temperature measurement showed a rise of 5°C and 50°C for the 500 ε/s and 1500 ε/s strain rates, respectively. Additionally, the kinetics of the phase transformation will also play an important role in the material response. Quasi-static stress-induced loading has shown the

generation of phase fronts (austenite to detwinned martensite) which move through the specimen under a near constant stress [8]. At high strain rates, a limitation may exist on the velocity of this phase front, which could then inhibit the development of detwinned martensite; however, this conclusion has yet to be experimentally validated.

Figure 5 Comparison of True Stress- True Strain results for NiTi under compressive quasi-static and high strain rate loading.

To study the influence of cyclic loading at high strain rates, the high strain rate samples shown in Figure 5 were repeatedly cycled with the attempt to maintain a constant strain rate. The resulting true stress-true strain curves are shown in Figure 6 and Figure 7. The stress-strain curves in Figure 6 were produced with strain rates nearly identical to the strain rate shown in Figure 4 for the 12.5 m/s striker bar velocity. This fact ensures that similar boundary conditions were present in each cycle. The stress-strain curves in Figure 7, however, were not performed at similar strain rates. The rate of hardening in the material due to the large plastic strain developed in the first cycle made it difficult to identify the required striker bar velocity to maintain a constant strain rate. Therefore, the second and third cycles in Figure 7 were at strain rates between 500-1000 ε/s.

Figure 6 Consecutive loading at nearly constant strain rate of 500 ϵ/s on annealed NiTi in the compressive SHPB.

Of particular interest in Figure 6 and Figure 7 is the change in the mechanical response as the cycle number and plastic strain level increases. The first change is reflected in the reduction of the threshold stress level for the onset of stress-induced martensite, delineated by the dotted line in Figure 6. As seen in Figure 2, if the slope of the martensitic start line is assumed constant, as in most thermodynamic NiTi models, then a reduction in the threshold stress implies an increase in the martensitic start temperature at zero stress. This result matches those observed in quasi-static loading [15, 16], and has been related to the development of dislocations that aid in the martensitic transformation. Comparing the reduction of the threshold stress seen in Figure 6 and Figure 7 shows that the test with the higher number of cycles shows the largest decrease in the threshold stress. Since the final plastic strain level is identical for both specimens it can be concluded that the threshold stress reduction is dependent on cycle number rather than level of plastic strain.

A second change in mechanical response identified in Figure 6 and Figure 7 is the material hardening during the phase transformation. As seen in Figure 6, the hardening slope in the inelastic region of the loading, which contains both the phase transformation and plastic deformation, increases with the number of cycles and plastic strain. However, the change in the hardening slope seen in Figure 7 is similar to that of Figure 6, such that the hardening slope measured in the final cycle of

both figures is almost identical. In summary, the data implies that the hardening slope of the material is dependent upon the level of plastic strain rather than the number of cycles.

Figure 7 Consecutive loading at nearly constant strain rate of 1500 ϵ/s on annealed NiTi in the compressive SHPB.

The final analysis performed on the specimens was a microstructural evaluation of the cycled specimens. Shown in Figure 8 are room temperature optical micrographs, at two different magnifications, of the NiTi specimen with mechanical results shown in Figure 7. The significance of these micrographs is the identification of martensitic plates in the microstructure. Table 1 shows that room temperature is 13°C above the austenitic finish temperature and therefore the microstructure should be completely austenitic. However, as described in the literature [17, 18], this retained martensite is due to dislocations locking the martensitic phase into the microstructure. It should also be noted that the specimen was heated at least 50°C above room temperature prior to examination under the optical microscope to ensure that the retained martensitic phase was not caused by an increase in the austenitic finish temperature induced by the deformation. The existence of the martensite and the measured temperature increase support the fact that a stress-induced phase transformation did occur, and the material was not simply loaded and deformed in the austenitic phase.

Figure 8 Optical micrographs at different magnifications of a NiTi specimen cycled three times in a SHPB with a total of 10% plastic strain.

Tensile Incipient Failure Tests

The engineering stress-strain results for three uni-axial specimens tested in the tensile SHPB are shown in Figure 9 showing one specimen tested to failure. The initial ringing of the curves around 500 MPa is associated with Pochhammer-Chree oscillations in the incident pulse and the time required to reach stress equilibrium in the specimen due to the complex specimen geometry. The ringing unfortunately masks the portion of the stress-strain curve that contains the martensitic stress-induced phase transformation. The oscillations shown in Figure 9 end with a region of very low strain hardening around 0.08-0.10 engineering strain, however, between 0.10-0.12 engineering strain the material begins to linearly harden. The unloading portions of the curves in Figure 9 also show the reverse phase transformation from martensite to austenite identified by the bilinear nature in the unloading. Figure 10 compares the results of a tensile SHPB specimen and a specimen tested quasi-statically in tension. It is difficult to identify the martensitic phase transformation and the martensitic yield stress in the quasi-static curve; however, an approximate transformation strain of 3.5% is delineated on the curve. The dynamic stress-strain curve lags the quasi-static curve at a strain equal to the transformation strain, which is similar to the result for the compression tests shown previously. The uniaxial specimens also showed a temperature rise with increases ranging from 22°C to 48°C.

The axial components of force and displacement for the “E” notch geometry, converted to values of engineering stress and engineering strain through the use of minimum cross-sectional area and a gage length equal to the notch radius, are shown in Figure 11. Due to the notch geometry, a more complex stress state is actually seen by the specimen than is represented in the figure. Three of the four specimens tested failed, with each failure occurring at different levels of engineering strain. The tests that failed near 30% engineering strain were unsuccessful attempts to arrest the void growth before specimen failure. The inconsistency in failure strain is

associated with the nature of the failure and will be discussed later. For the specimen that attained the highest level of stress, as seen in Figure 10, failure occurred after loading and during the reverse phase transformation from martensite to austenite. The mechanics of the reverse transformation, *i.e.* the large recovery of detwinned martensite induced strain, adds an additional level of complexity in the attempt to arrest damage prior to fast crack growth. If a large amount of damage is developed in the specimen during the loading, the specimen will not be capable of withstanding the forces applied to the end of specimens upon unloading and recovery of strain associated with the reverse transformation.

Figure 9 Engineering Stress-Strain for three uniaxial NiTi specimens loaded in the tensile SHPB.

Figure 10 Comparison of Engineering Stress-Strain curves for dynamic and quasistatic loading rates.

Figure 11 Engineering Stress-Strain in the axial direction for "E" notch specimens.

One specimen was tested to failure and a second was arrested for the "A" notch specimens. The engineering stress-strain curves for these specimens appear very similar in shape to those for the "E" notch seen in Figure 11 and are shown in Figure 12. The martensitic phase transformation began at a stress level of ~850 MPa in this specimen geometry, compared to ~750 MPa for the "E" notch and 600 MPa for the uniaxial specimens. This observation can be explained by the increased ratio of hydrostatic to deviatoric stresses created in the "A" notch geometry.

Observations of the failed specimen surfaces show very little cup and cone behavior associated with ductile fracture. Detailed analysis of the fracture surface is shown in Figure 13 and illustrates that the failure mechanism was mixed mode,

consisting of ductile void growth and transgranular cleavage that shifted planes along grain boundaries. However, the specimens that were arrested before failure did show void growth in all three geometries. A microphotograph of the voids developed in the "A" notch specimen is shown in Figure 14, which is representative of the voids seen in each specimen geometry. Figure 14 shows a close up view of the center of the specimen. The voids seen in the figure have an average size of approximately 10 μm and show a very random distribution across the specimen cross-section in the uniaxial specimen geometry, attributable to the lack of tensile instability (neck) prior to failure. The "E" notch specimen geometry showed enhanced porosity in the region of greatest stress triaxiality. The "A" notch specimen geometry exhibited enhanced porosity in the center and along one side of the center.

Figure 12 Engineering Stress-Strain in the axial direction for "A" notch specimens

Figure 13 SEM picture of a failed "E" notch specimen.

Figure 14 Post-test voids developed in arrested "A" notch specimen showing a. entire cross section and b. close-up of specimen center.

SUMMARY

In this research, NiTi specimens have been subjected to a dynamic compressive loading that induced a stress-induced martensitic transformation and plastically deformed the martensitic phase. Comparisons between quasi-static and dynamic loading stress-strain curves showed dramatic differences in the amount of detwinned strain developed in the specimen. Specimens loaded at strain rates of 500- 1500 ϵ/s showed no detwinning plateau as identified in quasi-static tests. Although the curves were shifted by the amount of detwinned strain, the rate of hardening in the martensitic phase was similar for both the quasi-static and high strain rate tests, therefore showing rate independence of the martensitic flow stress. Additionally, changes in the threshold stress for stress-induced martensite and the hardening rate during the phase transformation were identified with respect to cycle number and plastic strain level. Results showed that the reduction in the threshold stress for stress-induced martensite was dependent on cycle number and independent of plastic strain level. However, the hardening rate during the phase transformation was independent of cycle number and dependent on the plastic strain level.

NiTi specimens have also been subjected to dynamic tensile loading that induced a stress-induced martensitic transformation and plastically deformed the martensitic phase inducing damage through the formation of voids. The martensitic phase transformation was identified as having a higher strain rate than the remaining deformation and also a significant temperature increase associated with the latent heat of transformation was measured. The failure mechanisms of the NiTi specimens were identified as being mixed mode; void nucleation and growth followed by transgranular failure. Voids were also found in each arrested specimen. Voids occurred rather uniformly along the length of the uniaxial stress specimen geometry, suggesting that nucleation occurs by reaching a deviatoric strain threshold. The localization of voids in the "E" and "A" notch geometries in the region of highest stress triaxiality implies that increasing stress triaxiality decreases the amount of deviatoric strain necessary to nucleate voids.

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