

## Twinning Behaviour of Textured Polycrystalline Ni-Mn-Ga Alloy After Hot Extrusion

Robert Chulist<sup>1,a</sup>, Andrea Böhm<sup>2,b</sup>, Thomas Lippmann<sup>3,c</sup>, Werner Skrotzki<sup>1,d</sup>,  
Welf-Guntram Drossel<sup>2,e</sup> and Reimund Neugebauer<sup>2,f</sup>

<sup>1</sup> Institut für Strukturphysik, Technische Universität Dresden,  
D-01062 Dresden, Germany

<sup>2</sup> Fraunhofer-Institut für Werkzeugmaschinen und Umformtechnik, Reichenhainer Str. 88,  
D-09126 Chemnitz, Germany

<sup>3</sup> Institut für Werkstofforschung, GKSS Forschungszentrum Geesthacht,  
D-21502 Geesthacht, Germany

<sup>a</sup> robert.chulist@physik.tu-dresden.de, <sup>b</sup> andrea.boehm@iwu.fraunhofer.de,

<sup>c</sup> thomas.lippmann@gkss.de, <sup>d</sup> werner.skrotzki@physik.tu-dresden.de,

<sup>e</sup> welf-guntram.drossel@iwu.fraunhofer.de, <sup>f</sup> Reimund.Neugebauer@iwu.fraunhofer.de

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**Abstract.** Up to now most of the research on Ni-Mn-Ga alloys was concentrated on single crystals. However, there is a great interest in polycrystals for technical applications. Recently, polycrystalline Ni<sub>50</sub>Mn<sub>29</sub>Ga<sub>21</sub> rods of 25 mm diameter and 400 mm length were prepared by hot extrusion. The <100> <110> double fibre texture of the extruded rods was measured with high-energy synchrotron radiation. The microstructure after hot extrusion was analyzed with electron backscatter diffraction. Within the recrystallized grains all three martensitic variants have developed during cooling. Variants with twin boundaries preferentially aligned along the extrusion direction predominate.

### Introduction

Magnetically actuated shape memory alloys such as Ni-Mn-Ga have recently attracted special attention due to large strains achieved by the reorientation of martensitic twin variants. The movement of twin boundaries under a magnetic field resulting from the high magnetocrystalline anisotropy leads to a magnetic-field induced strain (MFIS) [1-3]. During cooling from high temperatures Ni-Mn-Ga alloys experience different phase transitions, from B2 (cubic) via L2<sub>1</sub> (cubic, austenite) to martensitic structures. Depending on the chemical composition and heat treatment at least three martensitic structures can be distinguished in the Ni-Mn-Ga system (7M modulated, monoclinic; 5M modulated and non-modulated, both tetragonal) [4-6]. However, MFIS only exists in the 5M and 7M modulated structures.

Until now, MFIS has been mainly reported for Ni-Mn-Ga single crystals [7]. But it was proven, that coarse grained Ni-Mn-Ga polycrystals with solidification texture show MFIS by twin boundary motion as large as 1 % after proper treatment including two step annealing and mechanical training [8]. Since for large-scale production, growth of single crystals is economically unfavourable, it is necessary to investigate polycrystalline samples on their suitability for MFIS. Therefore, to also obtain this effect in polycrystals, fabrication processes are needed to produce strong textures. One practical way to achieve a preferred crystallographic orientation in polycrystalline aggregates is hot extrusion.

This paper is focused on microstructure and texture analyses of Ni<sub>50</sub>Mn<sub>29</sub>Ga<sub>21</sub> polycrystals fabricated by hot extrusion. The texture of the samples was analyzed by diffraction of high-energy synchrotron radiation. Simultaneously, the crystal structure was determined. The microstructure

was characterized with electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM).

### Experimental procedure

A master alloy ingot of composition  $\text{Ni}_{50}\text{Mn}_{29}\text{Ga}_{21}$  was produced by induction melting in Ar atmosphere followed by casting into a cold copper mould. The mass loss was negligible, hence the composition is assumed to be the intended one. To decrease friction during extrusion, the cylindrical ingot (100 mm length, 50 mm diameter) was canned in stainless steel. Direct extrusion processing was done at  $1000^\circ\text{C}$  with an extrusion ratio of 4/1.

For the texture measurements three cylindrical samples with height and diameter of 10 mm were cut from the extruded rod at the front, middle and end. The texture was measured by diffraction of high-energy synchrotron radiation (100 keV) using the GKSS materials science beam line HARWI-II at DESY in Hamburg, Germany. Due to the high penetration depth of synchrotron radiation, texture measurements in transmission allow to collect orientation data from a relatively large sample volume, i.e. with a good grain statistics. The orientation distribution function (ODF) was calculated from the measured pole figures ( $\{400\}$ ,  $\{220\}$ ,  $\{224\}$ ) using LABOTEX software. The ODF was used to calculate the inverse pole figures of the extrusion direction.

The microstructure was analyzed by EBSD (Oxford system) in a SEM (Zeiss SUPRA 25). To do this, cuboid samples with an edge length of 5 mm were cut by spark erosion parallel to the extrusion direction which preferably is a  $[100]$  axis of the cubic high temperature phase. Subsequently, they were grinded and polished to achieve plane and parallel surfaces. To remove the highly damaged surface layer formed during grinding and polishing an electrolytic etching with 25 vol%  $\text{HNO}_3$  in ethanol was used.

All planes and directions mentioned in this paper are given in the cubic coordinate system which is related to the cubic axes of the parent  $\text{L2}_1$  phase. The tetragonal unit cell dimensions are given in following order  $a > c$ .

### Results and discussion

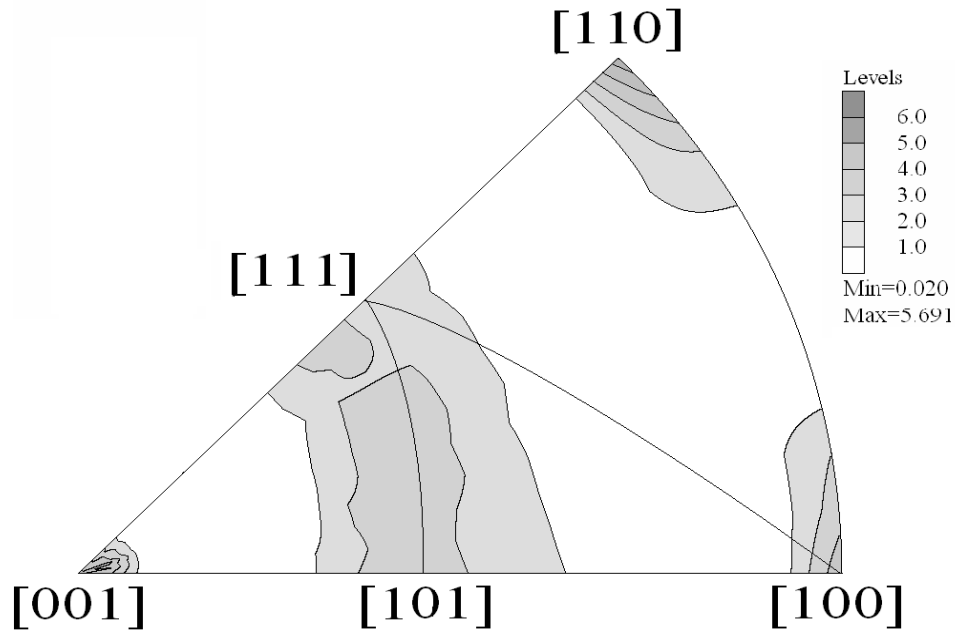
During extrusion the material mainly experiences axisymmetric tension. This deformation mode applied to Ni-Mn-Ga alloys in the B2 phase field produces a  $\langle 100 \rangle / \langle 110 \rangle$  double fibre texture (Fig. 1). This type of texture is characteristic for hot extruded B2 structured intermetallic compounds having experienced continuous dynamic recrystallization [9, 10].

During cooling the Ni-Mn-Ga alloy transforms to the  $\text{L2}_1$  cubic structure and finally to the 5M tetragonal structure. The latter transformation is such that the tetragonal (001) plane and  $[110]$  direction are parallel to the cubic (001) plane and  $[100]$  direction, respectively. Thus, three orientation variants of the tetragonal unit cell are possible. Figure 1 shows that a variant selection takes place with  $[110]$  preferentially aligned along the extrusion axis. The reason may be stresses developed during cooling. The textures of the front and end samples are comparable to that of the middle sample, however, their variant fractions are different.

A large uniaxial magnetic anisotropy, a low barrier against twin boundary motion, and an appropriate orientation of the twins are prerequisites to enable magnetic field-induced twin boundary motion. Both features not only depend on composition but also on crystal structure and microstructure. Only the modulated martensitic phases have an uniaxial magnetic anisotropy and are capable of MFIS. The crystal structure of  $\text{Ni}_{50}\text{Mn}_{29}\text{Ga}_{21}$  alloy is 5M modulated tetragonal and therefore fulfils this requirement.

Recent hot rolling experiments at  $1000^\circ\text{C}$  have shown that plastic deformation of polycrystalline Ni-Mn-Ga alloys above the ordering temperature is possible in the B2 structure without brittle fracture [11]. The same holds for hot extrusion. Orientation images obtained by EBSD (Fig. 2) show the grain structure after hot extrusion. It is seen, that dynamic recrystallization has taken place. Recrystallization leads to slightly elongated grains with a size of about  $100\text{ }\mu\text{m}$ . The

recrystallized grains contain a lot of twins with the trace of the twin boundaries preferentially aligned along the extrusion direction (Fig. 2b).

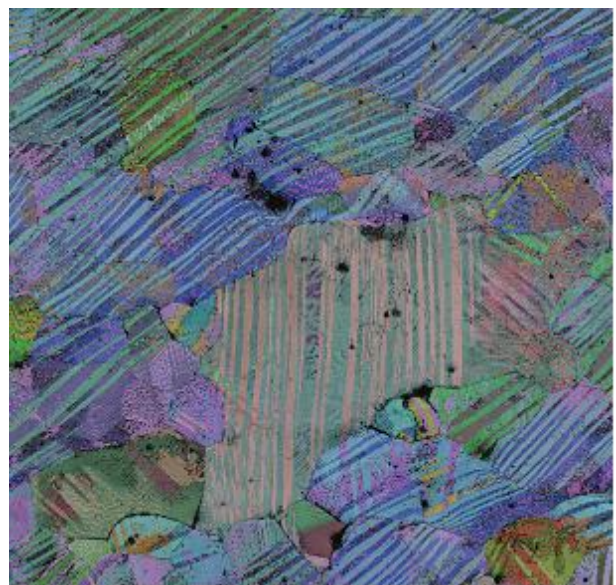
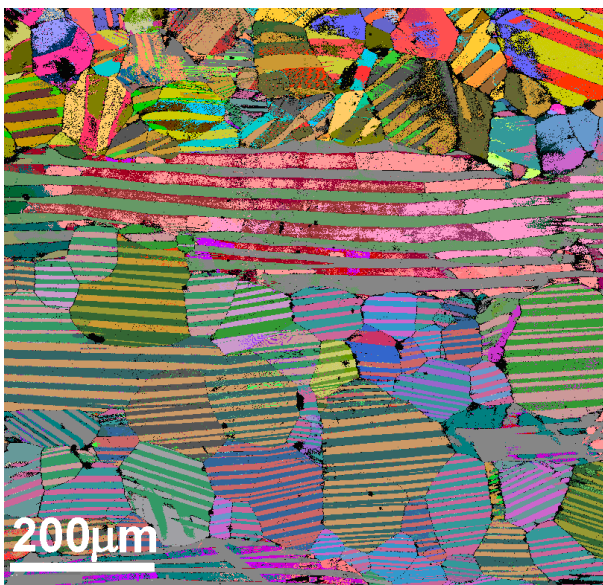


**Fig. 1:** Inverse pole figure of the extrusion direction of the tetragonal 5M Ni-Mn-Ga polycrystal measured in the middle region of the extruded rod (intensities are given in multiples of a random distribution, mrd).

Looking at higher magnification another twin variant is resolved as fine lamellae crossing the other twin variants in a zig-zag manner (Fig. 3a). It is obvious that a hierarchy in twinning formation exists. All three orientation variants of the tetragonal unit cell are revealed in the pole figures (Fig. 3b).

a)  $\Rightarrow$  extrusion direction

b)  $\otimes$  extrusion direction



**Fig. 2:** Microstructure of hot-extruded  $\text{Ni}_{50}\text{Mn}_{29}\text{Ga}_{21}$  alloy in the middle region of the rod

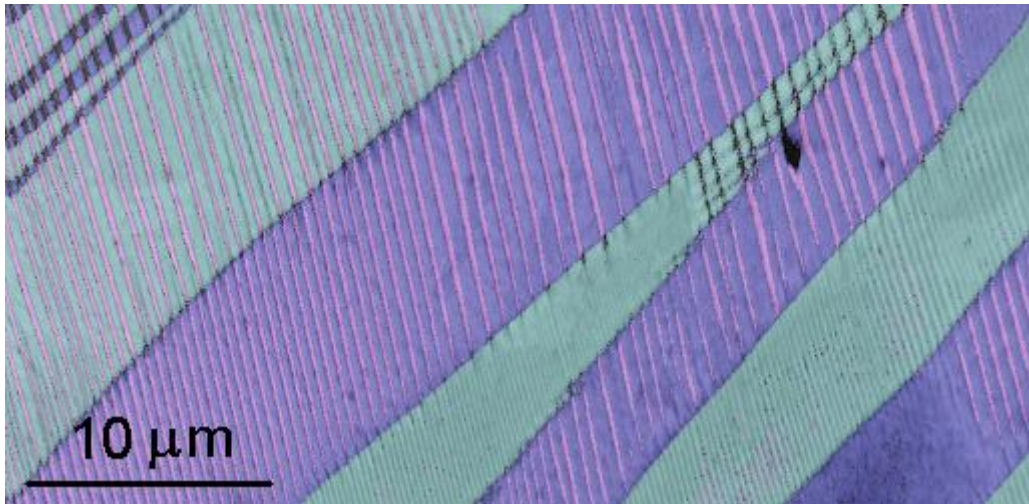
Further work is in progress to improve the microstructure and texture of hot extruded  $\text{Ni}_{50}\text{Mn}_{29}\text{Ga}_{21}$  alloys by adjusting the preparation technology with different post heat treatments and repeated mechanical loading cycles (training). Thus, MFIS in hot extruded Ni-Mn-Ga polycrystals may be achieved.

### Conclusions

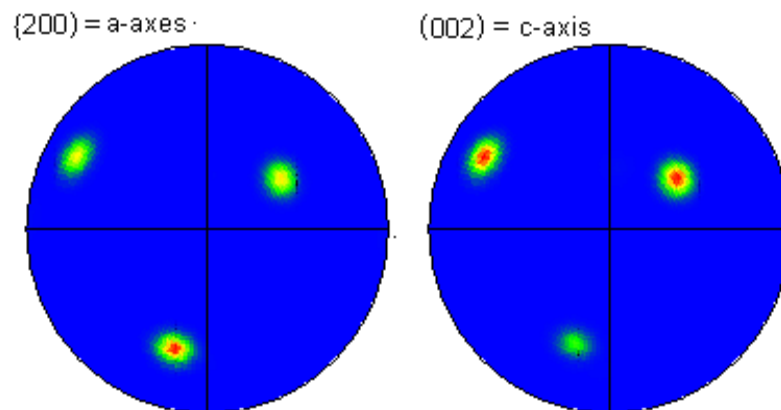
The results of hot extrusion experiments at  $1000^\circ\text{C}$  can be summarized as follows:

1. The plastic deformation of polycrystalline  $\text{Ni}_{50}\text{Mn}_{29}\text{Ga}_{21}$  alloys is possible in the B2 structure without brittle fracture.
2. The texture of the hot extruded material is characterized by a  $\langle 100 \rangle \langle 110 \rangle$  double fibre texture along the extrusion direction.
3. The microstructure of the hot extruded alloy is dynamically recrystallized with grains slightly elongated in the extrusion direction.
4. Within the grains the three twin variants consist of macro- and microlamellae.

a)  $\otimes$  extrusion direction



b)



**Fig. 3:** a) Twin boundary arrangement in polycrystalline  $\text{Ni}_{50}\text{Mn}_{29}\text{Ga}_{21}$  alloy after hot extrusion, b) pole figures of the tetragonal 5M phase with extrusion direction in the middle

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