Vol. **113** (2008) *ACTA PHYSICA POLONICA A* No. 1

Proceedings of the CSMAG'07 Conference, Košice, July 9–12, 2007

Structure and Magnetic Properties of Hot Pressed NiFe Powder

D. Olekšáková[a], P. Kollár[b], J. Füzer[b] and M. Kusý[c]

[a]Department of Applied Mathematics, Faculty of Mechanical Engineering
Technical University in Košice, Letná 9, 04200 Koišce, Slovakia
[b]Institute of Physics, Faculty of Sciences, P.J. Šafárik University
Park Angelinum 9, 04154 Košice, Slovakia
[c]Faculty of Material Science and Technology
Slovak University of Technology
Paulínska 16, 91724, Trnava, Slovakia

The aim of this work is to investigate the structure and magnetic properties of compacted microcrystalline NiFe (81 wt.% of Ni) powder. Bulk samples were prepared by compaction of milled NiFe (81 wt.% of Ni) ribbon. We found that after compaction of the powder displacement of domain walls becomes more dominant and the coercivity decreases and is comparable with the coercivity of conventional permalloy. The coercivity of the bulk material before heat treatment is lower than that for powder and that is why we can assume that the magnetic "contact" is restored after compaction. Annealing of bulk samples reduces the losses due to the relaxation of internal stresses induced by milling and compaction.

PACS numbers: 75.50.Bb, 81.20.Ev, 75.60.Ej

1. Introduction

The purpose of this work was to prepare and to investigate magnetic material by compaction of powder as alternative technology for preparing bulk and obtaining material with required soft magnetic properties [1]. It is well known that the alloy NiFe (with about 81 wt.% of Ni) exhibits zero magnetostriction [2]. We expected that in such alloy the residual stresses induced during the milling and consolidation of the powder do not cause any additional anisotropy and the alloys will have good soft magnetic properties.

2. Experimental

We have obtained powder by mechanical milling of microcrystalline NiFe (81 wt.% of Ni) ribbon (prepared by melt-spinning) in a high-energy planetary ball mill (RETSCH PM4000). The milling was performed in protective argon atmosphere with balls to powder mass ratio (BPMR) 6:1 and with a speed of 180 rpm. We have prepared bulk samples from these milled powders in the form of cylinders. The compaction was performed at a pressure of 800 MPa for 5 min at 300°C, 400°C, 500°C and 600°C in vacuum of 5×10^{-3} Pa. The parameters of cylinders were: height 2.5 mm, diameter 10 mm, and weight approximately 2 g. The cylinders were annealed at temperatures 500°C, 600°C, 700°C, 800°C, 900°C, 1000°C, 1200°C (first step) and then at 1100°C (second step). After each annealing the magnetic properties were investigated. The structure of all samples (in both powder and bulk form) was investigated by X-ray diffraction (XRD) (Philips PW 1050). The magnetostriction of the cylinder shaped samples was measured by strain-gauge method. The coercivity of the samples was assigned by a Förster Koerzimat. An axial hole with diameter of 5 mm was drilled by spark erosion into the compacted disc, which produced ring samples and we have prepared coils with number of primary turns 20 and number of secondary turns 20 for AC (AMH 401 POD WALKER) measurement.

2. Results and discussion

Investigating the structure and magnetic properties of ribbon NiFe (81 wt.% of Ni) and milled powder for 5, 10, and 25 h, we have obtained the samples consisting of fcc $FeNi_3$ phase (powder size was 30 μm, 10 μm, 5 μm, respectively) with the Curie temperature 550°C. The fact that all samples are single phase was confirmed by thermomagnetic curves, too [3, 4].

The powder prepared in such a way was suitable precursor for compaction, because it is well known that precipitates of "weak" phase of the two-phase system act as pinning centers for domain wall displacement resulting to the high coercivity [2].

XRD pattern of as-cast ribbon and of powder prepared by milling ribbon for 25 hours and the XRD pattern of compacted samples prepared from powder described in [4] revealed that the milling of the NiFe microcrystalline ribbon and the compaction of this powder have no significant influence on the phase composition of the material. All studied diffraction patterns documented in [4] prove presence of $FeNi_3$ as a major component with phase fraction at least 90%.

After preparation of bulk samples, we have confirmed that the saturation magnetostriction is really near to zero ($|\lambda_s| < 2$ ppm) and we can propose that the residual stress does not introduce any significant anisotropy to the bulk samples [4].

Figure 1 shows the increase in the coercivity with frequency for bulk samples prepared by compaction of 25 h milled ribbon. The annealing temperatures of 500°C and 1200°C caused lowering of the coercivity with the increase in the temperature. The coercivity after compaction decreased 10 times in comparison with the coercivity of the powder sample. The bulk sample is magnetized mostly by domain walls displacement, while in powder (with random oriented easy axes) rotation magnetization vector is dominated. The lowest value of the coercivity (11 A/m) was achieved for the sample annealed at 1200°C [4], comparable with the value of coercivity of the conventional permalloy (4 A/m) [5].

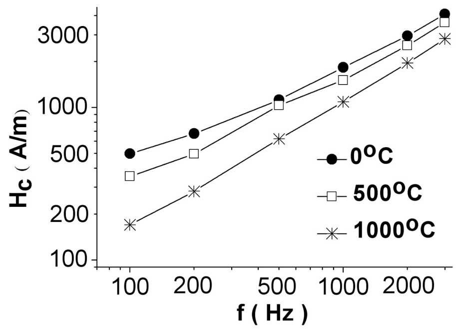

Fig. 1. AC coercivity dependence on frequency for bulk sample compacted from ribbon milled 25 h at temperature 600°C and annealed at different temperatures.

Further annealing at 1100°C led to the significant change of the frequency dependences of coercivity and losses, Fig. 2. Both quantities linearly increase with the frequency and the lowest coercivity and losses were detected for the sample prepared by compaction of broken ribbon. Samples prepared by compaction of powder milled 5 h, 10 h, and 25 h exhibit very similar values of coercivity and losses, so the powder size does not play significant role. The annealing leads to the creation of the magnetic contact between powder elements, through which domain wall can move. We assume that the pores between powder particles act as pinning centers for domain wall movement and determine the value of the coercivity.

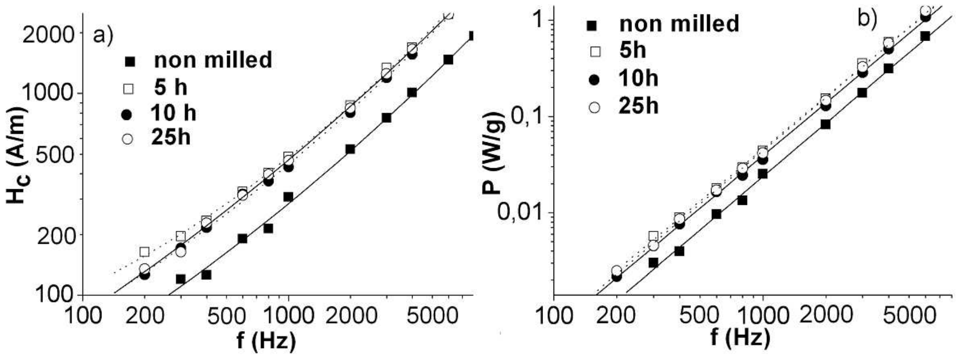

Fig. 2. Frequency dependence of (a) coercivity, (b) losses for bulk $Ni_{81}Fe_{19}$ consolidated broken ribbon and milled powder measured at 0.2 T.

4. Conclusion

Investigating of the structure and magnetic properties of NiFe bulk material obtained that single-phase compacts exhibit very good magnetic properties with coercivity of 11 A/m which is comparable with that for material prepared by convention way in the form of thin sheet ($H_c = 4$ A/m). The coercivity decreases more significantly at lower frequency than at higher frequency with increasing annealing temperature which is caused by improvement of electromagnetic contact between powder elements. At higher frequency coercivity decreases less significantly with annealing temperature because of increasing influence of eddy current. The absolute values of coercivity and losses of NiFe compacts are comparable (or slightly lower) to that many widely used Fe, Ni based materials in the form of thin sheets (which is usually about four times thinner than investigated bulk material) [2] and cannot be concurrence to thin ribbons and nanocomposites of various chemical composition and structural states [6].

Acknowledgments

This work was supported by the Science and Technology Assistance Agency (No. APVT-20-008404) and by Slovak Scientific Research Fund VEGA 1/4020/07.

References

[1] L.A. Dobrzański, Z. Brytan, M.A. Grande, M. Rosso, E.J. Pallavicini, *J. Mater. Process Technol.* **162-163**, 20 (2005).

[2] R.C. O'Handley, *Modern Magnetic Materials*, Wiley, New York 2000.

[3] O. Schneeweiss, B. David, T. Zak, R. Zboril, M. Mashlan, *J. Magn. Magn. Mater.* **310**, e858 (2007).

[4] P. Kollár, D. Olekšáková, J. Füzer, J. Kováč, S. Roth, K. Polański, *J. Magn. Magn. Mater.* **310**, 2609 (2007).

[5] W.F. Randal, *J. Inst. Elect. Eng.* **80**, 647 (1937).

[6] H. Shokrollahi, K. Janghorban, *J. Magn. Magn. Mater.* **317**, 61 (2007).