Materials Science Forum Vols. 660-661 (2010) pp 308-313
Online available since 2010/Oct/25 at www.scientific.net

doi:10.4028/www.scientific.net/MSF.660-661.308

CRYSTALLITE SIZE AND MAGNETIC PROPERTIES OF HDDR POWDERS OBTAINED FROM PrFeCoBNb ALLOYS

S. C. Silva[1, a], J.H. Duvaizem[1,b], R. N. Faria[1,c], H. Takiishi[1,d]
[1]Department of Metallurgy, Instituto de Pesquisas Energéticas e Nucleares, IPEN-CNEN, 05508900, São Paulo, Brazil
[a]scsilva@ipen.br, [b]jduvaizem@ipen.br, [c]rfaria@ipen.br, [d]takiishi@ipen.br

Keywords: HDDR magnets, crystallite sizes, Pr-Fe-B magnets

Abstract. The first goal of this work involved the study of HDDR powders obtained from annealed alloys with the general formula: $Pr_xFe_{77.9-x}Co_{16}B_6Nb_{0.1}$ (x = 12; 12.5; 13; 13.5 and 14). The alloys were processed at desorption / recombination temperature of 840ºC. The highest magnetic properties were obtained with 13.5 at. % Pr (Br= 1000mT and $\mu_{0i}H_c$= 890mT). The alloy with a minimum praseodymium content (12 at. %) exhibited the lowest magnetic properties (Br= 350mT e $_iH_c$= 120mT). The second aim of the work involved the characterization of HDDR powders using X-ray diffraction for phase quantification and mean crystallite sizes determination of the hard magnetic phase. The processed powders were characterized by scanning electron microscopy (SEM).

Introduction

The hydrogenation disproportionation desorption recombination (HDDR) process consists in a heat treatment of magnetic alloys under determined conditions in the presence of gas hydrogen. This process was reported in 1990 [1-4].

Desorption/recombination temperature affects the microstructure and magnetic properties of the bonded HDDR magnets. Each alloy requires an optimum reaction temperature and exhibits a particular microstructure according to the composition [5]. Studies presented that each alloy has an ideal crystallite size which is related with desorption/ recombination temperature [6]. This paper reports the effect of the Pr content (x = 12; 12.5; 13; 13.5 and 14) on the microstructure (crystallite mean size) and magnetic properties of the HDDR material.

Experimental Procedure

Commercial alloys were annealed in vacuum at 1100ºC during 20 h for iron elimination. Details of the HDDR magnets preparation and magnetic measurements have been described in previous papers [7-10]. Figure 1 shows a flowchart of the HDDR magnets preparation. In this case, the desorption/recombination temperature utilized was 840°C because in magnets containing 16 at.% Co the best magnetic properties were obtained with this reaction temperature [5]. Part of the HDDR powder was used to microstructural examination in a SEM and in X ray analysis. The phases identification was carried out by means of the ICDD-PDF2 database and mean crystallite sizes by using Scherrer method

$$D_p = \frac{0.94 \quad \lambda}{\beta \quad \frac{1}{2} \cos\theta} \tag{1}$$

Where D_p is the mean crystallite size, λ is the wavelength and β is the FWHM of the XRD profile.

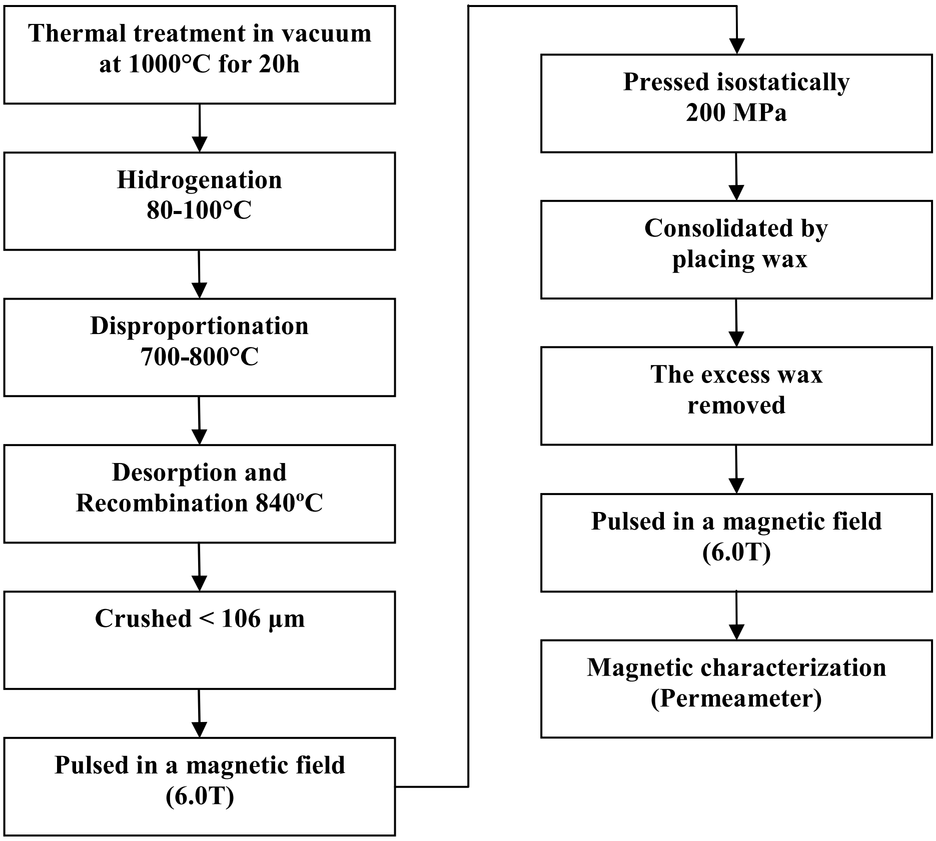

Figure 1 – HDDR process flowchart.

Results and Discussion

Table 1 lists a summary of magnetic properties of the HDDR magnets. The content of praseodymium varied considerably the magnetic properties. The HDDR magnet with 12% Pr had lowest magnetic properties.

Increasing the praseodymium content from 12 at.% to 13,5 at.% it has been verified an improvement in remanence (from 350 mT to 1000 mT). Magnets processed with 14% Pr exhibited a decrease in remanence, and increased the intrinsic coercivity value [1040 mT]. This discussion can be observed in Figure 2.

As well as the remanence, the maximum product of energy (BH_{max}) increased with the praseodymium percentage up to 13,5%; for 14% at. Pr there was a decrease in this property.

The Square Factor presented good result for HDDR produced by $Pr_{13,5}Fe_{bal}B_6Co_{16}Nb_{0.1}$ alloy (SF = 0.48). This is a reasonable result for HDDR magnets, and presented proximity to the values of SF mentioned in others papers [4, 10].

Table 1 - Magnetic properties of $Pr_xFe_{77.9-x}Co_{16}B_6Nb_{0.1}$ (x = 12; 12.5; 13; 13.5 and 14) HDDR magnets processed at 840°C (error ± 2%).

Composition	Br [mT]	$\mu_{0i}H_c$ [mT]	$\mu_{0b}H_c$ [mT]	BH_{Max} [kA/m]	SF [ratio]
$Pr_{12}Fe_{bal}B_6Co_{16}Nb_{0.1}$	350	120	100	8	0.33
$Pr_{12,5}Fe_{bal}B_6Co_{16}Nb_{0.1}$	780	900	520	96	0.33
$Pr_{13}Fe_{bal}B_6Co_{16}Nb_{0.1}$	960	890	640	160	0.42
$Pr_{13,5}Fe_{bal}B_6Co_{16}Nb_{0.1}$	1000	890	680	168	0.48
$Pr_{14}Fe_{bal}B_6Co_{16}Nb_{0.1}$	810	1040	630	120	0.43

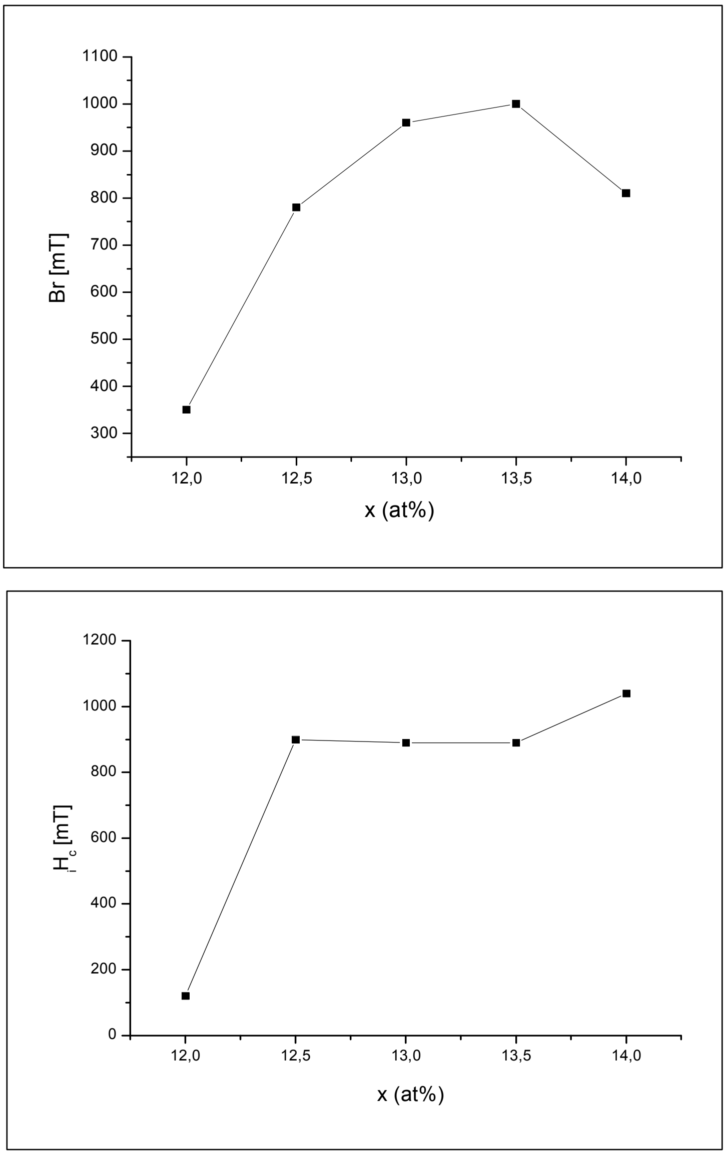

Figure 2 – Remanence and intrinsic coercivity versus Pr content for the $Pr_xFe_{77.9-x}Co_{16}B_6Nb_{0.1}$ (x = 12; 12.5; 13; 13.5 and 14) HDDR magnets.

The HDDR powders results were analyzed by X-ray diffraction and XRD spectra, shown in Figure 3, are very similar for all samples.

Table 2 presents the identified phases for the HDDR powders. All the samples presented the $Pr_2Fe_{14}B$ phase, but $Pr_{12}Fe_{bal}B_6Co_{16}Nb_{0.1}$ presented besides $Pr_2Fe_{14}B$ phase, one peak (2θ = 44.66) of Fe and few peaks (2θ = 28.02; 41.50; 53.17 e 58.16) of Pr_7O_{12}. The HDDR powders prepared by $Pr_{14}Fe_{bal}B_6Co_{16}Nb_{0.1}$ alloy shows one peak (2θ = 37.33) of Fe_2O_3 phase.

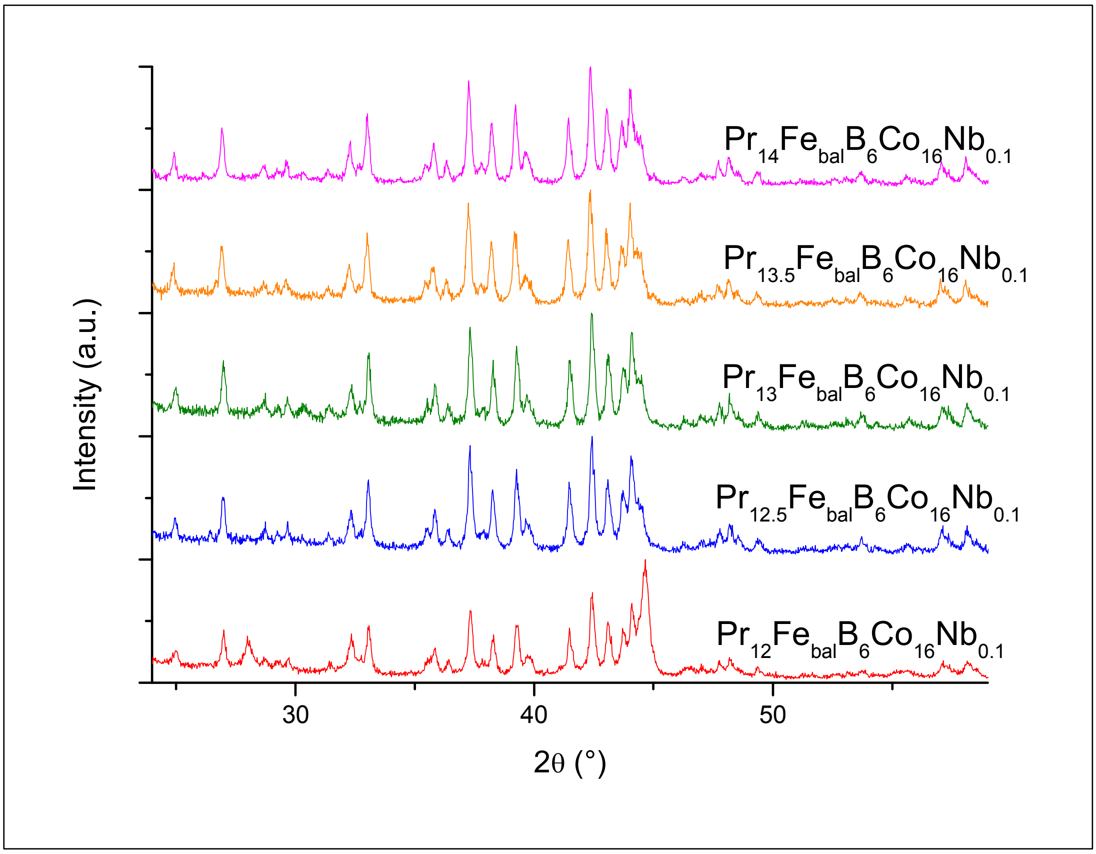

Figure 3 – X-Ray powder diffraction patterns for the $Pr_xFe_{77.9-x}Co_{16}B_6Nb_{0.1}$ ($12 \leq x \geq 14$) alloys. The powder specimens are obtained by HDDR process.

Figure 4 (a-e) shows the microstructures of $Pr_xFe_{77.9-x}Co_{16}B_6Nb_{0.1}$ ($12 \leq x \geq 14$) HDDR powders processed at 840ºC. It seems that there is no notable increase in the particle size with praseodymium content. With respect to the mean crystallite size, it has been found the same value for all magnetic powders (~ 60 nm).

Table 2 - Phases found in the HDDR powders resultants by X-ray diffraction.

Composition	**Phases**
$Pr_{12}Fe_{bal}B_6Co_{16}Nb_{0.1}$	$Pr_2Fe_{14}B$ Fe Pr_7O_{12}
$Pr_{12,5}Fe_{bal}B_6Co_{16}Nb_{0.1}$	$Pr_2Fe_{14}B$
$Pr_{13}Fe_{bal}B_6Co_{16}Nb_{0.1}$	$Pr_2Fe_{14}B$
$Pr_{13,5}Fe_{bal}B_6Co_{16}Nb_{0.1}$	$Pr_2Fe_{14}B$
$Pr_{14}Fe_{bal}B_6Co_{16}Nb_{0.1}$	$Pr_2Fe_{14}B$ Fe_2O_3

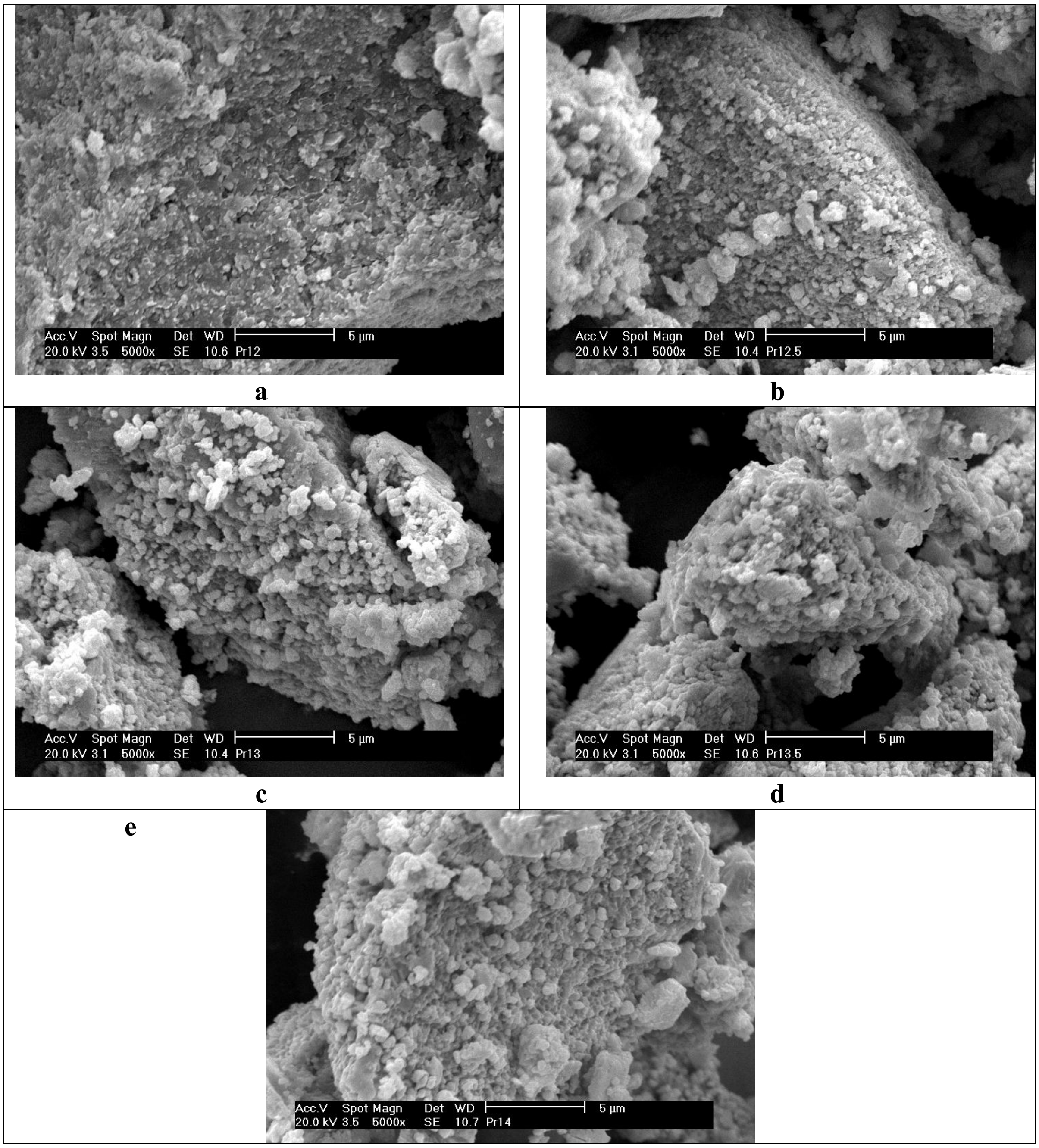

Figure 4 - Microstructures of $Pr_xFe_{77.9-x}Co_{16}B_6Nb_{0.1}$ HDDR powders: (a) x = 12; (b) x = 12.5; (c) x = 13; (d) x = 13.5 and (e) x = 14. (5000x).

Conclusion

HDDR powders processed at 840ºC presented the same value of mean crystallite size for all magnetic powders were approximately 60 nm. The best magnetic properties were obtained with the alloy with 13.5 at.% Pr.

Acknowledgements

The authors wish to thank FAPESP, CNPQ and IPEN-CNEN/SP for financial support and infrastructure made available to carry out this investigation.

References

[1] T.Takeshita and R. Nakayama: 10th International Workshop on RE Magnets and their applications,1989. Proceedings… p. 551.

[2] P. J. McGuiness, X. J. Zhang, X. J. Yin and I. R. Harris: J. Less-Commom Metals Vol. 158 (1990), p. 379.
[3] L. P. Barbosa, H. Takiishi and R. N. Faria: J. Magn. Magn. Mater Vol. 270 (2004), p. 291.
[4] L. P. Barbosa, H. Takiishi, L. F. C. P. Lima and R. N. Faria: J. Magn. Magn. Mater Vol. 285 (2005), p. 290.
[5] S. C. Silva, E. A. Ferreira, R. N. Faria and H. Takiishi: Mat. Science Forum Vols. 591-593 (2008), p. 108.
[6] S. C. Silva, J. H. Duvaizem, L. G. Martinez, M. T. D. Orlando, R. N. Faria and H. Takiishi, Mat. Science Forum Vols. 591-593 (2008), p. 42.
[7] Y. B. Kim and W. Y. Jeung: J. Appl. Phys. Vol. 83 (1) (1998) 6405.
[8] R. N. Faria, A. J. Williams and I. R. Harris: J. Alloy Compd, Vol. 287 (1999) L10.
[9] R. N. Faria, A. J. Williams and I. R. Harris: J. Magn. Magn. Mater. Vol. 202 (1999), p.349.
[10] R. N. Faria, D. N. Brown and I.R. Harris: J. Alloy Compd. Vol. 296 (2000), p. 219.

Advanced Powder Technology VII
doi:10.4028/www.scientific.net/MSF.660-661

Crystallite Size and Magnetic Properties of HDDR Powders Obtained from PrFeCoBNb Alloys

doi:10.4028/www.scientific.net/MSF.660-661.308