

Regular article

Effects of cyclic heat treatment and aging on superelasticity in oligocrystalline Fe-Mn-Al-Ni shape memory alloy wires

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ABSTRACT

The effects of nano-precipitation on stress-induced martensitic transformation (SIM) in Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} shape memory alloy wires were investigated. It was shown that a large grain size to wire diameter ratio, i.e. an oligocrystalline structure, is necessary to obtain superelasticity. The critical stress for SIM and tensile strength at room temperature increase with aging time at 200 °C without loss of superelasticity. A superelastic strain of 6.7%, with tunable transformation stress level as high as 600 MPa, and a low stress hysteresis, was obtained in the aged wires.

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Shape memory alloys (SMAs) are unique class of materials that display large reversible shape changes upon mechanical loading/unloading, i.e. superelasticity, enabled by a stress-induced martensitic transformation. NiTi alloys are the most well-known SMAs, which can exhibit up to 10% superelastic strain. They demonstrate reasonable ductility and relatively low elastic anisotropy [1,2]. However, they are difficult to cold work due to their ordered intermetallic structure [3]. In addition, they are highly composition-sensitive on the nickel-rich side of the stoichiometry, where 0.1 at% change in composition leads to a 20 °C change in transformation temperatures [4]. Besides the high cost of initial materials, difficulty of processing and the need to precisely control the chemistry further increases the cost and hinders the widespread use of NiTi.

Disordered Fe-based SMAs are alternatives to NiTi due to their inexpensive constituents and ease of processing. Since 1970s, many attempts have been made to develop Fe-based SMAs. However, only a few were successful in achieving superelasticity at room temperature. Maki et al. [5] were able to show reversible martensitic transformation by ausaging an FeNiCoTi alloy to form fine precipitates, however, they

observed superelasticity only at –100 °C. A major breakthrough came in 2010, in the form of a polycrystalline FeNiCoAlTaB alloy [6]. This material exhibits room temperature superelasticity of up to 13.5% strain, but requires a strong [100] texture, coherent nano-precipitates, and large grain size to achieve superelasticity. In 2011, a new FeMnAlNi alloy was developed [7] that exhibits 5% room temperature superelasticity, but its performance is also dependent on nano-precipitates and large grain size. In single crystalline FeMnAlNi alloys, the effects of aging on the superelastic properties were studied by Tseng et al. [8]. They found that superelasticity, recoverable strain level, and stress hysteresis are controlled by the size of the precipitates. A unique feature of this alloy is a small temperature dependence of the critical stress for stress-induced martensitic transformation (σ_{SIM}) of 0.41 MPa/°C, which is almost 15 times smaller than that of NiTi [9]. This characteristic is crucial for structural and civil infrastructure applications that require a stable superelastic response over a broad temperature range.

Fe-based SMAs, on the other hand, usually suffer from large elastic and transformation strain anisotropy similar to Cu-based SMAs [10–13]. This leads to high internal stresses at grain boundaries between grains with large orientation mismatches upon martensitic transformation [11,14]. Additionally, most of the Fe-based SMAs have a limited number of martensite variants [15]. Therefore, strain accommodation at grain boundaries during the transformation becomes more difficult

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and, together with high stresses, causes intergranular fracture and/or a suppression of superelastic behavior [11,14,16].

Thus, to obtain good superelasticity, it is necessary to decrease the total grain boundary area and triple junctions, and limit the effect of grain boundary constraint during the transformation [7,17–19]. Ueland et al. [17,18,20] and Chen et al. [21] produced bamboo structured microwires and obtained 7% superelasticity in Cu–Al–Ni microwires. Similarly, in Fe-based SMAs, it has been shown that grain size to wire diameter (d/D) [19] or grain size to sheet thickness ratios larger than one [7] is a necessary condition to obtain superelasticity.

Techniques to obtain large grain size through abnormal grain growth in FeMnAlNi SMAs involve cyclic heat treatments where samples are repeatedly cycled between the single phase and two phase regions in the phase diagram. Omori et al. [19] annealed FeMnAlNi wires and sheets at 1200 °C followed by air cooling, and repeated this process until they obtained large grain size. Vollmer et al. [16] have applied similar cyclic heat treatments, cycled between 1200 °C and 900 °C and obtained coarse grains in the FeMnAlNi flat, thin tension specimens.

A few studies on FeMnAlNi polycrystals have recently focused on suppressing intergranular fracture [16,19] and investigating the effect of grain size on superelastic behavior. Additionally, it has been shown that various aging treatments on FeMnAlNi single crystals improve the mechanical response and superelastic behavior [9]. However, the latter has not been studied in polycrystalline/oligocrystalline FeMnAlNi SMAs. In the present work, we investigate the effect of post-cyclic heat treatment and nano-precipitation on the superelastic behavior and mechanical properties of FeMnAlNi polycrystalline wires.

Ingots with a nominal composition of Fe–34%Mn–15%Al–7.5%Ni (at%) were prepared by vacuum induction melting. To enable wire drawing of the FeMnAlNi at room temperature it is necessary to create a two-phase microstructure consisting of a bcc alloy matrix containing ductile fcc second phase. To generate this microstructure, the ingots were first hot extruded at 1000 °C with an area reduction ratio of 7:1. The resulting rods were then solution treated at 1200 °C and annealed at 900 °C for 30 min under high purity argon followed by a water quench (Fig. 1a). The 900 °C treatment was performed to introduce the high volume fraction of ductile second phase particles necessary for wire drawing. The heat treated rod was then cold-drawn into wire with a final diameter of 0.5 mm, following an area reduction of 75%. Final chemical composition of the wires was determined using Wavelength Dispersive Spectroscopy (WDS) and compositional variations were found to be less than 0.2 at% from the nominal.

To promote abnormal grain growth, the wires were encapsulated in quartz tubes, evacuated, and filled with high-purity argon and then subjected to cyclic heat treatments. The encapsulated wires were cycled five times between the single phase region (1200 °C) and room temperature, by placing the encapsulated wires in a pre-heated furnace for 0.5 h and removing them from the furnace and cooling to room temperature in air. After these steps, the encapsulated wires were solution heat treated (SHT) at 1300 °C for 30 min and quenched in water. Finally, nano-sized precipitates were formed with precipitation heat treatments at 200 °C for 1, 3, 5 and 24 h.

Microstructure of the wires was investigated using a Keyence digital microscope. Specimens for optical microscopy were prepared by

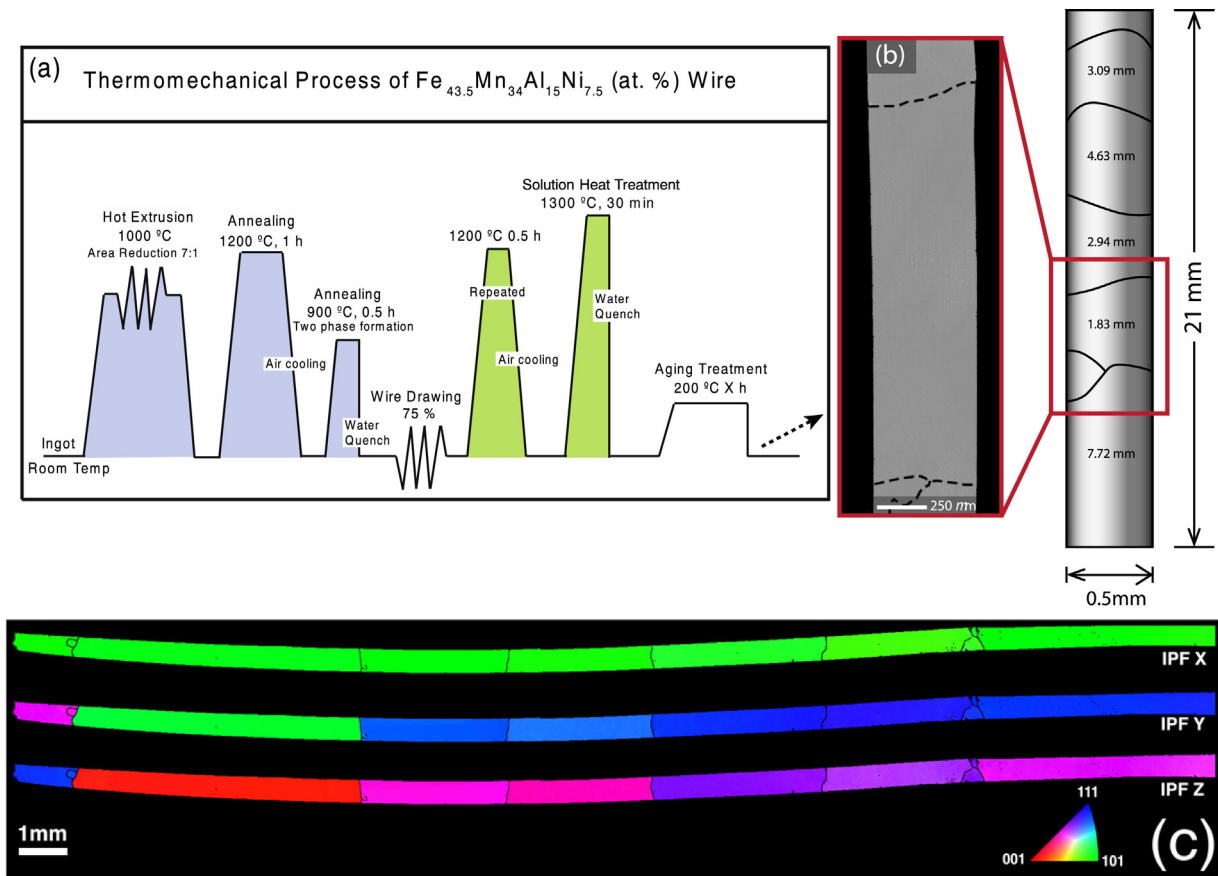


Fig. 1. (a) Thermomechanical processing history of Fe–34%Mn–15%Al–7.5%Ni (at%) superelastic wire. (b) Final grain sizes and distribution in a representative Fe–34%Mn–25%Al–7.5%Ni (at%) superelastic wire after cyclic grain growth treatment and final solution heat treatment at 1300 °C for 30 min (as defined in (a)). (c) EBSD orientation mapping of a Fe_{43.5}Mn₃₄Al₁₅Ni_{7.5} (at%) wire aged at 200 °C for 24 h after tensile superelastic test (the superelastic response is shown in Fig. 3c). The colors represent the crystal directions parallel to the drawing direction (IPFX), the transverse direction (IPFY) and the normal direction (IPFZ), given in the stereographic triangle. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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