



# Anisotropic bulk Nd<sub>2</sub>Fe<sub>14</sub>B/ $\alpha$ -Fe nanohybrid magnets with an enhanced energy product



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## ABSTRACT

Anisotropic bulk Nd<sub>2</sub>Fe<sub>14</sub>B/ $\alpha$ -Fe nanohybrid magnets have been prepared by high-pressure thermal compression (HPTC) of the blends of 70 wt% Nd-lean Nd<sub>9</sub>Fe<sub>85.5</sub>Cu<sub>1.5</sub>B<sub>4</sub> amorphous and 30 wt% Nd-rich Nd<sub>13.6</sub>Fe<sub>73.6</sub>Ga<sub>0.6</sub>Co<sub>6.6</sub>B<sub>5.6</sub> nanocrystalline alloys. The nanohybrid magnets have a high fraction ~24 wt% of  $\alpha$ -Fe phase and exhibit a (001) texture for Nd<sub>2</sub>Fe<sub>14</sub>B nanocrystals in both Nd-lean and Nd-rich regions. The Nd-lean region contains  $\alpha$ -Fe and Nd<sub>2</sub>Fe<sub>14</sub>B nanograins with equivalent sizes of 19 nm and 27 nm and the Nd-rich region comprises Nd<sub>2</sub>Fe<sub>14</sub>B phase having lath nanograins with 20–35 nm in thickness and 50–142 nm in length. This nanohybrid magnet exhibits a maximum energy product of 23 MGOe and an enhanced thermal stability. The present work provides a candidate route to fabricate high-performance bulk magnets for practical applications.

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## 1. Introduction

Nanocomposite magnets consisting of a fine mixture of nanoscale hard- and soft-magnetic phases have attracted tremendous attention for achieving high-performance magnets with less rare-earth metals [1–3]. For bulk nanocomposites, obtaining a crystallographic texture for hard-phase nanocrystals along their easy magnetization axis on the premise of a high fraction of soft phase (>20 wt%) with a small grain size ( $\leq 10$  nm) and a uniform phase distribution is a great challenge [4–6]. Many techniques such as severe plastic deformation [7], hot deformation [8] and bottom-up approaches [9] etc. have been employed to achieve texture for hard magnetic nanocrystals and a substantial progress has been made [10,11], but it still remains a challenge for Nd-Fe-B/ $\alpha$ -Fe bulk nanocomposites with high  $\alpha$ -Fe content ( $V_{\text{Fe}} > 20\%$ ) [8]. For these nanocomposites, a hybridization technique, in which a R-lean R-Fe-B and a R-rich R-Fe-B alloy (R = Nd or Pr) are hybridized to form a hybrid magnet by hot pressing and subsequent die-upsetting is an effective way to induce textures for the R<sub>2</sub>Fe<sub>14</sub>B phase [12]. Unfortunately, though a texture can be obtained in the host R-rich region, it is almost impossible to obtain texture in the R-lean region. Moreover, a rapid deterioration in texture and energy product with increasing the R-lean region, which leads to a low soft-phase content, and an obvious grain growth in both

R-lean and R-rich regions are ineluctable [12–15]. Hence, preparing a bulk nanohybrid magnet with crystallographic texture of R<sub>2</sub>Fe<sub>14</sub>B nanocrystals and fine grain size in both R-lean and R-rich regions is important to improve the energy product [4,5].

Here, we have prepared a bulk Nd<sub>2</sub>Fe<sub>14</sub>B/ $\alpha$ -Fe nanohybrid magnet possessing a (001) texture for Nd<sub>2</sub>Fe<sub>14</sub>B nanocrystals in both Nd-lean and Nd-rich regions and a fine grain size at a high fraction of  $\alpha$ -Fe phase ~24% by employing high-pressure thermal compression (HPTC) of the blends of 70 wt% Nd-lean amorphous and 30 wt% Nd-rich nanocrystalline alloys. This nanohybrid magnet exhibits a high energy product of 23 MGOe and a good thermal stability.

## 2. Material and methods

The Nd-rich Nd<sub>13.6</sub>Fe<sub>73.6</sub>Ga<sub>0.6</sub>Co<sub>6.6</sub>B<sub>5.6</sub> powders (<200  $\mu\text{m}$ ) were bought from Magnequench Inc. The Nd-lean Nd<sub>9</sub>Fe<sub>85.5</sub>Cu<sub>1.5</sub>B<sub>4</sub> powders (<50  $\mu\text{m}$ ) were prepared by arc melting of pure metals and melt spinning at a wheel speed of 26 m/s and then crushing of the ribbons. The Nd-lean and Nd-rich powders were uniformly mixed with a weight ratio of 70:30. The mixtures were consolidated into circular columns with a relative density of 80% in argon atmosphere at room temperature. These bulk samples were put into a steel tube and the high-pressure thermal compression (HPTC) was carried out on the sample units in vacuum by employing Gleeble 3800 machine. The deformation temperature,

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deformation time, height reduction and maximum stress were  $T = 650\text{ }^{\circ}\text{C}$ ,  $t = 45\text{ s}$ ,  $\varepsilon = 79\%$ , and  $\sigma = 820\text{ MPa}$ , respectively.

The microstructures of the magnets were characterized using X-ray diffractometer (XRD) with  $\text{Co K}\alpha$  radiation and transmission electron microscope (TEM). The weight fraction and grain size were determined from XRD patterns using the Rietveld refinement procedure with the HighScore Plus software. The room- and high-temperature magnetic properties were measured using a physical property measurement system and a vibrating sample magnetometer with a maximum magnetic field of 85 kOe and 20 kOe, respectively, and the demagnetization effect was corrected.

### 3. Results and discussion

Microstructure characterizations of the precursors show that the Nd-rich powders are crystalline with a grain size  $\sim 55\text{ nm}$  [Fig. 1(a) and inset] and the Nd-lean powders are completely amorphous (Fig. 1(b)). The deformed bulk magnet is composed of  $\alpha$ -Fe and  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phases with a grain size of 22 nm and 29 nm, respectively, determined from XRD pattern (Fig. 1(c)). The weight fraction of the  $\alpha$ -Fe phase was  $\sim 24\%$  calculated from the XRD pattern. This

demonstrates that the Nd-lean amorphous alloy transforms into  $\alpha$ -Fe and  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phases during HPTC process.

XRD pattern measured on the face perpendicular to the pressure direction [Fig. 1(c)] shows an obvious (00 $l$ ) texture of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  nanocrystals along the pressure direction, which is indicated by the enhanced intensity of (004), (105) and (008) peaks, where the intensity ratio  $I_{(004)}/I_{(220)} = 368\%$  (Fig. 1(d)) is much larger than that 68% for isotropic  $\text{Nd}_2\text{Fe}_{14}\text{B}$  crystals [see the standard powder diffraction file (ICSD): 067224]. This can be further confirmed by the XRD pattern measured on the face parallel to the pressure [Fig. 1(e)], in which reduced intensity of (004), (105) and (008) diffractions was obtained, where  $I_{(004)}/I_{(220)} = 31\%$  (Fig. 1(f)) is lower than that 68% for isotropic  $\text{Nd}_2\text{Fe}_{14}\text{B}$  crystals. These results demonstrate that a bulk nanohybrid magnet with a (00 $l$ ) texture of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phase has been synthesized by HPTC deformation.

TEM images show that the nanohybrid magnets are composed of two different microstructure regions. The major region comprises  $\text{Nd}_2\text{Fe}_{14}\text{B}$  and  $\alpha$ -Fe phases, which derive from the Nd-lean amorphous alloy (Figs. 2(a) and (b)). In this region, many lath-like nanocrystals (indicated by the black arrows in Fig. 2(a) with a thickness of 15–30 nm and a length of 26–45 nm are determined to be  $\text{Nd}_2\text{Fe}_{14}\text{B}$  nanocrystals (Fig. 2(b)). The equivalent sizes of  $\alpha$ -Fe

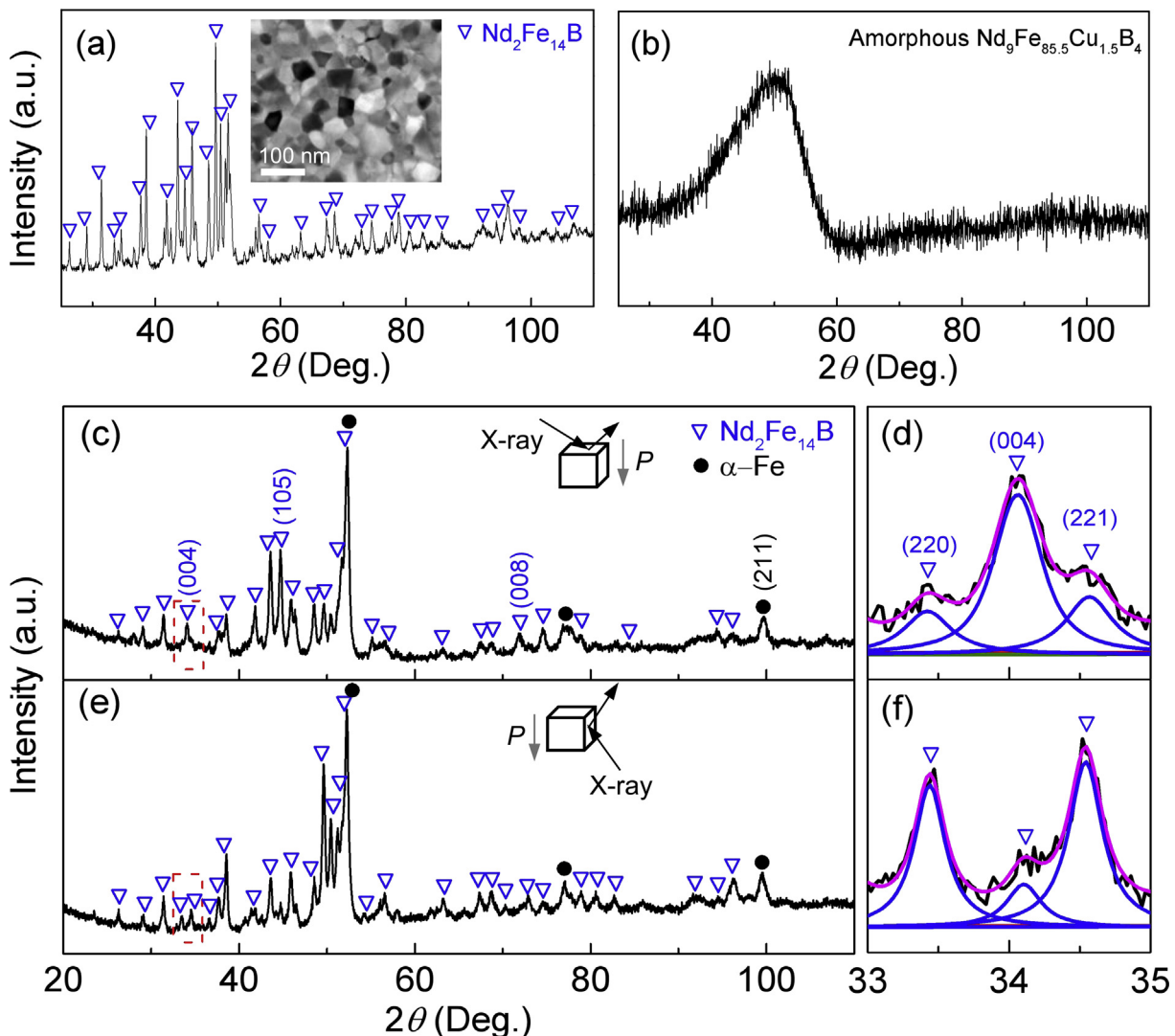


Fig. 1. XRD patterns of the precursors and deformed magnets. (a) Nd-rich powders, inset is the TEM image. (b) Nd-lean  $\text{Nd}_9\text{Fe}_{85.5}\text{Cu}_{1.5}\text{B}_4$  powders. Deformed magnets measured on the face (c) perpendicular and (e) parallel to the pressure direction. (d, f) The zoomed view of the XRD patterns in plane (c) and (e).

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