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Effect of variables the HDDR processing on magnetic properties and microstructure in permanent magnets based on Pr-Fe-B

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Abstract. The first goal of this work involved the study of the effect of variables the HDDR processing, such as: the added pressure of H_2 in the system, the time of heat treatment and recombination of $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ alloy with the aim of improving the magnetic properties like the magnetic properties of the $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ alloy (Br= 865mT and iHc= 790mT). The second aim of the work involved the characterization of HDDR powders that were analyzed by X-ray diffraction for identification and quantification of crystalline phases. These materials were analyzed by scanning electron microscopy (SEM).

Introduction

Permanent bonded magnets can be manufactured by the powder metallurgy using a resin or polymer material to the consolidation of this in a rigid sample [1] which aims to reduce production costs without reducing its performance considerably since there is no need of sintering. The HDDR process is influenced by a large number of variables that are interrelated and major efforts are being devoted to the study the complex processing of these materials [2,3,4]. The HDDR process allows the refinement of the matrix phase's grain providing a material for the preparation of permanent magnets with magnetic properties [1]. This process consists of four stages: hydrogenation, disproportionation, desorption and recombination [5-10]. In this work, Pr-Fe-B based bonded magnets were produced using this process. The work involved the study of the effect of variables the HDDR processing of a $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ alloy to improve the magnetic properties. A standard $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ magnet with good magnetic properties (Br= 865mT and iHc= 790mT) was used as a comparison.

Experimental

The cast alloys were annealed at 1100 ºC, with different times (20, 48 and 72 hours). The HDDR system [11] was subjected to vacuum to approximately 10^{-1} mbar, followed by the addition of hydrogen (H_2) with different values for each process. Various hydrogen pressures were used (400, 700, 930; 1400; 1900 or 8000 mbar). The temperature of the desorption and recombination stages was fixed at 840ºC (because this temperature showed good results previously [11]) and holding up time at these stages of 330, 900 and 1800 sec.. The magnetized HDDR magnets were magnetically measured and then characterized using SEM. The phases were determined by energy dispersive X-ray (EDX) and X-ray diffraction (XRD).

Results and discussions

The magnetic properties and processing conditions of the HDDR magnets are presented in Table 1. The second quadrant demagnetization curves are shown in Figure 1. It can be observed that the highest remanence values are for the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ magnet processed using a heat treatment of 20h, hydrogen pressure of 1900mbar and of 1800 sec. of desorption and recombination time (B_r=1.6 kG, $_iH_C$=0.21 kOe) and also using a heat treatment of 48h, hydrogen pressure of 930mbar and of 330 sec. of desorption and recombination time (B_r=1.6 kG, $_iH_c$= 0.25 kOe). The magnetic curves are shown in Figure 1a and b. The best intrinsic coercivity for the magnet prepared with the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ alloy was obtained with the heat treatment of 20h, hydrogen pressure of 1900mbar and of 330 sec. of desorption and recombination time (B_r=1.4 kG, $_iH_c$=0.6 kOe).The magnetic curve is shown in Figure 1c.

Table 1- Variations of each process in the permanent bonded magnets manufacture with their respective remanence and intrinsic coercive values.

Composition (at%)	Heat treatment time	H_2 pressure	Desorption and Recombination time (sec.)	B_r (kG)	$_iH_c$ (kOe)
$Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$	20h	700 mbar	330	0.8	0.2
	20h	1400 mbar	330	1.0	0.4
	20h	1900 mbar	330	1.4	0.6
	20h	8000 mbar	330	-	-
	20h	1900 mbar	900	1.5	0.3
	20h	1900 mbar	1800	1.6	0.21
	48h	930 mbar	330	1.6	0.25
	72h	400 mbar	330	1.1	0.2
	72h	930 mbar	330	0.8	0.09
$Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$	20h	930 mbar	330	8.65	7.9

Figure 2 presents X-ray diffraction patterns of the HDDR powder prepared using the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ alloy. A great similarity can be observed on the diffraction patterns which the same phases for all processing conditions: Fe (phase with cubic symmetry); $Pr_2Fe_{14}B$ (phase with tetragonal symmetry) and Pr_2O_3 (phase with hexagonal symmetry). The oxide of Pr_2O_3 phase was probably formed by alloy's rich phase reaction with oxygen in the atmosphere after the HDDR process.

Figure 3 presents X-ray diffraction patterns of $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ powder alloy processed using a heat treatment of 20h, hydrogen pressure of 930mbar and of 330 sec. of desorption and recombination time. In this pattern only the $Pr_2Fe_{14}B$ phase with tetragonal symmetry was observed.

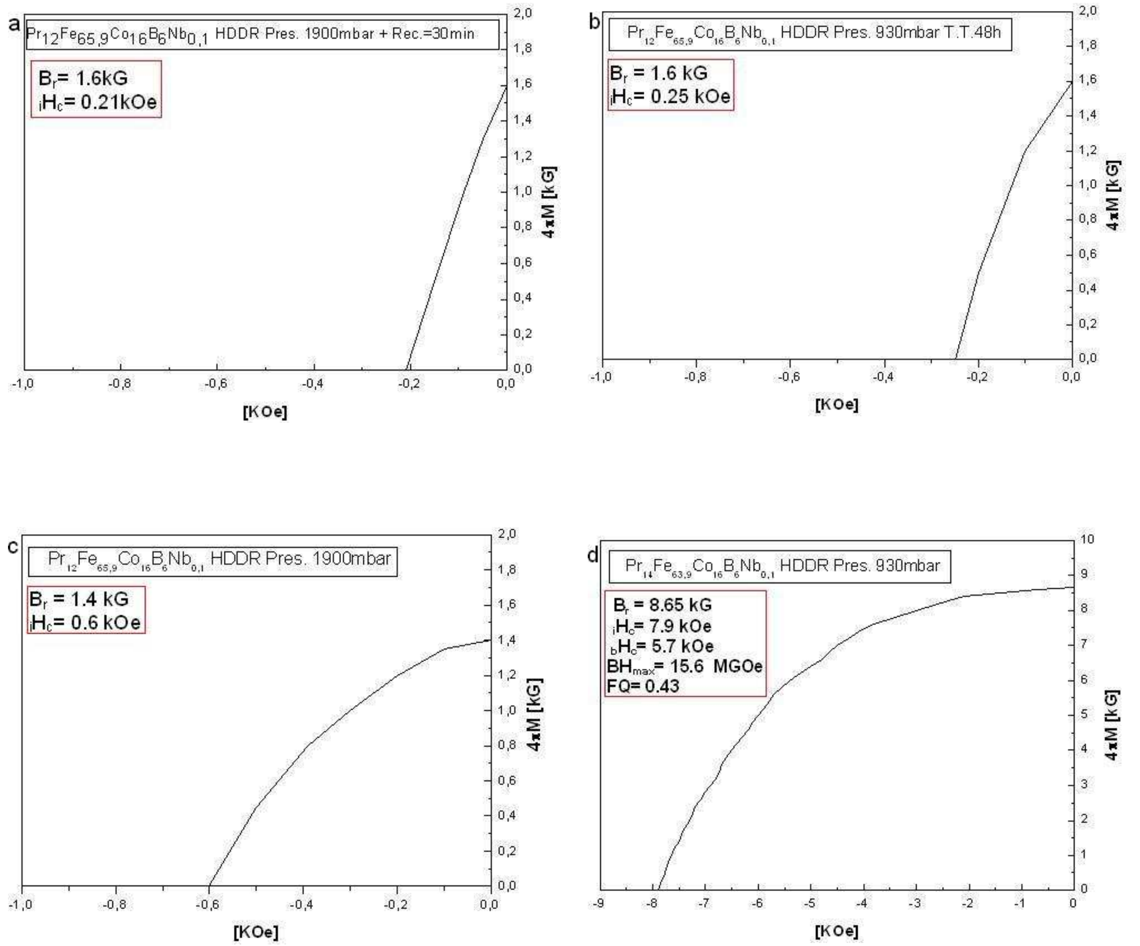

Fig. 1 - (a, b and c) Demagnetization curves of the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ bonded magnets with best values of B_r and ${}_iH_c$ values, (d) demagnetization curve of the standard $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ magnet.

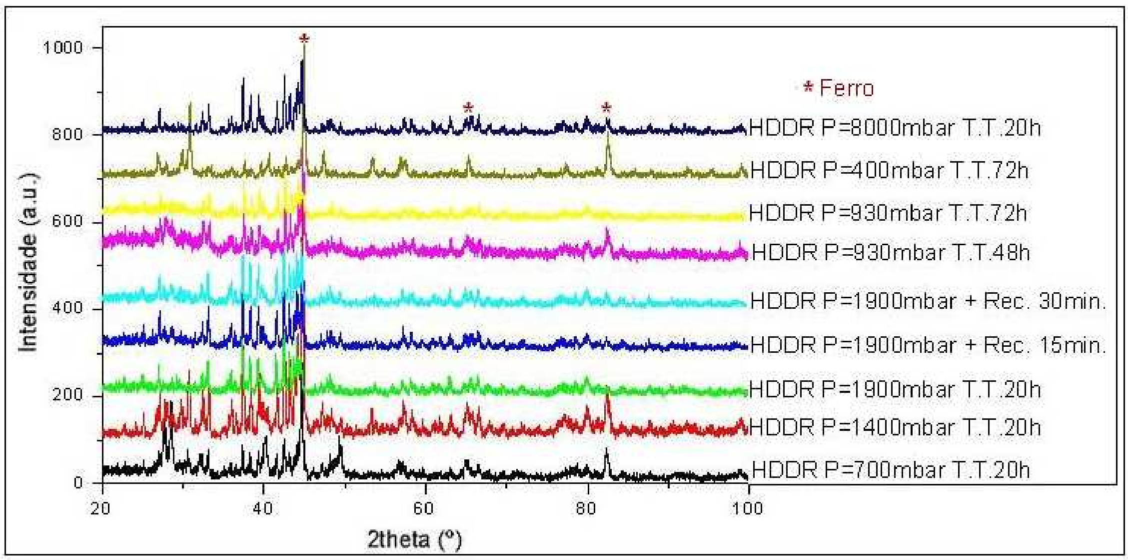

Fig. 2- X-ray diffraction patterns of the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ HDDR powders.

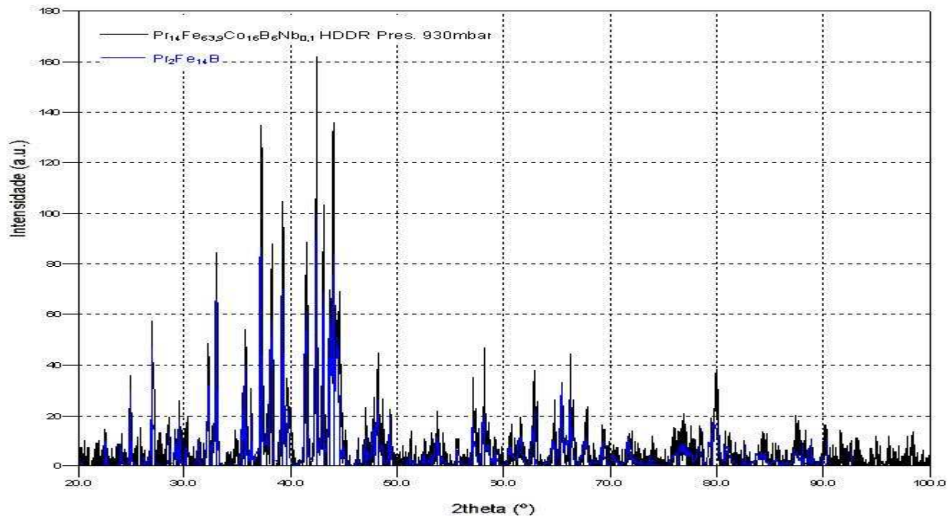

Fig. 3- X-ray diffraction patterns of $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ powders.

Figure 4 (a-d) shows the microstructures obtained by scanning electron microscopy of the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ alloy in the cast condition and after the heat treatments of 20, 48 and 72 hours at 1100 ºC. It can be observed that the as cast alloy showed large amounts of FeCo phase (black region). The alloys with heat treatment of 20, 48 and 72 hours also showed the FeCo phase, but in lesser amounts. Figure 5a and b show the SEM microstructures of the $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ alloy in the cast condition and after heat treatment of 20h at 1100 ºC. The as-cast $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ alloy showed large amounts of FeCo phase (black regions) whereas the treated alloy exhibited no presence of this phase. Thus, the homogenization treatment of 20 h was efficient to remove this deleterious phase.

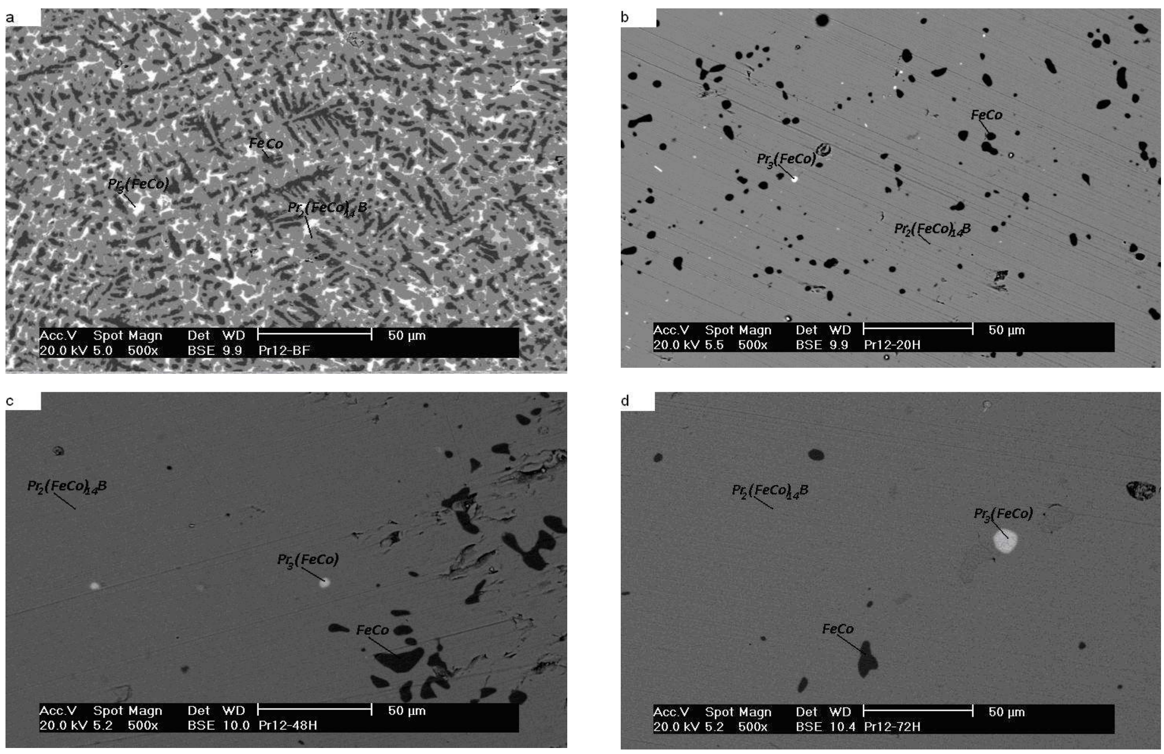

Fig. 4- SEM microstructures of the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ alloy; (a) cast alloy, treated for (b) 20 h, (c) 48 h and (d) 72 h.

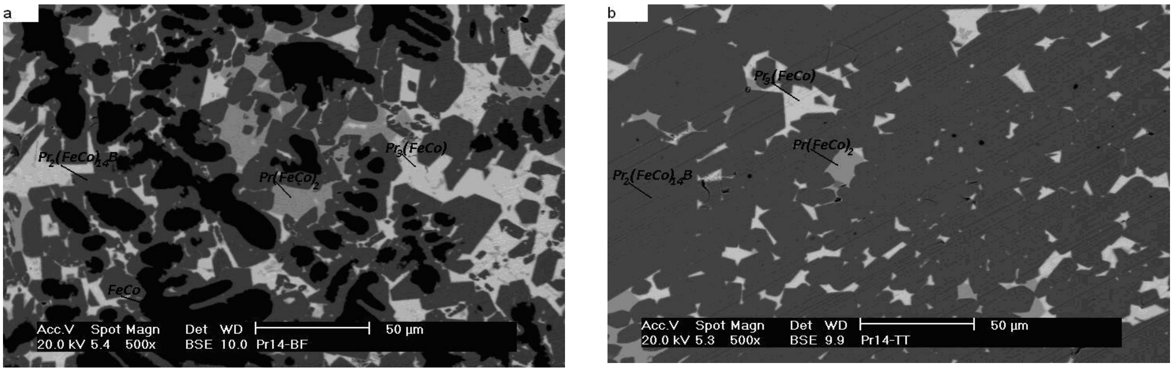

Fig. 5- SEM microstructures of the $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ alloy; (a) cast alloy and (b) with a heat treatment of 20 h.

Conclusions

- The best remanence values (B_r=1.6 kG) were obtained with $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ HDDR magnets with the alloy heat treated for 20h using a hydrogen pressure of 1900mbar and of 1800 sec. of desorption and recombination time. Similar results were obtained with a heat treatment of 48h using a hydrogen pressure of 930mbar and of 330 sec. of desorption and recombination time.
- The best value of intrinsic coercivity was obtained with the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ HDDR magnet using an alloy treatment of 20h, HDDR hydrogen pressure of 1900mbar and of 330 sec. of desorption and recombination time (B_r=1.4 kG, $_iH_c$=0.6 kOe).
- The lower magnetic properties of the $Pr_{12}Fe_{65.9}Co_{16}B_6Nb_{0.1}$ magnets compared with the standard $Pr_{14}Fe_{63.9}Co_{16}B_6Nb_{0.1}$ magnet has been attributed to the presence of the FeCo phase in the former even after the heat treatment.

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