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Thermomechanical cycling of a NiTi shape memory alloy-macroscopic response and microstructural evolution

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ABSTRACT

Thermomechanical cycling of a $Ni_{49.9}Ti_{50.1}$ (at.%) shape memory alloy was investigated. Combined *ex situ* macroscopic experiments and *in situ* neutron diffraction measurements were performed to relate the macroscopic evolution in behavior (e.g., dimensional instabilities) observed during thermal cycling to the responsible microscopic mechanism(s) through texture, internal strain, peak shape, and phase evolution from the neutron data. Pre-deformation in the austenite or martensite phases affected the macroscopic cyclic behavior (e.g., actuation strain), depending on the level of pre-strain and the associated microstructural changes. However, the pre-deformation did not completely stabilize the cyclic response. Subsequent thermomechanical cycling revealed that the martensite texture changed with continued thermal cycling, while the austenite texture did not. For the conditions investigated, stagnation of the martensite texture occurred around the eighth cycle, consistent with asymptotic saturation of the macroscopic transformation strains. Moreover, diffraction spectra peak shapes (broadening) were found to vary with cycling indicative of the accumulation of lattice defects, consistent with the constant increase in residual strain.

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1. Introduction

Near-equiatomic NiTi shape memory alloys (SMAs) display several unique properties associated with a thermo-elastic martensitic transformation between a high temperature austenite phase (B2 ordered structure) and a lower temperature martensite phase (B19' monoclinic structure). Of these properties, the most commonly used are the shape memory (thermally-induced) and superelastic (stress-induced) behaviors. Both transformations result in large strain recoveries or large stress generation depending on the mode of operation. These unique properties have the potential to benefit a large number of applications including aerospace (Hartl et al., 2010), medical (Ferčec et al., 2014), civil engineering (Abdulridha et al., 2013; Gajan and Saravanathiiban, 2011; Rahman Bhuiyan and Alam, 2013), and industrial technologies (Yamauchi et al., 2011). Nevertheless, when such applications require repeated actuation, SMAs have not been the primary materials of choice due to instabilities during thermal/mechanical cycling. As a result, widespread utilization of NiTi SMAs in cyclic actuation applications (particularly when employing the shape memory effect) has not occurred.

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Cyclic instabilities manifest as changes in the thermomechanical response of the SMA, namely the accumulation of irrecoverable strains, and changes in transformation temperatures, transformation strains and thermal hysteresis. To date, there are numerous studies that report on the effects of cycling on NiTi functional properties, but a very limited number of these studies have directly examined the microstructural mechanisms that govern these effects. Early work on cycling of NiTi alloys (Melton and Mercier, 1979; Miyazaki et al., 1986; Van Humbeeck, 1991; Wasilewski, 1971) and recent work on thermal cycling (Baruj et al., 1999; Chiang et al., 2008; Filip and Mazanec, 1994; He et al., 2006; Jones and Dye, 2011; Jordan et al., 1994; Kockar et al., 2008; Li et al., 2008, 2009; Liu et al., 2006, 2008; Manchiraju et al., 2011; Matsumoto, 1991, 2003; McCormick and Liu, 1994; Morin and Trivero, 1995; Motemani et al., 2011; Pattabi et al., 2007; Paula et al., 2004; Pelosin and Riviere, 1998; Pelton et al., 2012; Saikrishna et al., 2006; Tadaki et al., 1987; Uchil et al., 2001, 2002; Urbina et al., 2009; Wagner et al., 2010) and mechanical cycling (De la Flor et al., 2009; Delville et al., 2011; Gall and Maier, 2002; Nemat-Nasser and Guo, 2006; Sakamoto, 1983; Strnadel et al., 1995a,b; Tabanli et al., 1999; Tang and Sandstrom, 1993; Wagner et al., 2008; Wang et al., 2010) of SMAs have attributed the evolution of the macroscopic response to defect generation (dislocation and ordering effects), formation of intermediate phases (mostly the R-phase), precipitation, and martensite/austenite texture evolution. From a modeling perspective, there have also been a number of studies that have attempted to capture the effects of transformation cycling and the resulting evolutionary response (Chemisky et al., 2014; Hallai and Kyriakides, 2013; Kan and Kang, 2010; Morin et al., 2011; Saleeb and Kumar, 2009; Saleeb et al., 2013a,b,c,d, 2011; Suresh et al., 2013; Yu et al., 2013; Zhang et al., 2014). Despite all of these studies and others not reported here, the microscopic mechanisms responsible for the instabilities in NiTi SMAs are still not fully known. Until the underlying mechanisms are better understood, the means to properly stabilize, train, and ultimately employ NiTi actuators will be limited.

Therefore, the objective of this work was to examine the microstructural evolution of NiTi during thermomechanical cycling using combined *ex situ* macroscopic experiments and *in situ* neutron diffraction measurements. The high penetration of neutrons, compared to low energy X-rays, provides measurements from representative bulk polycrystalline behavior that is typically free of surface effects. Information from grains and variants of a specific orientation relative to the loading direction are obtained and analyzed. The goal of this investigation was twofold. The first was to study the role of pre-deformation in the austenite phase compared to pre-deformation in the martensite phase on the subsequent thermomechanical cyclic response of NiTi. Second was to follow the microstructural evolution including texture, internal strains, and phase fraction during pre-deformation and thermomechanical cycling, since the macroscopic behavior of NiTi is related to the underlying changes associated with the martensite and austenite microstructures. Moreover, this work is also aimed towards providing experimental insights into the cyclic behavior of NiTi for SMA models verifications. Data obtained regarding the cyclic transformation behavior of NiTi (i.e., transformation strains, residual strains, etc.) can be used to calibrate and validate constitutive SMA models that account for the evolutionary nature of these materials (e.g., Saleeb et al. (2013d)). Similarly, the microscopic data (i.e., texture, phase fractions, etc.) is useful to the formulation of micromechanical models that consider such constraints (e.g., Manchiraju et al. (2011)).

2. Material and experimental procedures

2.1. Material

The material investigated in this work was a binary $Ni_{49.9}Ti_{50.1}$ (at.%) alloy produced by Special Metals, New Hartford, New York (now SAES Smart Materials). The alloy was received in 10 mm diameter rods in the hot-rolled/hot-drawn and hot-straightened condition. Mechanical test samples were machined from the extruded rods into cylindrical, dog-bone tensile specimens with a 5.08 mm diameter and 15.24 mm long gauge section with threaded button ends. Thermomechanical and other properties of this alloy are available in the literature (Atli et al., 2013b; Benafan, 2012; Benafan et al., 2013a,b,c, 2012; Manchiraju et al., 2011; Padula II et al., 2012, 2008; Qiu et al., 2011, 2009; Raj and Noebe, 2013a,b; Stebner et al., 2013a,b). To relieve any residual stresses from the machining operation, all samples were subjected to two no-load thermal cycles (on the load frame) between room temperature and 230 °C. Stress-free transformation temperatures, martensite start (M_s), martensite finish (M_f), austenite start (A_s), and austenite finish (A_f), were measured from the second mechanical, no-load thermal cycle (from the strain–temperature response) using the intercept method (Benafan, 2012) and were found to be 71, 55, 92, and 105 ± 2 °C, respectively.

2.2. Thermomechanical testing

Thermomechanical *ex situ* experiments were performed on an 810 MTS servohydraulic load frame. Prior to thermal cycling, individual samples were deformed isothermally in uniaxial tension to strains between 0% and 18% in strain control at a rate of $1 \times 10^{-4}\ s^{-1}$ (using a high-temperature extensometer), and then unloaded to 0 MPa (Table 1). Table 1 illustrates the experimental scheme and test sequence of each specimen in order from the left column to the right column. The last two columns labeled "*Ex situ*" and "*In situ*" indicate which conditions were tested *ex situ* macroscopically only, and which conditions were examined both macroscopically and repeated using *in situ* neutron diffraction tests (under the same test conditions). Isothermal deformations were performed in both the austenite phase at 320 °C (samples 1 through 4) and in the martensite phase at room temperature (samples 5 through 10). A temperature of 320 °C was selected for deformation of