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Effects of crystallographic orientation on the superelastic response of FeMnAlNi single crystals

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ABSTRACT

The effects of crystal orientation on the tensile superelastic response of the Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} single crystalline shape memory alloy, oriented along the <100> and <123> directions were investigated. The single crystals aged at 200 °C for 3 h demonstrated superelastic strains of 3.6% along the <100> orientation and 7.8% along the <123> orientation. Although the theoretical transformation strain in the <100> orientation is higher than that in the <123> orientation, the <123> oriented single crystals demonstrate better superelastic recovery in tension due to a higher number of active martensite variants, which promote better accommodation of internal strains and help minimize defect formation during transformation.

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NiTi based shape memory alloys (SMAs) are widely used in industrial and medical applications. However, NiTi is expensive and difficult to cold work, therefore many attempts have been made to produce iron-based SMAs such as Fe–Ni and Fe–Mn based ones with lower material cost and better cold workability. However, superelastic recovery in FeMnSi, FeNiCoTi and FeMnAl SMAs [1–3] was historically poor. Recently, Tanaka et al. [4] and Omori et al. [5] discovered novel nano-precipitation hardened Fe_{40.95}Ni₂₈Co₁₇Al_{11.5}Ta_{2.5}B_{0.05} (at.%) and Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} (at.%) SMAs, respectively, with impressive superelastic recovery levels. The former showed 13.5% superelastic strain at room temperature through an fcc (austenite) to bct (martensite) martensitic transformation. The latter showed 5% superelastic strain through a bcc (austenite) to an fcc (martensite) martensitic transformation. Following these studies, several additional iron-based SMAs such as single crystal forms of Fe₄₁Ni₂₈Co₁₇Al_{11.5}Ta_{2.5} [6–9], Fe₄₁Ni₂₈Co₁₇Al_{11.5}Nb_{2.5} [10], Fe₄₁Ni₂₈Co₁₇Al_{11.5}Ti_{2.5} [11], Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} [12,13], and polycrystalline Fe_{40.95}Ni₂₈Co₁₇Al_{11.5}Ti_{2.5}B_{0.05} [14], Fe_{40.95}Ni₂₈Co₁₇Al_{11.5}Nb_{2.5}B_{0.05} [15], and Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} [16] were also reported to show 4–6% recoverable strain. Of particular interest is the Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} alloy, which shows a small temperature dependence of the superelastic stress for stress-induced martensitic transformation in a wide temperature range from –196 to 240 °C [5]. In this material, highly reversible superelasticity

is facilitated by the formation of precipitates around 10 nm that are coherent with both the austenite and martensite phases [17].

Crystal orientation, and thus crystallographic texture, is known to be one of the important factors that influence the superelastic response of the SMAs. Studies on NiTi single crystals by Gall et al. [18,19] showed that the superelastic response of NiTi SMAs is strongly dependent on the crystallographic orientation: under tension, the <100> orientation demonstrates smaller transformation strain and higher critical stress than those in the <111> orientation, and an opposite response is observed under compression [18,19]. In FeNiCoTi SMAs, Sehitoglu et al. [2] reported that the <123> oriented single crystal exhibits higher recoverable strain than that in the <100> orientation under both tension and compression. In our previous study, Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} (at.%) single crystals oriented along the <100> direction showed poor superelastic recovery in tension due to the large volume fraction of retained martensite pinned by dislocations both within the austenite matrix and at the austenite–martensite interfaces [12]. On the other hand, much better recovery was observed under compression. We demonstrated that the discrepancy was caused by a difference in martensite variant selection under tension and compression. Whereas only one martensite variant can accommodate tensile deformation along the <100> direction, two martensite variants can accommodate deformation in compression [12]. Since the martensite variants available for the accommodation of strain depend strongly on the direction of loading, it is important to characterize the FeMnAlNi single crystals in different crystallographic orientations to understand the orientation dependence of the superelastic response. This understanding will be

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beneficial for the prediction of the superelastic behavior in textured polycrystals. In this work, we study the $\text{Fe}_{43.5}\text{Mn}_{34}\text{Al}_{15}\text{Ni}_{7.5}$ (at.%) single crystals aged at 200 °C for 3 h along the $\langle 100 \rangle$ and $\langle 123 \rangle$ orientations to investigate the orientation dependence of superelastic response. The particular aging heat treatment was selected to obtain less than 10 nm nano-precipitates in the microstructure.

Single crystal samples with a nominal composition of $\text{Fe}_{43.5}\text{Mn}_{34}\text{Al}_{15}\text{Ni}_{7.5}$ (at.%) were grown using the Bridgman technique in a helium atmosphere. Flat dog-bone-shaped tensile samples with the gauge dimensions of 1.5 mm \times 3 mm \times 8 mm were cut using wire electro-discharge machining (EDM) from the single crystals with the tension axis parallel to the $\langle 100 \rangle$ or $\langle 123 \rangle$ orientations. The tension samples were sealed into quartz tubes under high purity argon atmosphere and solution heat treated at 1200 °C for 1 h followed by water quenching. Subsequently, the crystals were aged at 200 °C for 3 h under ultra-high purity argon atmosphere to introduce coherent B2 (bcc/NiAl) precipitates into the austenite matrix. Transmission electron microscopy (TEM) samples were prepared by twin-jet electro-polishing with a solution of 30% volume nitric acid and 70% volume methanol mixture at -20 °C. TEM investigations were conducted using a FEI Tecnai G² F20 electron microscope.

The superelastic response of the aged crystals was characterized via the incremental tensile testing at room temperature. Before performing the mechanical testing, the samples were mechanically polished. In this test, 2% tensile strain is first applied on the sample followed by complete unloading. Thereafter, the applied strain level was increased during each subsequent loading–unloading cycle until the sample fractured. These experiments were performed with a servo-hydraulic MTS test frame at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. An extensometer was directly attached to the gauge section of the samples to measure the axial strain.

The calculated transformation strains as function of crystal orientation in tension are displayed in Fig. 1. The transformation strains were calculated using energy minimization theory (for CVP strains, Fig. 1a) [20] and lattice deformation theory (for detwinning strains, Fig. 1b) [21]. The energy minimization approach assumes that the single crystal austenite transforms into martensite forming twinned martensite [20], while the lattice deformation approach assumes that the single crystal austenite transforms to single crystal martensite without internal twinning [21]. More detailed description of these theoretical frameworks can be found in [22–26]. Taking lattice parameters of austenite and martensite as 0.2903 nm and 0.3672 nm, respectively from [5], the calculated transformation strains with detwinning are found to be 26.5% and 14%, and the calculated CVP strains are 10.5% and 9% for the $\langle 100 \rangle$ and $\langle 123 \rangle$ orientations, respectively.

Fig. 2a and b show the tensile stress–strain responses of the $\text{Fe}_{43.5}\text{Mn}_{34}\text{Al}_{15}\text{Ni}_{7.5}$ single crystals with the $\langle 100 \rangle$ and $\langle 123 \rangle$ orientations. The sample with the $\langle 100 \rangle$ orientation shows partial recovery until a stress drop at about 6% applied strain, after which recovery is no longer observed. The result suggests a large amount of retained martensite [12]. On the other hand, the sample with the $\langle 123 \rangle$ orientation is almost fully reversible up to 8% applied strain. The sample was fractured at 9% applied strain. The relationship between the recoverable and applied strains in both $\langle 100 \rangle$ and $\langle 123 \rangle$ orientations is summarized in Fig. 2c. From these results, the recoverable strain in the $\langle 123 \rangle$ orientation is nearly perfect up to 8% of applied strain and the maximum recoverable strain is approximately 7.8%, comparable to the calculated 9% expected for the CVP strain. However, the maximum recoverable strain in the $\langle 100 \rangle$ orientation is only 3.5%. The experimental value is far smaller than the theoretical transformation strain in this orientation, which is 10.5% for CVP and 26.5% for the fully detwinned condition.

Although the $\langle 100 \rangle$ orientation has higher theoretical transformation strain than that in the $\langle 123 \rangle$ orientation under tension, the $\langle 123 \rangle$ orientation shows higher recoverable strain than the former. In order to study the reason behind this difference, TEM investigation is carried out on the tensile samples with the $\langle 100 \rangle$ and $\langle 123 \rangle$ orientations after the tensile tests at room temperature. Because the tension

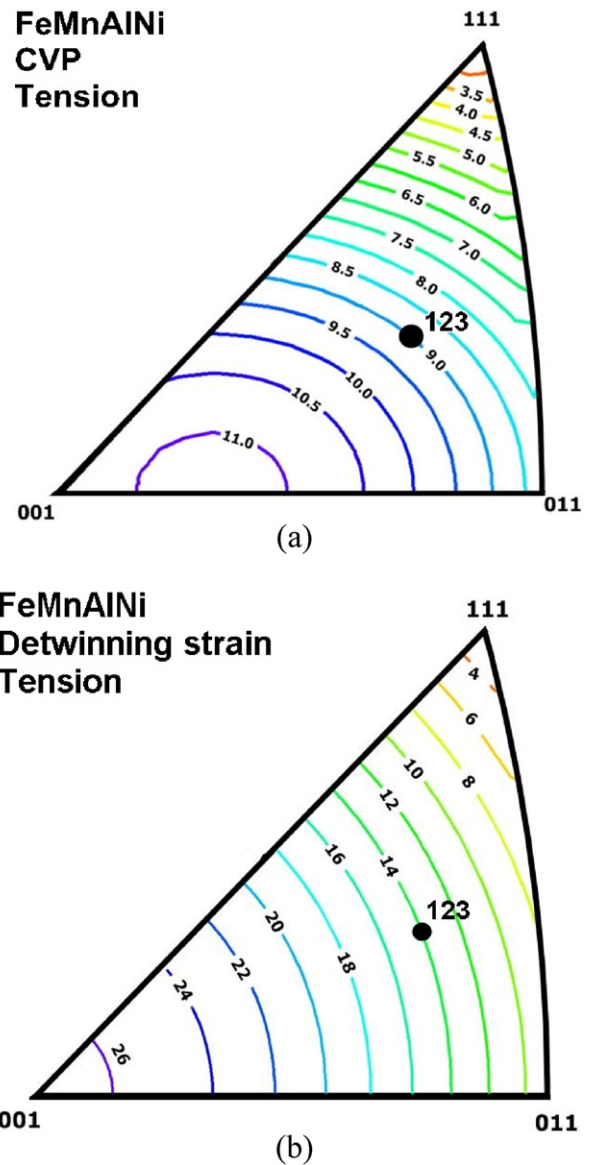


Fig. 1. Calculated orientation dependence of the transformation strains in tension for the $\text{Fe}_{43.5}\text{Mn}_{34}\text{Al}_{15}\text{Ni}_{7.5}$ single crystals assuming: (a) internally twinned martensite and (b) fully detwinned martensite. See text for details on the calculations.

sample with the $\langle 100 \rangle$ orientation after the completion of incremental strain test is almost fully martensitic, it is difficult to locate the austenite–martensite boundary. Therefore, we performed an experiment where a tensile sample was loaded to 8% instead, such that the martensite–austenite boundary could be more easily captured (inset, Fig. 2a). Fig. 3 shows the bright field TEM image of the sample with the $\langle 100 \rangle$ orientation after the 8% strain (inset, Fig. 2a). From the TEM results, high density of parallel dislocations and hairpin-shaped dislocations are generated at the austenite–martensite interface and in the austenite matrix, respectively. This kind of deformation microstructures were previously reported in studies on FeMnAlNi single crystals with the $\langle 100 \rangle$ orientation [12] and on FeNiCoTi polycrystals [27].

Fig. 4a shows the bright field TEM image of the solution-treated single crystals with the $\langle 123 \rangle$ orientation after 4% strain at room temperature (inset, Fig. 2b). A solution-treated sample was tested because aged samples show excellent superelastic recovery, making it difficult to obtain an austenite–martensite two-phase structure that makes it possible to study both the martensite structure and the austenite–martensite interface. The selection of 4% applied strain is based on the superelastic

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