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Change in microstructure during training of a $Ni_{50}Mn_{29}Ga_{21}$ bicrystal

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The change in microstructure during training of a $Ni_{50}Mn_{29}Ga_{21}$ bicrystal was investigated by electron backscatter diffraction in a scanning electron microscope. The martensitic variants form macro and micro twins which are twin related with each other. The twin plane is of type. To achieve magnetic field induced strain in Ni–Mn–Ga alloys, a training process was applied. This process consists of successively compressing the sample along two axes. As a result the twinning stress is reduced and the twinning strain is maximized.

Keywords: NiMnGa; Martensite; Training; Twinning

Large magnetic field induced strain (MFIS) [1–3] due to the high magnetocrystalline anisotropy and easy twin boundary motion in the martensitic state makes Ni–Mn–Ga magnetic shape memory alloys suitable for a large number of technological applications, including actuators or sensors. In a typical martensitic transformation the lattice of the high-temperature austenite phase has a higher crystallographic symmetry than that of the low-temperature martensite phase. Consequently, austenite may transform to several martensitic variants, the number of which depends on the change of symmetry during transformation. In a cubic to tetragonal transformation three variants can form with the so-called *c*-axis oriented close to the three main cubic axes of austenite [4]. Twin boundary motion increases the volume fraction of those variants with the *c*-axis (easy magnetization direction) aligned parallel to the direction of the magnetic field or the mechanical stress applied. To decrease twin boundary interactions, for technical applications Ni–Mn–Ga single crystals and polycrystals have to be trained [5]. The training process consists of successively compressing (mostly) cuboid samples along two or three axes up to a certain stress. Up to now, it is not really evident why this process is needed. Therefore, this paper studies the role of training on a $Ni_{50}Mn_{29}Ga_{21}$ bicrystal with 5 M modulated structure. The initial orientation distribution consists of three different martensitic variants separated by twin boundaries in each parent austenitic grain. Compression of these variants results in twin boundary motion, which changes the volume fraction of particular variants. Local orientation measurements by electron backscatter diffraction (EBSD) in a scanning electron microscope directly confirm the motion of the twin boundaries.

The bicrystal ($Ni_{50}Mn_{29}Ga_{21}$, 5 M modulated structure) was grown by the Bridgman method. A prealloy was prepared by induction melting of pure elements (Ni 99.98, Mn 99.8, Ga 99.999) [6]. The bicrystal with dimensions of $(5 \times 5 \times 3)$ mm^3 was cut by spark erosion from an as-grown ingot, and mechanically and electropolished for EBSD (system supplied by HKL) analysis. The cuboid was cut such that one grain (1) of the bicrystal (Fig. 1a) has cubic orientation with <1 0 0> related to the external shape of the specimen. The bicrystal is characterized by a general high-angle grain boundary with about 60° rotation about the <1 2 3> axis and different martensitic variants within the two grains. The volume fraction of grain 1 in the bicrystal is about 0.6. The training process was performed in an Instron 8562 testing machine at room temperature under a compressive stress up to 30 MPa at a strain rate of 10^{-3} s^{-1}. To

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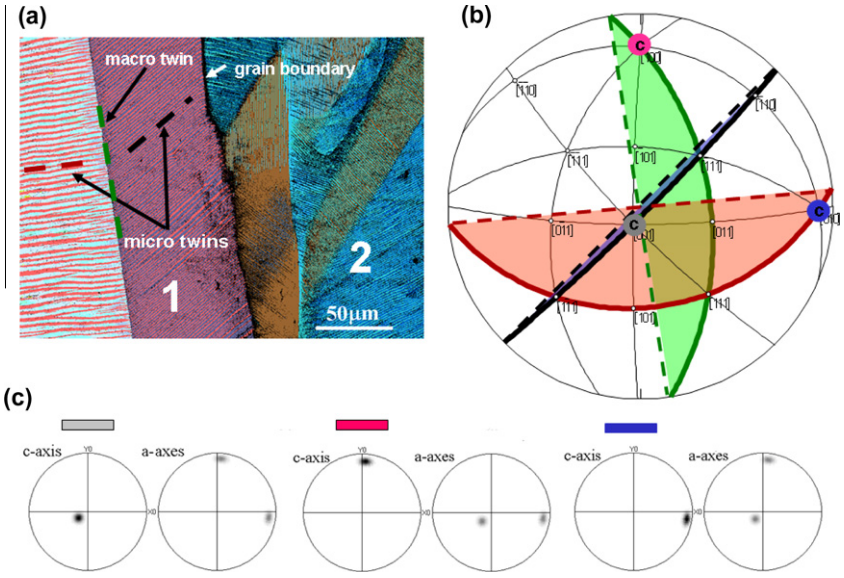

Figure 1. Orientation mapping (a) and stereographic projection with indication of twin traces in grain 1; (b) of the bicrystal. Pole figures of differently coloured areas of the tetragonal 5 M phase of grain and (c). The *c*-axis and *a*-axes (short and long lattice parameter) represent [0 0 1] and <1 0 0] directions of the tetragonal 5 M struc;ture, respectively. 1 and 2 denote the two grains of the bicrystal. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

achieve one type of twin, this process consisted of successively compressing the sample along two (or three) axes.

The microstructure was investigated by EBSD which allows crystallographic information to be obtained. In order to produce orientation mappings with EBSD, the samples were electropolished at 273 K using a mixture of nitric acid and ethanol (volume ratio 1:3). The twin plane was determined by surface trace analysis on two perpendicular mappings of the bicrystal. The Miller indices of the twin boundaries are given with respect to both martensitic variants creating this twin relationship.

The change of orientation during sequential compressions was measured by EBSD and further processed with LABOTEX software [7].

It should be noted that all planes and directions mentioned in this paper are given in the so-called "cubic coordinate system" which is related to the cubic axes of the parent $L2_1$ phase.

The bicrystal investigated consists of macro and micro twins [8,9] (Fig. 1a). The twin planes are within about 2° parallel to {1 0 1). The coarse twin lamellae extend through the whole parent austenitic grains, while the fine lamellae frequently cross the coarse variants in a zig-zag manner with mirror symmetry (Fig. 1a). It should be noted that the fine twin lamellae can only be resolved by EBSD at high magnification. It is obvious that there exists a hierarchy in twin formation. Coarse twin lamellae initially form and subsequently fine twins develop (Fig. 1a). This is deduced from the fact that the fine twins in general are not continuous across the coarse lamellae. Close examination of the microstructure (e.g. in Fig. 1a and b) shows that the fine lamellae are twin related with respect to the macro twins and create a so-called "twins within twins" structure. The initial orientation mapping reveals a distribution of three different martensitic variants in both parent austenitic grains. These martensitic variants with *c*-axis approximately parallel to the main cubic axes of the parent austenite originate from the cubic to tetragonal transformation according to the Bain model. All three variants of the tetragonal unit cell revealed in the pole figures of the bicrystal are displayed in Figure 2c. The *c*-axis and *a*-axes, the short (0.551 nm) and long (0.591 nm) lattice parameters, represent [0 0 1] and <1 0 0] directions, respectively. Since the twin plane is of type {1 0 1), the trace of this plane should be placed exactly in between two *c*-axes. To demonstrate this, a stereographic projection has been used. For example, within grain 1, the trace of the twin plane between the pink and blue orientation is indicated as a black line (Fig. 1b). The blue dashed line shows how this plane crosses the flat surface and corresponds very well to the orientation mapping on the left-hand side (Fig. 1a). Similarly, the other traces of twin planes (red and green) correspond to the orientation mapping. It should be noted that the green line marks the macro (main) twin and the red and black lines mark the micro twins.

In addition to the so-called "twins within twins" structure, another type of microstructure can be observed in $Ni_{50}Mn_{29}Ga_{21}$ alloys. Orientation mapping shows that the boundary plane of one of the micro twins is far away from the ideal position (Fig. 2a and b: the ideal position is marked with a red line), while the second micro twin and the main twin remain unchanged. In this configuration the micro twin planes look like a mirror image with respect to the main twin boundary on both perpendicular surfaces (Fig. 2c), but in fact one of them is not twin related. The second plane is about 3° away from {1 1 0), which is not equivalent to the {1 0 1) twin plane in the tetragonal system. However, the traces of these planes look like a mirror image with respect to the main twin boundary on both sides of this plane but crystallographically they are not twin related. Furthermore, in Figure 1a characteristic branching of the micro twins is observed due to lowering of the elastic energy at the macro twin boundary. Branching is favoured by the