Reversible structural transition in monocrystalline Ni₂FeGa microwires for shape-memory applications

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ABSTRACT

We report on the production and characterization of Heusler-based Ni₂FeGa microwires exhibiting two – way shape memory effect. The microwires are characterized by a monocrystalline structure with a strong preferred crystallographic orientation that shows [1 1 1] axis parallel to the wire's axis for high-temperature L2₁ austenite phase, while the [0 1 7] axis is preferred for low-temperature monoclinic phase. Variation of crystallographic axis (and corresponding easy magnetization axis) leads to 1600% variation of magnetic permeability due to a 2% strain in axial direction. Such straining is reversible immediately after production without the necessity of further thermal treatment. These properties give the microwire function of very sensitive SMART actuators that can be easily produced in a large amount.

1. Introduction

Shape memory alloys play a role in many practical and commercial applications in engineering, medicine, aerospace, automobile, etc., in which their possibility to recover strain up to 10% is employed [1,2]. Among many others, Ni-Ti-based alloys exhibit high tensile strength, and an excellent superelastic strain which makes this material available for practical applications [3]. Apart from Ni-Ti-based alloys, huge attention is focused on shape memory alloys with ferromagnetic nature due to their potential application as SMART materials. They are ferromagnetic materials with strong magneto-crystalline anisotropy exhibiting shape memory effect [4]. Magnetic shape memory alloys exhibiting shape memory effect also allows simultaneous sensing of strain due to a strong variation of magnetic properties induced by phase transition [5–7].

Ni-Mn-Ga Heusler alloy with chemical composition near to stoichiometry 2:1:1 is a prototype of ferromagnetic shape memory alloys. This alloy is known for its large recoverable deformation up to 12% strain and high-frequency response (up to 1 kHz) induced by magnetic or

mechanical loadings [8,9]. However, in spite of its remarkable properties, Ni₂MnGa has some drawbacks originating from the loss of Mn, relatively low Curie and martensitic transformation temperatures, high brittleness, etc. [10]. Production of Mn-based alloys is quite a tricky job due to the Mn evaporation during melting. This leads to the complex production of such materials, in which the composition cannot be controlled precisely. Since the chemical composition is crucial for the amplitude and temperature span of shape memory effect, such behaviour disqualifies Ni₂MnGa material from massive production. Therefore, it is necessary to develop new alloy systems to overcome such a disadvantage.

The elegant solution of the problem with Mn deals with replacement of Mn for Fe [11]. Ni₂FeGa Heusler based alloy shows recoverable strain up to 10%. However, the production of homogeneous Heusler alloy is still complex work that should be followed by long-term high-temperature thermal treatment [12]. It has been shown previously that rapid quenching is a suitable method for the production of a large amounts of material with increased homogeneity [13]. One possibility of producing a large amount of high-quality samples in suitable form is Taylor-

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Ulitovsky method [14]. This method allows the production of glass-coated microwires from a small amount of master alloy (typically 1–2 g) by rapid quenching method. Glass-coated amorphous microwires offer various functionality for sensing applications [15–18]. Recently, Taylor – Ulitovsky method has been employed for the production of Ni_2MnGa – based microwires [19]. In some cases, the first order transition has not been observed due to the problem with Mn evaporation [20]. Usually, Heusler alloys based on Ni and Fe with a crystal structure are brittle. The glass improves the brittleness of the metallic core and provides biocompatibility in glass-coated microwires. Moreover, the Taylor Ulitovsky method allows production microwires with oligocrystalline structure [21,22], which increases the amplitude of strain. The martensite transformation morphology in shape memory alloys becomes more complex with increasing grain restriction. In unconstrained monocrystalline regions, the transformation is simple, single variant, and complete. On the contrary, near grain boundaries, the transformation is only partial, containing regions of untransformed austenite [23]. Metal core diameter comparable to the grain size leads to resolution of the problem with single crystal.

In the given contribution, the production and characterization of Ni_2FeGa glass-coated microwire is shown. Such a wire is characterized by monocrystalline structure along the entire length and structural transition in the temperature range from 250 to 292 K immediately after production (without the necessity of further thermal treatment), confirmed by structural, magnetic, and mechanical measurements. Mechanical measurement reveals a 2% recoverable strain due to the change of temperature under applied force 0.10. In addition, different forces lead to a change in the strain value, but the transformation temperatures remain unchanged. As a result of the strong variation of well-defined magnetic anisotropy, such strain is accompanied by a 1600% change of permeability. Such properties lead to a SMART actuator that is a sensor by itself. The Taylor-Ulitovski method of microwire production allows for the simple production of up to kilometres of microwire within few minutes (<10 min). Such microwire could be directly (without post-production thermal treatment or training) used in bioengineering, aerospace engineering, CNC machines, robotics, or in various unconventional actuators [24,25].

2. Materials and methods

Shape memory glass-coated microwires were fabricated by the Taylor-Ulitovsky technique. The master alloy is placed into the glassy tube, which is placed into the induction coil. High-frequency electromagnetic field melts the sample by eddy current until the alloy softens the glass. When the glass reaches the softening point, the glassy capillary filled-up with the metallic nucleus is drawn out by the wounding coil. Glass-coated microwires with the metallic nucleus having a radius from 100 nm to 150 μm and glass thickness from 2 to 100 μm can be easily produced.

The ferromagnetic Heusler-based master alloy Ni_2FeGa was prepared from high-purity elements (Ni = 99.9%, Fe = 99.9%, Ga = 99.9%) by arc-melting under a protective argon atmosphere. In order to achieve a good homogeneity, the ingot was re-melted four times.

The microstructure has been studied by scanning electron microscope (SEM) TESCAN VEGA 3 XMU, which includes the energy-dispersive X-ray spectroscopy (EDS). EDS analysis was used to investigate the chemical composition of the Ni_2FeGa microwire.

In-situ crystal structure behaviour during the heating of microwire has been studied by X-ray diffraction at P02.1 undulator beamline of PETRA III storage ring situated in synchrotron facility DESY, Hamburg with the $\lambda = 0.20727 \text{ \AA}$. Microwire was placed into boron capillary and heated from 90 K up to 560 K using a combination of liquid N_2 cryostreamer and hot air blower. Diffraction patterns were recorded by two-dimensional detector PerkinElmer 1621 (2048×2048 pixels; $200 \times 200 \mu\text{m}^2$; 16-bit) during the heating experiment.

Crystallographic orientation along the wire has been studied by SEM

TESCAN MIRA 3, which includes a detector to analyse back-scattered electrons. The glass was mechanically removed from the microwire by compressive stress perpendicular to the sample axis. The surface of the cross-section was polished by ion slicer. EBSD analysis was possible in a limited area that complied with the diffraction condition of back-scattered electrons. The EBSD measurements are presented in the form of inverse pole figure maps (IPF), indicating the crystallographic orientation of each individual point. The stereographic triangle next to the maps gives the colour - code employed in the maps. The texture analyses from the X-ray diffraction pattern was realized by Lutterotti's MAUD software [26].

The temperature dependence of magnetization was measured by SQUID magnetometer MPMS3 in the temperature range from 30 K to 400 K under a magnetic field of 50 Oe and 10 kOe. Hysteresis loops were measured at the temperatures 10 K and 400 K within the range of magnetic fields from –10 to 10 kOe.

Susceptibility measurements were done using LCR meter at frequency 1 kHz and a magnetic field of 2.6 Oe.

Finally, mechanical properties were studied by dynamic mechanical analyzer Q800 DMA in the temperature range from 175 to 375 K by applying various forces ($F = 0.01, 0.02, 0.05$, and 0.10 N).

3. Results and discussion

Glass – coated microwires are produced from master alloy prepared by arc melting of pure elements of the desired composition. The easy production process consists of induction melting of master alloy placed inside the SIMAX® glass crucible until the temperature of molten master alloy reaches the working point of glass [27]. Once the working point of the glass is reached, the glassy-capillary is drawn from the crucible, in which the molten master alloy enters. It is possible to control the diameter of the wire by the wounding speed of the wounding coil [19].

Various glass-coated Ni_2FeGa microwires were produced having a metallic nucleus with the diameter in the range from 30 to 160 μm covered by SIMAX® glass with thickness in the range from 30 to 110 μm (one of them is shown in Fig. 1). Chemical analysis (obtained by averaging the measurements at 4 points) shows almost excellent agreement with the desired composition being $\text{Ni}_{50.0}\text{Fe}_{25.6}\text{Ga}_{24.4}$ (at. %). It points to the fact that Ni–Fe–Ga composition is very appropriate for the reproducible production of microwires with the required chemical composition.

XRD experiment was performed in the temperature range from 90 K

Fig. 1. SEM image of glass-coated Ni_2FeGa microwire.

up to 560 K for the phase transition temperature determination. Collected diffraction patterns are seen in Fig. 2. The start and end of the phase transformation were determined by a simple method of changing the integral structure factor value. It is not necessary to identify a phase by mention method, and this makes the procedure easy and independent on knowing of crystallographic data to determine the temperature of structural transformations. The phase transition temperature range occurs between 252 and 292 K visibly from lower to higher crystallographic symmetry. The high-temperature phase was identified as cubic $L2_1$ ordered phase, and low-temperature phase was determined by the best match with the monoclinic “5M” martensitic phase published by authors [28].

Fig. 3 presents a very strong preferred crystallographic orientation that was observed in the form of spot diffraction patterns, which is documented by pole figures (PF) and inverse pole figures (IPF). PF and IPF showed that the strongest pole intensity becomes from (0 1 7) that crystallographic orientation [0 1 7] of the monoclinic martensite phase is parallel with the long wire axis. The next strong intensity parallel with the wire axis belongs to [1 2 1]. This type of texture is present throughout the entire temperature range of the martensite phase existence. Ordered $L2_1$ austenite also exhibits very strong texture with crystallographic direction [1 1 1] parallel to microwire long axis in the whole temperature range of the phase existence.

It can be assumed that the unit cell volume of microwire core increases with rising temperature, which is typical for metallic materials. Fig. 4 shows the lattice parameter evolution in the examined temperature range. The cubic lattice parameter of austenite is predictably increased. However, in the case of the monoclinic phase of the martensite, not all lattice parameters grow with temperature. It should be noted that the examined metallic core is covered and bonded by the glass during the production. Therefore, internal stresses can be assumed in axial and also radial direction due to different thermal expansion coefficient of the metallic core and covering glass. If it is taking into account the results from the texture analysis that the monoclinic c parameter is mostly oriented parallel ([0 1 7] direction) to the long wire axis, it is evident that the lattice expansion of c parameter is dampened. Decreasing of the lattice parameter b and also changing of the monoclinic angle β are most likely to indicate a compensation of the stresses in the radial direction. Nevertheless, the unit cell volume of martensite

calculated by the refined parameters increases with temperature (shown in the inset of Fig. 4).

To confirm the well-defined orientation of the crystalline structure, EBSD analysis was carried out on various randomly selected parts of the surface of the microwire without glass. Firstly, microstructural analysis in Fig. 5 shows the single crystal structure across the section of the wire. The orientation map characteristic for all pieces of Ni_2FeGa microwire obtained from EBSD measurements at room temperature is also shown in Fig. 5, in which the colour variants are coloured according to IPF in directions X, Y, and Z, being X parallel to the wire's axis. Here, the crystallographic orientation is given by the color-coded stereographic triangle next to the maps. Analysis of randomly selected pieces of microwire shows the same figures, which points to the monocrystalline structure of the entire microwire with [1 1 1] axis parallel to the wire's axis. Such preferred orientation can be understood in terms of the tensile stresses applied to the metallic nucleus during microwire production. During crystallization, the metallic nucleus undergoes strong axial stresses applied by the glass due to different thermal expansion coefficient of glass and metallic nucleus. The easiest way how to strain cubic crystalline structure (with the lowest variation of lattice parameter a) is to strain it in the direction of [1 1 1].

The temperature dependence of magnetization measured under a small magnetic field (50 Oe) shows an unusual increase of magnetization in the temperature span where the structural transition takes place (shown in Fig. 6). Application of high field (10 kOe) suppresses the variation of magnetization in this range. However, small discontinuity is still observed in the same temperature range. The reason why the structural transition is visible at low but not at high fields is hidden in the different anisotropy and saturation magnetization of both phases. While the low-temperature phase has slightly higher saturation magnetization, it also has a higher saturation field due to the higher anisotropy of low-temperature martensitic phase. It is clearly visible from hysteresis loops measured at 200 and 300 K as can be seen in Fig. 7. While saturation magnetization is lower for high-temperature phase, the magnetization below 375 Oe is higher for monocrystalline austenite.

Thick glass-coating hinders the strain variation due to the phase transition in glass-coated microwires. However, mechanical analysis (seen in Fig. 8) of as-cast Ni_2FeGa microwire after glass-removal shows reversible strain under various applied forces. The first cycle shows a 1.3% elongation of the microwire at a tensile force of 0.01 N. The second and third cycles show 1.45% and 1.55% elongation at 0.02 N and 0.05 N, respectively. The fourth cycle was control and served as a comparison to the first cycle. It can be seen that both curves overlapped, and therefore, no irreversible phase was induced during the martensitic transformation. Finally, the microwire was loaded with a force of 0.10 N, and the elongation reached 2%. All transformations are reversible and have the same transition temperatures (martensite is induced at the temperature 217 K, and austenite is induced at 252 K. This strain is reversible and does not decrease with training.

The difference in the anisotropy of both phases drastically affects their field dependence of permeability (shown in Fig. 9). Due to the high anisotropy, the martensitic phase shows low permeability. On the other hand, the field dependence of permeability of austenite is higher, showing a sharp maximum at low fields [29]. Using properly selected amplitude of the excitation field (2.6 Oe in our case), huge variations of permeability can be achieved by phase transformation. As a result, Ni_2FeGa microwire shows a 1600% increase of permeability within the range of martensitic phase transformation. Such variation gives to microwires additional functionality to serve as a very sensitive sensor of strain, which makes them SMART actuators.

4. Conclusion

We have studied the structural, magnetic, and mechanical properties of shape memory Ni_2FeGa microwire with monocrystalline structure. The chemical composition of the as-cast Ni_2FeGa alloy is in excellent

Fig. 2. Collected diffraction data (up) and calculated change of the integral value of the structure factor (bottom) in dependence of temperature during heating.

Fig. 3. Pole figures and inverse pole figures of microwire Ni_2FeGa showed very strong preferred crystallographic orientation of austenite (top) and also martensite phase (bottom).

Fig. 4. Lattice parameters evolution of the phases in temperature range 100–550 K confirm the structural transition between 250 and 300 K accompanied with the shape memory effect.

Fig. 5. Cross section analysis showing single crystal structure of microwire (right up), EBSD analysis of Ni_2FeGa microwire (left) and sketch of microwire with preferred orientation of austenitic cubic phase with $[1\ 1\ 1]$ axis parallel to the wire's axis (right down).

Fig. 6. Temperature dependence of magnetic moment for Ni_2FeGa at 50 Oe and 10 kOe during heating.

Fig. 7. Hysteresis loops of glass-coated Ni_2FeGa microwire measured for low (200 K) and high (300 K) temperature phases.

Fig. 8. Dynamic mechanical analysis of as-cast Ni_2FeGa microwire after glass-removal measured under various forces.

Fig. 9. Temperature dependence of strain measured under constant force 0.10 N (purple line) and temperature dependence of initial permeability $\mu/\mu_{\min}$ (black line) measured under magnetic field 2.6 Oe.

agreement with the desired composition, which points to the very good repeatability of sample production.

XRD analysis shows the structural transition from monoclinic structures at low temperatures (below 250 K) to high-temperature austenite cubic $L2_1$ phase (above 290 K). The texture analysis from the X-ray diffraction pattern shows a very strong preferred crystallographic orientation for both phases. The EBSD analysis confirmed the martensite phase with crystallographic orientation $[0\ 1\ 7]$ parallel to the wire's axis, while the austenite exhibits strong texture with crystallographic direction $[1\ 1\ 1]$ parallel to the axis of the microwire.

Magnetic measurements reveal an unusual increase of magnetization measured at low fields (50 Oe) due to the decrease of magnetic anisotropy during phase transformation to the austenitic phase. Hysteresis loops measured in the parallel direction to the wire's axis show that austenite has an easy axis parallel to the wire axis, while the easy axis of the martensitic phase is tilted out of the wire's axis.

The structural transition is accompanied by 2% reversible strain in the microwire axis under the applied force $F = 0.10$ N. We have also performed cyclic measurement that shows reversibility under various force applied. As the force increases from 0.01 N to 0.1 N, the strain increases from 1.3 to 2%, but the transition temperatures remain unchanged. At different tensile forces, the strain is reversible and does not decrease with training.

Due to the different amplitude and direction of magnetic anisotropies

of both phases, such straining results in 1600% variation of initial permeability measured under correct conditions. Such straining was obtained without any additional thermal treatment or training.

The observed properties of the magnetic microwire Ni_2FeGa make this material SMART for practical applications like actuator and sensor, simultaneously in bioengineering, aerospace engineering, CNC machines, robotics, etc. Additionally, magnetic microwires coated by glass provide several further advantages: easy and fast production, low brittleness, biocompatibility, reduced geometrical dimensions connected with the miniaturization of magnetic sensors, and SMART actuators.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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