Journal of ELECTRICAL ENGINEERING, VOL. 58, NO. 2, 2007, 118–120

COMMUNICATIONS

(FeSm) —– (FeALPCBSIGa) COMPOSITES PREPARED BY HOT–PRESSING OF BALL MILLED POWDERS

Jarmila Degmová [*] — Jörg Finnberg [**] — Stefan Roth [**]

The present study was aimed to prepare a material which combines a high magnetostrictive strain in low magnetic fields with a high mechanical strength. Therefore we prepared composites which consist of soft magnetic $Fe_{77}Al_{2.4}P_{8.64}C_5B_4Si_{2.6}Ga_{0.8}$ and high magnetostrictive (Fe_2Sm) alloys. Ball milling of the amorphous FeAlPCBSiGa ribbon and a crystalline Fe_2Sm ingot and subsequent hot pressing of the resulting powders were used to prepare disc shaped composites. A maximum magnetostrictive susceptibility of 930 ppm T^{-1} at a field of $\mu_0 H = 0.1$ T was obtained. The hardness varied from 150 HV10 to 340 HV10 with increasing amount of the amorphous phase.

K e y w o r d s: high magnetostrictive material, soft magnetic material, magnetostrictive strain, ball-milling, hot pressing

1 INTRODUCTION

Permanent magnets achieve an increase in the energy product due to improvements in coercivity and in magnetization *ie* the coercivity is as high as necessary (of the order $H_c \sim J_r/\mu_0$, where J_r is the remanent magnetization) and the remanent magnetization is as high as possible. Therefore, it was proposed to prepare permanent magnets of composite materials consisting of two suitably dispersed ferromagnetic and exchange-coupled phases, one of which is of high coercivity (e.g. a rare earths transition-metal-compound), whilst the other may be soft magnetic just providing a high saturation polarization [1, 2]. Consequently the magnetization of the soft phase is polarized by the hard phase. This leads to an increase of the remanent magnetization caused by the exchange coupling between the phases as well as by the large magnetic moment of the soft magnetic phase. From the microstructural point of view the essential condition for such materials is a fine and regular dispersion of phases which ensures effective coupling between both, hard and soft magnetic phases.

The possibility of exchange coupling a highly magnetostrictive material to a soft magnetic material in a similar way as in the case of permanent magnets was demonstrated for thin films [3]. Pinkerton *et al* described in [4] (high magnetostriction rare earth) — (soft magnetic material) composites developed for automotive application. No dense composites were achieved in this study due to the low processing temperature necessary to avoid the decomposition of the intermetallic rare earth phase. In a recent study [5, 6] we have demonstrated the possibility to prepare a bulk amorphous material from powders of amorphous FeAlPCBSiGa multicomponent alloys with good soft magnetic and mechanical properties at a rather low compaction temperature. The successful preparation of disc-shaped bulk amorphous Fe-AlPCBSiGa samples at temperatures lower than the temperature of decomposition of the rare earth iron intermetallic phase encouraged us to use the FeAlPCBSiGa amorphous multicomponent alloy as a soft magnetic material in (rare earth — iron) — (soft magnetic material) composites.

2 EXPERIMENTAL

A Sm-Fe ingot was prepared by arc melting of pure elements and subsequent homogenization by annealing at 1103 K for 186 hours in an evacuated fused silica tube. An ingot of nominal composition $Fe_{77}Al_{2.14}Ga_{0.86}P_{8.4}C_5B_4Si_{2.6}$ was prepared by induction melting of a mixture of Fe, Al, Si (or Fe-Si) metals (purity 99.9 %) and pre-alloyed Fe-C, Fe-B, Fe-P, Fe-Ga (purity 99.9 %) under argon atmosphere. Amorphous ribbons with a cross-section of about 0.02 mm $\times$10 mm were prepared from this material by single roller melt spinning on a copper wheel with a fixed surface speed of 21 m s^{-1} under argon flow.

Finally, disc-shaped samples (see Fig. 1) were prepared in two different ways: (1) ribbons and ingots were ball-milled together (T-milling) and the resulting powders were subsequently compacted, and (2) ribbons and ingots were ball-milled separately (S-milling), the powders were subsequently thoroughly mixed by hand in chosen proportions and then compacted. Duration of T-milling varied between 1 and 64 h with the same ratio of phases (the SmFe fill fraction was about 67 %). S-milling lasted

[*] Department of Nuclear Physics and Technology, Faculty of Electrical Engineering and Information Technology, Slovak University of Technology, Ilkovičova 3, 812 19 Bratislava, current address: JRC-IE, Joint Research Centre, Institute for Energy, P.O. Box 2, Petten 1755 ZG, The Netherlands

[**] IFW Dresden, Institute for Metallic Materials, P.O. Box. 270016, D-01171 Dresden, Germany

ISSN 1335-3632

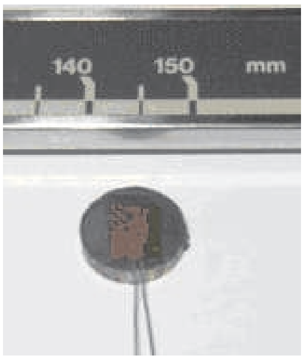

Fig. 1. Hot pressed sample.

Table 1. Properties of cylindrical composites prepared by 0.5 h and 1 h of S-millings. $SmFe_2$ characterises the fill fractions of high magnetostrictive phase in composites and $\Delta\lambda$ is the differential (maximal) magnetostrictive strain, which is a maximal difference between parallel, λ_{pl}, and perpendicular, λ_{pn}, magnetostrictive strain.

$SmFe_2$ (%)	17	33	50	67	83
$\Delta\lambda_{0.5}$ (ppm)	170	120	88	237	300
$\Delta\lambda_1$ (ppm)	123	78	68	182	274

Table 2. Properties of cylindrical composites prepared by T-millings. BM indicates ball-milling time, $SmFe_2$ characterises the fill fractions of high magnetostrictive phase in composites and $\Delta\lambda$ is the differential (maximal) magnetostrictive strain.

BM (h)	1	2	4	8	16	32	64
$SmFe_2$ (%)	67						
$\Delta\lambda$ (ppm)	259	428	390	207	155	77	37

0.5 and 1 h and the content of $SmFe_2$ in the composite varied between 20 and 80 mass%. The magnetostriction, λ, of the bulk samples was measured by the strain gauge method in a maximum magnetic field of 0.9 T. The hardness test was carried out according to Vickers (HV). For all samples a test load of 10 N was chosen.

3 RESULTS AND DISCUSSION

Table 1 shows the differential magnetostrictive strain, $\Delta\lambda$, as a function of $SmFe_2$ fill fraction for the composites prepared by compaction of hand mixed powders (S-milling) for 2 different milling times. As expected, the composites with a higher amount of $SmFe_2$ showed a higher magnetostrictive strain. Slight differences between the two ball-milling times can be found. The values corresponding to the composites prepared from powders ball-milled for shorter time (0.5 h) are in some cases as much as 50 ppm higher. The maximal value of magnetostrictive strain (of about 300 ppm) is obtained for the composites made from 17 % of the soft magnetic and 83 % of the high magnetostrictive phase.

Table 2 describes the magnetostrictive strain of samples compacted from T-milled powders, *ie* the compositions of powders ball-milled together for 1 up to 64 hours. In the case of a mixture with the 67 mass% of $SmFe_2$ a maximal magnetostrictive strain, $\Delta\lambda$, of about 428 ppm is achieved after 2 h of ball-milling and then it decreases again for longer milling times rapidly. The magnetostrictive suceptibility, $\Delta\lambda/\Delta H$, was measured for two samples showing the largest magnetostrictive strain *ie* for 2 and 4 hours T-milled powders (Fig. 2). The largest magnetostrictive susceptibility (about $930\,\mathrm{ppm\,T^{-1}}$ at a field of approximately 0.1 T) was found for the sample pressed from 4 hour T-milled powder. In the case of samples compacted from S-milled powder the variation of the magnetostriction as a function of the magnetic field is almost linear. Figure 3 shows the hardness of the samples prepared from S-milled powder as a function of the $SmFe_2$ fraction together with the values measured by Pinkerton et al. [7].

Original literature data were given as Rockwell B hardness so they were transformed to Vickers hardness in order to have comparable data. Such transformation is generally adopted for steel and might be slightly incorrect in the case of composites. Nevertheless it can be seen, that in the case of the amorphous alloy as a soft magnetic material, the hardness is much higher than for the composite with iron as the soft magnetic material, especially for lower amounts of the $SmFe_2$-phase. From the concentration dependencies we can conclude, that the hardness of the amorphous matrix is higher than the hardness of $SmFe_2$ whilst the hardness of Fe is smaller than that of $SmFe_2$.

4 CONCLUSION

$Fe_{77}Al_{2.14}Ga_{0.86}P_{8.4}C_5B_4Si_{2.6}$– $SmFe_2$ disc shaped composites with a diameter of 10 mm and a thickness of 1 mm were prepared by hot pressing of ball-milled powders. The powders were obtained by ball milling of cut pieces of the melt-spun ribbon and of crushed ingots of $SmFe_2$ at different conditions: S-milling (the ribbons and the ingot were milled separately) and T-milling (the ribbons and the ingots were milled together). S-milling (hand mixed powders) leads to an improvement of the magnetostriction which increases linearly with increasing amount of the high magnetostrictive phase. A maximum of about 300 ppm is reached at 83 % fill fraction of the magnetostrictive phase. In the case of T-milling (the SmFe fill fraction was about 67 %) a maximum of magnetostriction (= 428 ppm) appeared after 2 h of ball milling. The highest magnetostrictive susceptibility of $930\,\mathrm{ppm\,T^{-1}}$ at a field of about 0.1 T was achieved for ball milling 67 % of $SmFe_2$ together with amorphous $Fe_{77}Al_{2.14}Ga_{0.86}P_{8.4}C_5B_4Si_{2.6}$ for 4 h followed by hot pressing. This behaviour could be explained as an increased coupling between the two phases after milling

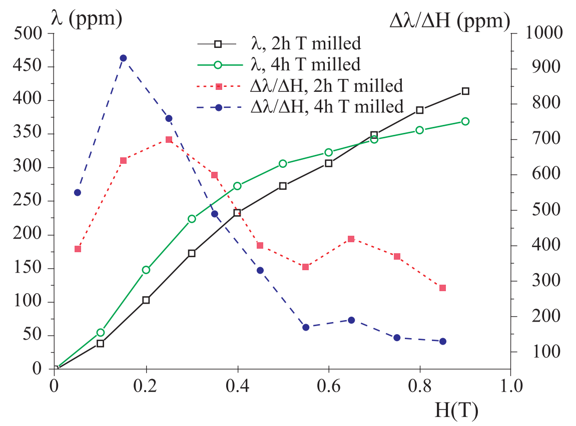

Fig. 2. Field dependence of the magnetostrictive strain and magnetostrictive susceptibility.

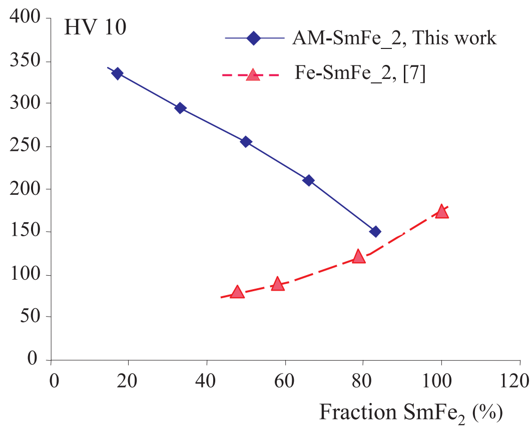

Fig. 3. Hardness as a function of $SmFe_2$ fill fraction.

together for 2 to 4 h. Prolonged milling decreases the magnetostrictive strain probably due to a degradation of the $SmFe_2$ phase. The increased magnetostrictive susceptibility after 2 to 4 h of T-milling can not be probably ascribed to exchange coupling but may be due to magnetostatic (dipolar) interaction. The hardness of the amorphous metal – $SmFe_2$ composites is higher then that of Fe–$SmFe_2$ composites. This is due to the superior mechanical properties of amorphous ferromagnets compared to iron [8]. Furthermore, the magnetostrictive susceptibility is enhanced due to the lower amount of pores in the composite which can be reached because of the low processing temperature of the amorphous alloy. A further improvement of the magnetostrictive behaviour of the composites may be expected by adjusting the initial amount of the magnetostrictive phase and the milling conditions as milling time and milling intensity.

Acknowledgements

The authors thank to H. Schulze for technical assistance. This work was supported by the Saxonian ministry of science and arts and partly by grant VEGA 1/3189/06.

References

[1] ECKERT, D.—MÜLLER, K. H.—HANDSTEIN, A.—SCHNEIDER, J.—GRÖSSINGER, R.—KREWENKA, R.: IEEE Trans. Magn. **26** (1990), 1834.

[2] KNELLER, E. F.—HAWING, R.: IEEE Trans. Magn. **27** (1991), 3588.

[3] QUANDT, E.—LUDWIG, A.: J. Appl. Phys. **85** (1999), 6232.

[4] PINKERTON, F. E.—CAPENHART, T. W.—HERBST, J. F.—BREWER, E. G.—MURPHY, C. B.: Appl. Phys. Letter **70** (1997), 2601.

[5] DEGMOVÁ, J.—ROTH, S.—ECKERT, J.—SCHULTZ, L.: Journal of Metastable and Nanocrystal. Mat. **12** (2002), 85.

[6] DEGMOVÁ, J.—ROTH, S.—ECKERT, J.—GRAHL, H.—SCHULTZ, L.: Mat. Sci. Eng. **A 375-377** (2004), 265–269.

[7] PINKERTON, F. E.—CAPERHART, T. W.—HERBST, J. F.—BREWER, E. G.—MURPHY, C. B.: J. Appl. Phys. **83** (1998), 7252.

[8] STOICA, M.—ECKERT, J.—ROTH, S.—ZHANG, Z. F.—SCHULTZ, L.—WANG, W. H.: Intermetallics **13** No. 7 (2005), 764–769.

Received 11 November 2006

Jarmila Degmová (MSc, PhD), graduated in electromaterial engineering from the Slovak University of Technology in Bratislava in 1995, gained her PhD in electrotechnology and materials from the same university in 2001. At present she is a researcher at the Faculty of Electrical Engineering and Information Technology SUT in Bratislava and she is doing her post-doc at Joint Research Centre EC Petten in Netherlands. Her research is focused on the construction materials of nuclear power plants.

Jörg Finnberg (MSc), graduated from the Dresden Institute of Technology in 2006 in the field of materials research.

Stefan Roth (PhD), graduated in physics from the Dresden University of Technology in 1969 and gained his PhD in theoretical physics in 1973 also from the Dresden University of Technology. Since 1972 he has worked in the Central Institute of Solid State Physics and Materials Research which is now named the Leibniz Institute for Solid State and Materials Research (IFW Dresden).He has worked in the field of soft magnetic materials since that time. His current interests are focussed on bulk amorphous soft magnetic materials and magnetic shape memory alloys.