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Title: The Effect of Repeated Compressive Dynamic Loading on the Stress-Induced Martensitic Transformation in NITI Shape Memory Alloys

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The effect of repeated compressive dynamic loading on the stress-induced martensitic transformation in NiTi shape memory alloys

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It has been shown that quasi-static, cyclic, isothermal mechanical loading influences the mechanical response of the stress-induced martensitic transformation in fully annealed NiTi Shape Memory Alloys (SMAs). As the cycle number increases, hardening of the stress-strain response during the martensitic phase transformation is seen along with a decrease in the threshold stress for initiation of stress-induced martensite. Also, the amount of plastic strain and detwinned martensitic strain decreases as the cycle number increases. However, NiTi SMAs have not been experimentally explored under high compressive strain rates. This research explores the cyclic near-adiabatic stress-induced martensitic loading using a 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. The results from the high strain rate tests show similarities with the quasi-static results in the hardening of the stress-strain response and shifting of the threshold stress for initiation of stress-induced martensite.

1. 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 [4-9].

Mechanical 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 are related to the presence of lattice defects introduced by cold working [10, 11]. There are also many experimental results on 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 on the transformation characteristics [12-15]. In these works, plastic deformation is developed during the loading as a result of the phase transformation process and the amount of plastic strain remains small compared to the overall applied strain. Miyazaki *et al.* [16] investigates large plastic deformations for isothermal mechanical loading, and Miller and Lagoudas [17] has studied the effect of plastic strain induced during cyclic pseudoelastic loading on the phase transformation temperatures and thermomechanical response.

Even though the stress-induced martensitic transformation has been studied for multiple loading paths and material conditions, it has not been experimentally characterized for high strain rates. In

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this research, dynamic loading tests are performed on NiTi specimens using a Split Hopkinson Pressure Bar (SHPB) which allows for investigation of strain rates from $10^2 \leq \dot{\epsilon} \leq 10^4$.

2. 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 relative high concentration of oxygen is present. Cylindrical specimens with a 5.0 mm diameter and 5.0 mm long were electro-discharge machined from the bar. Differential Scanning Calorimetry (DSC) results of the as-drawn material confirmed that the high level of dislocations imparted by the cold work resulted in suppression of the thermally-induced martensitic phase transformation. Therefore, the cold work in the specimen was removed through an annealing process of 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.

The room temperature loading path on the NiTi SMA specimens, as identified by the

transformation temperatures 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 [18]. Figure 2 shows an isothermal loading, represented by the dotted line, in which stress-induced martensite is formed upon loading and returns to the austenitic phase upon unloading when certain criteria are met, as described by Liu and Galvin [19]. However, in the SHPB the specimen will not remain isothermal due to the near adiabatic conditions associated with the dynamic loading and the release of the latent heat from the stress-induced martensitic phase transformation. A temperature increase in the specimen raises 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. Future studies on this material system are planned which will improve the temperature measurement capabilities to better capture the temperature rise during the phase transformation.

To study the response of the NiTi SMA under cyclic loading, specimens were repeatedly loaded in the 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 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 , mean rate of 1500 ϵ/s and quasi-statically are all compared.

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 for 1 hr. and water quenched

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



Figure 1 Microphotograph of annealed NiTi showing precipitant microstructures.

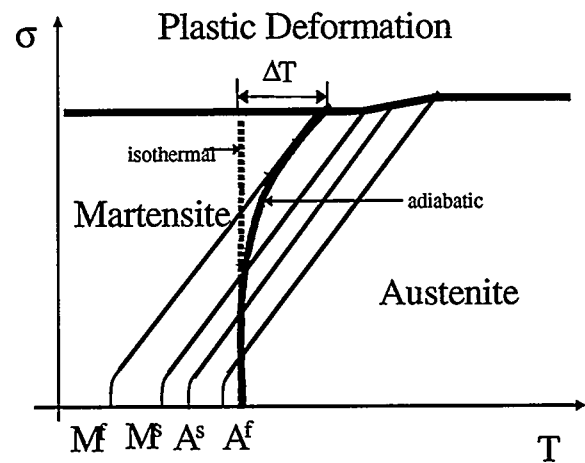


Figure 2 Stress-Temperature phase diagram for fully annealed NiTi.

3. RESULTS AND DISCUSSION

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 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 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 stress-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 1/s and 500 1/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 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 seen in the loading. Assurance that a martensitic transformation actually occurred during the loading is shown in the 12.5 m/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 18.5 m/s test, have been shown to inhibit the reverse transformation in quasi-static tensile tests [20, 21]. However, dynamic tensile tests with large-scale plastic deformation, ~45% plastic strain, did not inhibit the reverse transformations [22].

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.

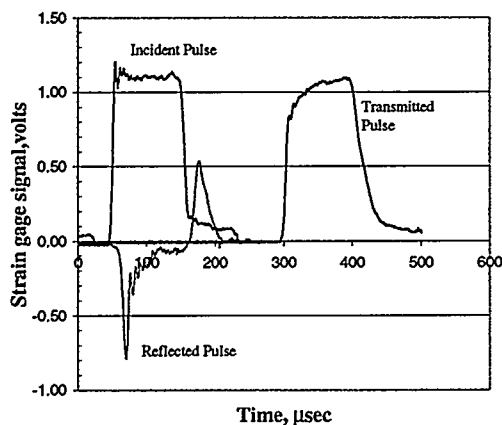


Figure 3 Strain gage output signals for NiTi specimen with striker bar velocity of 12.5 m/s.

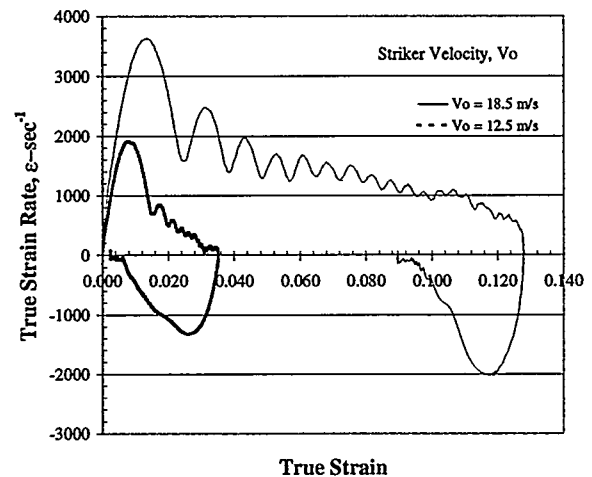


Figure 4 True Strain Rate-True Strain results for two striker velocities on NiTi specimens.

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 as discussed earlier. There are several possible reasons for which these phenomena might not occur in the high strain rate 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, a crude temperature measurement showed a rise of 5°C and 50°C for the 12.5 m/s and 18.5 m/s impact velocities, 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 [12]. 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.

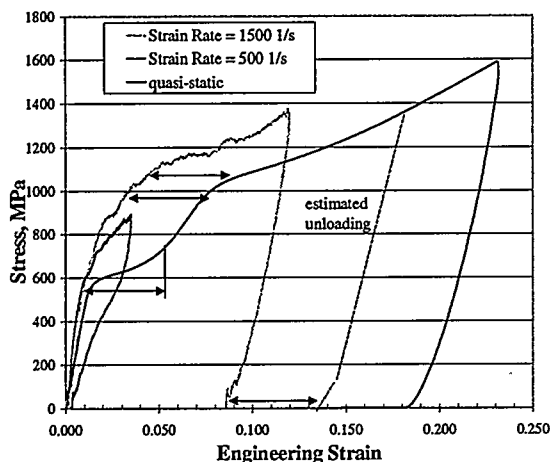


Figure 5 Comparison of Stress- Engineering Strain results for NiTi under quasi-static and high strain rate loading.

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 stress-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 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 1/s.

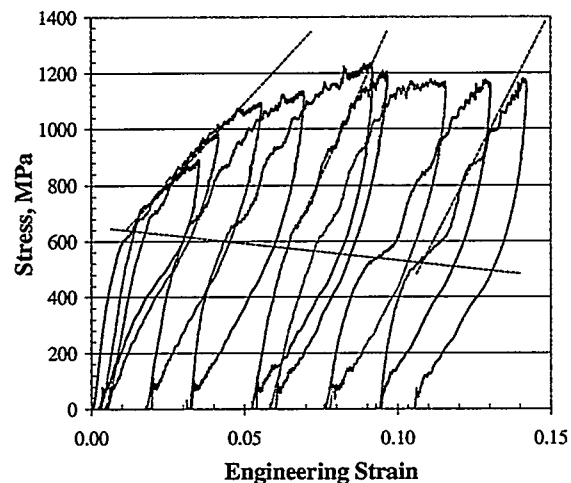


Figure 6 Consecutive loading at nearly constant strain rate of 500 1/s on annealed NiTi in the SHPB.

Also 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 [15-17, 21], 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 plastic strain.

A second change in mechanical response identified in Figure 6 and Figure 7 is the material hardening during the phase transformation and plastic strain development. As seen in Figure 6, the hardening slope of the inelastic region of the loading, which contains both the phase

transformation and the plastic deformation, increases as the number of cycles and plastic strain increases. 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.

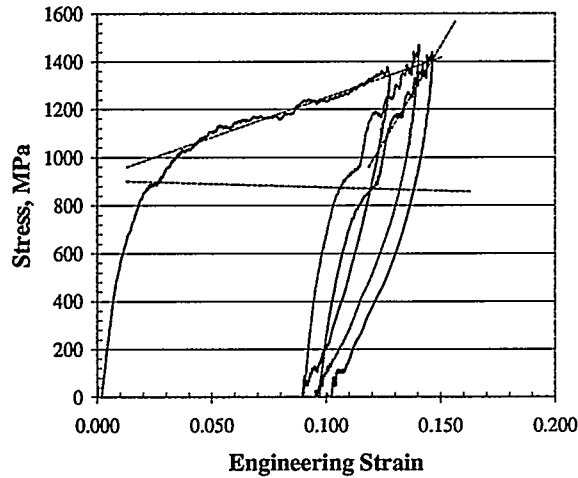


Figure 7 Consecutive loading at nearly constant strain rate of 1500 1/s on annealed NiTi in the SHPB.

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, 23, 24], 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 optical microscope to ensure that the retained martensitic phase was not caused by an increase in the austenitic finish temperature induced by the deformation.

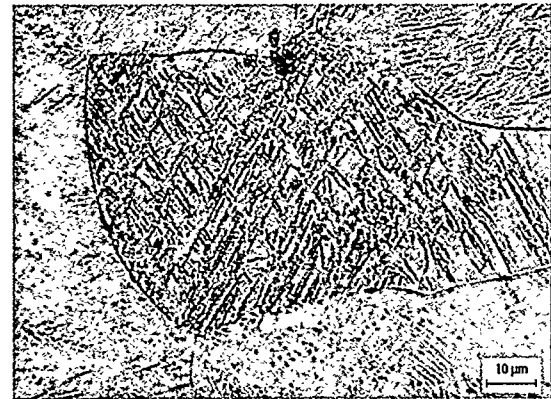


Figure 8 Optical micrographs at different magnifications of a NiTi specimen cycled three times in a SHPB with a total of 10% plastic strain.

4. SUMMARY

In this research, NiTi specimens have been subjected to a dynamic tensile 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 1/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, and thereby 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.

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