Materials Science Forum Vols. 591-593 (2008) pp 108-113
Online available since 2008/Aug/19 at www.scientific.net

doi:10.4028/www.scientific.net/MSF.591-593.108

The effect of the processing temperature on the microstructures of Pr-Fe-Co-B-Nb HDDR magnets

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Keywords: HDDR magnets, Hidrogenation, Pr-Fe-B magnets, Permanent magnets.

Abstract. $Pr_{14}Fe_{bal}Co_xB_6Nb_{0.1}$ magnets have been produced using the hidrogenation disproportionation desorption recombination (HDDR) process. The effect of the Co content (x= 0, 4, 8, 10, 12, 16) and the reaction temperature (800-900 ºC) on the microstructure and magnetic properties of the HDDR material have been investigated. The processing temperature (desorption/recombination) affected the microstructure and magnetic properties of the bonded magnets. The alloy with low cobalt content (4 at.%) required the highest reaction temperature (880°C) to yield anisotropic bonded magnets. The optimum temperature for alloys with 8 at.% Co and 10 at.% Co were 840°C and 820°C, respectively. Alloys with high cobalt content (12 at.% and 16 at.%) were processed at 840°C. Each alloy required an optimum reaction temperature and exhibited a particular microstructure according to the composition. $Pr_{14}Fe_{80}B_6Nb_{0.1}$ magnets have been processed for comparison.

Introduction

The HDDR process is well known as an effective method for producing anisotropic Nd-Fe-B magnetic powders [1]. In the last seven years extensive research has been carried out to study Pr-Fe-B-Co magnets produced using the HDDR process with promising results [2-6]. It has been shown that the percentage of cobalt in $Pr_{14}Fe_{bal}Co_xB_6Nb_{0.1}$ alloys has a significant influence on the alloys microstructures and on the properties of the HDDR magnets [7]. Additions of 0.1 at% niobium are necessary to develop optimum anisotropic magnetic properties of bonded magnets [8]. Heat treatments to homogenize the alloy $Pr_{14}Fe_{bal}Co_{16}B_6Nb_{0.1}$ were studied and observed that better condition to complete elimination of free phase Fe-Co content is in vacuum (10^{-4}mbar) at 1100°C for 20 hours [8]. This paper reports the results of investigations carried out on $Pr_{14}Fe_{bal}Co_xB_6Nb_{0.1}$ (where x= 0, 4, 8, 10, 12, 16) alloys to determine the optimum desorption/recombination temperature. A magnetic $Pr_{14}Fe_{bal}B_6Nb_{0.1}$ powder has been studied for comparison. The magnetic properties of the HDDR magnets were determined in a permeameter. The HDDR powder was examined using scanning electron microscopy (SEM).

Experimental procedure

Commercial alloys in the as-cast state were submitted to a heat treatment in high vacuum (10^{-4} mbar) at 1100°C for 20h. The chemical analyses of the as-cast alloys are given in Table 1.

Details of the HDDR magnets preparation and magnetic measurements have been described in previous papers [2-5; 9-10]. The processing temperature (desorption/recombination) was varied from 800 – 900°C in order to determine the optimum temperature of reaction for each alloy. Part of the HDDR material was used to microstructural examination in a SEM.

Table 1 - Composition of the as-cast alloys.

Nominal composition [at%]	Composition [wt%]					
	Pr	**Fe**	**Co**	**B**	**Nb**	**Al**
$Pr_{14}Fe_{80}B_6Nb_{0.1}$	30.11	68.68	-	0.97	0.14	0.10
$Pr_{14}Fe_{bal}B_6Co_4Nb_{0.1}$	30.05	65.16	3.58	0.97	0.15	0.09
$Pr_{14}Fe_{bal}B_6Co_8Nb_{0.1}$	30.29	61.27	7.15	0.96	0.15	0.09
$Pr_{14}Fe_{bal}B_6Co_{10}Nb_{0.1}$	30.47	59.20	9.03	1.02	0.16	0.12
$Pr_{14}Fe_{bal}B_6Co_{12}Nb_{0.1}$	30.14	57.80	10.83	0.98	0.15	0.10
$Pr_{14}Fe_{bal}B_6Co_{16}Nb_{0.1}$	30.35	54.11	14.34	0.96	0.14	0.10

Results and discussions

The Figure 1 shows the optimum temperature of reaction in the HDDR process as a function of the cobalt content for obtaining high remanence. The best temperature of desorption/ recombination varied considerably with the cobalt content in the alloys. The reference for comparison was the $Pr_{14}Fe_{80}B_6Nb_{0.1}$ alloy. Clearly, each alloy has an ideal desorption/ recombination temperature to yield magnets with excellent magnetic properties.

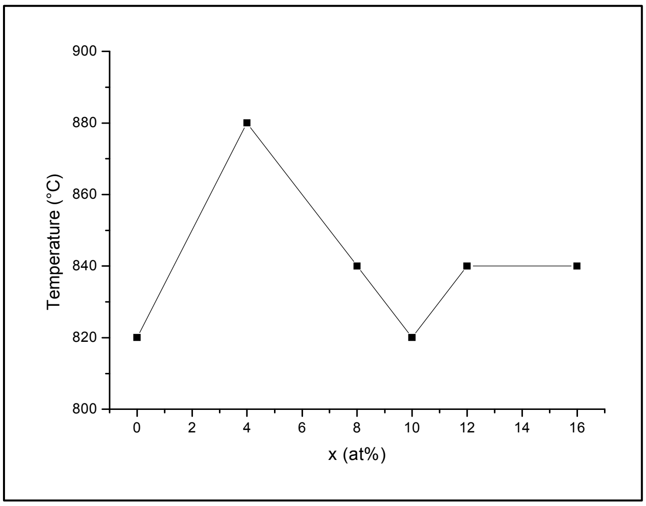
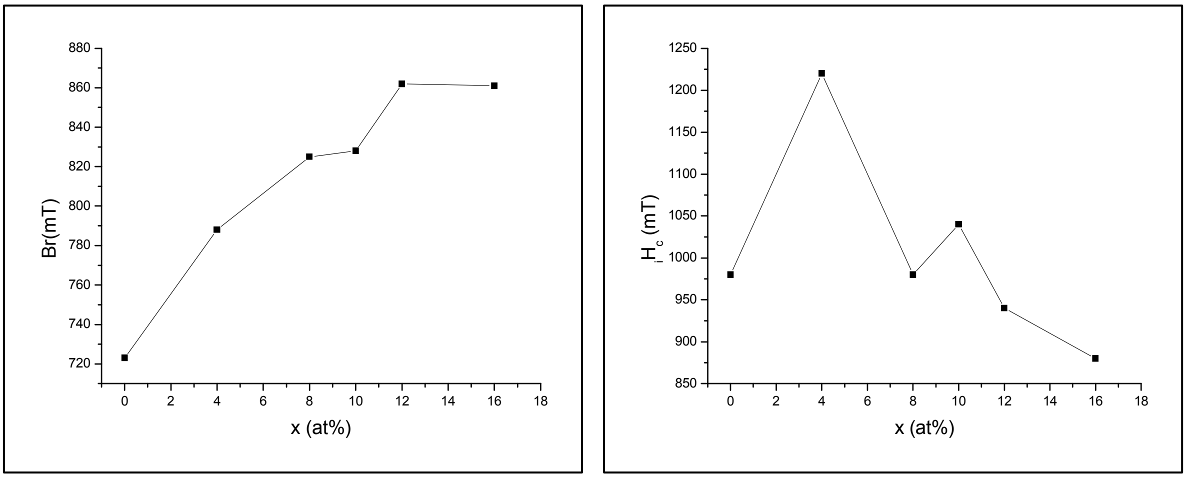


Fig. 1 – Cobalt content versus optimum temperature in the HDDR process for obtaining high remanence in the $Pr_{14}Fe_{bal}Co_xB_6Nb_{0.1}$ magnets.

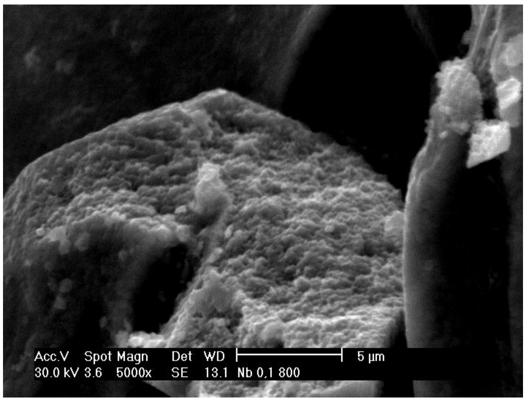
Increasing the cobalt content to 4 at.% this optimum temperature increased to 880°C. HDDR magnets with 8 at.% Co were processed at 840°C for achieving good remanence and magnets containing 10 at.% Co had to be processed at 820°C. The magnets containing 12 at.% Co and 16 at.% Co had a similar reaction temperature (840°C). The variation in the remanence of the HDDR magnets produced from homogenized Pr-based alloys as a function of cobalt content is shown in Figure 2a. The intrinsic coercivity of HDDR magnets prepared from homogenized alloys as a function of cobalt content is shows in the Figure 2b. Reasonable remanence (725 mT) was achieved in the Co-free magnet ($Pr_{14}Fe_{80}B_6Nb_{0.1}$) processed at 820°C. Increasing the cobalt content in the magnets led to an increase in the remanence values up to 12 at.% of Co. Remanence values were practically constant (860 mT) for 12 at.% and 16 at.% Co. Highest coercivity values (1220 mT) were obtained in magnets prepared using the $Pr_{14}Fe_{bal}B_6Co_4Nb_{0.1}$ alloy.



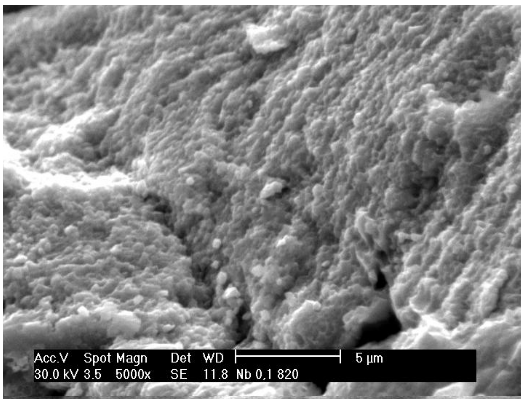
(a) (b)

Fig. 2 – Remanence and intrinsic coercivity versus Co content for the $Pr_{14}Fe_{bal}Co_xB_6Nb_{0.1}$ HDDR magnets.

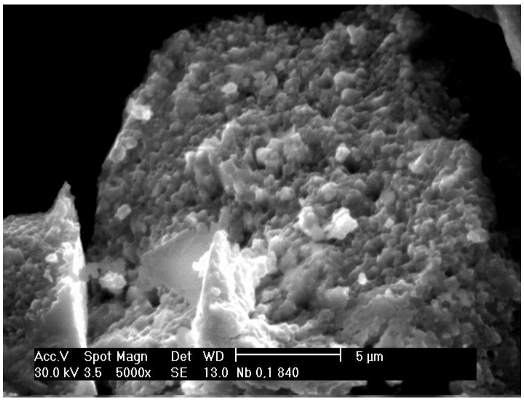
The Figure 3 (a-f) shows the microstructures of $Pr_{14}Fe_{bal}B_6Nb_{0.1}$ HDDR powders processed at distinct temperatures. It can be observed that, with the increase of the reaction temperature from 800°C up to 900°C there was also a stead increase in the grain size. The microstructures of the $Pr_{14}Fe_{bal}B_6Co_{16}Nb_{0.1}$ HDDR powders are shown in Figures 4 (a-f). A comparison of these microstructures reveals that the addition of niobium in the alloys refines the magnetic grains in HDDR powders. Cobalt addition in the alloys led to an increase in grain size and also modified the optimum reaction (desorption/recombination) temperature for each alloy, according to the amount of cobalt that was added.



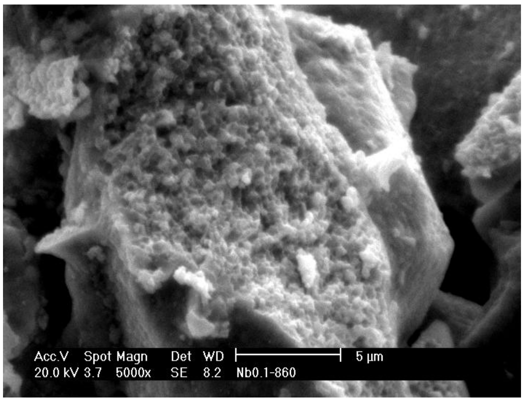
(a)



(b)



(c)



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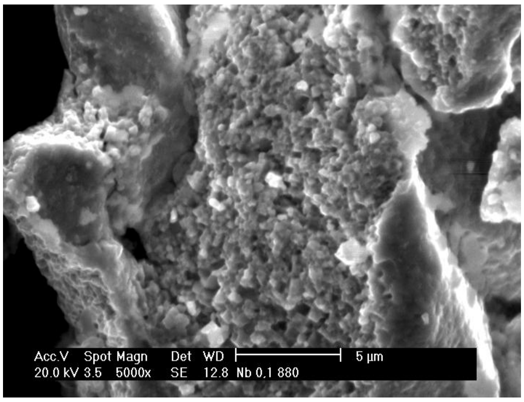
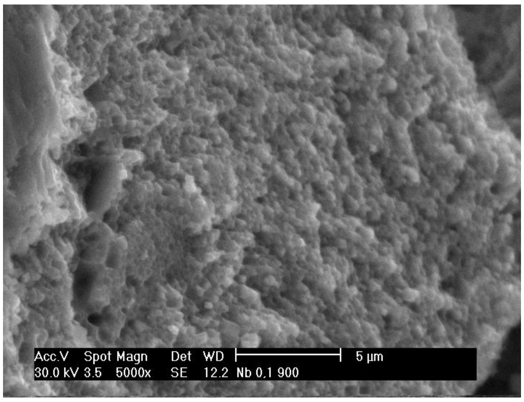


(e)



(f)

Fig. 3 – Microstructures of the $Pr_{14}Fe_{bal}B_6Nb_{0.1}$ HDDR powders: (a) 800°C; (b) 820°C; (c) 840°C; (d) 860°C; (e) 880°C and (f) 900°C. (5000x).

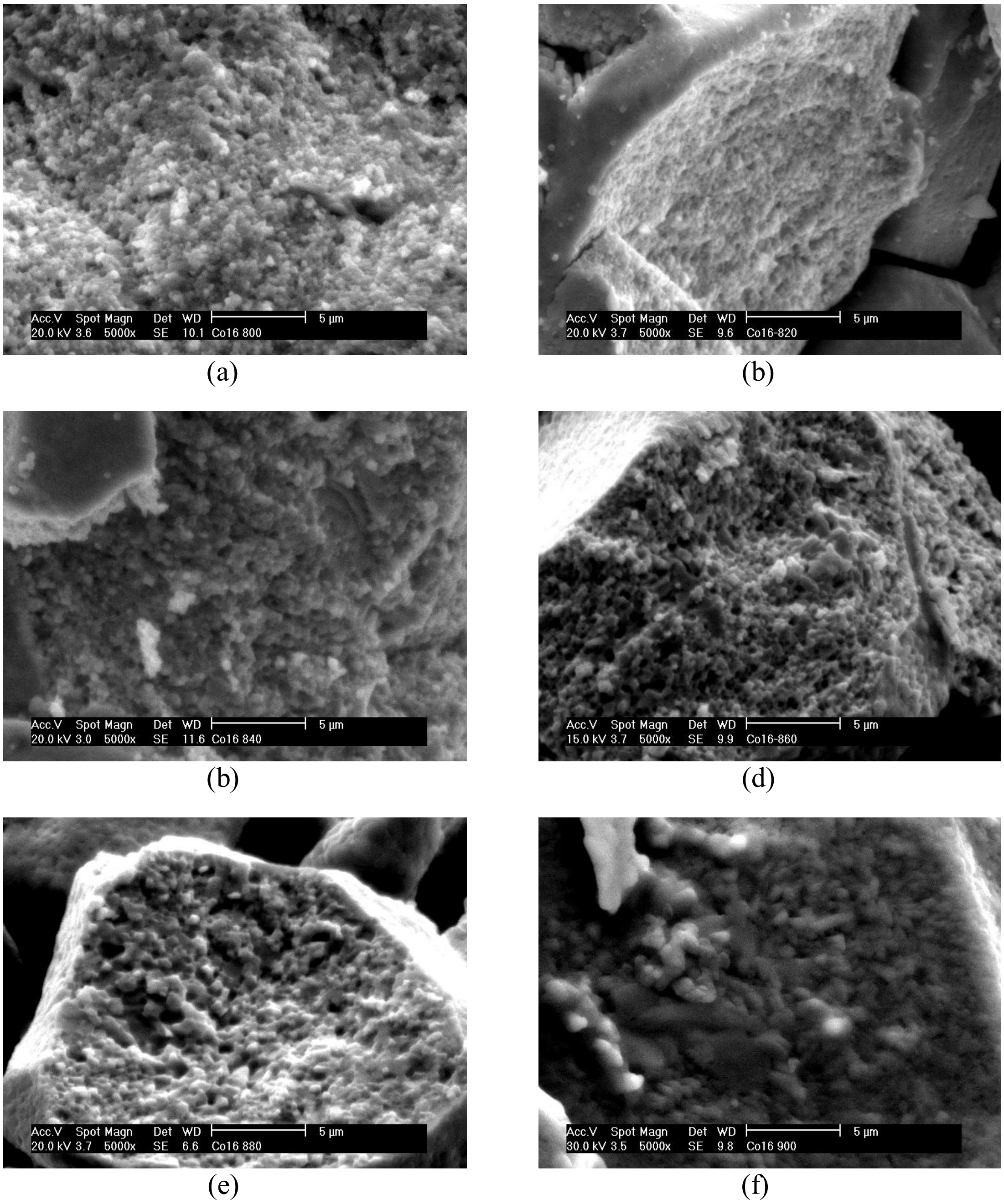


Fig. 4 – Microstructures of the $Pr_{14}Fe_{bal}B_6Co_{16}Nb_{0.1}$ HDDR powders: (a) 800°C; (b) 820°C; (c) 840°C; (d) 860°C; (e) 880°C and (f) 900°C. (5000x).

Conclusions

The temperature of desorption/recombination affected the microstructure and magnetic properties of the bonded HDDR magnets. According to the cobalt content, each alloy required a particular reaction temperature for achieving an optimum remanence and exhibited a distinct microstructure. Increasing the Co content in the HDDR magnet up to 16 at.% induced a higher anisotropy. However, the $Pr_{14}Fe_{bal}B_6Co_4Nb_{0.1}$ HDDR magnet exhibited the highest intrinsic coercivity (1220mT).

Acknowledgments

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

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Advanced Powder Technology VI
doi:10.4028/www.scientific.net/MSF.591-593

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doi:10.4028/www.scientific.net/MSF.591-593.108