Journal of Magnetism and Magnetic Materials 324 (2012) 569–577

Contents lists available at SciVerse ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Surface microstructure and magnetic behavior in FeSiB amorphous ribbons from magneto-optical Kerr effect

O. Životský [a,*], A. Hendrych [a], L. Klimša [a], Y. Jirásková [b], J. Buršík [b], J.A.M. Gómez [c], D. Janičkovič [d]

[a] Department of Physics, Technical University of Ostrava, 17.listopadu 15, 708 33 Ostrava-Poruba, Czech Republic
[b] Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Žižkova 22, 616 62 Brno, Czech Republic
[c] Department of Physics and Astronomy, Division of Nuclear Material Physics, Ghent University, Proeftuinstraat 86, 9000 Ghent, Belgium
[d] Institute of Physics, Slovak Academy of Sciences, Dúbravská cesta 9, 845 11 Bratislava, Slovakia

ARTICLE INFO

Article history:
Received 3 May 2011
Received in revised form
27 July 2011
Available online 3 September 2011

Keywords:
Surface magnetism
Magnetooptic Kerr effect
Magneto-optical microscopy
ILEEMS and CEMS
Nanoscale phase separation

ABSTRACT

The magneto-optical Kerr effect (MOKE) completed by other surface sensitive methods as integral low-energy and conversion electron Mössbauer spectroscopy, scanning and transmission electron microscopy and by X-ray diffraction have been used with the aim to trace the surface microstructure and magnetic properties of FeSiB amorphous ribbons prepared by planar flow casting. The general composition of studied samples is $Fe_{80}Si_xB_{20-x}$, where $x=4$, 6, 8, 10 at.%.

It is shown that MOKE used for magnetization, hysteresis loop, and domain structure determination is highly beneficial in a detection of both surface crystallization and local ordering of atoms into magnetically different clusters of amorphous structure. Moreover, a combination of blue and red laser with different penetration depths yields unique results concerning the surface anisotropy and depth sensitivity. In the case of samples with 4, 6, and 8 at.% Si MOKE detects two magnetically different phases diverging in coercivity values H_c, however, not varying with the sample composition. These phases have been identified by Mössbauer measurements as FeSi and FeB clusters. Their relationship changes with Si concentration. On the other hand, a strong increase in the surface H_c found for the sample with 10 at.% Si has indicated a nanocrystallization. It was confirmed by electron microscopy, Mössbauer and X-ray diffraction results. The size of nanocrystals has varied between 200 nm and 500 nm.

1. Introduction

Since the discovery of amorphous materials, their microstructure and magnetic properties have been extensively studied because of great interest for fundamental investigations and important potential in technological applications [1–3]. The planar flow casting is one of the possibilities to produce amorphous alloys in the form of thin ribbons. As the quenching rate slightly changes along the ribbon thickness, the ribbon can show different microstructure and magnetic properties at its surfaces with respect to the bulk as well as between both surfaces due to technology alone. The understanding and checking the surface magnetic properties in these materials are important in order to obtain magnetic devices with improved performance namely in the field of high frequency applications. Therefore, deeper knowledge of magnetic phenomena, i.e. magnetization process in the surface of amorphous, eventually nanocrystalline, material still belongs to the topic of present interest.

The surface magnetism can be effectively characterized by magneto-optical Kerr effect (MOKE) that is also used for imaging domains and magnetic singularities. The first system studied was an ultrathin Fe film epitaxially grown on the Au single crystal and the results were published approximately 25 years ago [4,5]. Since then MOKE has became a frequently used technique predominantly in physical characterizations of thin films produced by various technologies [6–8], multilayered systems [9,10], wires and microwires [11–14], and somewhat less also in studies of the amorphous and/or nanocrystalline ribbons [15–17].

The basic principle of Kerr effect is the interaction between the incident light and the magnetized surface which results in a rotation of the plane of polarization. The amplitude of this rotation is proportional to the sample magnetization. The surface thickness, from which the information about magnetic interactions is obtained, depends on the penetration depth of the light. It is directly proportional to the light wavelength and inversely proportional to the absorption coefficient of the sample material. Beside the wavelength the light penetration depth can be varied also by changing the incident angle. An important property for surface investigations is the so called magneto-optic additivity. Using MOKE two or more magnetically different phases can be detected and visually distinguished at measured hysteresis loops. Methods for separation of magnetic contributions from different depths for various materials were proposed by Hamrle et al. [9]

* Corresponding author.
E-mail address: ondrej.zivotsky@post.cz (O. Životský).

0304-8853/$ - see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.jmmm.2011.08.037

using a Babinet–Soleil compensator and by Postava et al. [18,19] using numerical linear combinations of measured signals. Recently, this approach is often generalized to selectivity from different materials in nanostructures [20].

The magnetization processes can be better understood by studying the magnetic domain structure yielding substantial information on magnetization and anisotropy distributions. Indeed, the magnetic domain pattern on the ribbon surface can be a key parameter predicting the microstructural inhomogeneities, crystalline inclusions and/or partial crystallization. In addition, the amorphous ribbons usually carry an induced anisotropy that is, in the as-prepared state, more or less random oriented. This induced anisotropy is based on small deviations from random pair orientations of the components of the material, and it is induced either by the magnetization pattern existing during cooling through the Curie temperature and possibly also by the flow pattern during quenching. Both contributions embody a random nature and therefore they are difficult to separate.

A unique possibility of studying, to a certain extent and resolution, local physical properties of iron-containing materials is offered by two nondestructive surface sensitive techniques, namely, Integral Low-Energy Electrons Mössbauer Spectroscopy (ILEEMS) [21] and Conversion Electrons Mössbauer Spectroscopy (CEMS) [22]. Both methods benefit from the fact that electrons emitted from a Mössbauer event in the sample (Mössbauer absorber) have a certain probability of reaching the surface and being detected as a function of Mössbauer source velocity. In such a way a Mössbauer spectrum can be obtained and its analysis yields information on the local atomic ordering, hyperfine magnetic and electrical interactions, chemical environment, etc. An important parameter determining the depth from which information concerning the electronic and atomic surrounding of the ^{57}Fe Mössbauer nucleus can be obtained is energy of the electrons. While the ILEEMS technique is based on detection of electrons of energy less than 15 eV, the CEMS uses the 7.3 keV K-shell internal conversion electrons. This energy difference indicates that the probing surface depth differs as well. At ILEEMS it is 10 up to 15 nm while at CEMS it is approximately 200 nm. Both Mössbauer techniques were used for micro-magnetic investigations of ribbon surfaces in this study but it is to note that the results obtained by ILEEMS support better the MOKE measurements due to their similar depth sensitivity.

It should be emphasized here that experimental methods like Transmission Mössbauer Spectroscopy, Vibrating Sample Magnetometer or SQUID made it possible to measure mainly bulk magnetic properties that are often completely different from the surface ones [23,24]. The combination of MOKE and ILEEMS methods was originally used for FeNiMoCuB ribbons [25].

The main objective of this paper is to bring new insight into the phenomena called nanoscale phase separation at the surfaces of amorphous alloys [26,27]. For this purpose the mentioned surface sensitive methods have been combined in systematic investigations of surface magnetic anisotropy, microstructure, magnetization reversal, and eventually surface crystallization. As a case study, the $Fe_{80}Si_xB_{20-x}$, $x=4$, 6, 8, and 10 at.%, amorphous alloys were chosen because they attract ongoing interest for themselves [28,29] and they constitute a basis of a broad class of FINEMET-type nanocrystalline materials [30].

2. Experimental

2.1. Sample preparation

The conventional planar flow casting (PFC) method in air was used for preparation of amorphous ribbon samples with the composition of $Fe_{80}Si_xB_{20-x}$, where $x=4$, 6, 8, and 10 at.%. The ribbons were 10 mm wide and 20 μm thick. Square samples 10 × 10 mm were cut out from the ribbons for subsequent experiments. Due to the technology used, the ribbon sides visually differ from each other. The side which is in contact with the quenching wheel is matt and is usually denoted as the wheel side, the opposite free one is generally shiny and is denoted as the air side, respectively. Both surfaces differ, as exposed the different cooling rate, often in their structural morphology, surface roughness and consequently physical properties as well, as it was shown many times at various ribbons prepared by PFC [31–33].

2.2. Surface magneto-optical methods

The surface magnetic properties were investigated by application of two magneto-optical methods. The first experimental arrangement is based on hysteresis loop measurements using the laser box schematically illustrated in Fig. 1. Two laser sources, red laser diode (wavelength $\lambda = 670$ nm) and the blue laser diode ($\lambda = 405$ nm), are situated perpendicularly to each other and their light incidents on the dichroic mirror/beam splitter (M/BS) that reflects beams in the range of 380–490 nm and transmits the light of wavelengths 520–700 nm. Due to precise micrometric stages at the output of M/BS both beams overlap and therefore impinge on the same place of the sample at an identical incidence angle. Minimal beam trace on the surface is ensured by a focusing lens that decreases the beam diameter down to 100 μm for blue and 200 μm for red light. The advantage of the blue laser to detect the magnetic properties from a smaller surface area is, on the other hand, compensated by approximately 10 times lower value of intensity in comparison to the red light source. Even though the magneto-optic effect at the wavelength of 405 nm is higher than for 670 nm, the measured "blue" hysteresis loops are more susceptible to be influenced by noise.

The amplitude of light outcoming from the laser box is modulated using photo-elastic modulator at frequency of 50 kHz and additionally linearly polarized (s or p polarization). The beam is then reflected from the ribbon surface, whereas its penetration depth (PD) being defined as $\lambda/(2\pi i N_z)$, where λ denotes wavelength of incident light and N_z is normalized wavevector in the surface normal direction expressed using the refractive indices of sample N and air N_a, and light incident angle ϕ as $N_z = \sqrt{N^2 - (N_a \sin(\phi))^2}$. The PD values for pure Fe, the refractive index of which is close to the investigated ribbons [34], and for $\phi = 60^\circ$ were estimated to be 33.47 nm and 23.91 nm for red and blue light, respectively. In the case of metals the influence of incident angle on PD is, however, almost negligible. In reality, the light directs both into and out of the sample and therefore MOKE PD is exactly half value of PD. The same incident angles were used for all investigated samples and a very similar depth of penetration for both lights can be expected. After reflection the light is generally elliptically polarized carrying information about the magnetization behavior from the inspected surface area. By transmitting through the Wollaston prism, we

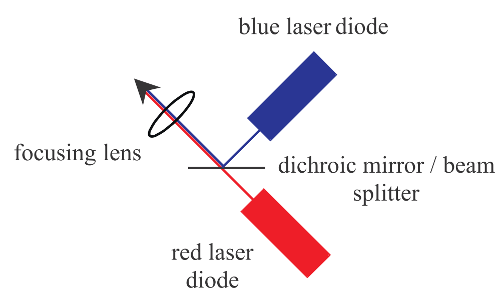

Fig. 1. Schematic illustration of laser box in the MOKE differential intensity method.