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Effect of grain size on the superelastic response of a FeMnAlNi polycrystalline shape memory alloy

L.W. Tseng [a], Ji Ma [b], M. Vollmer [c], P. Krooß [c], T. Niendorf [c], I. Karaman [a,b,*]

[a] Department of Mechanical Engineering, Texas A&M University, MS 3123, College Station, TX 77843, USA
[b] Department of Materials Science and Engineering, Texas A&M University, College Station, TX 77843, USA
[c] Institut für Werkstofftechnik (Materials Engineering), Universität Kassel, 34125 Kassel, Germany

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ABSTRACT

The effect of heat treatments on superelasticity was investigated in a FeMnAlNi polycrystalline alloy using incremental tensile strain tests at room temperature. Large grains of more than several millimeters can be obtained by a cyclic heat treatment process and the average grain size can be controlled by the number of heat treatment cycles. Improved superelastic response is observed in samples with large relative grain sizes, exhibiting increased elongation to fracture, lower critical stress for transformation, and higher reversibility.

Common superelastic materials recover large applied strains after unloading due to reversible stress-induced martensitic phase transformation. NiTi-based shape memory alloys (SMAs) are commonly used in industrial applications because of high superelastic strains up to 8%; however, high material costs and poor cold-workability have limited their wide-spread use in industrial applications. By comparison, Fe-based SMAs attracted much attention over the last few decades due to low material cost and outstanding cold-workability. However, the superelastic properties of most Fe-based SMAs, in terms of the level of transformation strains and shape recovery are poor [1–6].

In 2010, Tanaka et al. [7] reported that addition of Ta and B into the FeNiCoAl alloy system changes the martensitic transformation from non-thermoelastic to thermoelastic resulting in excellent superelasticity up to 13.5% strain in a $Fe_{40.95}Ni_{28}Co_{17}Al_{11.5}Ta_{2.5}B_{0.05}$ SMA. Since then several similar alloy systems such as $Fe_{40.95}Ni_{28}Co_{17}Al_{11.5}Ti_{2.5}B_{0.05}$ [8], $Fe_{40.95}Ni_{28}Co_{17}Al_{11.5}Nb_{2.5}B_{0.05}$ [9], $Fe_{41}Ni_{28}Co_{17}Al_{11.5}Ta_{2.5}$ [10–13], $Fe_{41}Ni_{28}Co_{17}Al_{11.5}Ti_{2.5}$ [14,15] and $Fe_{41}Ni_{28}Co_{17}Al_{11.5}Nb_{2.5}$ [16] have been reported showing up to 5–6% superelastic strain at room temperature.

In 2011, Omori et al. [17] reported thermoelastic martensitic transformation in FeMnAl alloys after the addition of Ni and formation of high volume fraction of very small (3–5 nm) nano-precipitates. $Fe_{43.5}Mn_{34}Al_{15}Ni_{7.5}$ alloy demonstrated up to 5% superelastic strain in both single crystalline and polycrystalline conditions [18–23]. Probably the most interesting characteristic of this material is the small temperature dependence of the critical stress for stress-induced martensitic transformation [17]. This means that the required stress to trigger superelasticity does not change appreciably with temperature, and a large superelastic temperature window from −196 °C to 240 °C is possible.

In NiTi alloys, the incompatibility between grains upon martensitic transformation is easily accommodated by the formation of corresponding variant pairs (CVP) involving two or four variants during deformation. This is due to twenty-four martensite variants which can theoretically be activated during B2 to B19′ martensitic transformation [24]. Therefore, large grain size is not required to obtain superelastic behavior in NiTi and similar SMAs [24]. Unlike NiTi alloys, only three martensite variants can be activated in FCC to BCT transformation of Fe-based SMAs [10,11,19,23], therefore the grain boundary incompatibility cannot be easily accommodated as the Taylor criterion for generalized deformation is not satisfied. Related to this, the superelastic properties strongly depend on the average grain size (d) relative to the thickness or width (t), or diameter (D) of the test sample. For example, FeNiCoAlTaB polycrystals with large mean relative grain size (d/t = 400 μm/220 μm) about 1.8, were reported to show over 13% recoverable strain [7]. In FeMnAlNi polycrystalline sheets and wires, 5% recoverable strain was obtained with relative grain size about 15 in sheets [17] and 2.19 in wires [18]. Although the d/t ratio of FeMnAlNi polycrystals is similar to that in a single crystal, the gauge section of the samples is nevertheless made up of multiple grains. The texture intensity in the polycrystal is weak [17] and some grains are oriented near the ⟨100⟩ direction. In the FeMnAlNi single crystal studies, samples along the

* Corresponding author at: Department of Mechanical Engineering, Texas A&M University, MS 3123, College Station, TX 77843, USA.
E-mail address: ikaraman@tamu.edu (I. Karaman).

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⟨100⟩ orientation show poor reversibility in tension [23]. As such, the superelastic response of the bamboo-like polycrystal samples is worse compared to favorably-oriented single crystals such as ⟨110⟩, but better compared to unfavorably-oriented single crystals such as ⟨100⟩. In the present study, the effect of grain size on the characteristics of superelasticity such as critical stress, superelastic strain, elongation and fracture in FeMnAlNi polycrystalline alloys with two different average grain sizes are investigated, and a thermo-mechanical treatment by which grain size can be controlled is proposed.

As-cast $Fe_{43.5}Mn_{34}Al_{15}Ni_{7.5}$ (at.%) ingots were thermo-mechanically processed to obtain polycrystalline sheets following the procedures mentioned below:

(1) hot rolling (HR) at 1200 °C with a reduction ratio of 60%;

(2) annealing heat treatment (AHT) at 900 °C for 1 h in the fcc + bcc two phase region;

(3) cold rolling (CR) at room temperature with a reduction ratio of 75% to a thickness of 1 mm; subsequently, dog-bone shaped tensile samples with the gauge dimension of 1 mm × 3 mm × 8 mm were cut from the cold-rolled sheet;

(4) heat treatment at 1200 °C for 0.5 h followed by air cooling (AC) to room temperature; this process was repeated several times in order to induce abnormal grain growth;

(5) solution heat treatment (SHT) at 1200 °C for 1 h followed by water quench (WQ);

(6) aging heat treatment (PHT) at 200 °C for 3 h to introduce B2 nano-precipitates followed by water quench.

All heat treatments and air cooling were conducted under high-purity argon atmosphere. Fig. 1 shows the illustration of the thermo-mechanical process used to obtain the FeMnAlNi polycrystalline sheets.

The grain size after thermo-mechanical processing was studied by optical microscopy (OM). The etching solution used is composed of 7% nitric acid and 93% ethanol by volume. The crystallographic texture in the tension samples after the superelastic tests was examined using electron backscatter diffraction (EBSD) method employing a scanning electron microscope operated at 20 kV. EBSD samples were polished using colloidal SiO_2 suspension with 0.02 μm particle size. The superelastic responses were characterized by isothermal incremental strain tests at room temperature in tension. In these tests, the samples were first loaded to 1% strain and unloaded, and strain levels were then increased by 1% during each loading-unloading cycle until failure. The tension tests were conducted with a servo-hydraulic MTS test frame at a strain rate of $5 \times 10^{-4}\ s^{-1}$. The extensometer was directly attached onto the gauge section of the tensile samples.

The microstructures of the $Fe_{43.5}Mn_{34}Al_{15}Ni_{7.5}$ polycrystalline alloys following two times air cooling (2AC) and five times air cooling (5AC) down from 1200 °C are shown in Fig. 2a and b. The OM images were taken at the gauge section of the tensile samples. It can be seen that grain sizes grow continuously with increasing number of air cooling cycles applied to the samples, and very large grains are obtained after 5AC. It should be noted that the fcc second phase forms along the grain boundaries or inside the austenite bcc grains after the air cooling process. The same observation is also reported by Vollmer et al. [21]. Furthermore, it is well known that the fcc second phase is required to introduce abnormal grain growth (AGG) [21,25]. However, due to the fact that the fcc second phase does not transform with the matrix, it can reduce the maximum transformation strain during martensitic transformation, and thus, the second phase regions are not desirable. Furthermore, since the fcc second phase is soft and ductile, it can negatively affect the reversibility of the martensitic transformation in the bcc matrix. Thus, in order to remove the second phase regions, the samples were solution heat treated at 1200 °C for 1 h (SHT) followed by water quenching (WQ) after the repeated air cooling processes. The average grain size in the two investigated samples is 2 mm (d/t ~ 0.67; t: the width) for 2AC + SHT and 5 mm (d/t ~ 1.67) for 5AC + SHT samples, as shown in Fig. 2c and d, respectively.

Fig. 3a and b present the tensile stress-strain curves of these samples. The recoverable strain (ε_{rec}) as a function of applied strain ($\varepsilon_{applied}$) from these tests is shown in Fig. 3c. Table 1 summarizes the grain size (d) to sample width (t) ratio, the critical stress for the onset of martensitic transformation (σ_c), fracture stress (σ_f), maximum recoverable strain (ε_{max}) and elongation to failure (ε_{el}) of both tensile samples (2AC + SHT + PHT and 5AC + SHT + PHT) after deformation. σ_c is high (around 740 MPa) in the tension sample 2AC + SHT + PHT with the d/t ratio around 0.67 and maximum ε_{max} (1.5%) is small. On the other hand, the 5AC + SHT + PHT sample with the d/t ratio about 1.67 exhibits a lower σ_c of 420 MPa, a larger strain to failure (9% vs. 4%), and higher ε_{max} of about 3%.

At least two mechanisms are responsible for these differences. First, the different critical stress levels in the 2AC and 5AC samples are related to the activation of differently oriented grains during superelastic transformation. Second, the small grain size leads to greater resistance to the superelastic response with less reversibility due to more significant grain boundary constraints. The smaller average grain size is reflected in the higher σ_c and the lower recoverable strain at a given applied strain level in the 2AC sample. When the grain size becomes large, the grain constraints decrease and each grain can transform almost independently to the martensite [17,18]. That means, the elevated grain constraints in the

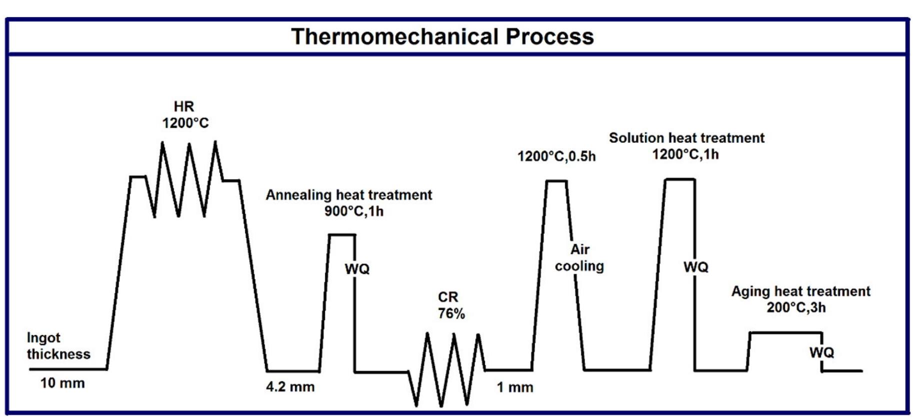

Fig. 1. Schematic illustration of the thermo-mechanical processes used in this study to obtain FeMnAlNi SMA polycrystalline sheets. HR, WQ and CR indicate hot rolling, water quenching and cold rolling, respectively. The solution treatment at 1200 °C for 0.5 h was repeated for inducing abnormal grain growth.