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Domain imaging in FINEMET ribbons

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ABSTRACT

The magnetization behaviour of a ferromagnetic material depends on its domain structure, which in turn is largely determined by magnetic anisotropies. In this work, domain patterns were observed by a quite forgotten but still the simplest and the cheapest technique: the Bitter method. A systematic study of the evolution of the domain structure in FINEMET ribbons after thermal annealing is presented, correlating the results with the crystalline structure, magnetostriction and coercivity measurements.

1. Introduction

There has been a great scientific and technological interest on the development of magnetic materials for more than a century.

In 1988, Yoshizawa et al. [1] opened a whole new area for research and application purposes by developing the nanocrystalline alloy, the so-called FINEMET ($Fe_{73.5}Si_{13.5}$ $B_9Nb_3Cu_1$), which due to its special two phase structures, i.e., crystals 10–20 nm size embedded in an amorphous matrix exhibits extremely soft magnetic properties (low coercivity $H_C \leq 1$ A/m and high permeability ($\mu_{r(1\ kHz)} \geq 10^4$) together with high saturation magnetization. Such properties make them suitable for a wide variety of technological applications such as transformer cores, inductive devices, magnetic shielding, sensors, etc. [2].

The extrinsic magnetization behaviour of ferromagnetic materials depends strongly on their domain structure, which in turn is largely determined by magnetic anisotropies. However, the knowledge of such structure was not exhaustively considered on the development of soft magnetic materials. Two other magnitudes that play an important role on the domain structure are magnetostriction (which relates stresses with domains orientation) and coercivity (which indicates the mobility of domain walls).

Therefore, it was of great practical interest to the authors to characterize and understand FINEMET's domain structure together with the crystalline structure, magnetostriction and coercivity, not only in the as-quenched but also in the annealed state at 540 and 650 °C.

Nowadays, new imaging methods such as MFM (magnetic force microscopy) [3] and MOKE (magneto-optical Kerr effect) [4,5] became very popular. However, on the one hand, these techniques are neither suitable for studies of rough surfaces nor to sharply disclose maze structures present in these ribbons. On the other hand, latest advances in digital imaging techniques made the old and almost forgotten Bitter method much more practical and powerful.

The evolution of FINEMET's domain patterns with heat treatments at different temperatures was observed with this method and compared with magnetostriction and coercivity variation.

2. Principles of Bitter method

This is the oldest domain imaging technique which owes its name to Francis Bitter, who reported it in 1931 [6]. Although he was not able to deeply understand the observed patterns, some conclusions could be stated: domains were static, they could be rather wide and had usually a periodic appearance.

Although it was discovered almost eight decades ago, it is still the simplest method. The magnetic surface is covered by a very thin and uniform layer of ferrofluid, i.e. a colloidal suspension of Fe_3O_4 particles, which can be home-made or commercial (e.g. ferrofluids from Ferrofluidics Corp., Nashua, New Hampshire, and lignosite FML from Georgia Pacific Corp., Tacoma, Washington).

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The particles collect and agglomerate in regions where stray fields are present, typically on the domain walls. Therefore, with this technique it is not possible to directly observe domains as with MOKE, but only domain walls. It is often possible to indirectly determine the domains, but not the magnitude of the magnetization or its direction. Then, the decorated domain walls can be observed with a direct or inverse optical microscope. New advances on digital imaging techniques turn this traditional technique into a more practical and powerful tool. A weak field (~50 mT) may be perpendicularly applied in order to enhance the image contrast, but it should be done carefully so that it will not change the sample magnetostructure. In fact, the applied fields (with magnets or electromagnets) should be used to clearly distinguish magnetic domain patterns from the topography of the sample.

Since with the Bitter technique it is only necessary to wait for the colloid to stabilize for few minutes, the acquisition time is rather fast compared with MFM, which can take half an hour per image. However, it is not as fast to observe dynamic domain behaviour as with MOKE. The resolution depends on the quality of the colloid and the resolution of the microscope (typically 500 nm), and it is better than MOKE (1000 nm) but not as good as MFM (100 nm) [7]. A disadvantage is that it is a destructive technique: after the domain observation, a dirty layer will remain on the sample, which is difficult to remove. This might be a problem for some samples as for instance, thin layers fabricated by sputtering. Still, it remains the simplest and the cheapest technique for domain imaging.

3. Experimental

A FINEMET ($Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$) master alloy was prepared in an induction furnace. Ribbons 10 mm wide and 20 μm thick were obtained from these ingots by planar flow casting technique in air. The chemical composition was checked by inductively coupled plasma spectroscopy.

The samples were studied in their as-quenched form, as well as in an annealed state at 540 and 650 °C (1 h). The annealings were performed in vacuum in an electric resistance furnace.

X-ray diffraction (XRD) was performed using a HZG 4 with graphite monochromator Cu $K\alpha_1$ radiation.

A special device for direct measurement of magnetostriction (λ_s), designed and constructed at the Institute of Physics SAS [8], was used.

Coercivity values were obtained using a quasistatic fluxmetric method by applying a longitudinal magnetic field to the sample.

Domain observations with the Bitter technique were carried out with the set-up shown in Fig. 1 with an inverted microscope in the absence of applied magnetic field. A cover glass was used for two purposes: to spread the very small ferrofluid drop on the sample surface and to avoid the rapid drying of the colloid. The samples did not require any surface preparation.

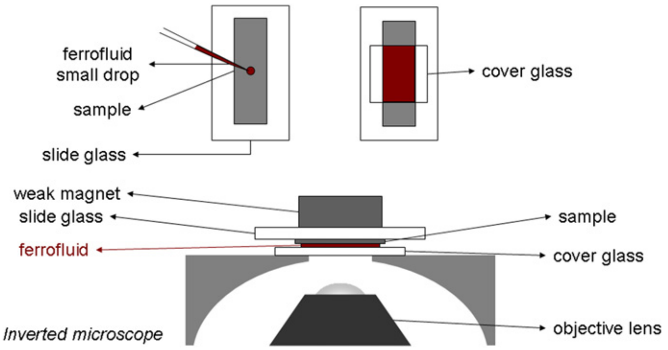

Fig. 1. Experimental set-up used for the Bitter technique.

4. Results and discussion

4.1. As-quenched ribbons

The amorphous structure of the rapidly quenched ribbons was checked on both sides by XRD (Fig. 2). Because of the random atomic arrangement, local magnetocrystalline anisotropies average out over large atomic distances, as explained by the random anisotropy model (RAM) [9]. Nevertheless, there is still an exchange coupling between the local moments that causes a long-range magnetic order. Thus, the amorphous material has good soft magnetic properties resulting in a very low coercivity value: $H_C=(3\pm2)$ A/m. Despite the lack of crystalline anisotropy of these materials, well defined domain patterns with locally fluctuating easy axes are observed as a result of residual anisotropies produced by internal stresses. The fabrication process is responsible for these stresses since there are differences in the quenching speed between the air and the wheel side, and at the same time, air bubbles are trapped between the ribbon and the wheel during the casting. The magnetization is coupled to the stress by the magnetostriction constant (λ_s). A value of λ_s equal to $(19\pm3)\times10^{-6}$ was measured. The disordered magnetic microstructure of the as-quenched FINEMET is shown in Fig. 3 with the corresponding scale and ribbon axis (which are rather important for domain pattern analysis but not always shown [10]).

Two different kinds of patterns were observed: (i) wide curved in-plane domains with 180° walls, i.e. Bloch walls, product of dominating tensile stress for the positive magnetostriction and (ii) narrow fingerprint domains. These fingerprints are also called “stress patterns” (even though both (i) and (ii) are due to stresses in the material), “stripe” or “labyrinth”. They are closure domains of underlying perpendicular domains, caused by planar compressive stress that induces an easy axis perpendicular to the surface. At increasing perpendicular anisotropy, i.e. stress level, the energy of the hard-axis closure domains rises and the “branching” mechanism appears in several “generations” (zoom in Fig. 3(b)). This occurs by a continuous modulation of the magnetization in a three-dimensional way [11]. Zig-zag walls are usually

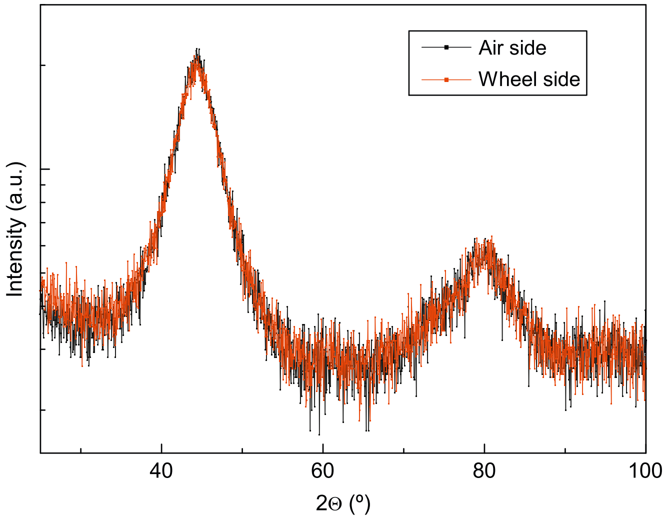

Fig. 2. X-ray diffraction pattern of as-quenched ribbon corresponding to the air and the wheel side.