A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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D. PŁUSA*, M. DOŚPIAŁ*, D. DEREWNICKA-KRAWCZYŃSKA**, P. WIECZOREK*** , U. KOTLARCZYK*

THE DOMAIN STRUCTURE OF DIE-UPSET ANISOTROPIC MAGNET BASED ON Nd–(Fe, Co)–B ALLOY

STRUKTURA DOMENOWA ANIZOTROPOWEGO ODKSZTAŁCONEGO NA GORĄCO MAGNESU NA BAZIE ZWIĄZKU Nd–(Fe, Co)–B

The measurements of the recoil curves for the die-upset Nd-(Fe,Co)-B based magnets from different points on the magnetization and demagnetization curves have been carried out by means of the LakeShore vibrating sample magnetometer in an applied magnetic fields up to 2 T. From the recoil curves the so-called Wohlfarth's remanence relationship has been derived. From this it was deduced that the magnetic interaction existing between the magnet grains has a dipolar nature. The existence of the magnetic interaction has been confirmed by magnetic domain observations by using the magnetic force microscopy (MFM). In the area of interaction domains there is the fine scale magnetic contrast resulting from the dipolar interaction between neighboring grains.

Keywords: exchange interactions, high coercivity materials, permanent magnets, domain walls and domain structure, magnetization reversal mechanisms

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Za pomocą magnetometru wibracyjnego firmy LakeShore wyznaczono krzywe powrotne dla spęczanych magnesów Nd-(Fe, Co)-B w polu magnetycznym do 2 T. Na podstawie krzywych powrotnych wyznaczono zależność remeanencyjną Wohlfartha. Z zależności tej wynika, że oddziaływania magnetyczne pomiędzy ziarnami to dalekozasięgowe oddziaływania dipolowe. Obecność tych oddziaływań została potwierdzona przez obserwację struktury domenowej za pomocą mikroskopu sił magnetycznych (MFM). Oscylacyjne zmiany kontrastu, występujące w obszarach zbliżonych do wielkości ziaren, świadczą o istnieniu uporządkowania magnetycznego w tych obszarach, które jest wynikiem oddziaływania dipolowego między sąsiednimi ziarnami.

1. Introduction

The Nd-Fe-B permanent magnets prepared from melt-quenched ribbons are isotropic. One of the methods for producing the anisotropic magnets and improving their magnetic properties is the hot deformation (die upset forging) process. The magnets produced by using this technique possess the higher remanence $\mu_0 M_R$ and the maximum energy product $(BH)_{max}$ and lower coercivity $\mu_0 H_C$ compared with the isotropic ones. The higher maximum energy product the smaller magnet size and device using it. Therefore the efforts of scientists and engineers have focused on increasing the $(BH)_{max}$ depending on both the coercivity and remanence.

The reason for decreasing in coercivity is the anisotropic grain growth in the direction perpendicular to the deformation one. Another reason is the interactions (magnetostatic or exchange) between the neighboring hard magnetic grains. These interactions result in the existence of the so-called interaction domains. The most powerful technique to investigate the interaction domains is the magnetic force microscope MFM. There are only a few works dealing with the investigation of interaction domains in die- upset magnets [1-4].

Therefore the main purpose of this paper is to investigate the nature of magnetic interactions in die upset magnets and their relation with the magnetic domain structure.

* INSTITUTE OF PHYSICS, CZESTOCHOWA UNIVERSITY OF TECHNOLOGY, 42-200 CZĘSTOCHOWA, 19 ARMII KRAJOWEJ STR., POLAND
** INSTITUTE OF PRECISION MECHANICS, 01-796 WARSAW, 3 DUCHNICKA STR., POLAND
*** INSTITUTE OF MATERIAL SCIENCE ENGINEERING, CZESTOCHOWA UNIVERSITY OF TECHNOLOGY, 42-200 CZĘSTOCHOWA, 19 ARMII KRAJOWEJ STR., POLAND

2. Material and experimental details

In this work the anisotropic $Nd_2(Fe, Co)_{14}$ B-type magnet with the composition of $Nd_{13,6}Fe_{73,7}Co_{6,6}Ga_{0,6}B_{5,5}$ has been investigated. The magnet was produced using the hot pressed Magnequench powder obtained from melt spun ribbons. Material prepared by this route was then hot deformed at 750^0C in a steel capsule by means of the apparatus INSTRON 8035 with maximum pressure force of 500 kN. The hot deformation process was performed at a strain rate (ε') of 0.002 s^{-1}. A deformation degree defined as $\varepsilon = (\Delta h/h_0)\cdot 100\%$ was equal to 65% (Δh is the change in the sample high after deformation, h_0 is the starting high). The strain rate $\varepsilon' = \Delta\varepsilon/\Delta t$ [s^{-1}].

Using the LakeShore vibrating sample magnetometer (VSM) the sets of recoil curves have been measured in an external magnetic field up to 2 T. These curves were obtained after applying the certain magnetizing field of $\mu_0 H_i$ (i = 1, 2, … 40), reducing it to zero (magnetization remanence state of $\mu_0 M^r(\mu_0 H_i)$) and increasing back to $\mu_0 H_i$. In the next step the value of magnetizing field was increased by 0.05 T and the procedure was repeated. The same procedures have been performed in a negative field direction -$\mu_0 H_i$ starting from a remanence $\mu_0 M_R$ state after saturating by a positive field of 14 T. After reducing a negative field to zero value the sample was in the demagnetization remanence state of $\mu_0 M^d(\mu_0 H_i)$. From the sets of recoil curves the relationship between the reduced demagnetization remanence M^d/M_R and reduced magnetization remanence M^r/M_R have been derived.

Magnetic domain structure has been observed by means of the magnetic force microscopy (MFM) MESP Nanoscope V, Multimode-type with the Co/Cr tip. The magnetic contrast imaging was performed in tapping (Atomic Force Microscopy)/lift (Magnetic Force Microscopy) mode at the tip high of 100 nm. The signal was obtained by measuring the phase shift of an oscillating cantilever. The oscillation phase changes which occur under the influence of magnetic force gradient acting on the tip were processed into the contrast changes.

The surface of the studied magnet were polished using abrasive paper, diamond pastes and diamond powder of the different gradations. Domain structure was observed for an ac-demagnetized sample.

3. Results and discussion

Fig. 1 shows the dependence of the reduced demagnetization remanence M^d/M_R versus the reduced magnetization remanence M^r/M_R along with the Wohlfarth relation (1), which is obeyed for any system of noninteracting single domain particles [5]:

$$\frac{M^d}{M_R}(H_i) = 1 - 2\frac{M^r}{M_R}(H_i), \tag{1}$$

Any deviations from the straight line are interpreted as being due to the magnetic interactions between the neighboring grains. All experimental points lie below the line (1) certifying that in the magnet investigated there exist the long range dipolar magnetic interaction.

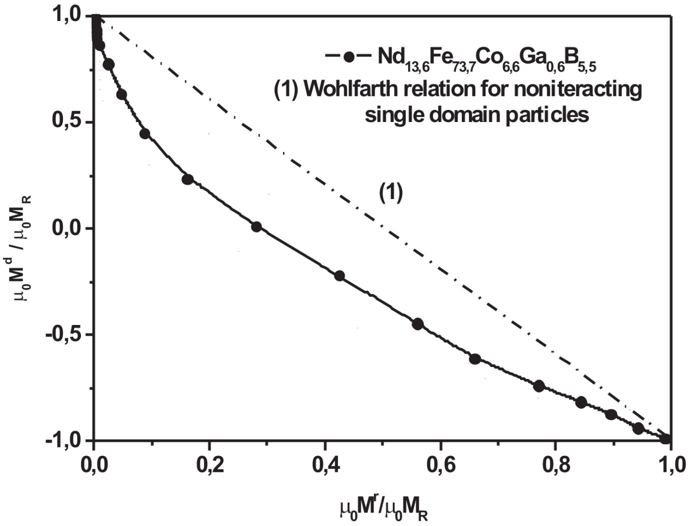

Fig. 1. Wohlfarth remanence curve for $Nd_{13,6}Fe_{73,7}Co_{6,6}Ga_{0,6}B_{5,5}$ magnet

The interactions between the grains and particles are the reason for forming the so-called interaction domains. For hot deformed $Nd_{13,6}Fe_{73,7}Co_{6,6}Ga_{0,6}B_{5,5}$ magnet at the deformation degree of 65% the majority of grains on the surface perpendicular to the deformation direction have diameter from 360nm to 500 nm [1]. Fig. 2 illustrates the MFM image with a scan size 5μm×5μm on such surface. Fig. 3 shows the change of magnetic contrast along the scanning line marked in Fig. 2. The width of bright domain amounts to 1.778 μm. However the dark domains on the left-hand side and the right-hand side of the bright one have the width of 0.994 μm and 0.783 μm, respectively. The domain sizes are larger than the grain size of the sample. One can suppose that observed domains are the interaction domains. The characteristic feature of MFM image is the oscillating change of magnetic contrast with significant amplitude of 6-7° occurring in both the bright and dark areas.

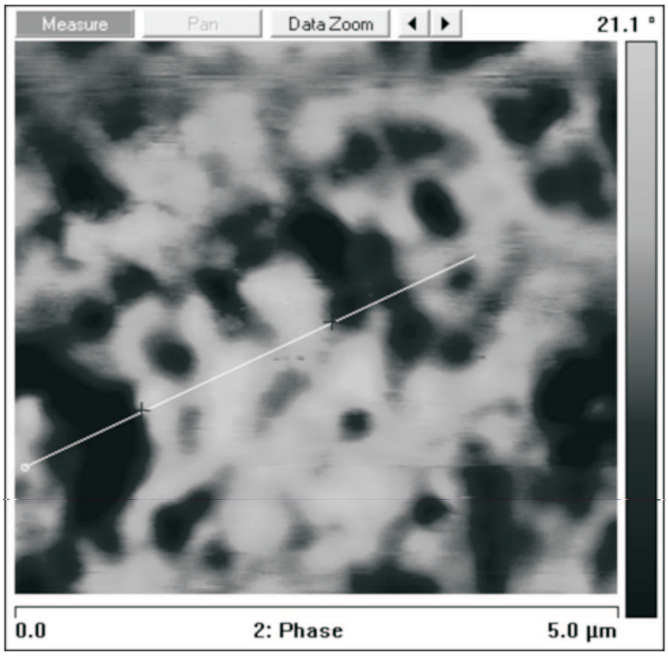

Fig. 2. MFM image of the sample $Nd_{13,6}Fe_{73,7}Co_{6,6}Ga_{0,6}B_{5,5}$ on the surface perpendicular to the deformation direction. Scan size is 5 µm×5 µm

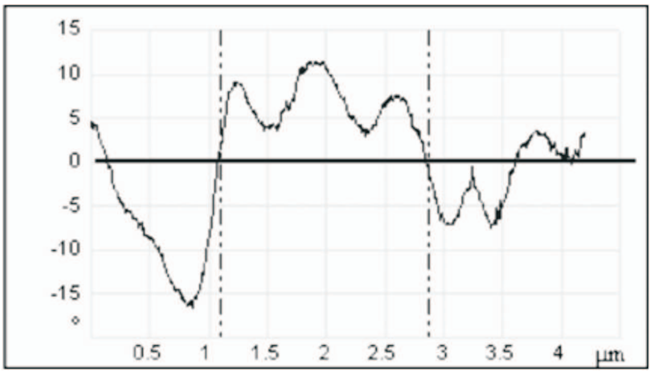

Fig. 3. Magnetic contrast changes along the line marked in Fig. 2

These changes occur in the area comparable with the grain size. Similar changes of contrast have been observed in magnets produced on the base of Nd-Fe-B alloys but an amplitude of changes was smaller [4, 6]. Such behaviour of magnetic contrast can be attributed to the existence of certain order of the grains as a result of dipolar magnetic interaction. For the magnet composed of grains with size of a few hundreds nanometers exchange coupling interaction is very weak and can not make the grains align.

4. Conclusions

From Wohlfarth's remanence curve results that between the neighboring grains there exist the magnetostatic (dipolar) interactions. The interaction domains observed in the investigated magnet confirm this fact. In both the bright and dark domain area the magnetic contrast changes oscillatory as a result of dipolar (magnetostatic) interaction between the adjacent grains.

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