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Influence of Milling and Compaction Processes on Magnetic Properties of FeCo Powder

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Magnetic and structural studies were performed on $Fe_{50}Co_{50}$ material. The samples (disk-shaped, diameter: 10 mm, thickness: 2.5 mm) were fabricated by compaction of powder under pressure of 800 MPa for 5 min at temperatures 300–600°C. The powder was obtained by milling of $Fe_{50}Co_{50}$ alloy swarfs in high-energy planetary mill. The milling time varied from 1 h to 40 h. In the course of milling process the mean size of alloy pieces was decreasing from about 0.5 mm to 0.05 mm (scanning electron microscopy), which provided more compact structure after compression. The annealing process during compaction strongly reduces a coercive field of the samples. Parameters of conversion electron Mössbauer spectra are almost the same for all samples, which points to not significant changes of atomic and magnetic order after milling and compacting.

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1. Introduction

Iron–cobalt alloys are well known to have many applications due to their soft magnetic properties (high Curie temperatures, high saturation magnetization, high permeability) [1]. These properties, significantly dependent on grain size, internal strain and crystal structure, are shown to be superior in the nanostructured alloys, for example Moumeni et al. [2, 3] reported that after 24 h of high-energy ball milling between elemental Fe and Co the homogeneous bcc $Fe_{50}Co_{50}$ solid solution was formed due to diffusion of Co into α-Fe. In the present work, the polycrystalline powder samples were prepared by ball milling of $Fe_{50}Co_{50}$ alloy swarfs. Ball milling technique has been also successfully used to prepare many alloys in powder form which are therefore suitable for compaction into a variety of shapes [4]. The structural and magnetic characteristics of the bulk samples fabricated by the compaction of $Fe_{50}Co_{50}$ powder are analyzed as a dependence on the temperature of consolidation and milling time. A main goal of present work was to check out the importance of possible grain surface effects and intergrain interactions after above-mentioned processing of samples. A choosing of the initial material as pure $Fe_{50}Co_{50}$ alloy makes such analysis more simple than in the case of mechanically alloyed samples [3].

2. Experimental

It is possible, in laboratory conditions, to mill medium hard material whose pieces have at least one dimension small enough (powder with particle size below 1 mm or small pieces of thin ribbon). Since it was impossible to prepare ribbons of $Fe_{50}Co_{50}$ alloy, the initial sample was fabricated in the form of swarfs (approximately 0.1 mm thick, also suitable for milling) from the ingot by turning. The milling was performed up to 40 h in liquid nitrogen by means of a high-energy planetary ball mill (RETSCH PM4000 with hardened steel vials and balls) with ball-to-powder ratio of 6:1 and speed of 180 rpm. The powder consolidation into bulk samples (cylindrical disks: 10 mm in diameter and approximately 2.5 mm in thickness) was done in a uniaxial hot press under argon atmosphere. During the consolidation process the powders were heated with constant rate of 60 K/min to the consolidation temperature of 300°C, 400°C, 500°C and 600°C, held isothermally at this temperature and pressed for 5 min using a pressure of 800 MPa.

The surfaces of disk-shaped $Fe_{50}Co_{50}$ samples had been smoothed and polished before the Mössbauer measurements were carried out with conversion electron Mössbauer spectrometer (CEMS). The experimental system consisted of 50 mCi ^{57}Co(Rh) source of γ-radiation, a vibrator (working in constant acceleration mode) as well as a gas-flow detector (96% He + 4% CH_4). Numerical analysis of spectra was realized with the use of MOSFIT program based on the Varret method [5].

3. Results and discussion

The size of the particles decreases with milling time. The average size of the particle (of irregular shape) after 1 h milling is approximately 0.5 mm, while after 40 h is 0.2 mm (shape of the particle is more spherical), Fig. 1. The porosity of the bulk sample prepared by compaction of 40 h milled powder is significantly lower than that for 1 h milled one.

The absolute values of coercivity are unexpectable high (Table). The milling time dependences of coercivity for different temperatures of consolidation have tendency to decrease. In the range from 0 h to 15 h of milling time the coercivity increases for the sample compacted at 300°C, does not change for the sample compacted at 400°C and decreases for samples compacted at 500°C and 600°C. We suppose that stresses introduced to the material during milling cause an additional anisotropy, through large enough value of saturation magnetostriction (about 60 ppm for polycrystalline $Fe_{50}Co_{50}$ alloy [6]) and the increase in coercivity. The higher temperature of consolidation causes relaxation of these stresses and the coercivity decreases with milling time due to decrease in the resulting porosity of the material [7]. Further tendency of the coercivity as a function of milling time (the particle size becomes smaller) indicates that the process of magnetization rotation dominates over domain wall displacement.

Room temperature CEMS spectra for compressed $Fe_{50}Co_{50}$ powder samples after 1 h and 40 h of milling and annealed at 500°C are presented in Fig. 2a and c, respectively. The spectra were fitted with histogram-like hyperfine field distribution (HFD) of the Zeeman sextets. The obtained HFDs for two mentioned samples are displayed in Fig. 2b and d, respectively. Mean values of hyperfine field $\langle B_{\mathrm{HF}} \rangle \approx 34.5$ T as well as width of HFD $\Delta B_{\mathrm{HF}} \approx 1$ T (statistical dispersion) are almost the same for all samples (Table). They are typical of disordered $Fe_{50}Co_{50}$ conventional alloy [8–10]. Mean hyperfine field value is lower than those reported for mechanically alloyed $Fe_{50}Co_{50}$ system [11, 12, 2, 13, 14]. Dispersion of HFD is about 3 times larger than in the case of the ordered $Fe_{50}Co_{50}$ alloy ([8] — ^{59}Co spin-echo NMR data, 30 days annealed samples). Isomer shift IS (of the order of -0.03 mm/s — relative to α-Fe reference foil) was weakly correlated with HFD, whereas quadrupole splitting QS revealed a linear correlation with hyperfine field value (correlation coefficient ≈ 0.012 mm/(s T)). The

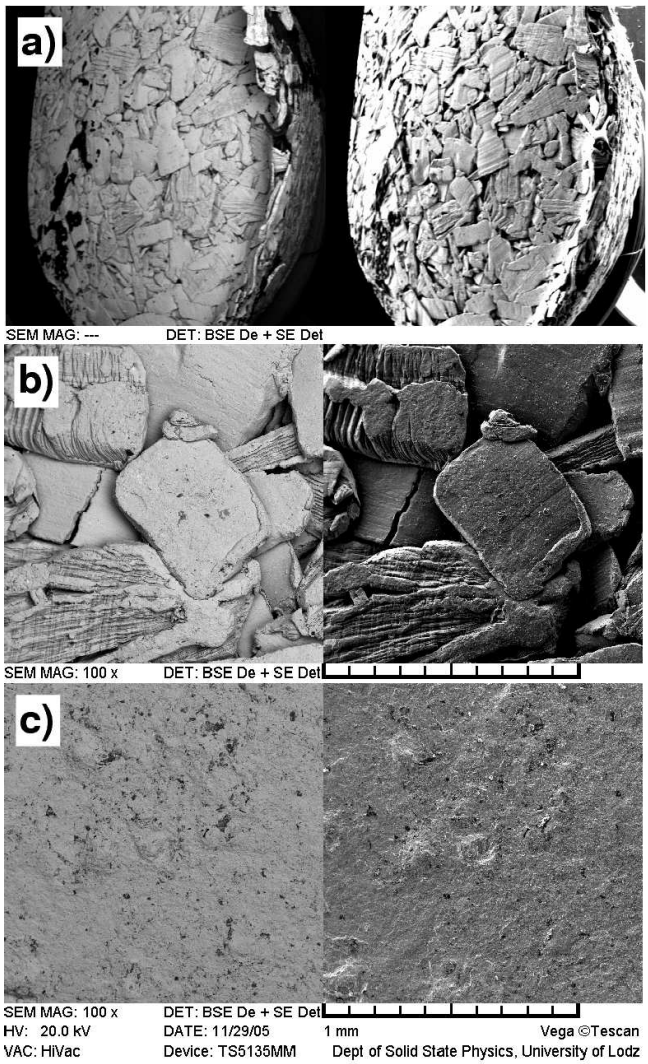

Fig. 1. Scanning electron microscopy (SEM) micrographs of compressed $Fe_{50}Co_{50}$ samples (consolidation temperature: 500°C): (a) milling time $t = 1$ h — "fish eye" view, (b) milling time $t = 1$ h — magnified view, and (c) milling time $t = 40$ h — magnified view.

Mössbauer spectra do not contain any noticeable paramagnetic component. Corresponding features and parameters of all the spectra are the same within an experimental error limit.

4. Conclusions

The milling and compaction process of $Fe_{50}Co_{50}$ alloy swarfs does not change significantly internal magnetic microstructure verified by means of CEMS technique. The parameters of the Mössbauer spectra are almost independent of milling time and their values are typical of disordered $Fe_{50}Co_{50}$ alloy. No grain surface contribution has been detected. Coercivity of compacted, disk-shaped samples is unexpectably high but its value is strongly reduced by annealing during compaction process (due to

TABLE

Dependence of coercive field H_c, mean hyperfine field values $\langle B_{HF} \rangle$ and width of HFD ΔB_{HF} (statistical dispersion) of compacted samples on milling time t of $Fe_{50}Co_{50}$ alloy swarfs and annealing temperature T_a during compaction process.

Sample number	t [h]	$T_a =$ 300°C H_c [A/m]	$T_a =$ 400°C H_c [A/m]	$T_a =$ 500°C H_c [A/m]	$T_a =$ 600°C H_c [A/m]	$T_a =$ 500°C $\langle B_{HF} \rangle$ [T]	$T_a =$ 500°C ΔB_{HF} [T]
FeCo/3	1	3760	3500	1840	1035	34.48	1.12
FeCo/7	5	3855	3900	1520	960	34.43	1.15
FeCo/11	10	4245	3810	1190	820	34.48	1.08
FeCo/15	15	4085	3465	1310	660	34.53	0.90
FeCo/19	20	4035	3675	1820	1015	34.45	0.85
FeCo/23	40	4035	3715	2105	1435	34.51	1.18

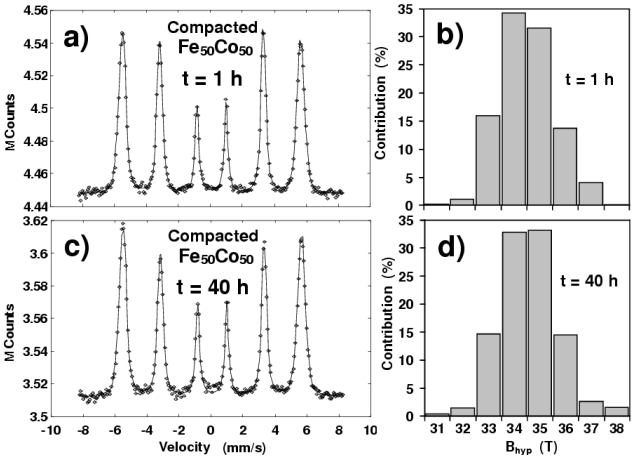

Fig. 2. CEMS results for samples consolidated at 500°C after 1 h milling of the alloy swarfs: (a) spectrum, (b) hyperfine field distribution, as well as after 40 h milling; (c) spectrum, (d) hyperfine field distribution.

mechanical strain relaxation). The observed evolution of coercivity with milling time should not be associated with microstructure changes, but could be attributed to variation of inter-grain interaction as well as internal strain, which is a crucial factor influencing the magnetic anisotropy due to the high value of magnetostriction constant in the case of equiatomic $Fe_{50}Co_{50}$ alloy. Milling and compaction of this alloy seem to be a good way of forming elements of different shape without significant worsening of magnetic properties.

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