Proceedings of the 15th Czech and Slovak Conference on Magnetism, Košice, Slovakia, June 17–21 2013

Mössbauer and Magnetic Study of Fe+Vitroperm+Plastic System

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Phase composition and magnetization curves of the soft magnetic composites, fabricated by compaction of several kinds of powders mixed in various proportions, have been investigated by means of conversion electron Mössbauer spectroscopy and an alternating gradient force magnetometry. The results point to significant quantity of iron oxides – hematite and magnetite – at the surface of the samples. After the rubbing of thin surface layer, the relative content of oxides was distinctly reduced. Magnetic measurements revealed very similar characteristics of hysteresis curves for all the investigated materials.

DOI: 10.12693/APhysPolA.126.148

PACS: 75.50.Bb, 68.35.bd, 76.80.+y, 75.60.–d, 72.80.Tm

Soft magnetic composites (SMCs) based on electrically insulated iron powders are a group of materials which give prospects for technical applications [1]. Due to good magnetic and mechanical properties, large electrical resistivity, sensibility to the parameters of the manufacturing procedure and flexibility to formation into 3-D structures, they are recommended for employment in electrical machines [2]. The present article provides data on atomic structure and magnetic properties of the surface and bulk regions of complex systems, composed of crystalline iron powders mixed with amorphous alloy particles. The soft magnetic properties of similar bulk samples were reported in earlier paper [3].

Iron based materials: Somaloy® 700 [4], provided by Höganäs AB, Sweden (with percentage p_S) and Vitroperm® 500 amorphous alloy (with percentage $p_V = 100\% - p_S$) in the form of flakes, provided by Vacuumschmelze, GmbH & Co. KG, Germany, were mixed and compacted together with plastic. The details of the production process are described in [3]. The specimens had form of 5 mm high cylinders of diameter equal to 2 cm. Five composite samples, containing 5%, 10%, 20%, 30%, 50% of Vitroperm in relation to the whole magnetic material, have been prepared. Conversion electron Mössbauer spectroscopy (CEMS) based on ^{57}Fe isotope was employed in order to identify phase composition of the surface regions. As source of gamma radiation, ^{57}Co(Rh) was used, the energy of which was modified by the source movement with linearly changing velocity. Magnetic properties were investigated at room temperature by use of an alternating gradient force magnetometer (AGFM), with the magnetic field applied in the plane of the sample. The results were complemented by data of mass density and electrical resistivity (collected in Table).

TABLE

Introductory characteristics of the investigated samples: percentage of iron powder – p_S, electrical resistivity – ρ, mass density – d, relative volume of the pores – V_p, mass – m.

	p_S (%)	ρ ($\mu\Omega$·m)	d (g/cm^3)	V_p (%)	m (g)
S95-5VPM	95	32	7.08	5.51	0.976
S90-10VPM	90	35	7.09	5.51	1.040
S80-20VPM	80	84	6.87	8.03	-
S70-30VPM	70	105	6.77	9.19	0.980
S50-50VPM	50	135	5.80	21.89	0.918

An exemplary spectrum of the original specimen, obtained using CEMS, is presented in Fig. 1a. This method yields information about the surface layer of investigated samples. The analysis of spectra has proved that the component characteristic of iron oxides: Fe_2O_3- hematite and Fe_3O_4- magnetite, composed of three sextets with hyperfine magnetic fields (hmf) larger than 45 T, dominates in most cases. It was superimposed with a set of four Zeeman sextets (with hmf from the range 24 T ÷ 33.1 T), characteristic of Fe-Si alloy [5] and α-iron. Moreover, a small smeared subspectrum, characterized by a continuous distribution of hmf, attributed to the disordered regions of both Vitroperm particles and iron ones, has been found. Percentage of the individual components derived for both sides of specimens (called arbitrarily A and B) differed considerably, and varied without any regularity, as shown in Fig. 2a. The results revealed poor resistance of the surface layer to oxidation. The differences in results, obtained for both sides, have arisen presumably from the fact that the samples rested for the long time on the one side (A or B, casually).

In order to remove 0.2 mm thick surface layer with oxides, the cylinders were subjected to the manual rubbing just before CEMS measurements. We expected properties of the surface after rubbing to be similar to those of the bulk material. We observed that the samples were fragile, especially the specimens with large content of Vitroperm, so the sample with $p_S = 50\%$ was damaged due to the rubbing.

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A typical resulting Mössbauer spectrum is presented in Fig. 1b. It has been stated that the contribution of iron-oxides-component was reduced or fully eliminated, enabling more precise analysis of the other phases. As shown in Fig. 2b, the percentage of the oxides increased with the Vitroperm content $p_V = (100\% - p_S)$ from 0 for $p_V = 5\%$ up to 15% for $p_V = 30\%$. The relative content of the smeared sextet (the "continuous" component) also increased from about 4% for $p_V = 5\%$ up to 20% for $p_V = 30\%$.

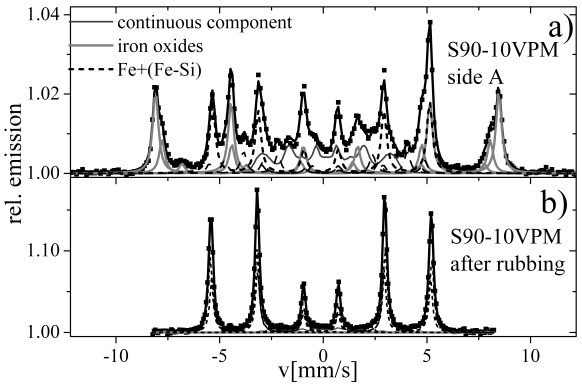

Fig. 1. Typical CEMS spectra collected before (a) and after (b) rubbing of the surface layer, at room temperature.

When we did not take into account the oxides component, a slight decrease of mean hyperfine magnetic field was observed with the rise of Vitroperm content: from 32.0 T for $p_V = 5\%$ up to 27.5 T for $p_V = 30\%$.

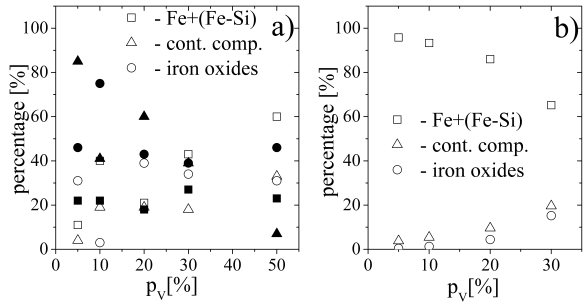

Fig. 2. Percentage of the components of composites CEMS spectra before rubbing (a) (open symbols – side A, solid symbols – side B) and after rubbing of the surface layer (b).

On the basis of AGFM investigations the magnetization curves have been derived. In Fig. 3 only normalized values of magnetization are presented, due to the fact that in each sample the amount of magnetic phase is different. It has been found that the magnetic saturation field had the similar value for all the samples – about 500 mT. Moreover, a trend of slightly faster reaching of the saturation state by magnetization has been observed for samples with smaller content of the magnetic phase. This result is probably related to the demagnetization effects. The magnetization curves do not reveal hysteresis – it means that *de facto* the coercive field induction is smaller than 0.5 mT, the value corresponding to the accuracy limit of AGFM system.

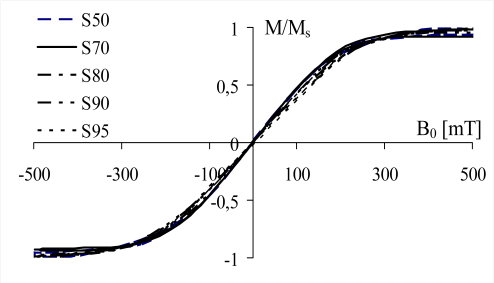

Fig. 3. Magnetization curves obtained by use of AGFM system, at room temperature.

The results of presented study have proved that the surfaces of the samples were very susceptible to oxidation. The layer of oxides was rather thin (about several tenths of mm). Oxidation seems to be related mainly to the Vitroperm particles. In the investigated systems, the magnetic properties are weakly dependent on the proportion between both magnetic constituents, thus the amount of nanocrystalline alloy (Vitroperm) can be reduced in final product.

Acknowledgments

This work was supported by the Grant Agency of the Slovak Academy of Sciences through project APVV-0222-10 MAGCOMP, by the projects, nanoCEXmat I, ITMS 26220120019 and nanoCEXmat II, ITMS: 26220120035, supported by the Operational Program "Research and Development" financed through European Regional Development Fund and by the Scientific Grant Agency of the Ministry of Education of Slovak Republic and the Slovak Academy of Sciences, projects Nos. VEGA 1/0861/12, No. 1/0862/12, No 1/0148/12 and 2/0155/12. Special thanks to Höganäs AB Sweden for providing Somaloy® 700 powder and to Mr. M. Vitovský, Vacuumschmelze, GmbH & Co. KG, Germany for providing Vitroperm® 500.

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