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Magnetic Properties of Crystalline NiFe Alloy Prepared by High-Energy Ball Milling and Compacting

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The structure and magnetic properties of compacted microcrystalline NiFe (81 wt% of Ni) powder is investigated. The powder of NiFe alloy prepared by ball milling of ribbon (prepared by melt spinning) remains single phase material suitable for compaction in order to prepare soft magnetic material. The bulk samples were consolidated by uniaxial compaction of the powder in vacuum. By measuring of AC and DC magnetic properties it was found out that in bulk samples the displacement of domain walls is the dominant magnetization process, while rotation of magnetization vectors prevails in powder material.

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1. Introduction

There is no doubt that the magnetic materials represent today one of the important issues of the research. Permalloy is a name which has been given to a series of nickel–iron alloys, which after heat treatment have an initial permeability much larger than that one of pure iron and are produced usually in the form of thin sheet [1]. Therefore it is logical to attempt to prepare such material in a more "bulk" form, for example in the form of a cylinder or a ring, which would be more convenient for some industrial applications.

Over the past several years the method of mechanical milling and mechanical alloying has been widely spread in order to exploit it to produce a variety of equilibrium and non-equilibrium alloy phases and it possesses further possibility for research work and application of permalloy [2–4]. One of the methods how to prepare material in bulk is to compact the powder. The aim of this work was to investigate the influence of powder size on AC and DC magnetic properties of the bulk samples prepared by hot compaction of the powder.

2. Sample preparation

The precursor for compaction is NiFe (81 wt% of Ni) very brittle microcrystalline ribbon (27 μm thick) obtained by melt spinning in Ar-protective atmosphere. Small pieces (several mm^2) of the ribbon prepared by crushing in the hands were used for preparation of the sample NiFe(I). The powder (the mean size of the powder particle is 10 μm) sample (NiFe(II)) was prepared by mechanical milling of the ribbon in a high-energy planetary ball mill with ball to powder ratio (BPR) of 6:1 at a speed of 180 rpm for 30 h. The bulk samples were prepared by uniaxial compaction of the small pieces of the ribbon (NiFe(I)) and of the powder (NiFe(II)) in the form of cylinders (diameter 10 mm, height 2.5 mm, weight approximately 2 g). The compaction was performed at a pressure of 800 MPa for 5 min at 600 °C in a vacuum of 5×10^3 Pa (in order to prevent oxidation and to remove free gases from a powder before the compaction). The mean value of the particle size of both samples was reduced during compaction to 5 μm for NiFe(I) and below 1 μm for NiFe(II) sample. The cylinders were annealed at temperatures between 500 °C and 1200 °C for 1 h. In order to prepare ring-shaped samples more suitable for AC measurements, the cylinders of bulk samples were drilled using spark plasma erosion, the diameter of the hole is 5 mm [5, 6].

3. The structure and morphology

In order to prepare the bulk soft magnetic sample by compaction of the powder it is necessary to have a powder which consists of one ferromagnetic phase only (in the opposite case, the resulting compact could exhibit hard magnetic properties for example due to domain walls pinning on minority phase). Figure 1 shows XRD pattern of 30 h milled NiFe ribbon and verifies our assumption of stability of the NiFe alloy during milling. The studied diffraction pattern documented in Fig. 1 proves presence of $FeNi_3$ as a major component with phase fraction at

least 90%. Minor phases are represented with FeO, because the powder sample was partially oxidized before X-ray investigation during exposition of oxygen for a relatively long time.

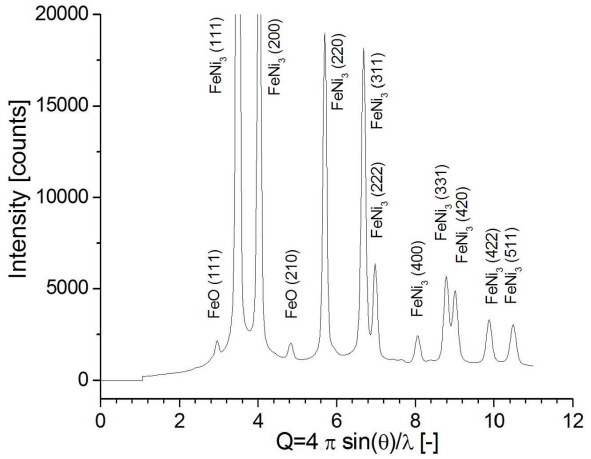

Fig. 1. XRD pattern of 30 h milled NiFe ribbon performed on the Wiggler beamline BW5 at the DORIS III positron storage ring (HASYLAB Hamburg, Germany) using monochromatic photon beam with energy of 99 keV ($\lambda = 0.01252$ nm).

4. The magnetic properties

For soft magnetic materials, it is convenient to have the absolute value of magnetostriction constant as low as possible, to get rid of the stresses effect. The magnetostriction represents a stress that has to be taken into account by the designer, especially because it may induce anisotropy when the material is liable to stresses. The absolute value of the magnetostriction of the NiFe bulk samples was checked by the strain gauge method to be below 2 ppm [6], so it was assumed that residual stresses introduced during milling and compaction process do not cause a significant additional anisotropy and further increase of the coercivity. The coercivity of bulk samples decreases up to 10 times in comparison to powder samples due to renewing of the "magnetic contact" between the powder particles. The compact is magnetized dominantly by domain walls displacement in contrast with powder in which magnetization vector rotation is a dominant magnetization process [7]. The annealing at higher temperatures causes relaxation of residual stresses introduced during milling and compaction, improving the contact between powder particles, causing lowering of the coercivity [5], Fig. 2. The lowest value of coercivity (11 A/m) was achieved for the sample prepared by compaction of broken ribbon, annealed at 1200 °C. The relief of the mechanical stress induced during milling and pressing influences also the hysteresis loops. Annealing at 1200 °C decreases the total power losses in both alloys by approximately a factor 2.

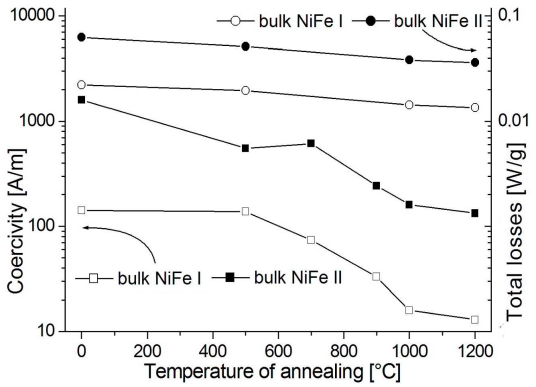

Fig. 2. DC coercivity and total losses ($f = 1$ kHz, $B_m = 0.2$ T) versus temperature of annealing for the NiFe(I) and NiFe(II) samples.

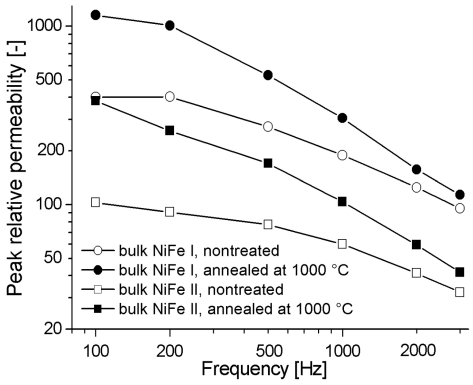

Fig. 3. Frequency dependence of the peak permeability for NiFe(I) and NiFe (II) samples for $B_m = 0.2$ T before heat treatment and after annealing at 1000 °C.

Figure 3 shows the peak permeability μ_p (determined as $\mu_p = B_{max}/H_{max}$ was from B–H loops), as a function of frequency for NiFe(I) and NiFe(II) samples. The NiFe(I) sample consisting of larger powder elements offers longer trajectories for domain walls displacement, which leads to the larger values of peak relative permeability in comparison with NiFe(II) sample. It can be supposed that annealing reduces the internal stresses in the material acting as pinning centres for domain walls motion. This phenomenon results in the increase of the values of the peak relative permeability for both samples in the frequency dependence as a consequence of the increase of domains walls mobility.

5. Conclusions

NiFe-based microcrystalline cores were prepared by hot pressing of small pieces of broken ribbon and of the 30 h milled ribbon, respectively. The powder remains single phased after 30 h milling. Magnetic properties of the bulk permalloys show strong dependence to its initial, master powder and annealing conditions. The lowest value of the

coercivity of the cylinder-shaped NiFe sample is 11 A/m which is comparable with the value of the coercivity of conventional permalloy. The compaction at higher temperatures and further annealing results in increase of the peak relative permeability and the decrease of the DC coercivity and core losses.

Acknowledgments

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