STRUCTURE AND MAGNETIC PROPERTIES OF POWDERED AND COMPACTED FeNi ALLOYS

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Received 10 May 2016; accepted 17 May 2016

1. Introduction

The FeNi based alloy (called permalloy) system shows excellent soft magnetic properties and these alloys have been widely applied in the field of electronic devices and industry. The soft magnetic properties of permalloy are significantly dependent on Ni content: lower coercivity (alloy with about 80 at. % Ni), higher-saturation magnetic induction (50 at.% Ni) and lower permeability but higher electrical resistivity (about 35 at.% Ni) [1].

It was found that the addition of a small amount of molybdenum to FeNi alloy can have a strong effect on structural properties of resulting FeNiMo alloys (called supermalloy) leading to the improvement of soft magnetic properties with very high relative permeability and low eddy current losses [1].

The structure of permalloys and supermalloys on atomic level is important for achievement soft magnetic properties of the alloys. Suitable structure of the alloys (produced usually in the form of thin sheet) having initial permeability much larger than that of pure iron arises after proper heat treatment. The form of a sheet is not suitable for some applications and therefore it is logical to attempt to prepare such material in more "bulk" form, for example in the form of a cylinder or a ring, which would be more convenient for construction of some type of devices. One of the suitable methods for preparation of 3-D samples is compaction of the powder prepared by mechanical milling or mechanical alloying [2].

Mechanical milling is a useful powder processing technique that can produce a variety of equilibrium and non-equilibrium alloy phases [3]. Some researches concerning FeNi and FeNiMo powders system produced by mechanical milling were reported in [4].

The aim of this work was to investigate the structure and magnetic properties of powdered and compacted $Fe_{19}Ni_{81}$(wt. %) and $Fe_{16}Ni_{79}Mo_5$(wt. %) alloys prepared by the mechanical milling and subsequently by a hot compaction of the powder.

2. Experimental methods

We have prepared two types of powder samples. The sample $Fe_{19}Ni_{81}$ was prepared by mechanical milling of microcrystalline ribbon FeNi (81 wt.% of Ni) obtained by melt-spinning, which is suitable for milling. The sample$Fe_{16}Ni_{79}Mo_5$ was prepared by the milling of the swarfs of FeNiMo (79 wt.% of Ni, 16 wt.% of Fe), which were prepared from an ingot by turning. We used swarfs, because it was not possible to prepare ribbons with this chemical composition.

The mechanical milling was performed in a protective argon atmosphere in a high-energy planetary ball mill (RETSCH PM4000) with ball-to-powder-ratio of 6:1in hardened steel vials and with a speed of 180 rpm. The $Fe_{16}Ni_{79}Mo_5$ sample was milled in a vialcooled in liquid nitrogen. The handling of the powder was performed in a glove box with a controlled atmosphere (O_2< 1 ppm, H_2O < 1 ppm)[4, 5].

Fig.1:*Ribbon$Fe_{19}Ni_{81}$(a), swarfs$Fe_{16}Ni_{79}Mo_5$(b), milled ribbon $Fe_{19}Ni_{81}$ (c),milledswarfs$Fe_{16}Ni_{79}Mo_5$ (d) and bulk samples (e) prepared by the compaction.*

The powder samples were consolidated at 800 MPa for 5 min. at 600 °C into discs with a diameter of 10 mm and a height of approximately 2.5 mm. An axial hole with a diameter of 5 mm was drilled into the disc to produce ring samples. We have wound primary coil with 35 turns and secondary coil with 50turns for ac and dc measurements. The magnetic induction B is sinusoidal when measuring the frequency dependent hysteresis cycle.

The structure and morphology of the samples were characterised by the XRD investigations (Philips PW 1050 diffractometer with Co-Kα radiation).

The dc hysteresis loops at maximum induction of 0.2 T were measured by a fluxmeter-based hysteresis graph. The ac hysteresis loops were measured in the frequency range from 1 z to 50 Hz. The coercivityof the powder and bulk samples was measured by a Förster Koerzimat at room temperature.

3. Structure

The structure and morphology of the powder and compacted samples$Fe_{19}Ni_{81}$ and $Fe_{16}Ni_{79}Mo_5$ were investigated by x-ray diffraction.

XRD patterns of milled $Fe_{19}Ni_{81}$ ribbon and $Fe_{19}Ni_{81}$ compacted powder verifies our assumption of stability of the FeNi alloy during milling. There are present only peaks of $FeNi_3$, Fig. 2[5]. It means that no additional ferromagnetic FeNi phase is created during milling. The same results were founded for the $Fe_{16}Ni_{79}Mo_5$ powder and $Fe_{16}Ni_{79}Mo_5$ compacted samples (Fig. 2) and these results were reported in [6].

The XRD analysis revealed that the milling of the $Fe_{19}Ni_{81}$microcrystalline ribbon and $Fe_{16}Ni_{79}Mo_5$swarfs and the compaction of these powders have no significant influence on the structure of the material [4].

4. Coercivity

The coercivity of the powder sample increases with milling time and we assume that displacement of the domain walls becomes less and less important magnetization process with milling time and the rotation of magnetization vector becomes more dominant. The magnetization process of the powder material is realized moreor less separately for each powder element[5, 6].

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. The annealing at higher temperatures causes relaxation of residual stresses introduced during milling and compaction and improves contact between powder particles, causing lowering of the coercivity. The lowest coercivity11A.m^{-1} was achieved for $Fe_{19}Ni_{81}$sample prepared by

compaction (600°C) of broken ribbon, annealed at 1200 °C [4], and it is comparable with that for material prepared by convention way in the form of thin sheet [7], Fig. 3.

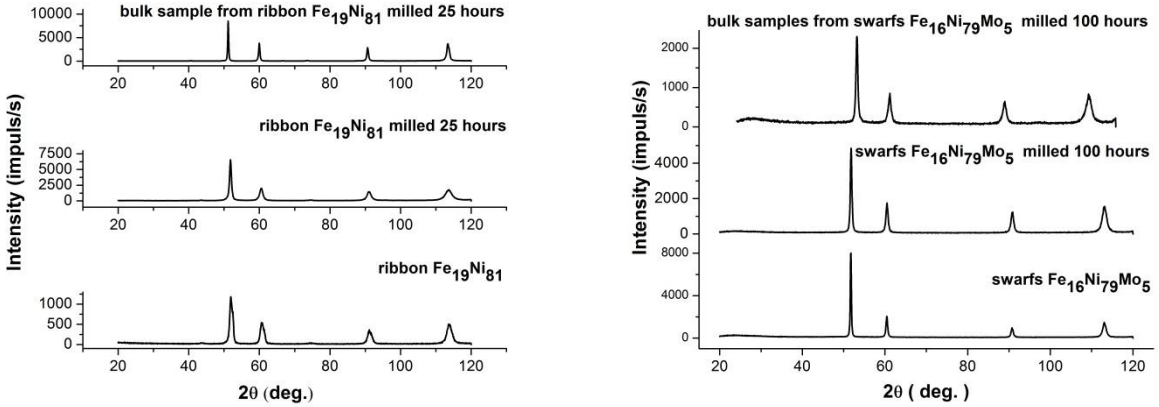

Fig.2:*XRD analysis for $Fe_{19}Ni_{81}$ and $Fe_{16}Ni_{79}Mo_5$ powdered and compacted samples.*

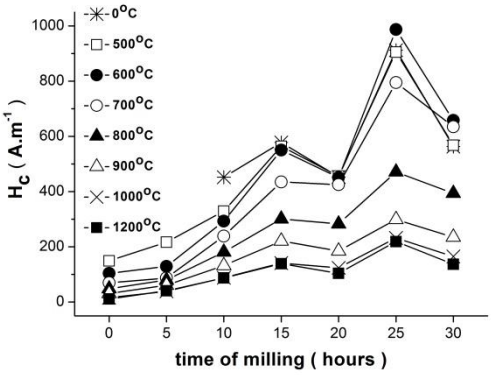

Fig.3:*The coercivity of bulk samples prepared by the compaction of powder $Fe_{19}Ni_{81}$ milled from 0 to 30 hours and compacted at the range 30°C - 1200°C.*

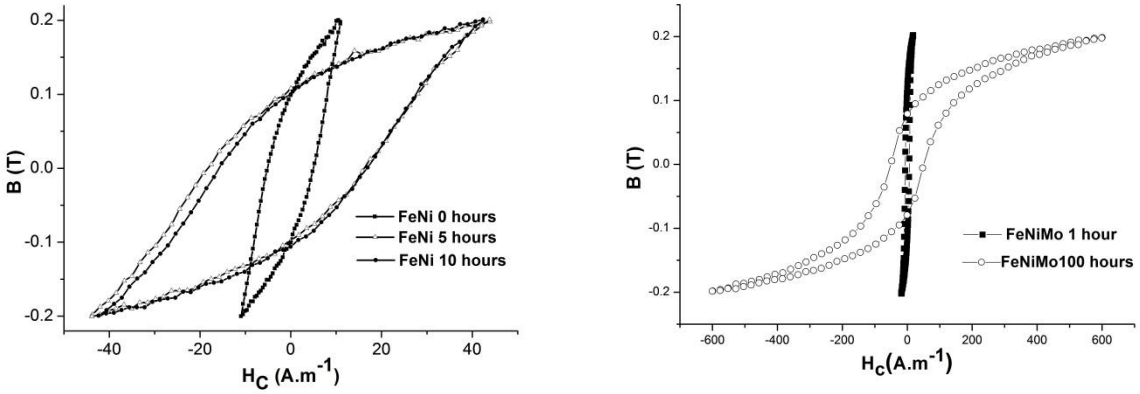

Fig.4:*The dc hysteresis loops of bulk samples $Fe_{19}Ni_{81}$ and $Fe_{16}Ni_{79}Mo_5$ prepared by the compaction of powder miller the different time.*

In the case of $Fe_{16}Ni_{79}Mo_5$samples the coercivity decreases with increasing annealing temperature, reaching a minimum value of 11.2 $A.m^{-1}$ (swarfs $Fe_{16}Ni_{79}Mo_5$milled 1 hour and compacted at 600 °C and annealed at 1100 °C) [8,9].

In order to prepare ring-shaped samples more suitable for AC and DC measurements, the cylinders of bulk samples were drilled using spark plasma erosion (the diameter of the holeis 5 mm). The coercive field increases with milling time for both types of samples as can be followed from DC hysteresis loops measured at 0.2 T, Fig. 4.

5. Conclusion

The soft magnetic properties of the $Fe_{19}Ni_{81}$ and $Fe_{16}Ni_{79}Mo_5$ alloys have been investigated. From the above study, we conclude that the magnetic properties of the permalloys and supermalloys show strong dependence to their initial master powder and annealing conditions. The bulk sample $Fe_{16}Ni_{79}Mo_5$ is found to show better soft magnetic properties over the $Fe_{19}Ni_{81}$ bulk sample as a function of different process parameters. We have prepared bulk samples in the form of the small cylinders with coercivity down to approximately 11 A/m. The discussed alloys show very good soft magnetic properties that can be tailored to the need of certain requirements by adjusting the chemical composition, the processing routes such as compaction, and heat treatment conditions. They have more degrees of freedom for tailoring their magnetic properties due to their flexibility in composition, shape and dimensions.

Acknowledgement

This work was part of the project "The progressive technology for the preparation of microcomposite materials for electrotechnics", ITMS:26220220105 supported by the Operational Program "Research and Development" financed through the European Regional Development Fund. by the Scientific Grant Agency of the Ministry of Education of the Slovak Republic and the Slovak Academy of Sciences, project No. VEGA 1/0330/15 and No.VEGA 1/0377/16 and KEGA 072TUKE-4/2014.

References:

[1] J. M. D. Coey: Magnetism and Magnetic Materials, Camridge University Press (2014).
[2] A. P. Hammersley, S. O. Svensson, M. Hanfland, A. N. Fitch, D. Häusermann: *High Press. Res.*, **14,** 235 (1996).
[3] C. Suryanarayana: *Progress in Materials Science*, **46**, 1 (2001).
[4] D. Olekšáková, P. Kollár, J. Füzer, M. Kusý, S. Roth, K. Polanski: *Journal of Magnetism and Magnetic Materials*, **316**, e838 (2007).
[5] D. Olekšáková, S. Roth, P. Kollár, J. Füzer: *Journal of Magnetism and Magnetic Materials*, **304,** e730 (2006).
[6] D. Olekšáková, J. Füzer, P. Kollár, J. Bednarčík, C. Lathe: *Acta Physica Polonica A*, **126**, 128 (2014).
[7] A. H. Morrish, The physical Principles of Magnetism, E. Krieger Publ. Co, Huntington (1980).
[8] J. Füzer, P. Kollár, D. Olekšáková, S. Roth: *Acta Physica Polonica A*, **113**, 59 (2008).
[9] J. Füzer, P. Kollár, D. Olekšáková, S. Roth: *J. Alloys and Compd.*, **483**, 557 (2009).