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Variant organization and mechanical detwinning of modulated martensite in Ni–Mn–In metamagnetic shape-memory alloys

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ABSTRACT

Based upon microstructural and crystallographic characterizations, the variant organization and mechanical detwinning of 6M modulated martensite in Ni–Mn–In alloys have been studied. In the stress-free circumstance, each martensite colony is composed of four distinct orientation variants that are related to one another in three twin relations. Adjacent variants with type-I twin relation possess straight interfaces, whereas those with type-II or compound twin relation have stepped interfaces at atomic scale. Schmid factor (SF) calculations show that under uniaxial compression condition, the loading orientation zones with high SFs for the type-I and type-II detwinning systems are close to each other, being at about 90° away from those of the compound detwinning system. The detwinning resistances of the different types of twins are considered to be associated with their twinning shears and twin boundary structures. Type-I twin possesses much larger detwinning resistance than that of type-II twin, though they have the same amount of twinning shear (0.2392). Compound twin has the smallest detwinning resistance, which is benefited from its tiny twinning shear (0.0277) and stepped twin interface. Under external loading, martensite variants react locally within colonies through detwinning if the loading orientation is favorable. It is possible to obtain single variant state in some colonies when the loading orientation is located in the common positive SF zone of the three detwinning systems. This study is expected to provide fundamental information on local variant reorientation of 6M Ni–Mn–In martensite during deformation.

1. Introduction

Over the last two decades, Ni–Mn-based magnetic shape-memory alloys have attracted considerable attention due to their large magnetic-field-induced strains [1–14]. Two different mechanisms have been discovered to account for the magnetic shape memory effect (MSME) in these alloys. The first mechanism, as typically observed in Ni–Mn–Ga alloys, involves the rearrangement of martensite variants through interface movement driven by magnetic field [2,15,16]. When the magnetocrystalline anisotropy energies (MAEs) of some favored variants are larger than their interface movement resistances, they will grow at the expense of other unfavored variants and thus generate giant macroscopic strain. However, due to the quick saturation of magnetization, the output magnetostress is restricted to only a few MPa [10,17]. Thus, the applications are limited to the low environment resistant conditions. The second mechanism, as typically found in Ni–Mn–In alloys, refers to the magnetic-field-induced inverse martensitic transformation (MIMT) [4,18]. Due to the giant difference in magnetization between the parent and product phase, the Zeeman energy (ZE) could significantly influence the phase stability and result in a structural change under external magnetic field. As the ZE continuously increases with magnetic field, a large output magnetostress may be achieved [10,15]. Moreover, unlike the MAE, the ZE does not strongly depend on crystal orientation. This character would allow utilizing polycrystalline Ni–Mn–In

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alloys for actuator/sensor applications.

In order to obtain MSME in Ni–Mn–In alloys, an appropriate predeformation at the martensite state is generally required, prior to applying an actuating magnetic field to induce the inverse martensitic transformation and realize the shape recovery. The aim of the predeformation process is to rearrange martensite variants by external loading [19–21]. Due to strong anisotropic mechanical response of martensite, the loading orientation should have significant impacts on the prestrain attainability and hence the resultant MSME triggered by magnetic field. In this context, it is essential to explore the mechanical behaviors of martensite variants during deformation. Apparently, in-depth knowledge on the martensite variant organization is a prerequisite to accomplish this task. It should be mentioned that various modulated martensite phases in Ni–Mn–Ga alloys have been studied under different points of view [22–30]. As for Ni–Mn–In alloys, the microstructural and crystallographic characteristics of modulated martensite have not been properly addressed, due to the lack of precise structure information such as modulation period and atomic coordinates. It is only very recently that the complete crystal structure of the 6M modulated Ni–Mn–In martensite has been determined [31] in the frame of $(3+1)$-dimensional superspace theory [32,33]. This serves as a basis for in-depth microstructural and crystallographic explorations of this family of alloys.

In the present work, we selected one Mn-rich off-stoichiometric $Ni_2Mn_{1.44}In_{0.56}$ polycrystalline alloy with incommensurate 6M modulated martensite at room temperature. The microstructural and crystallographic characteristics of the martensite phase, such as variant configurations, twin types, twinning elements and variant interfaces, were determined with the aids of electron backscatter diffraction (EBSD) and high-resolution transmission electron microscope (HRTEM). Guided by the obtained crystallographic parameters, the detwinning behaviors of martensite variants within chosen colonies were analyzed in detail under uniaxial compressive loading. As a first step, this work is expected to provide some fundamental information on how martensite variants react locally with external force through detwinning process in Ni–Mn–In alloys.

2. Experimental procedures and characterization methods

A polycrystalline bulk alloy with nominal composition of $Ni_2Mn_{1.44}In_{0.56}$ was prepared by arc-melting pure constituent elements Ni (99.97 wt.%), Mn (99.95 wt.%) and In (99.995 wt.%) under Ar atmosphere in a water-cooled copper crucible. The alloy was remelted for four times to ensure composition homogeneity. It was further remelted and injected into a copper mold to obtain a cylindrical rod of 8 mm in diameter and ~50 mm in length. The as-cast rod was annealed in a sealed quartz tube under Ar atmosphere at 1173 K for 24 h, followed by water quenching. Rectangular parallelepiped samples with dimension of $5.5 \times 5.5 \times 7.5$ mm^3 were cut out of the middle portion of the annealed rod for microstructural observations, crystallographic orientation measurements and mechanical compression tests. The sample surfaces were mechanically ground and further electrolytically polished at room temperature with a solution of 20% nitric acid in methanol at 8 V.

The critical temperatures for the forward and backward martensitic transformation of the present alloy were measured by differential scanning calorimetry (DSC, TAQ100) in a temperature range from 183 K to 473 K at a heating and cooling rate of 5 K/min. The magnetic properties were examined by physical property measurement system (PPMS, Quantum Design) under a magnetic field of 1 T. The *ex-situ* uniaxial compression with a maximum strain of 5.44% along the length direction of the sample was carried out using a conventional uniaxial compression machine. The microstructural examinations and crystallographic orientation investigations were performed in a field emission gun scanning electron microscope (SEM, Jeol JSM 6500F) with an EBSD acquisition camera and the Aztec online acquisition software package (Oxford Instruments). During the EBSD measurements, the "beam-control" mode was applied with a step size of 0.125 μm. A threefold layered superstructure model of the monoclinic incommensurate 6M modulated martensite, as illustrated in Fig. 1, was used to index the EBSD Kikuchi patterns. The details of this superstructure were described in Ref. [31]. The interfaces between adjacent martensite variants were examined using a high-resolution transmission electron microscope (HRTEM, JEM 2100F).

In order to assess the crystalline perfectness of given martensite crystals, the orientation deviations from their mean orientations were calculated with individually measured EBSD orientation data, using a method described in Ref. [34]. The mean orientations were expressed in three Euler angles (φ_1, ϕ, φ_2) with respect to the macroscopic sample coordinate frame [35,36], and the mean and maximum orientation deviations were expressed in minimum rotation angles. The orientation relationships between adjacent martensite variants were determined from their mean orientations *via* misorientation calculations [37]. The twinning elements of different types of twins were derived on the basis of the minimum shear criterion and the Bilby-Crocker theory [38,39]. The detailed determination procedure is given in Appendix A. Moreover, the interfaces of twin-related martensite variants were determined by the indirect two-trace method [40]. The results are given in Appendix B.

3. Results and discussion

3.1. Characteristic temperatures of austenite-martensite transformation

The heating and cooling DSC curves for the polycrystalline $Ni_2Mn_{1.44}In_{0.56}$ bulk alloy are displayed in Fig. 2a. The start and finish temperatures of the martensitic and austenitic transformations are 339.7 K (M_s), 330.4 K (M_f), 339.4 K (A_s) and 349.2 K

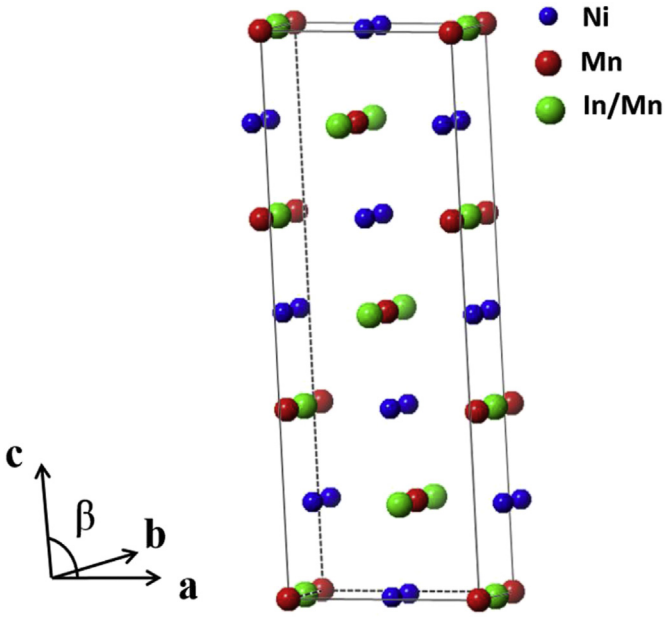

Fig. 1. Illustration of monoclinic 6M modulated superstructure of $Ni_2Mn_{1.44}In_{0.56}$ martensite.