Evolution of Phase Transformation Fronts and Associated Thermal Effects in a NiTi Tube Under a Biaxial Stress State

by

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ABSTRACT

This letter presents results from an experiment involving a pseudoelastic NiTi tube loaded under combined tension and internal pressure. Upon reaching a critical state of stress, the initially austenitic phase (A) transforms to the martensitic phase (M) resulting in macroscopic deformation of the material. The transformation-induced deformation is inhomogeneous, in this case in the form of localized helical bands of M that propagate, eventually consuming the whole specimen. On unloading, the material transforms back to A again in an inhomogeneous manner. The evolution of the transformations is captured using stereo digital image correlation. Because the rate of loading was somewhat higher than optimal, the latent heats of transformation caused a small amount of heating during transformation to M and cooling when the material reverted back to A. Infrared thermography showed heating and cooling fronts that develop to follow the transformation fronts. The local rise in temperature during loading resulted in a nearly linear increase in the transformation stresses and a similar decrease during unloading. The changes in stresses were shown to correlate quite well with the temperature dependence of the transformation stress established in independent experiments. The results illustrate that the effects of latent heats can distort the recorded stress-deformation responses under biaxial stress states.

1. INTRODUCTION

Pseudoelasticity describes the unique property exhibited by several shape memory alloys of being deformed to strains of a few percent and fully recovered on unloading ($\sim 7\%$ for nearly equiatomic NiTi). This property is derived from a diffusionless transformation between the *austenitic* (A) and *martensitic* (M) phases, which can be induced by either changes in temperature or stress. It is well-known that tension-induced phase transformation causes localized deformation with the “highly” strained M-phase coexisting with the low-strained A-phase at the same stress (e.g., Shaw and Kyriakides [1995, 1997], Li and Sun [2002], Iadicola and Shaw [2002, 2004], Feng and Sun [2006], Abeyaratne and Knowles [2006], Hallai and Kyriakides [2013], Reedlunn *et al.* [2013], Bechle and Kyriakides [2014]). We recently investigated how this reversible transformation varies under biaxial stress states using 6.35 mm NiTi tubes loaded under combined axial load and internal pressure (Bechle and Kyriakides [2015]). Radial paths with axial-to-hoop stress ratios ranging from -1.0 to uniaxial tension revealed that, but for a limited region near equibiaxial tension, transformation leads to localized helical deformation bands that generally propagate under essentially constant stress. The stress remains constant provided the experiment is conducted under isothermal conditions.

The aim of the present paper is to demonstrate that as is the case for uniaxial tension, under equibiaxial stress states the latent heat of transformation can alter the response if the test is not performed under isothermal conditions. This premise is demonstrated through a biaxial experiment in which a NiTi tube is loaded along a radial path with axial-to-hoop stress ratio of 2.0. Because of the anticipated instabilities associated with such phase transformations, the specimen is pressurized by prescribing the rate at which volume of liquid is pumped into the specimen. The axial load is run under load control and feedback is used to ensure that the applied tensile force maintains the required ratio to the measured pressure throughout the load/unload history. Even though the prescribed volume flux is small, the change in specimen internal volume associated with this stress ratio is very small and the induced rate at which material transforms is somewhat larger than ideal. Consequently, the latent heat released/absorbed during the M and A transformations could not be dissipated/supplied fast enough by the environment resulting in modest heating/cooling of the specimen. Stereo Digital Image Correlation (DIC) is used to monitor the full-field deformation history and infrared (IR) thermography the evolution

of temperature during loading and unloading. The measurements will be used to quantify the thermomechanical interaction that takes place and their effects.

2. EXPERIMENTAL

The experiment is conducted on a nearly equiatomic NiTi tube (50.9 at % Ni) with an outer diameter (D) of 6.323 mm and a wall thickness of 0.271 mm. The transformation temperatures, established via differential scanning calorimetry, are $A_s = -10$ °C and $A_f = 12$ °C. The experiment was performed at 24 °C thus the material was initially in the austenitic phase. The tube was loaded under combined axial tension and internal pressure in a custom biaxial test set-up shown schematically in Fig. 1. It consists of a screw-type axial testing machine that operates in conjunction with a 690 bar metering syringe pump that can dispense very small volumes of water at precise rates. The specimen with an overall length of 115 mm is mounted into the testing machine via carefully aligned custom stainless steel housing grips with locking clamps as shown in the figure. Steel plugs are inserted into the clamped ends of the tube to prevent crushing by the clamping. The setup leaves free a 50 mm test section. The axial force (F) is monitored by a 5 kN load cell and the pressure (P) by a 690 bar pressure transducer.

By connecting the axial and pressure loading systems through feedback, the tube is loaded along a radial stress path in the nominal axial (x) and hoop (θ) stress space so that

$$\sigma_x = 2.0\sigma_\theta. \quad (1)$$

The stresses are related to the measured force and pressure via

$$\sigma_x = \frac{F}{2\pi R t} + \frac{PR}{2t} \text{ and } \sigma_\theta = \frac{PR}{t}, \quad (2)$$

where R and t are respectively the mid-surface radius and wall thickness of the tube.

Pressurization is performed by prescribing the rate at which water is pumped into the closed system (0.02 ml/min). The axial testing machine is operated under *load control* with the pressure signal as external command using a TestProfiler method in Instron's Bluehill 3 software. The pressure is used in Eqs. (1) and (2) to establish a target value of force that corresponds to the stress ratio of 2.0. This target value is compared to the current set point and the difference becomes the command signal that ramps the force in the chosen time step. In this manner the force is continuously updated so that it follows the measured pressure at the desired ratio.

The dispensed fluid volume, pressure, axial load, and machine displacement are monitored using a LabVIEW based data acquisition system. The surface deformation history over the length of the test section and approximately one third of its circumference is recorded using stereo Digital Image Correlation (DIC—ARAMIS software with 5 MP cameras). To accommodate the DIC measurement of strains, one-half of the circumference of the specimen is covered with a fine black speckle pattern painted over a white undercoat. Images are recorded at 5 s intervals. The evolution of axial and circumferential strains, ε_x and ε_θ , is determined by post-processing the recorded images. Simultaneously, a FLIR ThermoVision SC6000 infrared camera is used to record full-field temperature profiles of the backside of the specimen. To this end, this half of the circumference is painted with flat black enamel. Infrared images are recorded every 2.5 s at a 620×512 pixel resolution. Post-processing of the data is performed with FLIR's ExaminIR data. The data acquisition system, the DIC and Infrared cameras are operated on a common time base for synchronization.

3. EXPERIMENTAL RESULTS

(a) Loading

In the experiment the nominal axial and hoop stresses follow a radial path so that a constant stress ratio of 2.0 is maintained ($\sigma_x = 2.0\sigma_\theta$). Figure 2a shows a plot of σ_θ against σ_x confirming that this stress ratio is maintained throughout the load/unload history. The specimen is loaded to a stress level that corresponds to just beyond the completion of the inhomogeneous part of austenite (A) to martensite (M) transformation and unloaded. Figure 2b shows the two recorded stress-average strain responses ($\sigma - \bar{\varepsilon}$) where $\bar{\varepsilon}_x$ and $\bar{\varepsilon}_\theta$ represent the spatial average of the strain in each direction over the full field of view recorded by DIC (FOV has an axial span of about 6D or 38 mm). The two responses trace closed hystereses common to pseudoelastic SMA behavior. Figure 3a shows 38 sets of full-field DIC contours of axial and hoop strain recorded during loading taken at 5 s intervals. The images numbered 1 to 38 correspond to the numbered bullets on the responses in Fig. 2b. Bullet and image 1 correspond approximately to the onset of M transformation and 38 to the point at which unloading commences. Included in Fig. 3a is a set of recorded infrared (IR) thermograms with color contours representing the change in temperature, ΔT , over the base temperature of 24 °C. The

images very closely correspond to the same times as the strain contours in the same figure (the IR images span about a length of 7D).

The results collectively clearly indicate that transformation occurs with modest change in stress, leads to inhomogeneous deformation, and to small but tractable changes of temperature. We discuss the events associated with loading through Figs. 2 and 3a with supporting results presented in Figs. 4-6. The A phase initially deforms in a linearly elastic manner with modulus and Poisson's ratio (E_A, ν_A) of about (66 GPa, 0.425) (see Fig. C1 in [Bechle and Kyriakides \[2015\]](#)). The deformation is homogeneous and the temperature remains uniform and essentially unchanged at 24 °C as depicted in images 1. In this neighborhood, the modulus starts to decrease with a sharp knee developing at an axial stress of 615 MPa. This is an indication that transformation to M has commenced. In fact, the DIC images following 1 show that higher strain localized deformation is entering the FOV from the RHS. Simultaneously, a somewhat higher temperature front has appeared on the right and, interestingly, a weaker one on the left. It appears that transformation nucleated nearly simultaneously at both ends due to the stress concentrations associated with the clamping of the specimen. However, initially these zones lie outside the DIC FOV, and no strain is recorded by the DIC until the transforming material enters the FOV.

Localized deformation patterns enter the FOV first on the RHS in the form of a multi-helical transformation front with sinistral bands. These can be seen in more detail in an expanded view of image 7 included in Fig. 4. Such a front is preferred as it tends to minimize structural distortions of the tubular specimen (see [Feng and Sun \[2006\]](#)). The mainly transformed martensite has an axial strain of just under 7%, a value that corresponds to the end of the stress plateau in the response in Fig. 2b. The corresponding hoop strain is at about -2.3% as it is mainly driven by the Poisson effect. The IR images in Fig. 3a show that the latent heat released by the transforming material has resulted in an increase in the temperature behind the front (see also [Favier et al. \[2007\]](#)). Figure 5 displays a more quantitative view of the evolution of temperature in the test specimen. Plotted are axial temperature change profiles at different times during loading. In concert with the IR images, the $\Delta T - x/L$ profiles show that both ends of the specimen experience local heating from the beginning of the stress plateau. This occurs before any deformation fronts enter the DIC FOV and continue once the front of the right appears. This supports the notion that transformation nucleated first at the ends outside the DIC FOV. The thermal fronts are somewhat diffuse and become visible ahead of the transformation front.

In images 2-12 the multi-helical transformation front on the right propagates towards the center of the specimen and concurrently a thermal front is seen in the IR images to follow suit. The thermal front has a finite width as some of the heat released by the transforming front gets dissipated behind it. In images 5 the transformation front on the left has also entered the DIC FOV. It is also multi-helical but with less pronounced prongs. The two fronts propagate towards each other in a nearly steady-state manner supported by the approximately linear time trajectories of both the strain and thermal fronts in Fig. 3a. With the propagation of the fronts the overall temperature of the specimen increases in the manner shown in Fig. 5 with the temperature at the location of transformation being higher. Consequently the stress of transformation increases causing the nearly linear increase in the recorded stresses observed in Fig. 2b.

As the two fronts approach each other, approximately from image 24 onwards, the helical prongs get weaker, which for example can be observed in the expanded view of the RHS front from image 24 in Fig. 4. At some point the helical bands disappear as shown in the expanded view of one of the fronts from image 29 in Fig. 4. This takes place first on the left end and then on the right leaving behind two fronts that form ellipses along inclined planes of constant angle (see images 28-34). The fronts interact thermally with the two temperature peaks eventually coalescing as shown in in Figs. 3a and 5. The maximum ΔT of about 5.75 °C is recorded in image 34 that also corresponds to the completion of transformation close to the end of the top stress plateau in Fig. 2b. Although the specimen loading continues until an axial stress of 689 MPa is reached at point 38, the specimen temperature in subsequent images is decreasing. The nearly linear increase in the stresses continues until images 34 with the axial stress reaching 647 MPa, which represents about a 30 MPa increase over a stress measured at the onset of transformation (for a full record of the response and associated events see movie alp_2.0_DIC+IR.mp4).

(b) Unloading

Unloading the specimen is achieved by reversing the flow of water into the specimen and proceeds at the same rate as that followed during loading. Figure 3b shows 33 sets of DIC contours of axial and hoop strain taken again at 5 s intervals. Included is a set of IR images of the specimen corresponding to the same times. The mainly M phase material initially unloads

homogeneously (see images 39) but follows a rather nonlinear stress-strain trajectory similar to that observed in unloading under uniaxial tension. At a stress of about 290 MPa, sharp knees develop in the responses in Fig. 2 indicating that transformation to A has commenced, albeit initially outside the DIC FOV. This transformation again leads to inhomogeneous deformation with the strain behind it being at the level of A at this stress (end of stress plateau $\sim 0.34\%$). A lower strain transformation front appears on the right starting in image 41, and is seen to propagate towards the center of the specimen in subsequent images. This front is also multi-pronged but has both sinistral and dextral finger-like features (see expanded view of image 50 in Fig. 6; see Shaw and Kyriakides [1998] for similar features in strips). The symmetry of this front minimizes structural distortions. The nucleation of A at the ends of the specimen is unusual as in most biaxial experiments reported in Bechle and Kyriakides [2015] A tended to nucleate at the sites at which M transformed last during loading, which in this case is near the center of the specimen. This difference may have been caused by non-uniform temperature outside the IR FOV when unloading commenced. Since the A transformation is endothermic, transformation at the ends has a local cooling effect that is discernible in the IR images, and is even more clearly seen in the $\Delta T - x/L$ profiles plotted in Fig. 7. The temperature reduction is smaller than the increase during loading, but it still leads to a gradual but noticeable decrease in the recorded stresses in Fig. 2b. In images 46 a second multi-pronged front enters the DIC FOV from the left. It also propagates towards the center and is followed by a cooling front (Figs. 3b and 7). As a result of the dual propagation of transformation fronts from the two ends, the center of the specimen remains at about 2 °C higher temperature (Fig. 7). Consequently, in image 49 transformation nucleates at a third site near the center of the specimen. It appears to be in the form of a helical band as its front end enters the FOV further on the right in image 51 (see Fig. 3b). This end subsequently intersects the first multi-prong front and stops while the rest of the helical band widens. The local cooling associated with the multiple propagating fronts results in the undulations observed in the $\Delta T - x/L$ profile of thermogram 50 and in subsequent ones in Fig. 7 and in axial temperature variations in the corresponding IR images in Fig. 3b.

By image 58 the multiple transformation fronts have reduced the remaining M to what appear as two "islands" near the middle of the FOV (may be part of a helical band). The M zones are close enough for them to interact thermally as seen in Fig. 7. Transformation continues and by images 67 the whole FOV has returned back to A and any further deformation is

homogeneous. Although transformation has been completed, cooling of the central part of the specimen continues for a while longer, an indication that heat transfer occurs at a slower rate than transformation. The stress is down to 264 MPa or 26 MPa lower than the level at which M nucleated. Further unloading occurs with the elastic modulus of A (a small residual strain remains after unloading).

(c) Thermomechanical Interaction

The data presented provide a vehicle for quantifying the thermomechanical interaction that has taken place. Concentrating on loading, in Fig. 8a we plot the maximum temperature increase time history for the 38 images in Fig. 3a. It is observed that following an initial transient, ΔT increases nearly linearly with time. Thus between images 13 and 31 the temperature increases at an essentially constant rate of 0.0192 °C/s. Between points 31 and 34, which represents the end of transformation, the temperature experiences a sudden increase due to thermal front interaction and is followed by a decay in ΔT .

The stress at which M nucleates at 23 °C for this NiTi alloy under biaxial stress states has been shown to trace an unconventional trajectory in the $\sigma_x - \sigma_\theta$ plane (see Fig. A1; see also tension/torsion results in Sun and Li [2002]). These transformation stresses were shown to be well represented using the Cazacu et al. [2006] anisotropic "yield" function that also accounts for the tension-compression asymmetry exhibited by the NiTi tubes studied. The calibrated function was adopted here to evaluate the equivalent stress (σ_e) recorded in the present biaxial experiment as a function of time. The σ_e time history plotted in Fig. 8b is seen to increase nearly linearly from the onset of M transformation at point 1 to the end of transformation at 34. The rate of increase of σ_e between points 13 and 31 is 0.147 MPa/s ($\dot{\sigma}_e$). We aim to correlate this increase in stress to the temperature increase. To this end we use results on the variation of transformation stresses with temperature reported in Bechle and Kyriakides [2014] from sets of isothermal uniaxial tension and compression experiments on tube of the same alloy. Their results are presented in Fig. A2 normalized by the stress at which M nucleated under tension at 23 °C ($\sigma_{NM23} = 381$ MPa). In the temperature range of interest here, the M transformation stress under tension, σ_{NM} , has a slope of 5.9 MPa/°C. Using this slope and the measured rate of change of temperature, the rate of change of equivalent stress in our experiment is calculated to be 0.113

MPa/s ($\dot{\sigma}_{eca}$). Included in Fig. 8b is a plot of equivalent stress vs. time between points 13 and 31 yielded by this calculation. Although this prediction yields slightly lower stress than what was measured, the general trend confirms the influence of latent heat on the measured stress-strain results for SMAs. The difference can be attributed to the fact that the temperatures reported are surface temperatures, which may be influenced by heat losses.

4. CONCLUDING REMARKS

A NiTi tube has been loaded under combined tension and internal pressure tracing a radial path with an axial-to-hoop stress ratio of 2.0 in a manner that allows for the inhomogeneous deformation associated with phase transformations to develop freely. The material is initially in the A phase, but transforms to the much higher strain M when a critical stress is reached. Stereo DIC enabled tracking of localized helical bands of M (A) that gradually consume the specimen during loading (unloading). Because the rate of loading was somewhat higher than optimal, the latent heats of transformation caused a small amount of heating during transformation to M and cooling when the material reverted back to A. Infrared thermography showed heating and cooling fronts that develop to follow the transformation fronts. The local rise in temperature during loading resulted in a nearly linear increase in the transformation stresses and a similar decrease during unloading. The changes in stresses were shown to correlate quite well with the temperature dependence of the transformation stress established in independent experiments. The results illustrate the influence of latent heats on recorded stress-deformation responses. They also support the notion that in order to avoid this thermomechanical interaction, material characterization experiments are best performed under isothermal conditions.

Appendix

Figure A1 shows a locus of martensite transformation stresses generated from combined axial load-internal pressure experiments using the same NiTi tube stock as the one used in the present experiment (from Bechle and Kyriakides [2015]). Included is a fit of the data using the anisotropic yield function of Cazacu et al. [2006]–CPB– that also includes the tension/compression asymmetry exhibited by the tubes tested. A fit of the data based on the von Mises yield function is seen not to be appropriate.

Bechle and Kyriakides [2014] conducted a series of isothermal uniaxial tension and compression experiments at several temperatures on similar alloy NiTi tubes. Fig. A2 shows the measured M and A nucleation stresses vs. temperature. The stresses are normalized by the M nucleation stress in tension at 23 °C ($\sigma_{NM23} = 381$ MPa). This batch of material had an $A_f = 21$ °C where as for the present batch $A_f = 12$ °C. We assume that the transformation stress-temperature slopes are the same for the two batches of tubes.

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Figure Legends

- Fig. 1 Schematic of the axial-internal pressure test facility used in the experiment. Included are the specimen and gripping, the feedback loop used to produce the radial stress path load/unload history and the DIC and IR diagnostics used to monitor the deformation and temperature of the test section.
- Fig. 2 (a) The radial stress history followed. (b) Axial stress-average strain and hoop stress-average strain recorded.
- Fig. 3a Sequences of axial and hoop strain contours from DIC and infrared thermograms corresponding to the numbered bullets marked on the responses in Fig. 2b during loading.
- Fig. 3b Sequences of axial and hoop strain contours from DIC and infrared thermograms corresponding to the numbered bullets marked on the responses in Fig. 2b during unloading.
- Fig. 4 Expanded views of martensitic transformation fronts during loading.

Fig. 5 Axial profiles of temperature changes at different times during loading.

Fig. 6 Expanded view of austenitic transformation front during unloading.

Fig. 7 Axial profiles of temperature changes at different times during unloading.

Fig. 8 (a) Maximum temperature in specimen vs. time during loading.
(b) Measured equivalent stress vs. time during loading.

Fig. A1 Locus of martensitic transformation stresses measured in biaxial experiments and fits based on Cazacu et al. [2006] and von Mises functions (from Bechle and Kyriakides [2015]).

Fig. A2 Transformation stress vs. temperature for loading and unloading from tension and compression tests on NiTi tubes (from Bechle and Kyriakides [2014]).

(a)

(b)

F3a

F3b

F4

F5

F6

FA2