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Inducing anisotropy in bulk Nd–Fe–Co–Al–B nanocrystalline alloys by quenching in magnetic field

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ABSTRACT

We have observed magnetic anisotropy in bulk $\rm{Nd}_{55-x}Co_xFe_{30}Al_{10}B_5$ (x $=$ 10, 15 and 20) alloys prepared by copper mold suction casting method with a presence of external magnetic field (quenching field) μ_0 H = 0.25 T. By changing direction of the measuring field from perpendicular to parallel one in comparison with that of the quenching field, coercive force of the alloys slightly decreases while remanent magnetization and squareness of hysteresis loop increase more clearly. It is also found that the higher Co-concentration in the alloys the larger magnetic anisotropy is induced. The structure analyses manifest nanocrystalline particles embedded in residual amorphous matrix of the alloys. The size of the particles is in range of 10–30 nm and their crystalline phases consist of $Nd_2(Fe, Co)_{14}B$, $Nd₃Co$, $Nd₃Al$, $NdAl₂$ and Nd .

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1. Introduction

A challenge in raising maximum energy product $(BH)_{\text{max}}$ of hard magnetic nanocomposites or exchange spring magnets is how to make magnetic anisotropy in these materials. That means if we find methods to produce nanocomposites with their magnetic nanocrystalline grains aligned together, the maximum energy product of these hard magnetic materials can approach theoretical limit (\sim 1000 kJ/m³). Up to now, there have been considerable efforts of scientists, which were devoted to overcome this challenge. Several methods have been used to fabricate anisotropic nanocomposites or anisotropic nanocrystalline materials such as hot deformation [\[1–3\]](#page--1-0), spark-plasma sintering [\[4,5\]](#page--1-0), addition of anisotropy-induced elements [\[6–8](#page--1-0)]. In this work, we induced the magnetic anisotropy for bulk Nd–Fe–Co–Al–B nanocrystalline alloys by casting in magnetic field. Although, there were publications reporting the possibility of inducing anisotropy by quenching melting materials in magnetic field [\[9–11](#page--1-0)], most of the investigated materials were neither nanocrystalline nor bulk materials. The alloys we investigated in this work have large glass

* Corresponding author. E-mail address: [dannh@ims.vast.ac.vn \(N.H. Dan\)](mailto:dannh@ims.vast.ac.vn). forming ability. That means they could be easily fabricated in bulk forms with amorphous or nanocrystalline structures [\[12–14\]](#page--1-0).

2. Experiment

The bulk $Nd_{55-x}Co_xFe_{30}Al_{10}B_5$ ($x=10$, 15 and 20) alloys in rod form with dimensions of $10 \times 1 \times 30$ mm³ were prepared from pure components of Nd, Fe, Co, Al and FeB by copper mold suction-casting method on an arc-melting system. To create the quenching field (the magnetic field applied to the alloys during the casting process), two Nd–Fe–B permanent magnets were placed beside the copper mold. A strength of 0.25 T of the magnetic field was measured inside the copper mold. [Fig. 1](#page-1-0) shows schematic illustration of experimental setup for preparing the anisotropic bulk nanocrystalline alloys by suction casting in magnetic field. The alloys were first melted several times to ensure their homogeneity and then were brought to the top of the copper mold by using the sample turning rod. After that, the alloys were arc-melted quickly, to avoid heating the copper mold, and sucked to the copper mold by a vacuum pump. The arcmelting process was performed under Ar atmosphere using Ti to gather gas-contaminations. The structure of the alloys was examined by X-ray diffraction and electron microscopy methods. The high resolution transmission electron microscopy (HRTEM)

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Fig. 1. Schematic illustration of experimental setup for preparing the anisotropic bulk nanocrystalline alloys by suction casting in magnetic field.

and selected area electron diffraction (SAED) images were carried out on a Philip CM20-FEG apparatus operated at $V = 200$ kV and $C_s=1.2$. The specimen preparation for the HRTEM and SAED investigations, was done by cross-section and plane-view methods, at the final step the specimens were milled by ion milling in Ar with a RES 100 system. The magnetic properties of the alloys were investigated by hysteresis measurement on a pulsed field magnetometer. The specimens for the magnetic hysteresis measurement were cut from the bulk alloys by electric spark technique. They are in cubic form with dimensions of $1 \times 1 \times 1$ mm³. One axis of the cubes was kept coincidentally with the direction of the quenching field.

3. Results and discussion

Fig. 2 shows hysteresis loops of a representative alloy, i.e. the alloy with $x=15$, which were measured in the magnetic field parallel and perpendicular to the quenching field. A difference between the hysteresis loops is clearly observed for the alloy quenched in the magnetic field of 0.25 T (Fig. 2a), while these two hysteresis loops of the alloy quenched in zero magnetic field are almost coincided (Fig. 2b). The difference between the demagnetization curves in the second quadrant of the former case can be observed more clearly in the inset of the Fig. 2a. Thus, the quenching field can induce magnetic anisotropy for the alloys. From Fig. 2a, one can also realize that coercive force of the alloy is slightly different, while remanent magnetization changes in a larger magnitude with changing direction of measuring field. It should be noted that the squareness of the hysteresis loop of the alloy is improved considerably by quenching in magnetic field. The squareness of the parallel-hysteresis loop is better than that of the perpendicular-one.

As for the other alloys ($x=10$ and 20), the anisotropy feature of them is similar to that of the alloy with $x=15$. For all the alloys the coercive force measured in the direction parallel to the quenching field is smaller than one measured in the perpendicular direction. On the contrary, the remanent magnetization of all the alloys increases considerably by measuring in the same way. [Table 1](#page--1-0) indicates values of the coercive forces, remanent magnetizations and maximum magnetizations (in the magnetic field of 5 T) of the alloys measured in the parallel- and perpendicular-magnetic field. We can realize that the change of these values from the perpendicular-case to the parallel-one, i.e. magnetic anisotropy of the alloys, slightly increases with increasing Co-concentration. This trend of the magnetic anisotropy as well as variation of the measured magnetic quantities versus Co-concentration of the alloys can be observed easier in [Fig. 3.](#page--1-0)

Fig. 2. Hysteresis loops of the $Nd_{40}Co_{15}Fe_{30}Al_{10}B_5$ alloy measured in the magnetic field parallel and perpendicular to the quenching field of 0.25 T (a) and 0 T (b). The inset shows demagnetization curves in the second quadrant of the hysteresis loops.

When the Co-concentration is increased from 10 at% to 20 at%, the change of the coercive force is raised from \sim 4% to \sim 7%. As for the remanent magnetization, the change is larger, from \sim 11% to \sim 15%. The magnetic anisotropy induced in the alloys probably is due to magnetic crystalline grains, which were crystallized orientationally under the presence of the external magnetic field during the solidification of the melts. The increase of the magnitude of the anisotropy with increasing Co-concentration in the alloys is supposedly caused by the nature of Co. This element usually makes its compounds such as Fe-Co, $Nd_2(Fe,Co)_{14}B$ phases have higher Curie temperature leading to the higher possibility of the orientational crystallization in the alloy during quenching process. [Fig. 4](#page--1-0) shows thermomagnetization curves measured in magnetic field of 10 mT of the alloys with various concentrations of Co. We can see that Curie temperature of the alloy is considerably elevated from \sim 555 K to \sim 612 K with increasing Co-concentrations from 10% to 20%, respectively.

The preferred orientation following direction of the quenching field of micrometer-grains of some different materials such as $YBa₂Cu₃O₇$, Sm₂Co₁₇ was observed by other authors [\[9,10](#page--1-0)]. The action of the quenching field on the anisotropic particles during solidification was assumed by these authors for preferred crystallization or texture in the materials. The quenching field could force the anisotropic particles in mixture state of solid and liquid

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