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Cyclic degradation in bamboo-like Fe–Mn–Al–Ni shape memory alloys — The role of grain orientation

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ABSTRACT

In the present study the cyclic deformation behavior within differently oriented grains in Fe–34.8Mn–13.5Al–7.4Ni (at.%) shape memory polycrystals featuring a bamboo-like structure was investigated. In cyclic tensile tests up to 50 cycles, the degree of degradation in pseudoelasticity was evaluated and contributing elementary mechanisms are discussed. The results reveal rapid cyclic degradation in the bamboo-like samples. The unexpected stabilization of parent phase in reverse transformed areas and the proceeding activation of new martensite variants in subsequent cycles were found to be the prevailing degradation mechanisms. Dislocation activity is found to be the most detrimental factor.

Since commercially available shape memory alloys (SMAs) like Ni–Ti suffer from high cost due to difficult workability, new alloys were introduced in last decade. Recently developed Fe–Ni–Co–Al–X (X = Nb, Ta, Ti) [1–4] and Fe–Mn–Al–Ni [5] SMAs are attractive due to combination of low cost processing and high transformation strains. In order to obtain a thermoelastic martensitic transformation in these iron-based SMAs the presence of finely dispersed coherent precipitates is crucial [1,6]. In case of Fe–Mn–Al–Ni Omori et al. added Ni to Fe–Mn–Al, which resulted in the formation of coherent β precipitates (B2) in the disordered α matrix (A2-bcc) by a low temperature heat treatment [5,7]. Thus, a thermoelastic transformation between an α austenitic high-temperature parent phase (A2-bcc) and a γ' martensitic product phase (A1/2 M-fcc) takes place.

Recent studies revealed that a high reversibility can be obtained in numerous SMAs like Cu-based SMAs, Fe-based SMAs or Co–Ni–Ga, when grains of large dimensions with respect to the cross section of the specimen are established, refered to bamboo structures [5,8–16]. For Cu-based SMAs, Ueland et al. [9–11] demonstrated that such bamboo structures minimize grain constraints, due to the almost exclusive formation of boundaries perpendicular to the loading direction. Based on the observations of abnormal grain growth (AGG) in a Cu–Al–Mn SMA [12] as well as grain coarsening in Fe–Mn–Al–Ni [5,8], a cyclic heat treatment was applied to Fe–Mn–Al–Ni SMA also resulting in an abnormal growth of favored grains [17]. In that study, Vollmer et al. [17] also reported that grain boundary cracking due to fast quenching from the solution heat treatment temperature can be suppressed by inducing a thin film of ductile γ phase at the grain boundaries.

For intended industrial applications, a high cyclic stability is indispensable. However, data reporting on the functional degradation in Fe–Mn–Al–Ni are still lacking in open literature. Some of the current authors already investigated elementary cyclic degradation mechanisms in Fe–Ni–Co–Al–Ta for ⟨001⟩ oriented single crystals in tension [18]. They found a high density of residual martensite upon fatigue [18]. This was attributed mainly to two different mechanisms: (i) self-triggered pinning induced by martensite variant–variant interactions resulting in an increased dislocation activity at the boundaries between the parent phase and the martensite and (ii) martensite–precipitate interaction resulting in strongly localized pinning of martensite variants.

Due to the highly anisotropic nature of SMAs especially in Fe–Mn–Al–Ni alloys [5,8,19], the characterization of functional properties in different crystallographic directions is essential. Therefore, in this study two different bamboo-like Fe–Mn–Al–Ni samples were characterized by using *in situ* scanning electron microscopy (SEM) in order to shed light on the initial martensite formation. *In situ* pseudoelastic cycling

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experiments up to 50 cycles in tension were conducted in order to correlate functional degradation and proceeding microstructure evolution. Post-mortem transmission electron microscopy (TEM) analyses were carried out to identify the prevailing degradation mechanisms in each crystallographic orientation.

From polycrystalline Fe–Mn–Al–Ni with a chemical composition of Fe–34.8%Mn–13.5%Al–7.4%Ni (at.%) dog-bone shaped samples with a gauge length of 12 mm and a cross section of 1.6 mm × 1.5 mm were electrodischarge machined, ground to 5 μm grit size and sealed into quartz tubes under argon atmosphere. Cyclic heat treatments between the single phase region (1200 °C) and the ($\alpha + \gamma$) two-phase region (900 °C) were conducted for AGG followed by quenching in tempered water to obtain a bamboo-like structure. Samples were aged at 200 °C for 3 h in air in order to introduce nano-sized β precipitates. Further details on heat treatment, sample preparation and testing can be found in [17]. For *in situ* testing, the samples were vibration-polished. A miniature load frame was used in combination with a scanning electron microscope (SEM) operated at 20 kV and an electron-backscatter diffraction (EBSD) system in order to characterize the microstructural evolution during the first two cycles *in situ* in high resolution. The tests were done under constant crosshead displacement at a rate of 5 μm s^{-1} up to 2.5% nominal strain (300 μm) in the first cycle and to 3.2% nominal strain (380 μm) in the second cycle. In this regard, nominal strains were calculated from displacement data. The functional fatigue tests were carried out using an MTS servo-hydraulic testing system employing the same settings (5 μm s^{-1}; 3.2% nominal strain) in displacement control. In these tests strains were measured using an extensometer directly attached to the tension grips. Surface images were taken in cycles 10, 25 and 50 at the maximum strain and upon unloading using a Keyence digital microscope equipped with a long working distance objective. All tests were done at room temperature. A high resolution transmission electron microscope (HRTEM) operating at 200 kV was employed to study the microstructural evolution in thin foils prepared from the gauge section of the samples by focused ion beam (FIB) preparation.

It is well known that pseudoelastic performance in iron-based SMAs is strongly related to its anisotropic behavior [18–22]. In account of this, two bamboo-like samples with selected grain orientations, i.e.

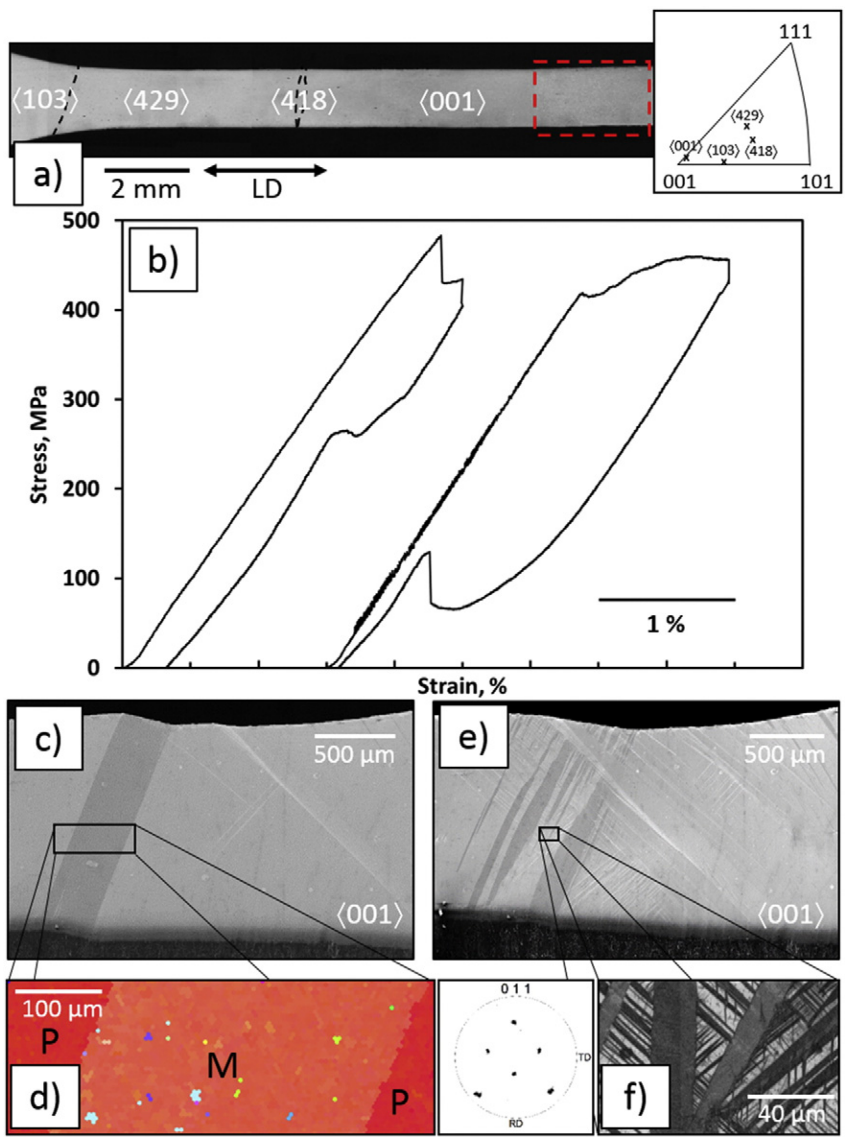

Fig. 1. *In situ* characterization of the bamboo-like sample shown in (a) during the two initial cycles. Orientations with respect to the loading direction (LD) are superimposed to the optical micrograph and highlighted in the corresponding Inverse Pole Figure (IPF). (b) Stress–strain curves for the first and the second cycle. SE overview image (c) of the region highlighted in (a) by the dashed red box and EBSD orientation mapping including parent phase (P) and martensite (M) as well as the pole figure for {011} parent phase (d) at maximum elongation (2.5% nominal strain) in the first cycle. SE overview image (e) of the same region as before and EBSD image quality (f) at maximum elongation (3.2% nominal strain) in the second cycle. Color coding for the EBSD orientation map is shown in Fig. 2.