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Journal of Alloys and Compounds 287 (1999) L10–L12

Journal of
ALLOYS
AND COMPOUNDS

Letter

High anisotropy in Pr–Fe–Co–B–Zr HDDR powders

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Received 21 January 1999

Abstract

The magnetic behaviour of HDDR powders prepared from a $\text{Pr}_{13.7}\text{Fe}_{63.5}\text{Co}_{16.7}\text{B}_6\text{Zr}_{0.1}$ alloy has been investigated. It has been shown that it is possible to produce highly anisotropic HDDR material from this alloy in the homogenised state (1373 K for 20 h). Remanent magnetisation in the easy direction of the aligned powder was 126 ± 2 J/Tkg whereas, in the hard direction, it was 30 ± 0.6 J/Tkg. HDDR powder based on the corresponding Nd-based alloy (subjected to the same heat treatments) and commercially available, HDDR powder, have both been used as a comparison. The degree of alignment for the HDDR powder based on the Pr alloy was higher (76%) than that of the commercial HDDR powder (69%). © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Permanent magnets; Coercivity; HDDR treatment; Anisotropic powder

1. Introduction

Over the past 10 years, extensive research has concentrated on the study of the hydrogenation disproportionation desorption and recombination (HDDR) process in Nd–Fe–B-type alloys (see, for example [1,2]). This has been described in detail in a recent review [3]. However, only recently, have any studies been reported on the HDDR process in praseodymium-based compounds [4]. In this work, HDDR powder based on the alloy $\text{Pr}_{13}\text{Fe}_{\text{bal}}\text{Co}_{24}\text{B}_6\text{Ga}_1\text{Zr}_{0.1}$, was reported to have a remanence of about 104 J/Tkg (9.8 kG; ~1T) and a coercivity of 382 kA/m (4.8 kOe). Although a number of advantages have been attributed to Pr–Fe–B-type alloys [5,6], the most important with respect to the HDDR process would be the possibility of a different grain growth mechanism compared to that of Nd-based systems [6], leading to greater control of the grain growth after the recombination stage.

In the present work, an investigation of HDDR powders based on the alloy $\text{Pr}_{13.7}\text{Fe}_{63.5}\text{Co}_{16.7}\text{B}_6\text{Zr}_{0.1}$ has been carried out using a vibrating sample magnetometer (VSM). Magnetic characterisation of the corresponding Nd-based

alloy ($\text{Nd}_{13.7}\text{Fe}_{63.5}\text{Co}_{16.7}\text{B}_6\text{Zr}_{0.1}$) and a batch of commercial HDDR powder (MQA-T) have also been included in the present work for comparison.

2. Experimental

The alloys investigated in this work have been provided by Rare Earth Products. Ingots were prepared in a rectangular water-cooled copper mould and the chemical analyses of the as-cast alloys are given in Table 1. The homogenisation heat treatment of the ingot was carried out under vacuum at 1373 K for 20 h.

For the HDDR treatment (as shown in Fig. 1), the annealed material was crushed into coarse lumps and 15 g batches placed into a HDDR reactor, which was evacuated to backing-pump pressure ($\sim 10^{-1}$ mbar) and hydrogen introduced to a pressure of 1 bar. The reactor was then heated to a temperature of 1133 K with a dwell time of 15 min. Subsequent desorption and recombination was carried out under vacuum at the same temperature until a pressure of 10^{-1} mbar was achieved (<10 min). Subsequent rapid cooling of the material was carried out by removing the furnace and fitting a water-cooled copper coil onto the Inconel reactor tube. The resultant HDDR powder was crushed readily with a mortar and pestle in air, such that all the material passed through a <106 μm sieve.

Magnetic characterisation of the HDDR powders was

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Table 1
Chemical analyses of the as-cast alloys

Alloy (at%)	Pr/Nd	Fe	Co	B	Zr (wt%)
Pr _{13.5} Fe _{63.4} Co _{16.5} B _{6.1} Zr _{0.1}	29.33	Bal.	15.08	1.03	0.15
Nd _{13.7} Fe _{63.0} Co _{17.0} B _{6.2} Zr _{0.1}	30.03	Bal.	15.26	1.02	0.14

carried out by mixing the powders with wax and aligning by melting the wax, applying a magnetic field (2 T) and allowing the sample to set in the field. VSM measurements were performed both parallel and perpendicular to the alignment directions after magnetising in a pulsed field of 4.5 T. No corrections were made for self demagnetising fields. The level of anisotropy in the powder has been estimated from the degree of alignment (α) which was calculated from the expression:

$$\alpha = [1 - (m_r/M_r)]100 (\%)$$

where M_r and m_r are the remanent magnetisations along the easy (parallel to alignment) and the hard (perpendicular to alignment) directions, respectively.

3. Results and discussion

In Fig. 2, the magnetic properties of the Pr_{13.7}Fe_{63.5}Co_{16.7}B₆Zr_{0.1} HDDR powder are shown along the easy and hard directions of magnetisation. A high degree of alignment (76%) has been achieved with this powder. Fig. 2 shows a good loop shape in the easy direction for this Pr-based HDDR material, along with a very good remanent magnetisation and a reasonable intrinsic coercivity. The present coercivity value is twice that reported for a Pr₁₃Fe_{bal}Co₂₄B₆Ga₁Zr_{0.1} HDDR magnet [4] and comparable with that reported for anisotropic bonded magnets of Nd_{12.6}Fe_{bal}Co_{11.5}B₆Zr_{0.1} HDDR powders [7].

Table 2 shows a comparison between the magnetic properties of the Pr_{13.7}Fe_{63.5}Co_{16.7}B₆Zr_{0.1} HDDR powder and those of a standard commercial HDDR powder

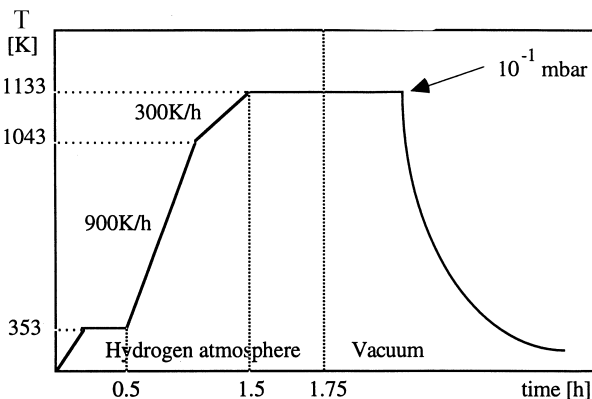


Fig. 1. Schematic diagram of the HDDR heat treatment.

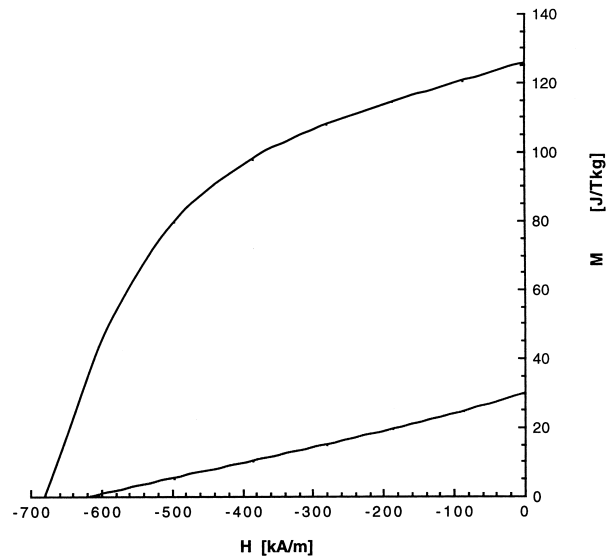


Fig. 2. VSM measurements of Pr_{13.7}Fe_{63.5}Co_{16.7}B₆Zr_{0.1} HDDR powder produced from a homogenised cast ingot.

measured using the same technique. The former shows better remanent magnetisation and degree of anisotropy whereas the latter has a higher intrinsic coercivity. Nd_{13.7}Fe_{63.5}Co_{16.7}B₆Zr_{0.1} HDDR powder processed using the present HDDR treatment has not been included in Table 2, because it showed no significant permanent magnetic properties. This could be due to the requirement for a higher quenching rate (Ar gas), after recombination, for these materials [7]. It appears therefore, that Pr-HDDR powders based on the present alloy are easier to produce with a relatively straight forward HDDR treatment.

Further investigations are underway in an attempt to explain the different magnetic behaviours of these two materials.

4. Conclusions

Magnetically anisotropic HDDR powder has been produced successfully from a heat treated alloy of composition Pr_{13.7}Fe_{63.5}Co_{16.7}B₆Zr_{0.1}. When the powder was aligned in molten wax in a field of 2 T, then a remanent magnetisation in the easy direction of 126 J/Tkg was achieved with a degree of alignment (α) of 76%. An equivalent Nd-based alloy processed under the same conditions gave no significant permanent magnetic properties. A Nd-based, commercial anisotropic HDDR powder (MQA-T) measured using the same technique yielded a lower remanent magnetisation of 119 J/Tkg in the easy direction and a lower degree of alignment (69%). The intrinsic coercivity of the Pr-based HDDR powder (680 kA/m) was lower than that of the MQA-T powder (916 kA/m), but the precise HDDR conditions have not yet been optimised.

Table 2
Magnetic properties of the Pr-based HDDR and MQA-T powders (error: $\pm 2\%$)

HDDR material	M_r J/Tkg	m_r J/Tkg	α %	B_r^a T	iH_c kA/m	$(B_r^2/4\mu_0)$ kJ/m ³
Pr _{13.7} Fe _{63.5} Co _{16.7} B ₆ Zr _{0.1}	126	30	76	1.2	680	286
MQA-T	119	37	69	1.1	916	241

^a Remanence: $B_r = 4\pi\rho_r M_r \cdot 10^{-4}$; theoretical density: $\rho_r = 7.5$ g/cc.

Acknowledgements

Many thanks are due to the FAPESP and IPEN-CNEN/SP for the provision of a research grant (R.N. Faria). Thanks are due to REP for the provision of the alloys. Thanks are also due to the EPSRC for the support of the general research programme of which this work forms a part.

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