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Comparison of local and global texture in HDDR processed Nd–Fe–B magnets

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Abstract

The local texture in polycrystalline Nd–Fe–B powder particles processed by hydrogenation disproportionation desorption and recombination (HDDR) has, for the first time, been observed directly by electron backscatter diffraction (EBSD). The local texture quality was found to vary strongly with the hydrogen pressure applied during HDDR processing. At 0.3 bar, a strong biaxial local texture occurs in the individual powder particles. Increasing the hydrogen pressure during HDDR results initially in a lower texture quality and finally leads to near-isotropic particles. Clusters of similarly oriented grains within near-isotropic particles were observed rather than a completely random spatial distribution of orientations. The local texture within the powder particles was shown to be critical in determining the magnetic properties of compacts produced by aligning the powder in an external magnetic field. A compact produced from the powder with the strongest local texture (processed at 0.3 bar) showed highly anisotropic magnetic properties with remanence (related to the density of $Nd_2Fe_{14}B$) parallel and perpendicular to the applied field direction of 1.24 T and 0.55 T, respectively.

Keywords: Hard magnets; Magnetic anisotropy; Electron backscattering diffraction; Texture; Hydrogen absorption

1. Introduction

Recently, research in high-performance permanent magnets based on $Nd_2Fe_{14}B$ has undergone something of a renaissance, owing to the application of such materials in electric motors for hybrid vehicles and in generators for wind turbines [1–3]. Besides the sintering route for high-performance permanent magnets [4], hydrogenation disproportionation desorption and recombination (HDDR) offers a lower-cost processing route based on resin bonded magnets, which additionally offers more complexity in the final shape [5–7].

Dynamic-HDDR (d-HDDR) is a unique method for producing highly anisotropic powder particles with a maximum energy product of ~340 kJ m^{-3} measured in a resin bonded magnet [8]. Here "dynamic" is used to describe the dynamic control of the (low) hydrogen pressure and temperature during the disproportionation and recombination steps, where the reaction is exothermic and endothermic, respectively. The role of hydrogen partial pressure in changing the thermodynamic phase equilibrium of disproportionation and recombination has been described by Sugimoto et al. [9]. The HDDR process transforms the single crystalline initial particles of tetragonal $Nd_2Fe_{14}B$ into polycrystals consisting of submicron grains with an average size between 200 and 400 nm [10–12]. This effect is demonstrated in the SEM images of Fig. 1. Under optimal conditions, the final grains within one particle show a strong preferential orientation which is explained by the texture memory effect (TME) model [13]. In the TME model, it is postulated that the orientation of the parent particle is transferred to the final grains via a textured intermediate phase (Fe_2B) during the d-HDDR process of NdFeBGaNb [14]. It is important to note that Fe_2B is the only tetragonal disproportionation product, in addition to face-centered cubic NdH_x and body-centered cubic Fe. It has been elucidated that the TME works for Nd–Fe–B

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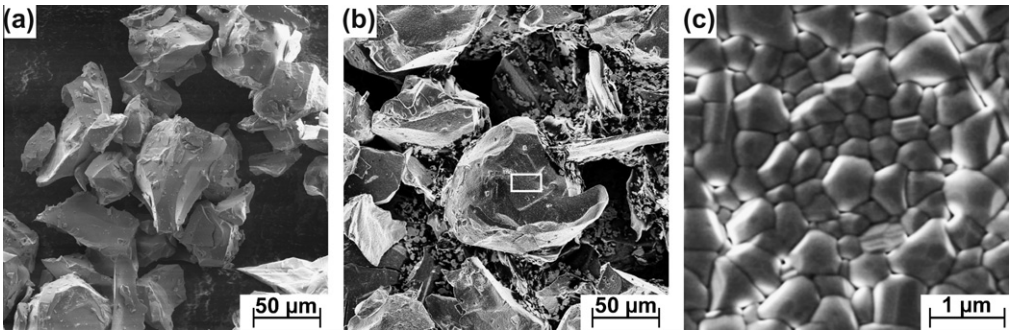

Fig. 1. Secondary electron images showing the morphology of the $Nd_{28.78}Fe_{bal}B_{1.1}Ga_{0.35}Nb_{0.26}$ powder particles in different states: (a) initial state: single crystalline particles; (b) polycrystalline particles following d-HDDR; (c) higher magnification view of the region marked with a white box in (b) showing the submicron grains within one powder particle.

alloys with additives such as Co, Ga and Nb, but also, very importantly, for the ternary compound without additives [13–15]. The TME is also operational for extended dwell times in hydrogen; at this stage the described rod-like structure occurring at the early stages of disproportionation has converted into equiaxed grains. Interestingly, and consistent with the TME model, no texture has been reported for HDDR processed Sm_2Fe_{17}- or $Nd(TM)_{12}$-type alloys [16,17]. For completeness, it should be mentioned that other works dealing with the inducement of texture report: (a) a tetragonal Fe_3B intermediate phase and precipitations of nanocrystalline $Nd_2Fe_{14}B$ which have simple orientation relationships with the original $Nd_2Fe_{14}B$ lattice in the early stage of disproportionation of conventionally HDDR-treated NdFeCoGaZrB [18] and (b) orientation relationships between NdH_2 and α-Fe with respect to the $Nd_2Fe_{14}B$ phase [19].

In resin bonded magnets, anisotropic magnetic properties are produced by aligning the powder in an external magnetic field. It has been shown that the degree of texture (DOT) in the magnetic properties achievable in resin bonded magnets varies strongly with the hydrogen pressure during processing of the powder [14]. It can be implied from these magnetic measurements that the texture quality in the powder particles must also change with hydrogen pressure, but no measurements of texture in the grains within HDDR powder particles on a local scale have been carried out to date. Here, electron backscatter diffraction (EBSD) has been used to achieve this and thus separate the contributions to the overall texture of the magnet from the local texture of the grains and the effectiveness of the magnetic field alignment process.

EBSD is a ubiquitous method for determining the local texture, e.g., in metals and minerals [20,21], but there are few publications demonstrating EBSD on permanent magnet materials, largely because sample surface preparation is non-trivial. A smooth, deformation-free surface is needed, because the information depth in EBSD is ~50 nm [22].

The first attempt at an EBSD investigation on Nd–Fe–B sintered magnets was done by Lillywhite et al. in 2001 [23]. In 2004, Khlopkov et al. [24] showed large ($120 \times 100\ \mu m^2$) crystal orientation maps of the $Nd_2Fe_{14}B$ grains. Woodcock and Gutfleisch extended the technique to include all the phases present in the microstructure of Nd–Fe–B sintered magnets [25]. EBSD has also been successfully applied to Nd–Fe–B thick films with grain size in the range 300–600 nm, intended for micro-electromechanical systems applications [26].

In this work, the powerful EBSD technique was adapted to submicron Nd–Fe–B powders in order to investigate the local orientation over large numbers of grain within one particle. These results were compared with global texture analysis in the context of the HDDR reaction scheme in order to elucidate the TME further.

2. Experimental

An arc melted $Nd_{28.78}Fe_{bal}B_{1.1}Ga_{0.35}Nb_{0.26}$ ingot was decrepitated by applying a hydrogen pressure of 0.5 bar at room temperature. After grinding with a pestle and mortar and sieving, the powder was shown by secondary electron images of random samples, to consist almost entirely of single crystalline particles with a size of ~100 µm (Fig. 1a). Batches of this powder were processed by d-HDDR at hydrogen pressures of 0.3, 0.5 and 1.0 bar at 840 °C for disproportionation.

Then, the powders were embedded in an electrically conductive polymer matrix for grinding and polishing. Standard metallographic preparation was performed using water-free suspensions to avoid oxidation of the Nd–Fe–B alloy.

A high-resolution thermal field emission gun scanning electron microscope (Zeiss Leo 1530 Gemini) coupled with an EBSD system (*H K L*) was used. Electrical charging and beam instabilities have to be minimized for acquisition of large orientation maps of embedded powders with submicron grain size. Sample areas up to $40 \times 30\ \mu m^2$ were scanned using a step size between 50 and 100 nm.

The orientation of the powder particles in the embedding material is random. This led to the angle between the axis of preferred orientation and the sample normal being different for each particle measured. In order to facilitate comparison of data sets from different particles and different samples, the crystal coordinate system was rotated