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Research articles

The role of structural properties on magnetic characteristics of glass-coated microwires

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ABSTRACT

We report on the influence of crystalline structure on the magnetic properties of glass-coated microwires with CoFe-based metallic core realised as an amorphous matrix with nanocrystals. Using X-ray diffraction analysis and transmission electron microscopy, we have examined the structural features of the core, and demonstrated a possibility to tailor magnetic response of microwires by changing their crystalline structure. Finally, we present the first attempt to "visualise" stress distribution in the core of microwires, using crystal orientation imaging obtained by transmission electron microscopy.

1. Introduction

Ferromagnetic microwires are among the most promising materials for different applications attractive for technology miniaturization. They show a number of outstanding effects depending on composition (Fe-, Co-, CoFe-based) and crystal state (amorphous, nanocrystalline or crystalline) of the ferromagnetic core [1,2]. These effects include magnetic bistability [3] used in coding, logic, and memory systems (for example, [4,5]), magnetoimpedance effect [7,8] used in high-performance sensors [6,9], shape memory and magnetocaloric effects [10,11] for magneto-mechanical actuators [12]. The ability to produce partially crystalline microwires was demonstrated several years ago [13,14]. For obtaining nanocrystals in amorphous matrix different methods were used, for example, adding new components to initial alloy [13], or changing the manufacturing conditions [14]. A partially crystallised structure was obtained when copper was added to the initial alloy (CoFeSiB) for manufacturing microwires [13] as well as by varying velocities of cooling and extraction of microwires during fabrication process [14]. The possibility to switch magnetisation of different phases by varying magnetic field strength leads to a step-wise hysteresis loops. Magnetic properties of microwires which can be controlled and tuned in this way, are used for sensing and logical devices [15–17].

To tune and control magnetic properties of glass coated microwires effectively, it is necessary to determine and control experimentally radial distribution and magnitudes of mechanical stress in metallic core of glass-coated microwires – one of major parameters which define micromagnetic structure.

Manufacturing process leads to internal mechanical stress in the metallic core of microwires which have different origin [18–21]: (i) solidification stress, (ii) stress arising due to the difference of thermal expansion coefficient for metal and glass, (iii) stress arising due to the drawing process. Co-existence of several types of internal stress leads to certain distribution of the stress components (radial, axial and cylindrical) along core radius. Significance of the internal mechanical stress is evident from measuring magnetic properties under applied stress or after microwire annealing [23]. Albeit there are some theoretical calculations for the magnitude and distribution of these stress components [22], they have never been, to the best of our knowledge, experimentally assessed for amorphous microwires. In case of crystalline microwires the magnitude and distribution of initial stresses components cannot be estimated, too. To achieve a required crystal structure in metallic cores of the microwires produced from, for example, Heusler alloys, annealing should be used. All the three stress types obtained during manufacturing are removed in the process of annealing, and they cannot be restored.

To overcome these difficulties, we propose to use a partially crystalline metallic core of microwires as a convenient material to study distribution of stress components, because orientation of

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nanocrystals formed during fabrication should be sensitive to magnitude and orientation of mechanical stress.

The purpose of this work is twofold: (i) to explore the effect of partial crystallisation in the metallic core on the magnetic properties of microwires, and (ii) to develop an approach to visualise internal stress in the core, using microwires consisting of an amorphous matrix with crystalline inclusions.

2. Experimental details

We investigated glass-coated $Co_{73.7}Fe_{3.8}B_{15}Si_{7.5}$ and $Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ microwires with metallic core diameter, *d*, of 17 μm and 24.6 μm, and total microwires diameter, *D*, of 23.6 μm and 33 μm, respectively. All samples were manufactured by quenching and drawing method [18] in the laboratory of Prof. M. Vazquez in the Institute of Material Science, Madrid. Changing technological parameters, such as a drawing velocity and cooling conditions, enables production of microwires with the nanocrystals inside the amorphous matrix [14].

The crystalline structure of the microwires was investigated by X-ray diffraction analysis (XRD) with Cu Ka radiation and transmission electron microscopy (TEM) using JEM2100 LaB6 microscope operating at 200 kV. A sample for XRD measurements comprised an 1 cm^2 area fully covered with microwires. Thin foils for TEM analysis were prepared from microwires by focused ion beam technique with Ga^+ ions milling and standard lift-out procedure. Using TEM in dark field regime provided the first visual impression about stresses distribution.

The Lake Shore vibrating sample magnetometer (VSM) series 7404 was used for evaluating magnetostatic properties at room temperature and for measuring temperature dependences of magnetic moment in the range from 300 K to 1000 K. Heating rate was 2 K per minute. The length of the samples was 5 mm. After complete cooling, hysteresis loops were once again measured at room temperature.

3. Results and discussion

3.1. Structural properties

Fig. 1 represents the XRD spectra for microwires with core compositions of $Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ and $Co_{73.7}Fe_{3.8}B_{15}Si_{7.5}$. Both spectra consist of a halo originating from the glass, amorphous halo from the amorphous phase and sharp peaks from crystalline phases, which are marked. The intensity of crystalline phase in the spectrum for $Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ is almost negligible indicating very small volume fraction of crystalline phase in the material. The intensity of reflections from the crystalline phase is much higher in the XRD spectrum of $Co_{73.7}Fe_{3.8}B_{15}Si_{7.5}$ microwire. The peaks on XRD spectrum are produced by fine crystals whereas the amorphous halo is almost undistinguishable in the background formed by an overlap of crystalline peaks at the same angles. This indicates a high volume fraction of fine crystals (crystalline phase) in the microwire. The peaks can be attributed to the Co-based phase of FCC and Hexagonal closed packaged (HCP) modifications. Some peaks cannot be attributed to any crystalline phase of the studied system and their origin is not clear. Further analysis of the microstructure was done by TEM.

The TEM micrograph of the $Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ microwire is represented in Fig. 2. Fig. 2a depicts selected area diffraction (SAED) which represents an amorphous halo. The dark-field observation in the first ring reflection (Fig. 2b) allowed us to find a fine crystal in the amorphous matrix. Only one 10 nm crystal was found in the inspected area. This makes a good agreement with XRD data

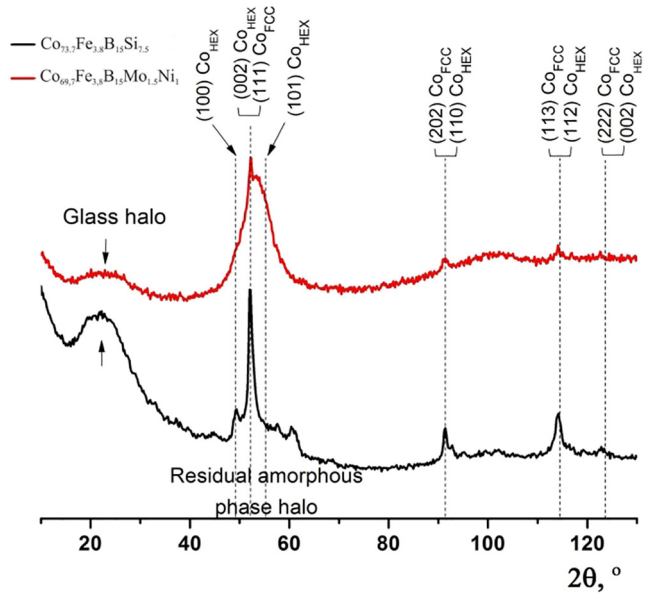

Fig. 1. XRD spectra for microwires with core compositions of $Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ (red spectrum) and $Co_{73.7}Fe_{3.8}B_{15}Si_{7.5}$ (black spectrum).

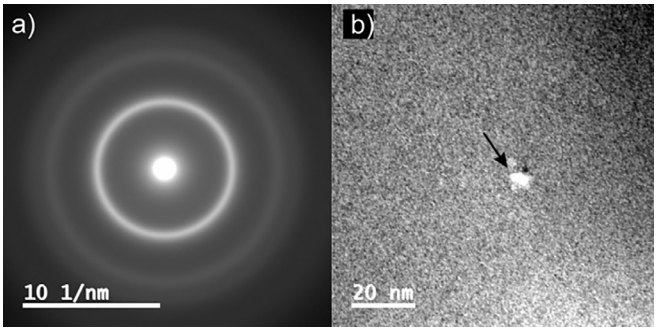

Fig. 2. a) A SAED from the $Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ microwire and b) subsequent dark-field image obtained in the first diffraction ring. The sample demonstrates amorphous structure, only one fine crystal was found in the inspected area.

indicating almost fully amorphous state of the $Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ microwire.

The TEM micrographs of the $Co_{73.7}Fe_{3.8}B_{15}Si_{7.5}$ sample are shown in Fig. 3. The bright field images at different scales (Fig. 3a, b) indicate a presence of fine elongated crystals (50–100 nm) with many planar defects inside. The SAED obtained from the area in Fig. 4a, represents a polycrystalline diffraction pattern with reflections corresponding to both FCC and HCP Co modifications. The presence of stacking faults of closed packaged {1 1 1} planes in originally FCC Co, lead to the appearance of thin HCP crystals inside. Thin HCP crystals cause long streaks in the SAED. The dark field image taken in the first ring demonstrates an existence of fine crystals (black and white) as well as almost discontinuous net of amorphous phase (grey – indicated by arrow). The volume fraction of amorphous phase estimated by grey color area is 30%, in a good agreement with XRD.

3.2. Magnetic properties

Normalised hysteresis loops of as-cast samples are shown in Fig. 4. The step-wise hysteresis loop in Fig. 4a ($Co_{69.7}Fe_{3.8}B_{15}Mo_{1.5}Ni_1$ microwire) is typical for biphase microwires [13,24]. The rectangular part of the hysteresis loop near 0 Oe corresponds to the

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