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Development of anisotropic Nd-Fe-B powder from isotropic gas atomized powder

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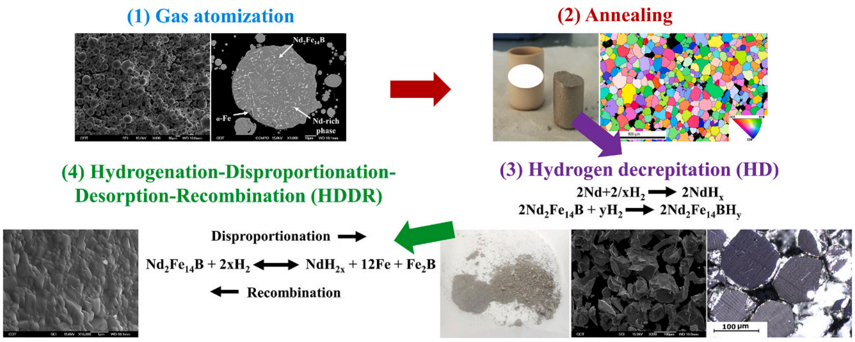
HIGHLIGHTS

- The particle shape has been improved compared to conventional HDDR powders from casting techniques.
- Gas atomization combined with HDDR is a promising method to produce NdFeB powders for anisotropic bonded magnets.
- Good magnetic properties were obtained in ternary alloys without heavy rare earths or other critical raw materials.

GRAPHICAL ABSTRACT



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ABSTRACT

This work presents an innovative approach to obtain anisotropic Nd-Fe-B powder from isotropic gas atomized powder. The new process was developed using a ternary Nd-Fe-B alloy, without the requirement for additional heavy rare earth or other critical raw materials. It comprises the following steps: (a) gas atomization to produce a polycrystalline isotropic powder; (b) annealing at high temperature to induce grain growth; (c) hydrogen decrepitation to obtain a monocrystalline powder; and (d) hydrogenation-disproportionation-desorption-recombination to obtain the final ultrafine anisotropic particles. The final particle shape is polygonal, which should improve the injection molding characteristics of current powder. The final powder exhibits both high remanence (0.97 T) and coercivity (1354 kA/m) for laboratory batch sizes, which is a result of its anisotropic ultrafine microstructure. Thus, gas atomization is considered a feasible alternative to casting methods as a first step to produce powders for anisotropic bonded magnet.

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1. Introduction

Magnets based on neodymium-iron-boron (Nd-Fe-B) alloys have become an integral part of many electrical devices since the discovery of the alloy in 1984 [1,2]. Nowadays, Nd-Fe-B magnets are used in applications such as hard-disk drives, microphones, speakers, sensors, permanent magnet motors or electric generators [3]. For such applications, Nd-Fe-B magnets provide high intrinsic coercivity (H_{cj}) and high maximum energy product (BH_{max}) [4–6]. BH_{max} can be significantly enhanced if the remanence (B_r) is increased by aligning the magnetic particles to have their easy magnetization axis parallel. In practice, the alignment is realized by applying an external magnetic field during shaping, resulting in an anisotropic magnet [6]. Anisotropic Nd-Fe-B magnets can be either sintered or bonded. Sintered magnets offer the highest magnetic properties, with BH_{max} in the range of 200 to 400 kJ/m^3, and hence, they are chosen for the most demanding applications. Bonded magnets are a cheaper option when the required BH_{max} is in the range of 80 to 150 kJ/m^3.

Resin bonded magnets can be manufactured by injection molding using either isotropic Nd-Fe-B powders, which are produced by melt spinning, or anisotropic HDDR powders (HDDR: hydrogenation, disproportionation, desorption, and recombination). The HDDR process, developed by Nakayama & Takeshita [7], utilizes the reversible reaction of materials such as Nd-Fe-B with hydrogen at elevated temperatures. Commercially, the HDDR process is performed on either book mold cast alloys or strip cast alloys after homogenization heat treatment. The absorption of hydrogen at temperatures of 750–950 °C leads to the disproportionation of $Nd_2Fe_{14}B$ matrix phase into an intimate mixture of NdH_2 and Fe_2B in an α-Fe matrix. The subsequent removal of hydrogen forces the extremely fine disproportionated mixture to recombine into $Nd_2Fe_{14}B$ grains approximately 0.3 μm in diameter [8–10]. The powder produced by HDDR processing can exhibit anisotropic behavior as the reformed $Nd_2Fe_{14}B$ grains remember the texture of the original $Nd_2Fe_{14}B$ grains in the cast alloy and grow in the same crystallographic direction. This results in particles of HDDR powder which contain many grains aligned in the same direction. This anisotropic behavior is achieved through careful control of the absorption and desorption of hydrogen during the HDDR process [11–13]. Alloying additions such as niobium (Nb) and gallium (Ga) can also be used to facilitate the inducement of anisotropy [11,14–16]. The anisotropic powder can then be formed into bonded magnets by mixing it with a resin and then either compression or injection molding the mixture in an aligning field.

Nd-Fe-B-Nb alloys are currently used to produce commercial grades of HDDR powder, such as MAGFINE, developed by Aichi Steel. This is currently used to produce the strongest anisotropic bonded magnet available on the market, with a BH_{max} of 175 kJ/m^3 [17]. This magnet was made from HDDR powders which are free of dysprosium (Dy) and Ga. According to the MAGFINE technical datasheet [18], the base alloy has 27–27.5 wt.% of Nd, the average particle size is about 100 μm, and particles have a granular shape. These HDDR powders have a remanence of 1.25 T, but a coercivity equal to or <200 kA/m. After HDDR, the powders are subsequently modified using the grain boundary diffusion process (GBDP). It is only after the GBDP, when the HDDR powder is mixed with 6 wt.% of 80Nd-10Cu-10Al wt.% powder and heat treated at 900 °C for 1 h under vacuum, that a H_{cj} of 1432 kA/m is achieved [19].

Concerning new routes to produce Nd-Fe-B powders, gas atomization is highly attractive due to its high productivity, the spherical particle shape, and the low oxygen content of the powder [20–23]. Another advantage of gas atomization is that solidification rates are high, so the formation of α-Fe is highly reduced. The microstructure of the powder depends on the cooling rate, which in turn depends on the particle size and atomizing gas. In general, particles bigger than about 30 μm are dendritic, particles between 30 and 5 μm contain equiaxial grains, and particles below about 5 μm are ultrafine, nanometric or amorphous. Since gas atomized particles tend to be polycrystalline with randomly oriented grains [24], they can only be used to manufacture isotropic bonded magnets [23], which limits the attainable BH_{max} and, subsequently, the number of practical applications it can be used for.

The flowability of a feedstock (powder mixed with organic binders) for injection molding depends strongly on the particle shape [25]. A parameter often used to evaluate powder morphology is the aspect ratio, which can be defined as the ratio of the minimum to the maximum Feret diameter, so that it is always a number comprised between 0 and 1. It measures the elongation of the particle, i.e. when the aspect ratio increases, the corresponding particle is more equiaxial (or less elongated) and, as a result, less irregular. The more equiaxial is a powder, the lower is the feedstock viscosity and higher volume fractions of magnetic material can be attained in bonded magnets, thus improving its performance. As a result, increasing the aspect ratio of HDDR powders has the potential of improving the performance of the magnets.

The objective of this article is to present a new process to obtain anisotropic Nd-Fe-B powder from isotropic gas atomized powder by annealing the polycrystalline isotropic gas atomized powder and then HDDR treating the powder to produce anisotropic particles with high aspect ratio.

2. Experimental procedure

Powders of several ternary Nd-Fe-B alloys with varying Nd content were produced by gas atomization in a laboratory unit PSI model Hermiga 75/3VI with a capacity of 3 kg of material per batch. Subsequently, annealing trials were performed on the powders to induce grain growth. Previous work by Sarriegui et al. [24] looked at optimizing both the gas atomizing step and the annealing process. The same annealing procedure was used in this work. A small sample of ~10 g was annealed at 1100 °C for 96 h under high purity argon to induce grain growth and obtain single crystal particles. Grain growth annealing resulted in the sintering of the sample, so the outcome was not loose powder, but a semi dense block.

Samples from these trials were exposed to hydrogen with the aim of determining the optimum amount of Nd required to enable the annealed blocks to break up into single crystal particles. Pieces of ~2 g were cut using a diamond wheel with an oil-based lubricant. After cleaning the semi dense sample with acetone, it was decrepitated in a vessel under 0.15 MPa of hydrogen at room temperature for 4 h. The sample with composition 31Nd-bal.Fe-1.1B wt.% demonstrated an optimum decrepitating behavior, so the resulting powder was submitted to stages 2 and 3 of the HDDR cycle illustrated in Fig. 1, which is based on reference [26], using a custom built HDDR furnace at the University of Birmingham.

For microstructural observation, the Nd-Fe-B powders were

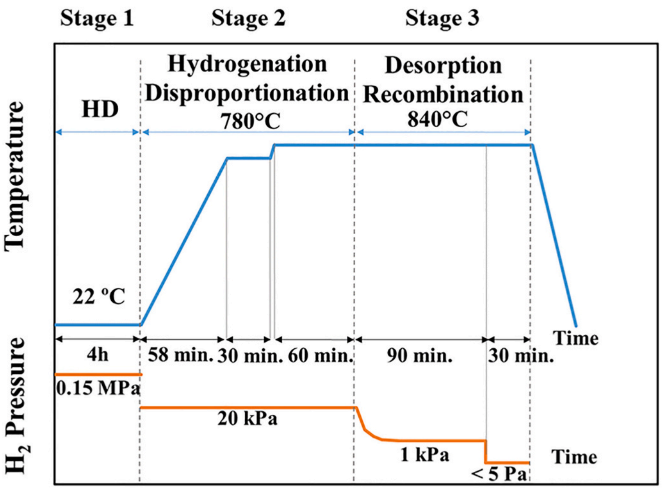

Fig. 1. HDDR cycle used in this work.

mounted, ground with SiC abrasive papers, and polished down to 0.04 μm SiO_2 particle size. The Nd-Fe-B particle shape and microstructure were studied via field emission gun scanning electron microscopy (SEM) using a JEOL JSM-7100F instrument equipped with energy-dispersive X-ray spectroscopy (EDS). Electron backscattered diffraction (EBSD) was performed on the same microscope with an accelerating voltage of 30 kV and a step size of 5 μm using a NORDLYS II camera and the OXFORD HKL CHANNEL 5 software. The microstructure was also examined by Kerr effect microscopy using a Carl Zeiss Jenavert metallographic microscope fitted with the corresponding polarizer and analyzer.

The magnetic properties were measured at room temperature using a vibrating sample magnetometer (VSM) Quantum Design PPMS-9T option Model P525 or LakeShore 7300 VSM. The anisotropic sample was prepared by mixing the powder with molten paraffin wax, aligning the particles under a field of about 1 T, and allowing the paraffin to solidify in order to fix the orientation of the particles. The results have been corrected to remove the effect of the demagnetizing field.

The particle size distribution and aspect ratio of HDDR processed Nd-Fe-B powder were measured by dynamic image analysis in an equipment SYMPATEC QICPIC VIBRI/L. The particle size is reported as the diameter of a circle of equal projection area. As explained above, aspect ratio is defined as the ratio of the minimum to the maximum Feret diameter, so that it is always a number comprised between 0 and 1. The commercial powder MAGFINE MF18P was measured as well for comparison.

3. Results and discussion

Fig. 2(a) shows the particle shape of the gas atomized powder. As is characteristic of this technology, the powder is spherical in shape and the particle size distribution is relatively broad. Fig. 2(b) illustrates the microstructure for a cross sectioned sample. The alloy displays a negligible amount of metastable α-Fe (dark gray phase), since it has a high Nd content (excess of 4.3 wt.% over the stoichiometric concentration) and was solidified under a high cooling rate [27]. The other phases in Fig. 2 (b) are $Nd_2Fe_{14}B$ (light gray contrast) and the Nd-rich phase (in white contrast). The particles are polycrystalline with an average grain size of 3.1 ± 0.1 μm and the grains are randomly oriented [24]. Consequently, applying HDDR on this powder would result in poor anisotropy of the processed material.

To achieve anisotropy in HDDR powder monocrystalline particles are required, as the recombined grains inherit the crystallographic texture of the prior grain [28–30]. This is the reason why the powder was subjected to a grain growth annealing stage. The microstructure of the material after this step is displayed in Fig. 3(a). As for the constituent phases, the main difference with the as-atomized powder is the absence of α-Fe and the formation of $NdFe_4B_4$ (indicated with an arrow in Fig. 3 (a)). The average grain size reached a value of 108 ± 3 μm, as is apparent in Fig. 3(b). In order to induce grain growth, the heat treatment temperature must be high, in the range of liquid phase sintering [6,31], where significant densification takes place over a range of 1000 to 1100 °C [32]. As a result, annealing also produced the formation of necks between the particles. Thus, the loose powder became a sintered block.

Hydrogen decrepitation (HD) of the sintered block was used to return the annealed material back into a powder [33]. During this step, the material reacts with hydrogen resulting in volume expansion of both the Nd-rich phase, at triple junctions and grain boundaries, and the $Nd_2Fe_{14}B$ matrix phase [34]. This expansion leads to cracking along the grain boundaries (i.e. intergranular cracking) and, to some extent, across the grains (i.e. transgranular cracking). As shown in Fig. 3(c), the particles are no longer spherical, but equiaxial and faceted. The annealing and subsequent HD trials performed on gas atomized powder with varying Nd content showed that the composition with at least 31 wt.% Nd was required to enable the block to break up into single crystal powder particles; below this Nd concentration, the block did not fully decrepitate.

The analysis of the microstructure of the 31 wt.% Nd composition shows a single and distinct magnetic domain pattern on the surface of each grain, indicating that the majority of the particles are monocrystalline (Fig. 3(d)). In addition, Fig. 4(a) demonstrates that the median particle size (D_{50}) of Nd-Fe-B powder after HDDR processing is in fact close to ~100 μm, i.e. similar to the size of the grains in the sintered block. This confirms that the dominant fracture mechanism during HD is intergranular cracking and that the majority of the particles observed in Fig. 3. (c) and (d) are monocrystalline. Fig. 4(b) compares the aspect ratio distribution of this powder with the commercial powder MF18P. The curve of the gas atomized powder is always on the right side, i.e. the aspect ratio of the gas atomized HDDR powder is higher than that of the commercial MF18P powder. MAGFINE powders use a base alloy with a Nd content close to the stoichiometric value in the $Nd_2Fe_{14}B$ phase (26.7 wt.%). Since high cooling rates are required to minimize the formation of α-Fe, strip casting is the preferred method to produce raw Nd-Fe-B-Nb material [35,36]. Strip casting is known for producing a columnar structure of hard magnetic grains due to a directional solidification [37]. Therefore, it is reasonable to expect that MF18P powder has a more elongated shape than the powder obtained by the gas atomization route.

The monocrystalline decrepitated powder was finally subjected to stages 2 and 3 of the HDDR process shown in Fig. 1. As expected, the particles exhibit a final ultrafine microstructure with a crystallite size <1 μm (Fig. 5(a)). The demagnetization curves of the initial gas atomized powder and the final HDDR powder are compared in Fig. 5(b). The gas atomized powder exhibits a remanence of 0.47 T and an intrinsic coercivity of only 144 kA/m. These low properties are a consequence of

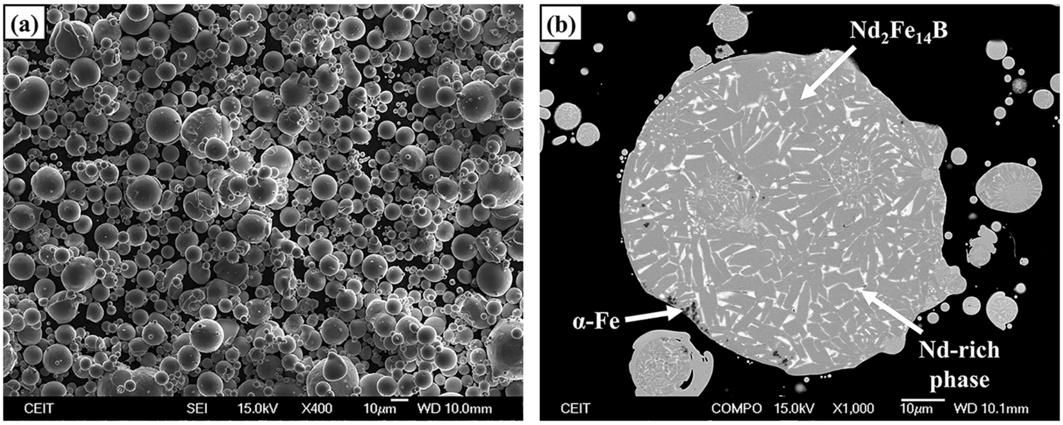

Fig. 2. (a) Secondary electron image showing the particle shape of the gas atomized powder and (b) Backscattered electron image showing the microstructure of the gas atomized powder.

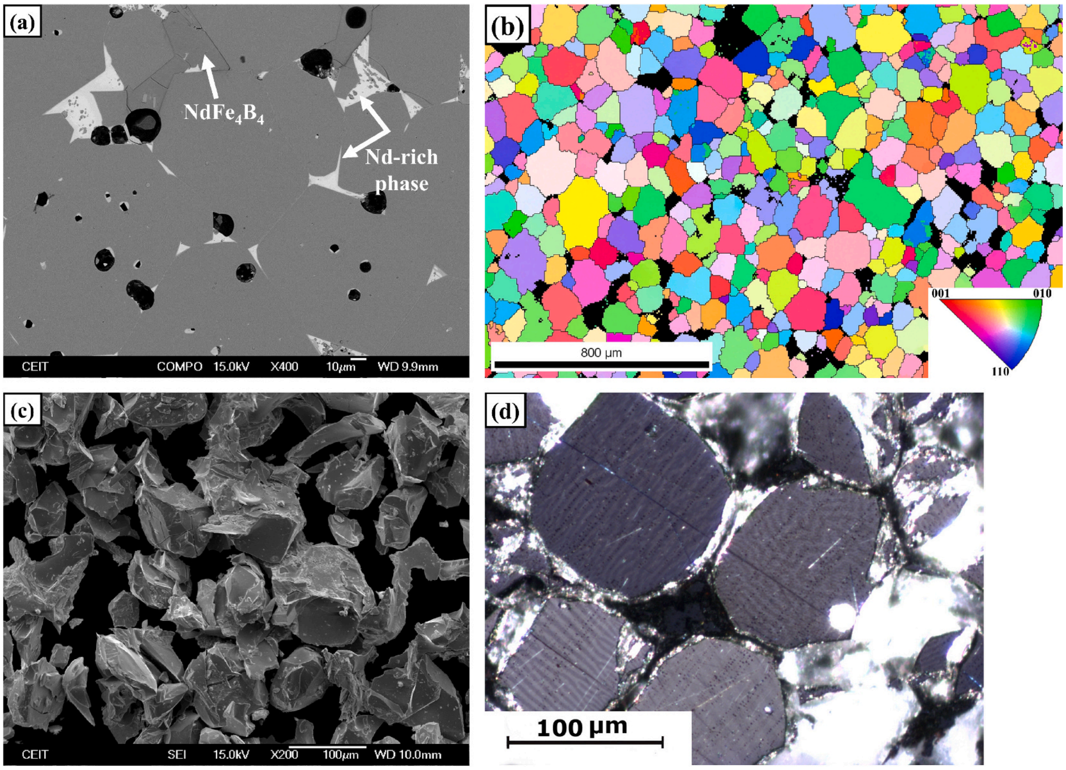

Fig. 3. (a) Backscattered electrons image and (b) Inverse pole figure (IPF) of the block after grain growth annealing; (c) Powder shape and (d) Kerr effect image showing that particles are monocrystalline after HD (note the single pattern of magnetic domains inside each particle).

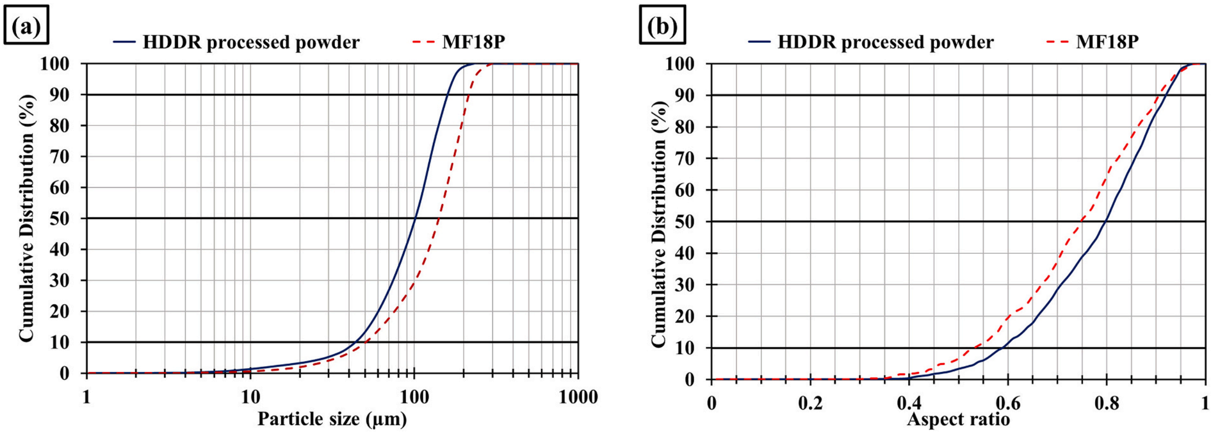

Fig. 4. (a) Particle size distribution and (b) Cumulative distribution of aspect ratio of Nd-Fe-B powder compared with commercial MF18P powder.

the microstructure of the powder, which is isotropic and has a grain size in the range of microns. In contrast, the HDDR powder has a remanence value of 0.97 T and an intrinsic coercivity of 1354 kA/m. This high remanence indicates that the powder is anisotropic (i.e. the ultrafine grains are oriented in a similar direction inside each particle). The degree of alignment has been estimated from magnetic measurements as $M_r/M_s = 0.97/1.32\ T = 0.7$, where M_r is the remanence and M_s the saturation magnetization. The good alignment, ultrafine grain size, and presence of the Nd-rich intergranular phase explain the high coercivity of the anisotropic powder.

Compared with other HDDR ternary alloys from the literature, the HDDR gas atomized powder exhibits a lower remanence, but higher coercivity. The literature indicates that ternary Nd-Fe-B powders processed through HDDR typically consist of alloys with Nd contents below 29 wt.%, exhibiting high remanence (>1 T), but low coercivity (<700 kA/m) [8,11,12]. In comparison, the ternary HDDR gas atomized powder introduced in this work has 31 wt.% of Nd. The volume fraction of the Nd-rich phase rises with higher Nd contents, reducing the magnetic coupling effect between grains and increasing the intrinsic coercivity as in this study. However, the main phase volume fraction of $Nd_2Fe_{14}B$ phase decreases, leading to a reduction in remanence.

4. Conclusions

The transformation of an isotropic polycrystalline powder produced by gas atomization into an anisotropic ultrafine powder has not been reported before. It resulted in a significant improvement of the magnetic properties. The remanence was increased from 0.47 to 0.97 T and coercivity rose from 144 to 1354 kA/m. It is worthy to point out that these results were obtained with a Nd-Fe-B ternary alloy, i.e. without

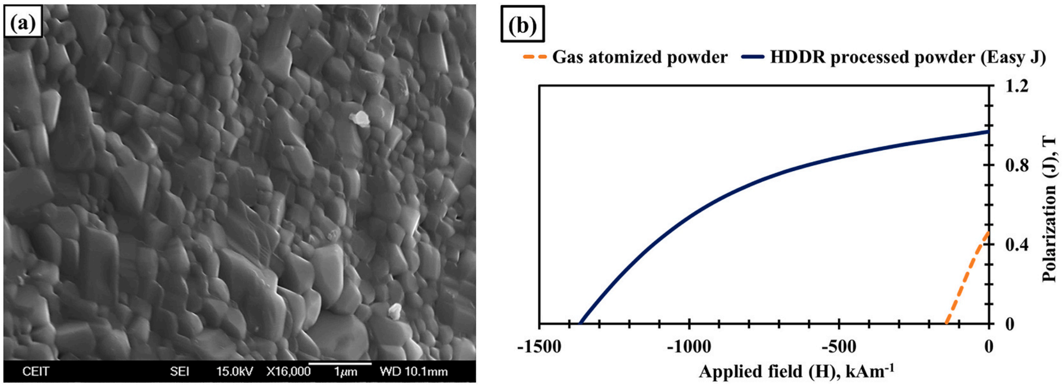

Fig. 5. Microstructure and magnetic properties of the HDDR processed powder: (a) Secondary electron image of the surface of a particle after HDDR process showing the final ultrafine grains and (b) Demagnetization traces of gas atomized and HDDR processed powders.

expensive heavy rare earths, cobalt, niobium or gallium (all of them are considered critical raw materials nowadays). The HDDR gas atomized powder is more equiaxial than conventional HDDR powders from casting methods, which would facilitate particle packing and reduce the viscosity of the feedstock during injection molding. The gas atomization is presented as a viable alternative to conventional strip casting as the initial step in the production of powders for anisotropic bonded magnets. This work demonstrates that anisotropic ternary alloys with comparable properties and a better particle shape can be produced.

CRediT authorship contribution statement

Gabriela Sarriegui: Methodology, Formal analysis, Data curation, Investigation, Writing – original draft, Writing – review & editing, Visualization. **Malik Degri:** Methodology, Formal analysis, Data curation, Investigation, Writing – review & editing. **Mihail Ipatov:** Methodology, Data curation, Investigation, Writing – review & editing. **Lydia Pickering:** Methodology, Formal analysis, Data curation, Investigation, Writing – review & editing. **Blanca Luna Checa:** Data curation, Investigation, Visualization. **Nerea Burgos:** Data curation, Investigation, Visualization. **Muhammad Awais:** Methodology, Formal analysis, Investigation. **Richard Sheridan:** Methodology, Formal analysis, Investigation. **Allan Walton:** Methodology, Formal analysis, Supervision, Funding acquisition, Writing – review & editing. **José Manuel Martín:** Conceptualization, Methodology, Formal analysis, Writing – original draft, Writing – review & editing, Supervision, Project administration, Funding acquisition. **Julián González:** Methodology, Formal analysis, Writing – review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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