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Texture analysis of melt-spun Ni-Mn-Ga tapes by means of electron backscatter diffraction (EBSD)

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Abstract. A texture analysis is performed by means of the electron-backscatter diffraction technique (EBSD) on melt-spun ribbon-like samples of the composition $Ni_{52.5}Mn_{24.5}Ga_{23}$ (at.-%) were prepared. A dedicated surface treatment is required in order to achieve high quality Kikuchi patterns. For this purpose, mechanical polishing plus ion polishing was employed. EBSD analysis and transmission electron microscopy revealed that the samples have a polycrystalline, granular morphology, with grain sizes around 1 – 2 µm. Several larger grains being present in the region selected for EBSD analysis, and many small grains are found, even embedded in the larger ones. The larger grains exhibit a common direction of elongation, yielding to a specific texture.

1. Introduction

The microstructure in ferromagnetic shape memory alloys (FSMA) and the respective details of it play an important role for the development of effective switching elements. A detailed analysis of the texture is, therefore, an important source of information [1,2]. The rapid solidification route has been explored to prepare Ni-Mn-Ga FSMA in the form of ribbons. The material preparation in this way is much faster than the conventional techniques and provides the possibility to tune the phase composition by means of controlling cooling rate and composition [3,4]. For the melt-spun FSMA samples, the texture analysis is even more important. The electron-backscatter diffraction (EBSD) technique is very suitable for a detailed microstructural analysis as also details within the individual grains can be resolved. In this way, individual grain orientations and the structure of the twin boundaries can be obtained.

2. Experimental procedure

Melt-spun ribbon-like samples of the composition $Ni_{52.5}Mn_{24.5}Ga_{23}$ (at.-%) were prepared at the National Metallurgic Laboratory [3,4]. TEM analysis revealed that the samples have a granular morphology, with grain sizes of the order of 1 – 2 µm. The as-spun ribbon showed dominant L2(1) austenitic (cubic) structure with a splitting of the primary peak in the X-ray diffractogram indicating the existence of a martensitic phase (see Fig. 3). The Curie temperature of the as-spun ribbon was determined to be 302 K. In order to further improve the surface quality, ion-polishing was carried out employing 5 keV Ar-ions for about 1 min. The resulting surfaces are then well suited for the EBSD measurements. The EBSD system employed here consists of a FEI dual beam workstation (Strata DB 235) equipped with a TSL EBSD unit [5]. The voltage is 20 kV, and the Kikuchi patterns are recorded using a DigiView camera system. To produce a crystallographic orientation map, the electron beam is scanned over a selected surface area and the resulting Kikuchi patterns are indexed and analysed automatically. An image quality (IQ) parameter and a confidence index (CI) are recorded for each Kikuchi pattern. The dimensionless IQ parameter is the sum of the detected peaks in the Hough transform employed in the image recording; the CI value yields information about how exact the indexation was carried out; the value ranges between 0 and 1. A detailed description of the measurement procedure can be found in Ref. [6]. The working distance was set to 10 mm.

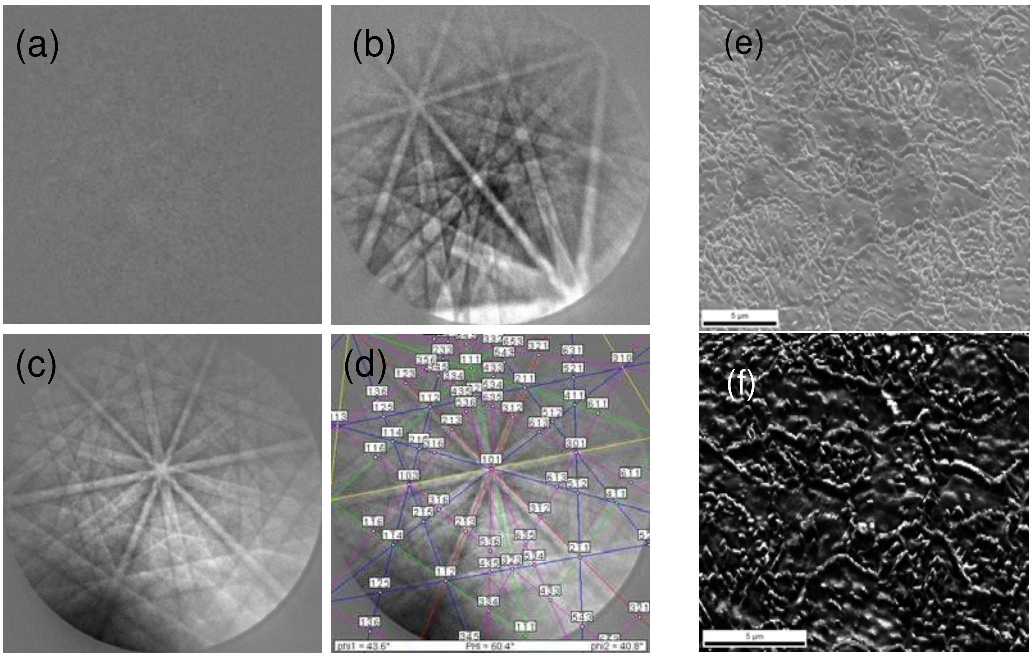

Figure 1. Kikuchi patterns of the austenitic cubic phase (a-d), SEM image under EBSD conditions (e) and IQ map (f).

3. Results and discussion

Figure 1 illustrates what can be achieved concerning the quality (intensity) of the recorded Kikuchi patterns. Image (a) is what is obtained from the sample after mechanical polishing. Figure 1 (b) presents the result after ion-beam polishing (5 keV Ar-ions, 30 s) and (c) additional image processing. Figure 1 (d) presents the indexation of the Kikuchi pattern. Figure 1 (e) presents a SEM image obtained under EBSD conditions (70° tilt to the electron beam), and (f) gives the image quality (IQ) map which resembles a backscattered electron image.

Figure 2 gives the inverse pole figure (IPF) maps in (0 0 1), (1 0 0) and (0 1 0) directions. The colour code for these maps is given in the stereographic triangle below the map. The chosen EBSD step size is 100 nm, so we do not expect to see a contribution of the martensitic phase, which is, according to the TEM analysis [3], located at the grain boundaries. This will be analysed in further experiments. Here, we can see from the maps that in (b,c) the colours are not so widespread as in (a), which indicates a different type of texture. This becomes even more pronounced when regarding the respective pole figures given in Fig. 3, where the [1 0 0] direction shows a ring, indicating a texture in the rolling direction (RD) as expected from a melt-spun tape.

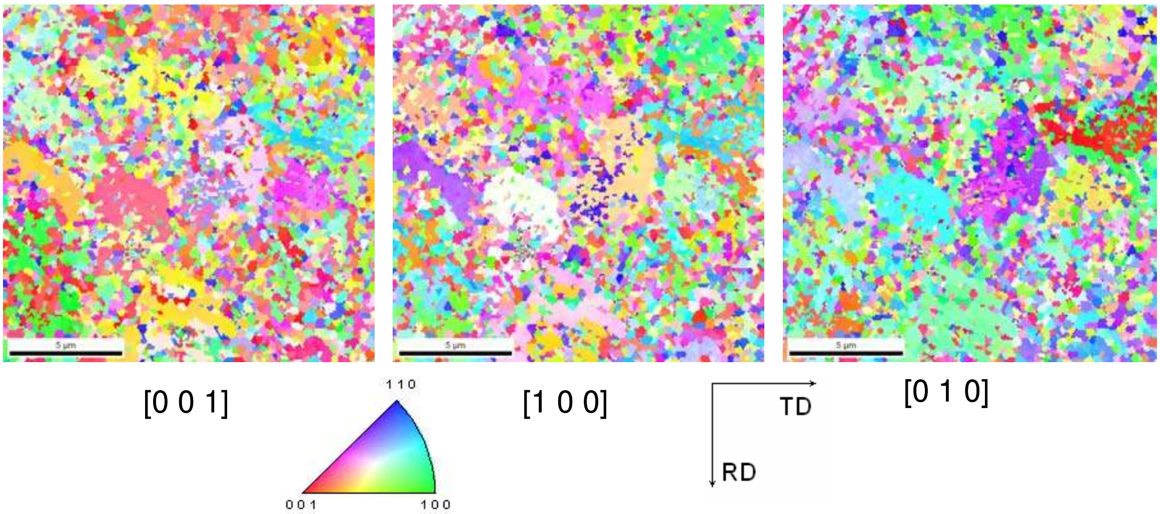

Figure 2. IPF maps in [0 0 1], [1 0 0] and [0 1 0] directions, respectively. The colour code for the orientation is given in the stereographic triangle.

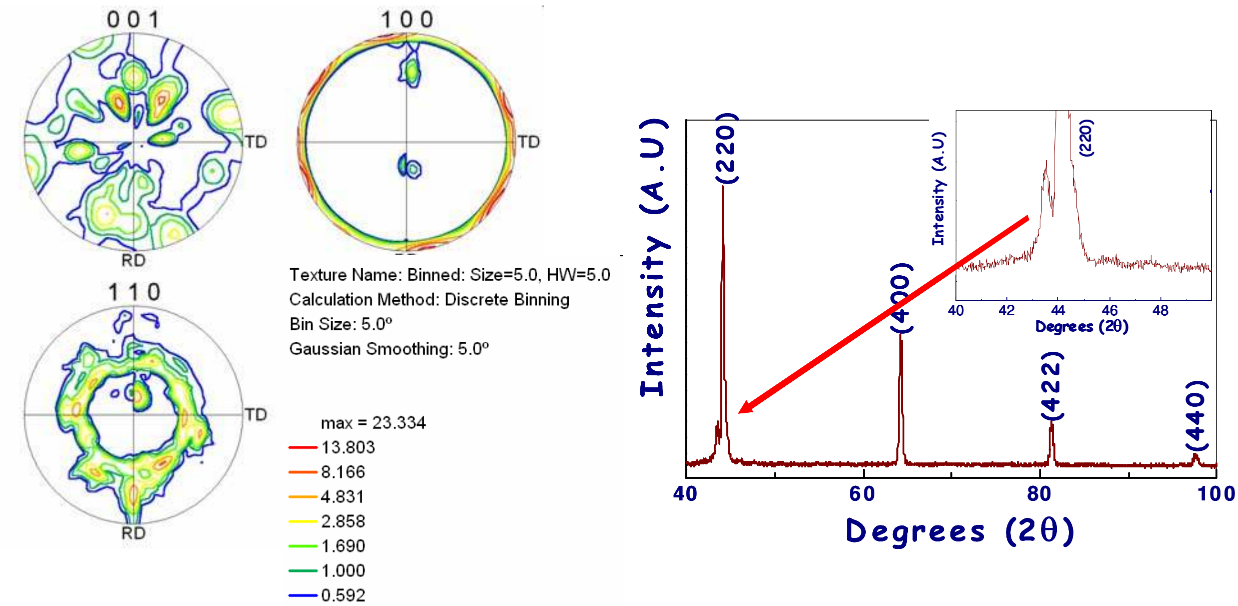

Figure 3. (left) Pole figures in [0 0 1], [1 0 0] and [1 1 0] directions, as determined from the EBSD maps, and (right) X-ray analysis, indicating a small contribution of the martensitic phase.

Figure 3 also gives the X-ray analysis of the investigated sample. Only a small contribution of the martensitic phase is seen here, which justifies the EBSD analysis of only the austenitic phase.

The grain size map of Fig. 4 reveals that there is a large number of grains with a grain diameter of about 0.3 μm (white/light gray), but there are several distinct larger grains with a grain diameter ranging between 1.2 – 1.7 μm (represented in black). The map of Fig. 4 further shows the EBSD-determined misorientation angles. The colour code for these misorientations is given in rainbow colors as given in the respective histogram. There is a large number of small-angle misorientations (blue) being present in the selected area, but also a distinct secondary maximum at about 45° – 50° misorientation (dark orange).

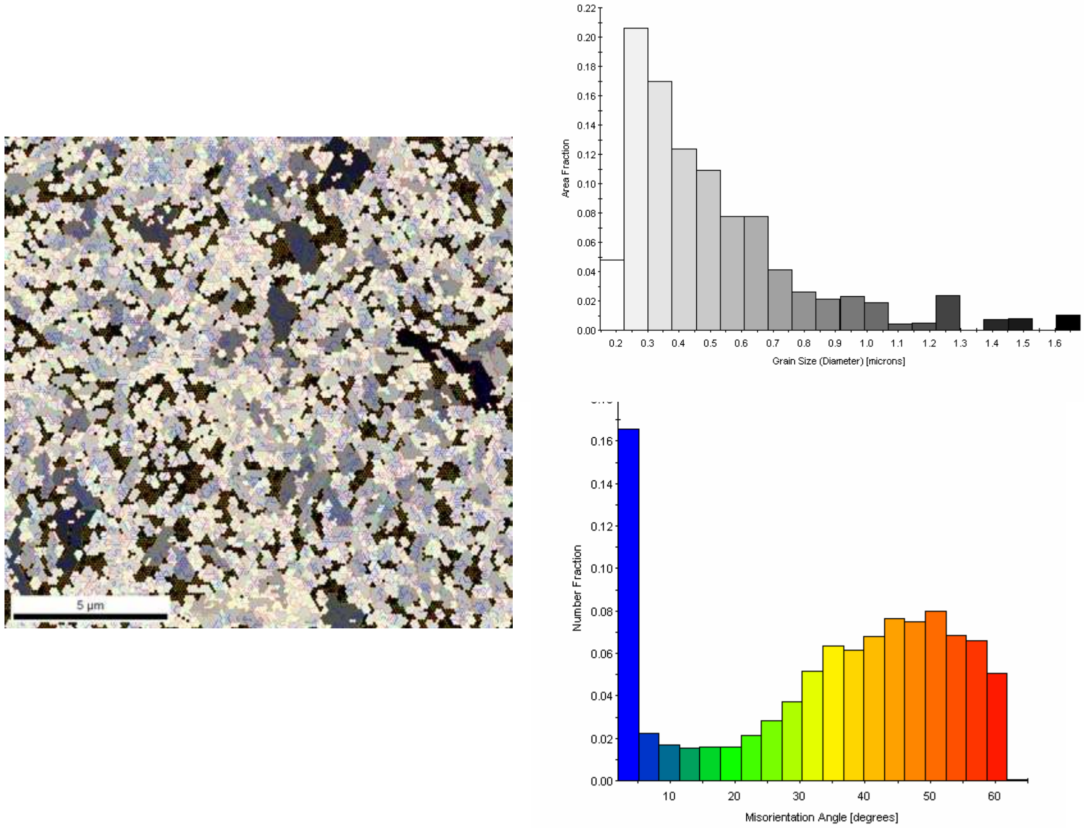

Figure 4. Grain size map (colour code given by the grain size histogram (represented in gray tones), together with the EBSD-determined misorientation angles (the colour code for this information is given in the coloured histogram below).

Conclusions

The advantage of the EBSD technique is even more visible in the analysis of a melt-spun ribbon-like sample with grain sizes ranging between 0.2 µm and 1.7 µm. In this case, the ion polishing step provides high-quality Kikuchi patterns, which form the base for a detailed EBSD analysis. The inverse pole figure (IPF) maps and pole figures reveal in this case the presence of a distinct texture, and the grain size and grain shape aspect ratio analysis provide even more information about the microstructure of such a sample.

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