

## MAGNETIC PROPERTIES OF NANOCRYSTALLINE BISTABLE FeNiMoB MICROWIRES

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### ABSTRACT

The structure evolution of the annealed amorphous microwires of composition  $Fe_{40}Ni_{38}Mo_4B_{18}$  and its interplay with magnetism has been studied. It has been shown by x-ray diffraction, that primary crystallization phase has a microstructure of  $\gamma$ -(Fe,Ni) crystallites of about 10 nm embedded in a residual amorphous matrix. Changes of the saturation magnetization and the switching field after different thermal treatments were observed. The dependence of the switching field on the applied mechanical stress was investigated.

**Keywords:** glass-coated magnetic microwire, amorphous alloy, nanocrystalline material, magnetic bistability, switching field, tensile stress

### 1. INTRODUCTION

Amorphous and nanocrystalline ferromagnetic microwires covered by a glass coating are attracting a great interest, due to their large potential for technical applications. This is a consequence of their outstanding magnetic characteristics (for example cylindrical symmetry, strong magnetic shape anisotropy and magnetic bistable behavior for microwires with positive magnetostriction), small dimensions and reliability on electrical, mechanical and corrosive external effects due to the Pyrex coating layer.

These microwires, prepared by quenching and drawing technique, consist of a magnetic nucleus (with diameter from 1-30  $\mu\text{m}$ ) coated by a Pyrex-like glass (thickness of 2-20  $\mu\text{m}$ ). Due to their amorphous nature, magnetoelastic and shape anisotropy contributions govern their magnetic properties. As a result of above-mentioned anisotropies, the domain structure of microwires with positive magnetostriction consists of single axial domain and their magnetization process is characteristic of the bistable behaviour [1, 2].

The nanocrystalline microwires have been subject of different studies starting from Finemet based microwires [3-7], as well as the Nanoperm based ones [8]. Such nanocrystalline microwires are usually prepared by the heat treatment. However, the microwires undergo strong stresses during the annealing due to the different thermal expansion coefficients of the metallic nucleus and glass coating. As a result, a  $\gamma$ -Fe phase appears in the Finemet based microwires instead of desired  $\alpha$ -(Fe,Si) phase [6, 7]. When the annealing is performed under high pressure, the formation of  $\gamma$ -Fe phase is energetically more favorable due to its higher pack density. Presence of  $\gamma$ -Fe crystallites could be the reason for the observed magnetic hardening in Finemet-type nanocrystalline glass-coated microwires.

Therefore we are looking for new chemical composition in which the problem will be solved. It has been recently shown that partial crystallization of  $Fe_{40}Ni_{38}Mo_4B_{18}$  ribbon leads to the formation of nanocrystalline material with  $\gamma$ -(Fe,Ni) nanocrystals [9].

Since the  $\gamma$ -(Fe,Ni) phase is ferromagnetic in contrary to the paramagnetic  $\gamma$ -Fe phase,  $Fe_{40}Ni_{38}Mo_4B_{18}$  alloy is promising candidate for preparation of soft magnetic nanocrystalline microwires.

### 2. METHODS

The study has been performed on glass-coated amorphous microwires with nominal composition  $Fe_{40}Ni_{38}Mo_4B_{18}$  prepared by the Taylor-Ulitovsky method. The diameter of the metal core was 8  $\mu\text{m}$  and the total diameter 12  $\mu\text{m}$ . In order to obtain nanostructured specimens, the amorphous microwires were annealed for 1 h at various temperatures  $T_a$  from 520 K to 820 K in helium atmosphere.

Microstructure of the annealed material was analyzed by x-ray diffraction. High-energy x-ray diffraction (XRD) measurements were performed at HASYLAB at DESY (Hamburg, Germany). The new sample was annealed for each X-ray measurement.

The saturation magnetization  $M$  was measured by the VSM magnetometer at the applied field of 1T. The same sample was used for magnetization measurement and four subsequent runs were performed in the temperature range of 4 - 800 K. The heating and cooling rate was 10 K/min.

Hysteresis loops were measured by the SQUID. The switching field  $H_{sw}$  has been measured at room temperature by the induction method [10], using triangular waveform at the frequency 100 Hz. Stress dependences of the switching fields were estimated for tensile stresses  $\sigma$  up to 140 MPa. The stress was applied on the annealed sample at room temperature.

### 3. RESULTS

As shown by x-ray diffraction, a primary crystallization process resulted into formation of  $\gamma$ -(Fe,Ni) nanocrystallites embedded in an amorphous matrix. Small precipitations of  $\gamma$ -(Fe,Ni) phase is indicated after annealing of the microwire at the temperature above 620 K. The intensity of observed peaks increases with the

annealing temperature up to 700 K. Grains of  $\gamma$ -(Fe,Ni) have size around 10 nm [11].

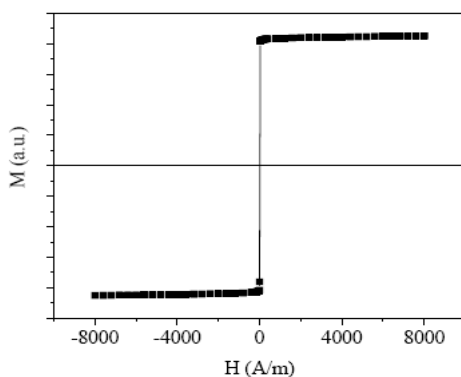
Since partial crystallization of initially amorphous microwire results into formation of ferromagnetic  $\gamma$ -(Fe,Ni) phase, its presence and temperature behavior can be also studied by measuring the temperature dependence of magnetization. Curie temperatures of the amorphous  $T_C(am)$  and crystalline  $T_C(cr)$  phases were obtained by the analysis of the thermomagnetic curves. The results are summarized in Tab.1.

**Table 1** The Curie temperatures of amorphous  $T_C(am)$  and crystalline  $T_C(cr)$  phase of  $Fe_{40}Ni_{38}Mo_4B_{18}$  microwire after different thermal treatments [11]

$T_a$ (K)	570	620	670	720	770
$T_C(am)$ (K)	696	672	621	623	622
$T_C(cr)$ (K)			739	735	724

Annealing at 670 K leads to the crystallization of  $\gamma$ -(Fe,Ni) phase, which is accompanied with a decrease of the Curie temperature of the amorphous phase, since the concentration of the Fe and Ni atoms in the amorphous phase decreases. The Curie temperature of the crystalline phase was found to be 739 K. contrary to the x-ray measurements, where annealing above 700 K leads to the disappearance of the crystalline phase, the thermomagnetic analysis shows further increase of the crystalline volume fraction that is accompanied by the increase of the magnetization above the Curie temperature of the amorphous phase. The Curie temperature of the amorphous phase is not affected by the further annealing, however the Curie temperature of the crystalline grains decreases slightly. The difference between x-ray and thermomagnetic measurements could be explained by the fact that in the case of magnetization measurements, the same sample was used.

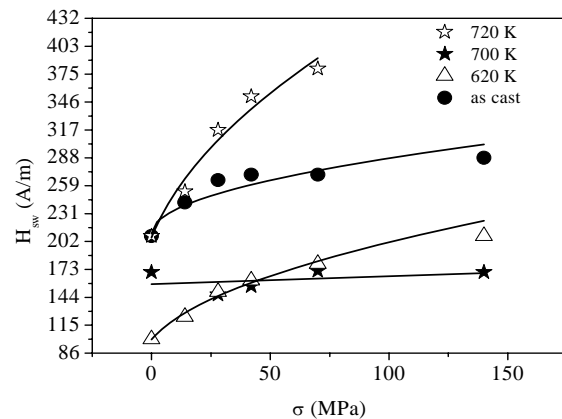
Hysteresis loop measurements exhibit bistability in the amorphous as well as in nanocrystalline  $Fe_{40}Ni_{38}Mo_4B_{18}$  microwires (Fig. 1).



**Fig. 1** Hysteresis loop of the nanocrystalline  $Fe_{40}Ni_{38}Mo_4B_{18}$  microwire annealed at 700K

Switching field  $H_{sw}$  in bistable microwires is mainly governed by the magnetoelastic interaction of the magnetic moments and the stresses induced during the microwire production. These cooling residual stresses can

be reduced by thermal treatments or external stresses  $\sigma$  applications.



**Fig. 2** Stress dependence of the switching field  $H_{sw}$  in  $FeNiMoB$  microwire after different thermal treatments [12]

As-cast sample shows strong dependence of the switching field on the applied stress  $\sigma$  where the switching field increases with the applied stress (Fig. 2). Annealing of the microwires at low temperatures causes the release of the internal stresses introduced during microwires production. This results in the decrease of the switching field as well as in the decrease of its stress dependence. The minimum value of the switching field was observed for the annealing temperature  $T_a = 620$  K. In the case of nanocrystalline  $Fe_{40}Ni_{38}Mo_4B_{18}$  microwire annealed at 700 K, the switching field shows no stress dependence, probably as a result of very low magnetostriction. On the other hand, the switching field does not reach the minimum value. In the case of glass-coated microwire, one should take into account the stresses introduced by the glass coating (as a result of the different thermal expansion coefficient of the glass and metallic nucleus) during cooling after annealing. The annealing at the temperature above the optimum (700K) annealing temperature ( $T_a = 720$  K) leads to the strong stress dependence of the switching field in the nanocrystalline  $Fe_{40}Ni_{38}Mo_4B_{18}$  microwire. The diameter of the grains increases and the therefore the switching field increases, too. It is known, that the coercivity of the nanocrystalline magnetic materials increases with the diameter of the crystalline grains ( $D$ ) following the  $D^6$ -law [9]. Moreover, after the treatment at  $T_a = 770$  K, the samples become brittle and the maximum applied stress can be no higher than 30 MPa. In generally, the switching field is proportional to the square root of the applied stress, which corresponds to the nucleation-propagation model of the coercivity mechanism, which is valid in the case of amorphous and nanocrystalline microwires [12].

#### 4. CONCLUSIONS

We demonstrate that  $Fe_{40}Ni_{38}Mo_4B_{18}$  alloy composition can be considered to prepare soft magnetic nanocrystalline microwires with positive magnetostriction.

Such microwires exhibit bistability even in the nanocrystalline state.

At low annealing temperatures, the strong switching field dependence on the stress can be obtained, which is useful for stress sensing elements. Close to the optimal annealing temperature (700 K), no stress dependence of the switching field was detected. Such behaviour is useful for the different sensing elements (temperature, current), which must not be stress sensitive.

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