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EFFECT OF HEAT-TREATMENT ON MAGNETIC HYSTERESIS IN Nd-Fe-B BASED MAGNETS

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Abstract. – The optimum magnetic properties of Nd(Dy)-Fe(Al)-B magnets are obtained after a post-sintering heating at 900 °C and a subsequent annealing at 600 °C. SEM studies show irregular grain shapes in sintered samples changing to regular polyhedra after the heat-treatment. The increase in coercivity may be related to these changes.

Introduction

In the technology of Nd-Fe-B based magnets, heat-treatment plays an important role in optimizing the magnetic properties and especially coercivity [1]. Much work has been already devoted to reveal the structural features at the grain boundaries [2] and the minority phases [3] which may control coercivity. In this paper we would like to focus more on the differences in grain morphology and magnetic properties between samples subjected to a different heat-treatment.

Experiment

The magnets were received from IG technologies. The green compacts were vacuum sintered for 1 hour at 1100 °C. After cooling some of them were heat treated for 1 hour at 900 °C, and finally some were annealed for 1 hour at 600 °C for optimum coercivity. Magnetic measurements were made using a VSM magnetometer in the temperature range of 300-550 K. Microstructure studies and composition analysis were performed with a Jeol 100C scanning transmission electron microscope equipped with an energy dispersive X-ray detector.

Results and discussion

MAGNETIC PROPERTIES. – The demagnetization curves of sintered, sintered and heat-treated, and sintered heat-treated and annealed samples are shown in figure 1. After magnetizing in a field of 30 kOe the three samples mentioned above had coercivities at room-temperature of 11.4, 12.2 and 18.5 kOe, respectively. The initial curves for all three samples were steep indicating a nucleation or localized pinning-type coercivity mechanism at grain boundaries [4, 5]. It is worth noticing that annealing at 600 °C leads to a substantial increase of coercivity (about 50 % ). On the contrary, the post-sintering heat treatment at 900 °C gives a minor increase in \( H_c \).

The field dependence of coercivity is shown in figure 2. The coercivities remain negligible up to an external field of about 8 kOe and then increase drastically. The steepest increase of coercivity is observed for annealed samples. It is clear from figure 2 that for annealed samples an applied field of 12 kOe leads to a much higher coercivity (\( H_c > 12 \) kOe). This behavior indicates a non-uniform domain wall pinning and can

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only be explained by nucleation or localization pinning at grain boundaries [5].

Figure 3 shows the temperature dependence of the normalized to room temperature coercivity $H_c(\text{RT})$ in the temperature range between 300-500 K. The coercivity of the samples without annealing drops very rapidly with increasing temperature. The post-sintering heat treatment does not change the temperature dependence of coercivity. The annealed samples show a much slower decrease of coercivity with temperature which is more desirable for practical applications.

**Microstructure Studies.** — Studies of the differently heat-treated magnets performed with scanning electron microscopy on fractured and polished samples, revealed a morphology consisting of the majority 2:14:1 phase grains ($\sim$ 8-15 μm) and smaller grains of minority B-rich and Nd-rich phases (Fig. 4). The biggest difference in morphology between the different kind of samples was in the shape of grains of Nd$_2$Fe$_{14}$B phase. The shape of the grains after sintering is irregular with a lot of sharp edges, corners and concave surfaces. The post-sintering and subsequent annealing processes change the grain shapes to more regular polyhedra having flat surfaces and less sharp edges and corners. In our opinion the irregular shapes, sharp edges or corners might be the places of higher demagnetizing fields and therefore easier nucleation or unpinning of domain walls.

Transmission electron microscope studies show a rather perfect crystal structure of the main phase for all samples. Interactions between domain walls in the main phase and the spherulites of Nd-rich phase were observed. However, we do not think that this interaction is strong enough to explain the high coercivity.

In conclusion we think that the changes of coercivity which occur after heat-treatment are related to macroscopic changes of the grain morphology rather than microscopic changes at around grain boundaries.

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