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In situ neutron diffraction study of magnetic field induced martensite reorientation in Ni–Mn–Ga under constant stress

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Abstract

The magnetic field induced reorientation of martensite twins as a function of a compressive stress and crystallographic direction was studied on a single-crystalline $Ni_{49.7}Mn_{29.3}Ga_{21}$ magnetic shape memory alloy by the *in situ* neutron diffraction technique. The compressive stress 0–3 MPa and magnetic field 0–3.5 T were applied perpendicularly to each other along the [001] and [100] ([010]) crystallographic directions of tetragonal martensite, respectively. The neutron diffraction method provides integrated information about the presence and volume fractions of individual martensite variants involved in magnetic actuation. It has been found that (i) martensite variant reorientation is not completed by applying magnetic field as high as 2.5 T, (ii) recoverable strain of about 3% due to switching between two variant microstructures is observed upon cyclic application of magnetic field under external compression stress 0.9 MPa, (iii) the magnetic field induced reorientation was suppressed by the bias external stress of 3 MPa and (iv) mechanical training by successive compression deformation on two different faces of the cuboid Ni–Mn–Ga single crystal is essential to achieve the magnetic field induced cyclic reorientation.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

The compounds close to Ni_2MnGa are ferromagnetic shape memory alloys, which is exhibited in the large martensite state deformations (up to 10%) induced by an external magnetic field or applied stress [1, 2]. Applying the magnetic field along the hard magnetization axis causes rotation of the lattice (twinning) in such a way that the easy magnetization axis is aligned with the field. The twinning is accompanied by the change of the sample length. The twinning interfaces, however, move only if the equivalent magnetic stress determined by magnetocrystalline anisotropy energy is larger than the twinning stress needed to activate the twinning [3–6]. If one wants to use the Ni_2MnGa as an actuator, a bias compression stress of suitable value must be applied to restore the shape of the crystal when magnetic field decreases. This bias stress is known to affect the maximum achievable actuation strains. The magnetic field induced strains are related to the actually existing martensite variant microstructures in the specimen which are sensitive to the bias load. Though one

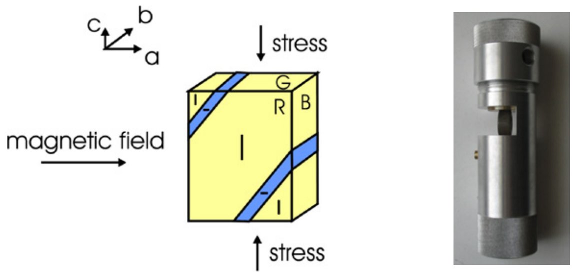

Figure 1. Schematic view of the Ni–Mn–Ga single-crystal sample existing mainly in the martensitic variant 1 (yellow) and with a small volume fraction of variant 3 (blue) together with the directions of applied magnetic field and compression stress applied by a spring in the miniature deformation rig.

Table 1. Transformation matrices R for the planes A $\rightarrow$ M for three lattice correspondence variants (LCVs).

LCV	1	2	3
Transformation matrix R for planes A $\rightarrow$ M	$\begin{pmatrix}1&0&0\\0&1&0\\0&0&1\end{pmatrix}$	$\begin{pmatrix}0&1&0\\0&0&1\\1&0&0\end{pmatrix}$	$\begin{pmatrix}0&0&1\\1&0&0\\0&1&0\end{pmatrix}$

Table 2. Corresponding martensitic planes (CMPs) to the $(200)_A$ austenitic plane for all three martensitic variants.

LCV	CMP to $(200)_A$ austenitic plane	d_{hkl} martensite (nm)
1	$(200)_M$	0.2975
2	$(020)_M$	0.2975
3	$(002)_M$	0.2805

can anticipate the maximum strains are, in addition to the crystallography and lattice constants related to the actual twin microstructures, twinning stress and bias stress, detailed information on twinning stresses and variant microstructures existing under stress and magnetic field are missing due to the lack of reliable data. In addition, it has been found empirically that a compressive training prior to the actuation (e.g. by the application of several compression cycles to different faces of the sample) is extremely beneficial for the magnetic actuation [7].

In this work, we have investigated microstructure evolutions in a Ni_2MnGa single crystal subjected simultaneously to a magnetic field and a mechanical force by a recently developed *in situ* neutron single-crystal diffraction method [8]. The essential advantage of this method is that it gives, unlike the other structural methods, information from the whole volume of the studied crystal. Results of dedicated experiments simulating conditions of a magneto-mechanic Ni_2MnGa single-crystal actuator working under various bias stresses are discussed in section 4, and the effect of compressing training on magnetic actuation is discussed in section 5.

2. Experimental material and experimental techniques

A single crystal of $Ni_{49.7}Mn_{29.3}Ga_{21}$ magnetic shape memory alloy was cut from an ingot produced by AdaptaMat Oy. A rectangular specimen with dimensions of 5 × 5 × 10 mm was cut along the {100} faces of the austenite crystal lattice. The details of heat treatment and sample preparation have been published elsewhere [5]. The transformation temperatures were determined by DSC as $M_s = 305$ K and $M_f = 301$ K. The parent phase of the Ni–Mn–Ga single crystal is a cubic $L2_1$ structure with lattice parameter $a = 0.584$ nm. The structure of the martensite is tetragonal with five-layered modulation (5M) [9]. The lattice parameters of the 5M martensite were measured by x-ray diffraction as $a_M = b_M = 0.595$ nm, $c_M = 0.561$ nm, with easy axis magnetization along the short c-axis of the martensite.

The neutron diffraction experiment was performed at ILL Grenoble on the single-crystal diffractometer D10 in the two-axis configuration equipped with the horizontal cryomagnet (3.8 T) and 80 × 80 mm^2 two-dimensional detector using monochromatic neutron wavelength $\lambda = 1.26$ Å. For the *in situ* diffraction experiments under applied load, a small deformation rig (figure 1) was constructed. The deformation rig consists of a cylindrically shaped aluminium body with a sample stage in the centre, a compression spring and a load cell. The body has a window for the neutron beam. The diameter of the cylinder was 34 mm and the length 115 mm (figure 1). The sample stage and spring were made from bronze. The spring is used to maintain the desired stress which is tuned by the screw from the bottom part of the deformation rig. The load cell is attached in the top of the rig. It is essential that the materials used for the construction of this small deformation rig are non-magnetic so any unwanted effects due to the applied magnetic field are avoided. This deformation set-up with a sample exposed to particular force was mounted in a horizontal field cryomagnet. The magnetic field and stress were applied in mutually perpendicular directions.

3. Single-crystal neutron diffraction method

Due to the difference in crystal symmetry between the cubic structure of austenite and tetragonal martensite, there are three lattice correspondence variants of martensite. These martensitic variants have the same tetragonal structure but different orientations with respect to the parent austenitic phase. The lattice correspondence between cubic austenite and tetragonal martensite can be chosen in two different ways [9, 10]. In this paper, we use the lattice correspondence where the crystallographic axes of tetragonal martensite are parallel to the crystallographic axes of the cubic lattice [9]. Each martensitic variant is described by a transformation matrix R (see table 1).

Using the transformation matrix R, it is possible to calculate martensitic planes in each of the three variants corresponding to a particular austenitic plane (see table 2):

$$(hkl)_M^T = R^T * (HKL)_A^T, \quad (1)$$

where $(hkl)_M$ are Miller indices of the plane in the martensite and $(HKL)_A$ are the indices of the corresponding austenitic plane. T means the transpose of the row vector.

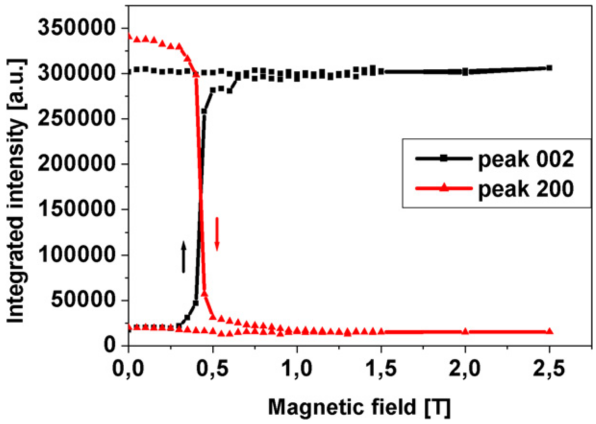

Figure 2. Variation of the integrated intensity of diffraction peaks 200_M (V1) and 002_M (V3) with magnetic field (geometry shown in figure 1) measured by the *in situ* neutron diffraction experiment under zero applied stress.

However, the lattice corresponding martensitic planes are not exactly parallel to the austenitic plane and have lattice spacings different from the original austenitic plane $(200)_A$ due to the Bain distortion characteristic for the cubic to tetragonal transformation in Ni–Mn–Ga (see table 2). The martensitic plane $(002)_M$ in variant 3 has the shortest lattice spacing, 0.2805 nm. The martensitic plane $(200)_M$ in variant 1 and the martensitic plane $(020)_M$ in variant 2 have the same lattice spacing, 0.2975 nm. From the diffraction point of view, these three martensitic planes will yield two different diffraction peaks at different diffraction angles 2θ. In the following text, the $(002)_M$ diffraction peak (short c-axis) will characterize the variant whose volume fraction increases under the effect of increasing magnetic field (variant 3 or 2). The second diffraction peak ($(200)_M$ or $(020)_M$) at different 2θ angle originates from the martensitic planes $(200)_M$ or $(020)_M$ in two distinct tetragonal variants 1 and 2, respectively, the volume fraction of which decreases under the effect of increasing magnetic field. In order to find out in which variant the crystal actually exists, it is always possible to take advantage of the fact that the martensite planes $(200)_M$ and $(020)_M$ are not aligned in space and perform an omega scan (the detector is left stationary while the crystal rotates) to distinguish between them. The essential advantage of the neutron diffraction single-crystal method is that it provides information about the presence and volume fractions of individual martensite variants involved in cyclic magnetic actuation under stress that is integrated over the whole volume of the sample.

4. *In situ* magnetomechanical loading experiments

The diffraction experiment consisted in the investigation of magnetic field induced reorientation in a Ni–Mn–Ga single crystal under different stress values (0, 0.9 and 3 MPa). As common for any mechanical experiment on a martensite single crystal [11], it is of key importance to have the initial microstructure in the sample well defined. In order to achieve this, the sample was deformed alternately several times on two different faces denoted B and G in figure 1. Following such a simple training, the sample is assumed to consist mostly of one martensitic variant [8, 11] with short c-axis in the direction of the last compression direction. In the beginning of the experiment, the tetragonal c-axis of the crystal was always oriented in the direction perpendicular to the magnetic field (applied in the martensitic direction $[100]_M$).

Firstly, the experiment with magnetic actuation without stress was performed (figure 2). The microstructure changes during magnetic field induced reorientation were investigated using the principles outlined in section 3. The $(200)_M \rightarrow (002)_M$ and $(002)_M \rightarrow (200)_M$ peak intensity changes were followed as evidence for the martensite reorientation. Initially, the sample was mainly in the martensitic variant 1 (V1), that corresponds to the $(200)_M$ martensitic reflection in zero magnetic field (figure 2). Besides, the presence of the martensitic reflection $(002)_M$ indicates a small volume fraction of another martensitic variant 3 (V3). The variant 2 $(020)_M$ was not detected with the help of the omega scan.

Figure 2 shows the sharp decrease of the $(200)_M$ martensitic reflection and the sharp increase of the $(002)_M$ martensitic reflection at the magnetic field value of 0.3 T. This indicates the onset of the magnetic field induced reorientation. Since the $(200)_M$ never disappears completely, the martensite reorientation is not completed even at the maximal magnetic field of 2.5 T, which is well above magnetic saturation. This means that we did not get a true single crystal (single variant) of the martensite and the sample contains twin interfaces.

Figure 3 shows a detail at low magnetic field. It is clearly seen that the intensity of the $(200)_M$ martensitic reflection

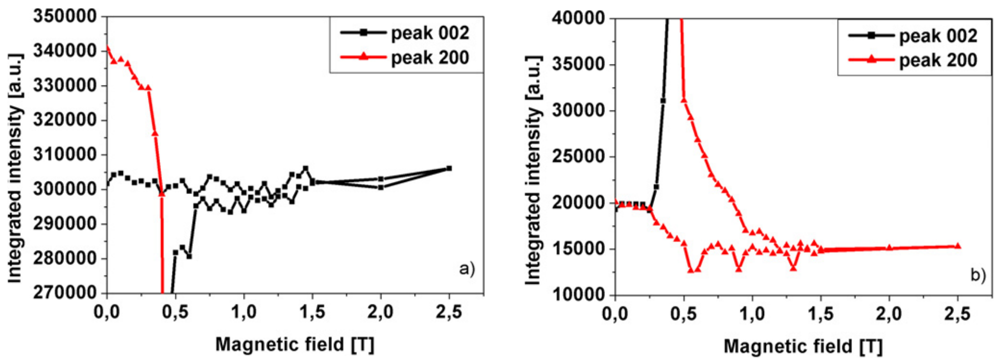

Figure 3. Detailed views showing parts of figure 2.

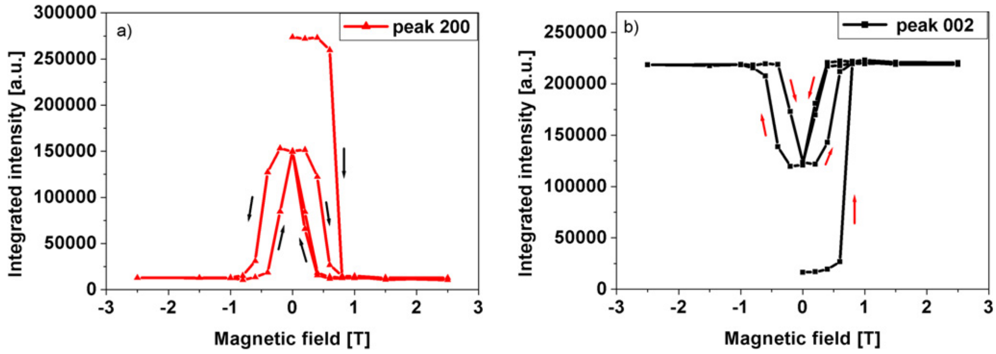

Figure 4. Variation of the integrated intensity of diffraction peaks 200_M (V1) and 002_M (V3) with magnetic field (geometry shown in figure 1) under applied stress 0.9 MPa measured by the *in situ* neutron diffraction experiment. Arrows show the test direction.

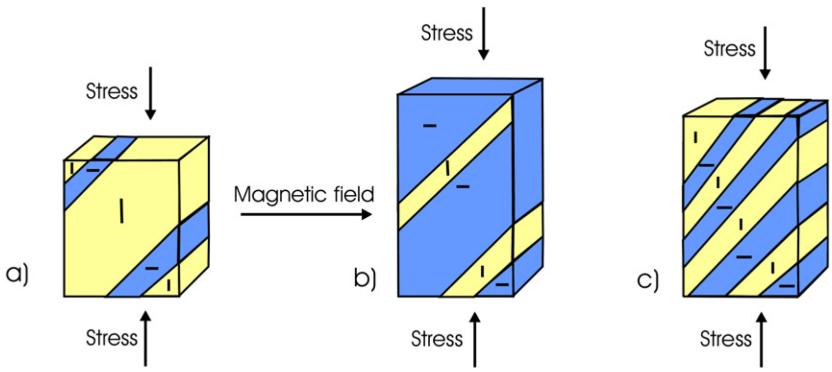

Figure 5. Schematic drawing of the microstructure evolution during the magnetic field induced reorientation of the Ni–Mn–Ga single crystal under 0.9 MPa stress: (a) initial microstructure of the sample after multiple B–G training (see figure 7), (b) variant microstructure after reorientation by magnetic field 2.5 T and (c) microstructure consisting of a mixture of variants 1 and 3 at a magnetic field of 0 T (see figure 4).

slowly decreases from the start of the magnetic field loading up to 0.3 T (figure 3(b)), where it starts to fall very quickly.

This early decrease is attributed to the rotation of magnetization vector, since only the magnetic moments perpendicular to the scattering vector gives rise to the magnetic contribution, i.e. to the intensity of the $(200)_M$ reflection. The magnetic moments rotate from the easy magnetization c-axis towards the direction of the increasing magnetic field before the magnetically induced twin motion is activated. This agrees with the magnetic measurements performed on Ni–Mn–Ga single crystals [1, 5]. The observed jump in intensity is well correlated with the jump of magnetization observed at 0.25 T in [5]. This abrupt change in magnetization corresponds to the reorientation of martensite lattice, as a result of which the c-axis becomes aligned with the direction of magnetic field [1, 5]. The intensity of the $(002)_M$ reflection, on the other hand, does not change remarkably before the field of 0.25 T is reached (figure 3(a)), since the magnetic moments in this case are parallel to the scattering vector of $(002)_M$ reflection and there is thus no magnetic contribution.

The decrease of the magnetic field causes no significant changes in the intensity of both martensitic reflections down to the magnetic field of 0.5 T (figure 3(b)). Below this value, however, the intensity of the $(002)_M$ reflection stays constant but that of the $(200)_M$ reflection increases. A question is whether this small change should be ascribed to the changes of partial lattice orientation or magnetization rotation. We believe this is not due to the lattice reorientations upon decrease of the magnetic field but due to the magnetic moment rotations back to the direction of easy magnetization as the magnetic anisotropy prevails at low magnetic fields. This interpretation is also supported by the fact that the intensity of the $(002)_M$ martensitic reflection did not change significantly with the decrease of the magnetic field.

Figure 4 shows the neutron diffraction results obtained in the second experiment with the magnetic field induced reorientation under constant stress of 0.9 MPa. The initial state of the sample was same as in the previous case—i.e. the sample was mainly in the martensitic variant 1, which corresponds to the large intensity of the $(200)_M$ martensitic reflection (figure 4(a)). The minority variant 3 (martensitic reflection $(002)_M$ (figure 4(b))) was also present. Magnetic field was applied in the same direction as for the previous case, as shown in figure 1.

Compared to the previous case, the martensite reorientation started at higher values of the applied magnetic field, ~0.7 T. Again, the reorientation was never totally completed and the small volume fraction of the martensite variant 1 remained in the sample even at maximal magnetic field of 2.5 T (figure 5(b)). Upon decreasing the magnetic field under applied stress, the reverse reorientation occurred at magnetic field of 0.4 T due to the effect of the bias stress. The 0.9 MPa stress,

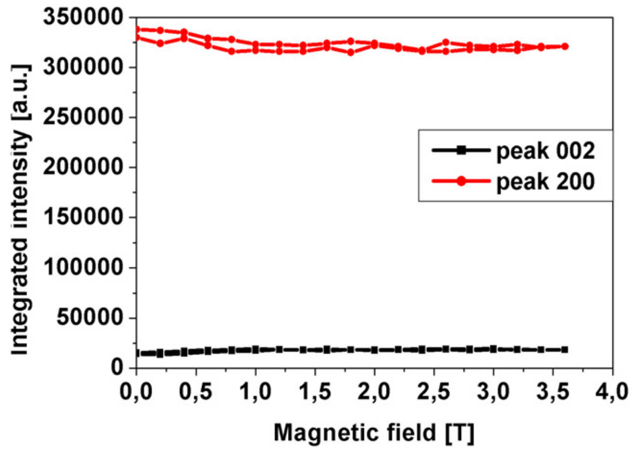

Figure 6. Variation of the integrated intensity of diffraction peaks 200_M (V1) and 002_M (V3) with magnetic field (geometry shown in figure 1) measured by the *in situ* neutron diffraction experiment under applied compression stress 3 MPa.

however, was too small to reorient fully the martensite crystal back to variant 1 (figure 5(a)). At zero magnetic field, the sample thus existed in the new microstructure state characterized by nearly equivalent volume fractions of martensitic variants 1 and 3 as can be inferred from the diffraction intensity (figure 4) and suggested schematically in figure 5(c). When magnetic field of the opposite direction is applied, variant 1 undergoes an analogical process of twinning reorientation to the martensitic variant 3 (figures 4 and 5). Finally, the decrease of the magnetic field down to 0 T again leads to the same microstructure consisting of nearly equal volume fractions of the martensitic variants 1 and 3. As found in parallel magnetic strain measurement on this crystal, periodical changes of the magnetic field under 0.9 MPa bias stress bring about reversible strain changes of approximately 3% similarly as in [5].

Figure 6 shows the neutron diffraction results obtained in the third experiment with the magnetic field induced reorientation under constant compression stress of 3 MPa. The results are different again. In this case, there were only very small intensity changes of $(200)_M$ and $(002)_M$ martensitic reflections, indicating that the reorientation of the martensite lattice is small if any. The magnetic moments, however, rotated (as known from earlier magnetization measurements [5]) towards the direction of the magnetic field, which gives rise to the minor intensity changes of the 200_M martensite reflection. However, very small but discernible change in the $(002)_M$ reflection suggests that a small volume of the crystal underwent the reorientation at this level of stress, again in agreement with previous magnetic observation [5].

When applying magnetic field to the ferromagnetic Ni–Mn–Ga crystal, the two processes—rotation of the magnetic moments from the *c*-axis and the twin reorientation—compete. If it is energetically favourable to rotate the magnetic moments (the mechanical energy for the twinning is higher than the energy needed to rotate the magnetic moments), the moments rotate and no magnetic-field reorientation occurs. The mechanical energy due to applied bias stress must be added to the mechanical energy for the twinning [4, 5]. The applied bias stress of 3 MPa is, however, already too large for magnetic actuation, since the magnetic field induced lattice reorientation was nearly completely suppressed as determined from the neutron diffraction results (figure 6).

5. The effect of compression training on magnetomechanical actuation

In order to obtain the magnetic field induced strain in magnetic shape memory alloy single crystal, it is generally believed that three basic conditions must be fulfilled [5, 6, 12]. The material has to be ferromagnetic, the twin boundaries must be highly mobile and the magnetic anisotropy energy must be higher than the elastic energy needed for the twin boundary motion.

Based on our own experience with shape memory alloys and looking carefully through the literature reports, however, there seems to be another very important factor. An appropriate mechanical training done before the magnetic actuation experiment on an Ni–Mn–Ga single crystal is beneficial for the actuation (larger strain and lower critical magnetic field) [1, 7, 7, 13]. In this context, the experiments described in section 4 were all performed on samples trained by multiple successive compressive deformations applied on two different faces B and G of the cuboid sample (training B–G in figure 7(a)).

The twinning stress recorded during the training cycles (compression on face B in figure 7(e) right) significantly decreases with increasing number of training cycles. After the training, the magnetic field had always been applied perpendicularly to the compressive loading axis in the direction where the deformation was previously applied (along direction B in figure 7(c)). Magnetic field induced reorientation was observed (figures 2–5) and the crystal changed its shape as suggested in figures 7(a) and (c).

On the other hand, if such a trained crystal was magnetized along the third untrained direction, where the compressive deformation training was not applied (along direction R in figure 7(b)), the reorientation process was not observed. This seems curious since the drastic difference between the crystal responses during the magnetic loading shown in figures 1 and 8 is only due to the training. In fact, compared with the multiple compression in the B–G directions (figure 7(a)), the sample was given just two training cycles in the R–G direction (figure 7(b)) in order to create the nuclei of variant 2 (V2) observed as appearance of the $(002)_M$ reflection. This variant V2 was set to grow under magnetic field in the diffraction experiment. The sample was mounted to the holder and only a small stress of 0.2 MPa was applied only to hold it in place. Variant V2, however, although its nuclei were present in the sample before the test (schematically shown in figure 7(b) and measured in figure 8), did not grow and did not yield the strain as suggested in figure 7(d) under the increasing magnetic field. Only the magnetic moment rotation towards the direction of the magnetic field was observed (figure 8).

Why the reorientation did not occur in this last case can be explained based on the results of the neutron diffraction experiments. Recall that the sample is not a martensite single crystal (there is also a small volume fraction of the

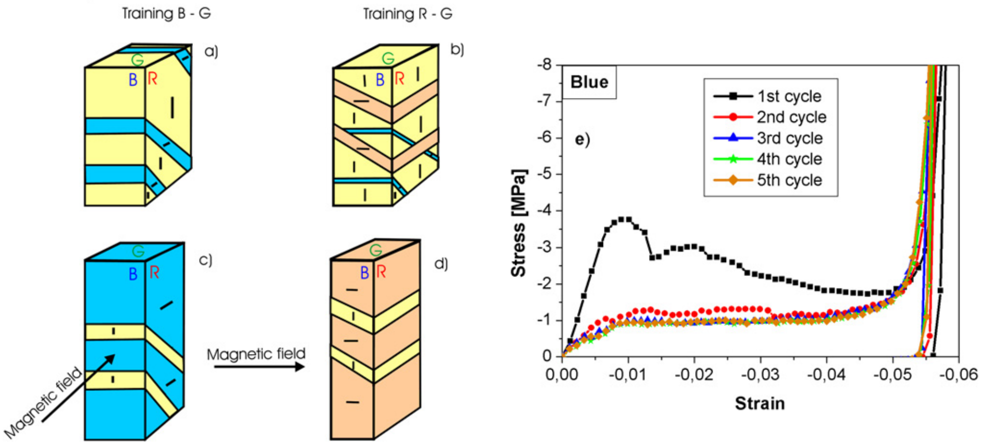

Figure 7. Schematic drawing showing the microstructure of the Ni–Mn–Ga single crystal given the compressive training B–G ((a), (e)) and R–G (b) and the effect of the magnetic field applied in the $[100]_M$ direction (c) and the $[010]_M$ direction (d) on it. (e) Stress–strain response of the crystal trained by multiple compression on faces B and G.

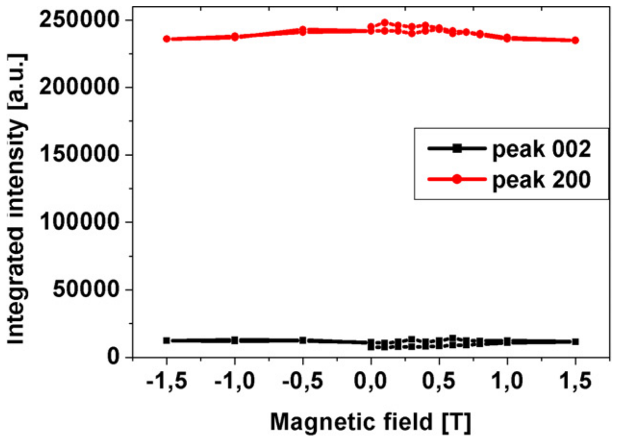

Figure 8. Variation of the integrated intensity of diffraction peaks 200_M (V1) and 002_M (V2) with magnetic field applied along the third untrained direction $[010]_M$ (geometry shown in figure 7(d)) measured by the *in situ* neutron diffraction experiment under applied stress 0.2 MPa.

second martensitic variant) before the experiment and that the magnetic field induced reorientation strain appears only when the magnetic field is applied in the direction parallel to the crystallographic *c*-axis of this minor martensitic variant (see figure 1). This variant with *c*-axis parallel to the magnetic field grows and becomes the major variant at maximum field. If we apply the magnetic field along direction R (figure 7(b)) along which the sample was not deformed before, the magnetic field is not oriented parallel to the *c*-axis of the minor martensitic variant and this is not thus expected to grow under the effect of magnetic field. Hence, a new twinning system is needed which will reorient the major variant in the favourite martensite variant with the *c*-axis parallel to the magnetic field. The existing minor variant may even act as an obstacle for the magnetically driven reorientation process (it increases the twinning stress).

It has been mentioned in the literature [7, 13] that the training is beneficial namely since it creates the nuclei (properly oriented minor variants) for the magnetically induced reorientation processes in the sample. This is probably true but is it only that? We succeeded in creating the nuclei (evidenced by the existence of the variant 2–$(002)_M$ reflection) by only two R–G training cycles before the last experiment but it did not help to achieve the magnetically induced reorientation (figure 8). More R–G training cycles to higher applied stresses might be necessary for this.

There is an analogy with the 'superelastic training' of shape memory alloys. The upper plateau stress of superelastic SMA materials decreases with increasing number of superelastic deformation cycles and, as a consequence of this, a partial two way memory strain appears in successive stress free thermal cycles [14]. The two way memory phenomenon can be hardly ascribed solely to the effect of martensite nuclei as the nuclei are wiped out during transformation; there must be something else which stabilizes the trained transformation paths (internal stress, dislocations, ?). A similar argument can also be put forward for the training of Ni–Mn–Ga single crystals by successive compression deformations. The more training cycles are performed the easier is the subsequent magnetically induced reorientation along the trained reorientation path, i.e. the reorientation occurs at lower magnetic field and results in larger strain.

Presented results thus show that a substantial training is essential for the crystal to exhibit the magnetic field induced variant reorientation. Training by just two cycles in the last experiment, although sufficient to create the nuclei of the minor variant destined to grow under the application of the magnetic field, was not sufficient to bring about magnetically induced reorientation and large strain (figure 8). There must be something else, in addition to the twin nuclei, which has been created during the compressive training, which assists the variant reorientation induced by the magnetic field applied along the proper crystal direction and prevents it when the field is applied along the third improper (untrained) direction. Results of the studies dedicated to the compressive training, particularly to the clarification of the physical mechanism behind it, are however beyond the scope of this paper and will be reported separately [15].

6. Conclusions

A neutron diffraction single-crystal method was applied to investigate the twinning processes driven by the application of the magnetic field to a Ni–Mn–Ga martensite single crystal exposed to bias compression stress. The essential advantage of the neutron diffraction method is that it provides relevant integrated information about the presence and volume fractions of individual martensite variants involved in magnetic actuation under external stress.

It has been found that (i) martensite variant reorientation is not completed by applying magnetic field as high as 2.5 T, (ii) recoverable strain of about 3% due to switching between two variant microstructures is observed upon cyclic application of magnetic field under external compression stress 0.9 MPa, (iii) the magnetic field induced reorientation was nearly fully suppressed by the bias external stress of 3 MPa and (iv) mechanical training by successive compression deformation on two different faces of the cuboid Ni–Mn–Ga single crystal is essential to achieve the magnetic field induced cyclic reorientation.

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