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Influence of Si Content on the Surface Magnetic Properties of $Fe_{80}Si_xB_{20-x}$ Metallic Glasses ($x = 6, 10$)

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The surface sensitive magneto-optical Kerr effect methods and conversion as well as integral low-energy electron Mössbauer spectroscopy are used to compare the surface magnetic and microstructural properties of two Metglas alloys with compositions $Fe_{80}Si_6B_{14}$ and $Fe_{80}Si_{10}B_{10}$. It is shown that the samples differ markedly both in the Mössbauer spectra and the surface hysteresis loops. While the ribbon with lower amount of Si is amorphous and characterized by three contributions corresponding to clusters with prevailing content of Fe and Si atoms, to clusters of mainly Fe and B atoms, and regions in between, the surface of the second sample is partially crystallized into bcc-FeSi crystals embedded in the residual amorphous matrix.

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1. Introduction

FeSiB metallic glasses have been investigated for many years but they still attract attention as recent extensive studies have evidenced [1, 2]. The reason lies in the evolution of their structure which has not been understood properly up to now. The low coercivity and high saturation magnetization of as-prepared amorphous samples can be further improved by annealing at temperatures well below the crystallization. Nevertheless, in addition to bulk physical properties, their surface properties are important as well. In particular, the surface crystallization and oxidation contribute to changes of surface magnetoelastic anisotropy, thus influencing the surface magnetization processes and the magnetic properties of the whole sample. These phenomena are known, however the systematic investigations of surface structural and physical properties are still insufficient.

Main objective of this paper is to find connection between the Si and B content and the surface magnetic behavior. For this purpose two samples $Fe_{80}Si_6B_{14}$ (denoted as A) and $Fe_{80}Si_{10}B_{10}$ (denoted as B) were prepared by conventional planar flow casting process. Ribbons are approximately 20 μm thick, 10 mm wide, and their length was cut down to 10 mm. For their shiny side (the side in contact with air during preparation) characterization we use the combination of the Mössbauer spectroscopy (MS) with the magneto-optical Kerr effect (MOKE) methods.

^{57}Fe Mössbauer spectra at room temperature (RT) were taken in scattering geometry by detecting 7.3 keV conversion electrons (CEMS, [3]) with the penetration depth of about 200 nm and by detecting 10 eV low-energy electrons (ILEEMS, [4]) the penetration depth of which is approximately 5–10 nm. Obtained spectra were analyzed using CONFIT program package [5] and calibration of velocity scale was done by a standard thin α-iron foil at RT. MOKE hysteresis loops were obtained in longitudinal configuration [6], i.e. external magnetic field applied along the original ribbon axis has direction parallel with the measured magnetization component. Light from the red semiconductor laser ($\lambda = 670$ nm) is incident at the angle of 60° and penetrates a few tens of nanometers into the ribbon depth. For homogeneity measurements the laser spot with diameter of about 0.3 mm was focused onto different places on the sample surface.

2. Results and discussions

The Mössbauer spectra of sample A (Fig. 1a) exhibit magnetic patterns of the broad lines characteristic for an amorphous structure. A satisfactory analysis of experimental results was obtained by the use of three Gaussian distributions of hyperfine inductions. Based on our as

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well as on other authors' investigations [7–9] each component reflects a certain local ordering of atoms into clusters. The component with highest value of mean hyperfine induction, $B_{\text{mean}} = 25$–26 T was ascribed to clusters with dominating Fe and Si atoms, slightly lower value of B_{mean} around 24 T is supposed to represent the clusters of prevailing Fe and B atoms. This value is very close to the value of hyperfine induction of crystalline bct-Fe_2B phase [10] which is, together with bcc-FeSi, the final crystalline phase after annealing of FeSiB amorphous alloys at appropriate temperatures. The third distribution with $B_{\text{mean}} = 10$–12 T represents the boundaries between the both types of clusters. The mentioned components are denoted in Fig. 1a for CEMS spectrum. The ILEEMS spectrum was analyzed with the same model. The main difference between the CEMS and ILEEMS results is in the ratio of second and first lines of sextets reflecting the magnetic moment orientation. While the magnetic moments in the very close surface layers (ILEEMS) are oriented out of ribbon plane, the spin direction obtained by CEMS measurements as well as measurements of bulk ribbon properties using γ-rays MS in transmission geometry (not presented here) is close to 85° from the γ-rays direction. It is a typical orientation induced by magnetic shape anisotropy in most ferromagnetic alloys prepared by planar flow casting. Slight differences between CEMS and ILEEMS results are also in the relative representation of components. While the CEMS yields approximately the same representation of FeSi and FeB clusters (44% and 53%, respectively), at the close surface (ILEEMS) the FeSi clusters prevail, 65% as compared to 28% of FeB clusters.

Figure 1b shows the results obtained for sample B. Here the surface is partially crystallized. The analysis of the CEMS as well as ILEEMS spectra yields crystalline bcc-FeSi phase represented by three six-line subcomponents of hyperfine inductions 33.5–30.8–27.7 T with a total amount of 32% (CEMS) and 27% (ILEEMS), respectively. Beside these subcomponents both remaining distributions with similar values of B_{mean} as those for A sample are present as well. The crystallization is exclusively localized at the surface because the transmission measurements (not presented here) have shown fully amorphous structure of the bulk. The higher tendency to the surface crystallization of sample B can be ascribed to lower content of glass forming B element.

Results of MS are in good agreement with MOKE measurements. Upper subplots of Fig. 2 show the surface hysteresis loops from two different places of sample A. Left curve indicates the place, where the MOKE signal detects the presence of one of mentioned clusters (either FeSi or FeB) with coercive field about 159 A/m. Conversely, right curve is composed of two minor loops representing two magnetically different phases confirming the existence of both clusters in near-surface region. Homogeneity measurements of the whole ribbon indicate that clusters are more or less randomly distributed on the ribbon surface, penetrate into the different depths and in this way contribute to marked inhomogeneity of the sample that is evidenced by the shapes of MOKE loops.

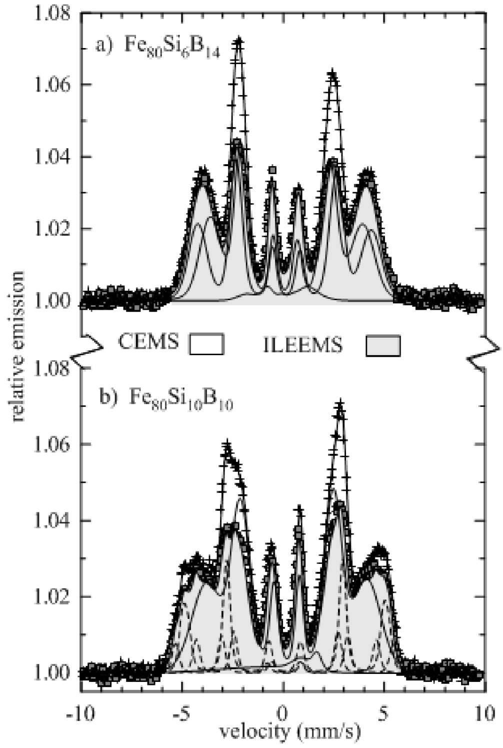

Fig. 1. CEMS and ILEEMS spectra of the $Fe_{80}Si_6B_{14}$ (a) and $Fe_{80}Si_{10}B_{10}$ (b) samples. Subcomponents, depicted for CEMS spectra only, represent the amorphous structure (full lines) and crystalline bcc-FeSi phase (dotted lines).

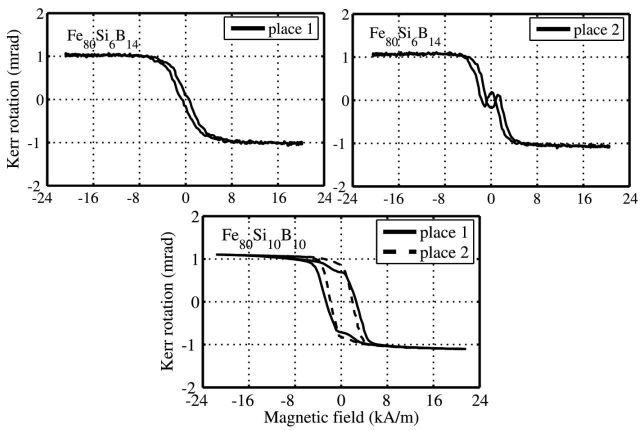

Fig. 2. MOKE hysteresis loops measured from near--surface region of $Fe_{80}Si_6B_{14}$ (upper subplots) and $Fe_{80}Si_{10}B_{10}$ (lower subplot) metallic glasses.

Partial surface crystallization of sample B is manifested on the hysteresis loops (lower subplot of Fig. 2) by approximately 10–15 times increase of coercive field in comparison with sample A. Similarly as in previous case, fluctuation of H_c around the value of 2.2 kA/m is mainly caused by the fact that texture of the ribbon surface is beside the bcc-FeSi phase composed of randomly distributed FeB clusters and their boundaries.

3. Conclusions

The Mössbauer and MOKE surface studies of two FeSiB samples with varying Si/B ratio, 6/14 and 10/10 in at.%, respectively, have shown that lower content of B as a glass forming element increases a tendency to surface crystallization. This resulted as in appearing three discrete narrow six-line components of bcc-FeSi phase superimposed on broad six-line Mössbauer spectrum of amorphous matrix as in a substantial increase in the surface coercivity detected by MOKE. Moreover, the measurements of the MOKE loops on various surface places have evidenced the marked inhomogeneity of the structure in the fully amorphous sample which comes out consequently in partially surface crystallized sample.

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