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MACROSCOPIC AND MICROSCOPIC OBSERVATIONS IN Fe-B-Si[1]

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The coercivity and hyperfine field parameters of Fe-B-Si alloy are discussed from a point of view of structure changes. A sharp minimum at 500 K in temperature dependence of coercivity of sample $Fe_{80}B_{10}Si_{10}$ annealed at 873 K correlates well with the temperature dependence of magnetocrystalline anisotropy constant K_1 in Fe_2B which becomes zero at about 524 K. No similar sharp minimum was observed in $Fe_{80}B_{20}$ after the same temperature treatment. It means that Si invokes a formation of Fe_2B phase during crystallization from an amorphous state. Using scanning and transmission electron microscopies the phase composition and structure were observed as well. Mössbauer phase analysis confirmed the presence of Fe_2B and Fe–Si with (12 ± 3) at.%Si.

1. Introduction

Magnetic properties of ferromagnetic materials are influenced by their composition, grain size, type, content of defects etc. One of the most exciting property is the coercivity H_c. Its experimental value may vary in a large range that is evoked by intrinsic properties as magnetocrystalline anisotropy constant, K_1, or spontaneous magnetization, M_s, and by microstructure, which depends on the preparation and pretreatment conditions.

The purpose of the present paper is to study correlations of magnetic properties with structure and, in particular, with grain morphology.

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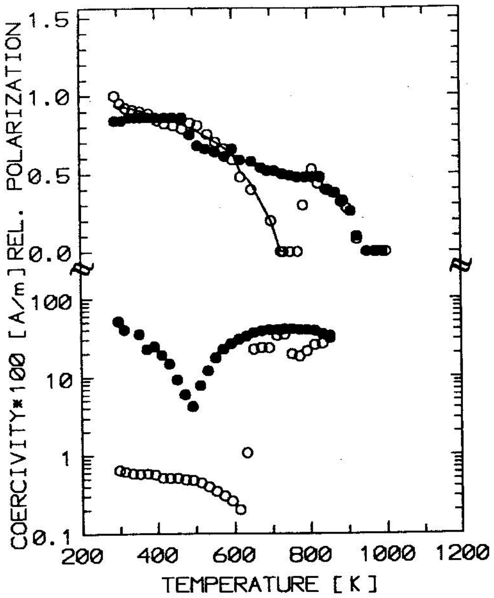

Fig. 1 Temperature dependences of the magnetic polarization (top) and the coercivity (bottom) of the as-prepared sample $Fe_{80}B_{10}Si_{10}$ at increasing (open points) and decreasing (full points) temperature. Full line is the Brillouin function fit of experimental data.

2. Methods

Metallic ribbons of nominal composition $Fe_{80}B_{10}Si_{10}$ 25 μm thick and 10 mm wide were produced at the Institute of Physics of Slovak Academy of Sciences by the conventional flow-casting method. The samples of (50 x 5) mm in dimension for magnetic and of 10 mm in diameter for Mössbauer measurements were prepared. For magnetic polarization and static coercivity investigation the earth-field compensated Förster coercimeter completed with vacuum furnace was used. The temperature dependences of both characteristics were measured in the regime of linear temperature increase or decrease 120 K/hour in the vacuum better than 0.2 Pa. The magnetic polarization was measured in the field of 2 kA/m.

Scanning and transmission electron microscopy observations were done on samples thinned by Ar^+ ion beam techniques.

The results of transmission electron microscopy became the starting point for the Mössbauer phase analysis. ^{57}Fe Mössbauer spectra were taken in transmission geometry at room temperature using ^{57}Co in Cr matrix as a source. The calibration was done against pure α-Fe. Computer processing of spectra was the same as in previous studies [1]. Hyperfine inductions and relative intensities of the multiplets for different nearest-neighbour configurations of the iron atoms in Fe-Si and Fe-B were calculated and compositions of crystalline phases were estimated.

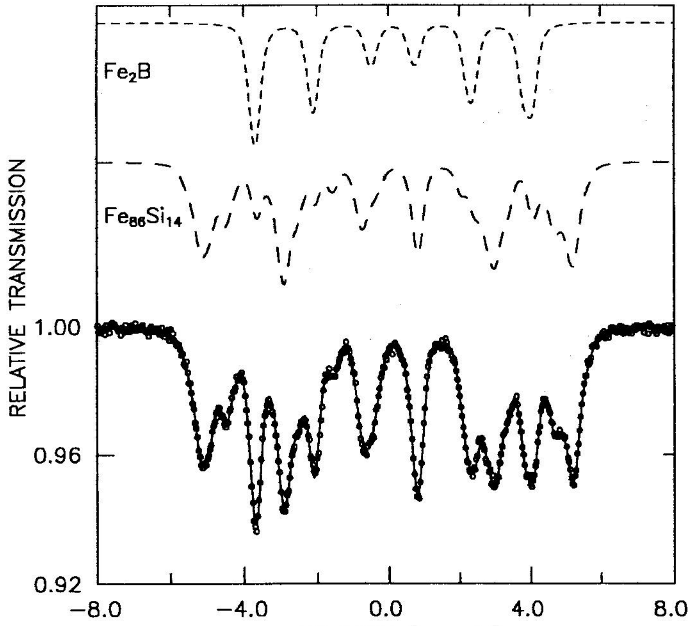

Fig. 2 Mössbauer spectrum of the crystallized $Fe_{80}B_{10}Si_{10}$ sample (bottom), subspectra of Fe_2B and $Fe_{86}Si_{14}$ phases (above).

3. Results and discussion

The temperature dependence of magnetic polarization and coercivity in $Fe_{80}B_{10}Si_{10}$ alloy are shown in Fig. 1. The Curie temperature 732 K of amorphous phase was determined using Brillouin function for extrapolation of experimental magnetic polarization data (full line in Fig. 1) [2]. The glass-to-crystalline state transition takes place around 783 K as indicated by a sudden rise of the magnetic polarization at this temperature corresponding to the crystallization of the Fe–Si solid solution [3]. At about 830 K, the steep rise abruptly ends indicating a formation of a new phase which was identified from the temperature dependence of coercivity. A steep decrease in cooling branch (full dots in Fig. 1 bottom) at about 500 K correlates well with the temperature, at which the magnetocrystalline anisotropy constant K_1 in Fe_2B becomes zero [4]. For comparison the similar temperature dependence of coercivity was measured on sample $Fe_{80}B_{20}$ annealed at the same conditions.

The cooling branch is smooth, without any anomaly at 500 K. It confirms that pure Fe–B alloy crystallizes in α–Fe structure and metastable Fe_3B which transforms into a stable Fe_2B phase at higher temperatures [5]. On the other hand the Si addition invokes direct formation of Fe_2B phase.

The annealing temperature of 873 K was about 90 K above the amorphous-to-crystalline transition and it could be assumed that all the crystallization and transformation events were complete. Both scanning and transmission electron microscopies reveal "cauliflower" like structure that indicates very fine grain size of the arising phases. The coercivity increased by about two orders in comparison to the original as-prepared amorphous sample. In the electron diffraction pattern the reflections of body-centered tetragonal Fe_2B phase were present. The other reflections corresponded to the Fe–Si phase with Si content of about 9 to 14 at.%. No Fe_3(Si,B) phase as in [6] was found in this sample. That was also confirmed by decomposition of Mössbauer spectrum (Fig. 2). The spectrum was fitted by 9 sextets with the hyperfine field parameters corresponding to the different nearest- and next-nearest-neighbourhoods of iron atoms. Proportioning the iron atoms according to their relative intensities in the sextet with the hyperfine induction of about 24 T corresponds to a Fe_2B phase [7]. Than the process of crystallization can be expressed by reaction $Fe_{80}B_{10}Si_{10} \rightarrow Fe_2B + Fe_{85.7}Si_{14.3}$.

5. Conclusions

Relations between magnetic properties and structure changes in sample prepared by crystallization of metallic glass $Fe_{80}B_{10}Si_{10}$ has been studied. Magnetic measurements determined the presence of Fe_2B phase in the sample after annealing at 873 K. The magnetocrystalline anisotropy K_1 of Fe_2B becomes zero near 524 K [4] where very sharp decrease of the coercivity was also observed. Comparison with the pure $Fe_{80}B_{20}$ alloy indicates that Si supports formation of Fe_2B phase. From diffraction studies only the presence of Fe_2B phase was confirmed and the content of Si in the α–FeSi phase of 9–~~14 a~~t.% was estimated. The results of the Mössbauer spectroscopy confirmed the presence of Fe_2B as well and determinated 12 ± 3 at.% of Si in the Fe–Si phase.

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