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

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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.

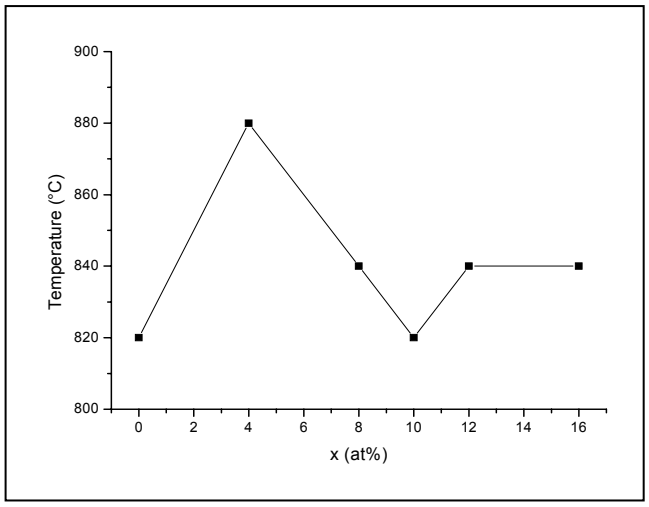
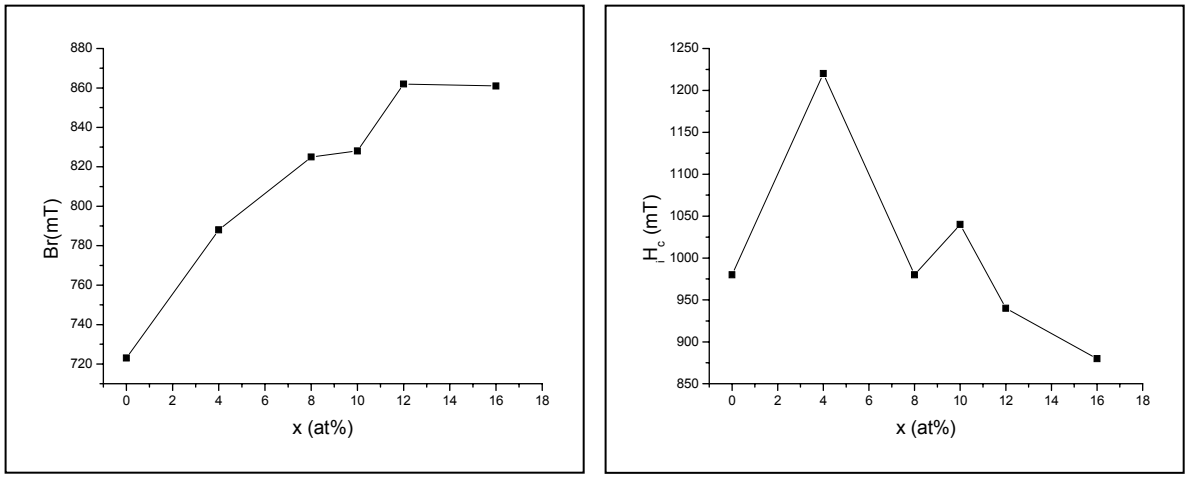


Figure 1 – Cobalt content versus optimum temperature in the HDDR process for obtaining high remanence in the $Pr_{14}Fe_{bal}Co_xB_6Nb_{0.1}$ magnets.

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)

Figure 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 and this behavior is consistent with a previous work [11]. 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.

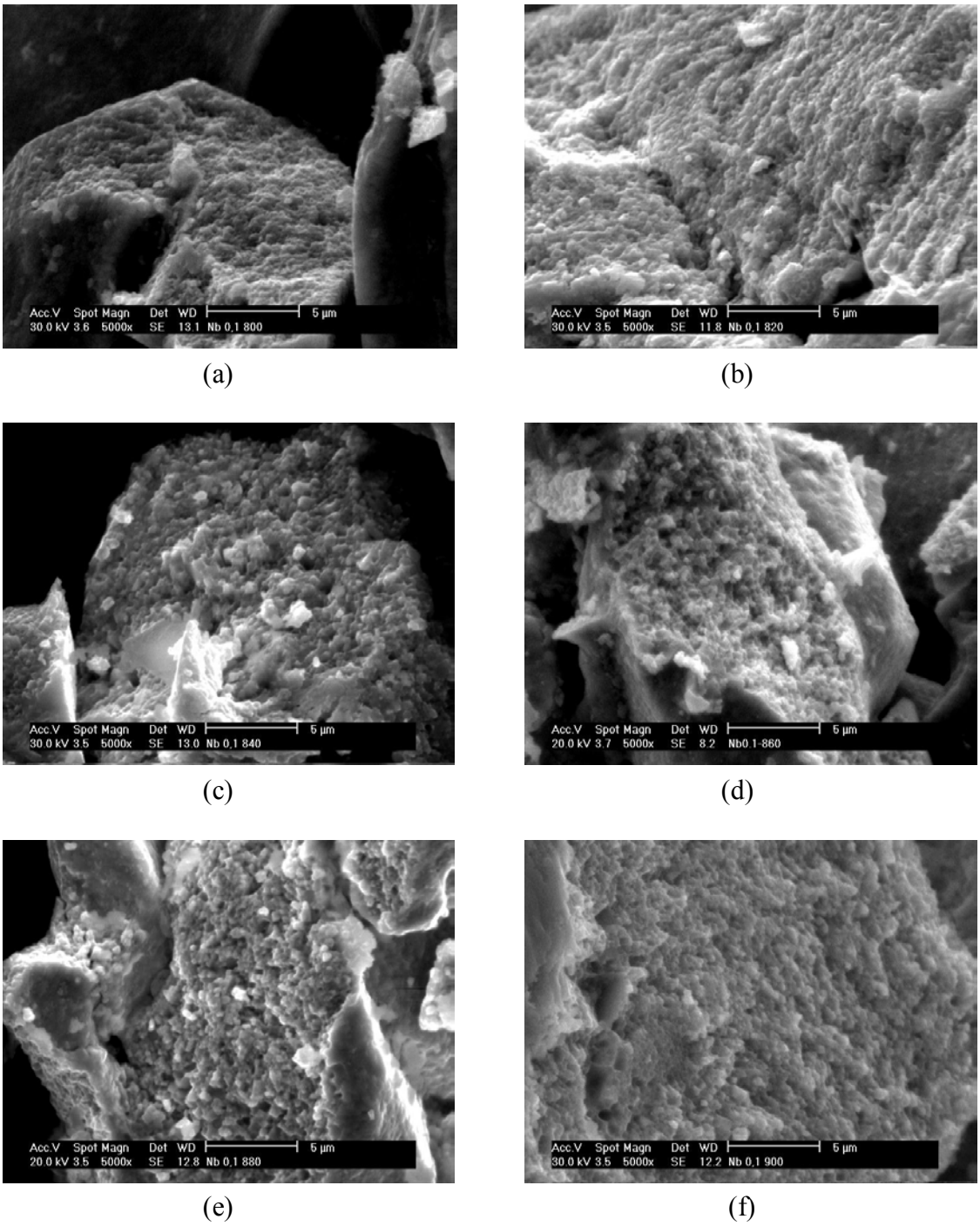


Figure 3 – Microstructures of the $Pr_{14}Fe_{bal}B_6Nb_{0.1}$ HDDR powders: (a) 800°C; (b) 820°C; (c) 840°C; (d) 860°C; (e) 880°C and (f) 900°C. (5000x).

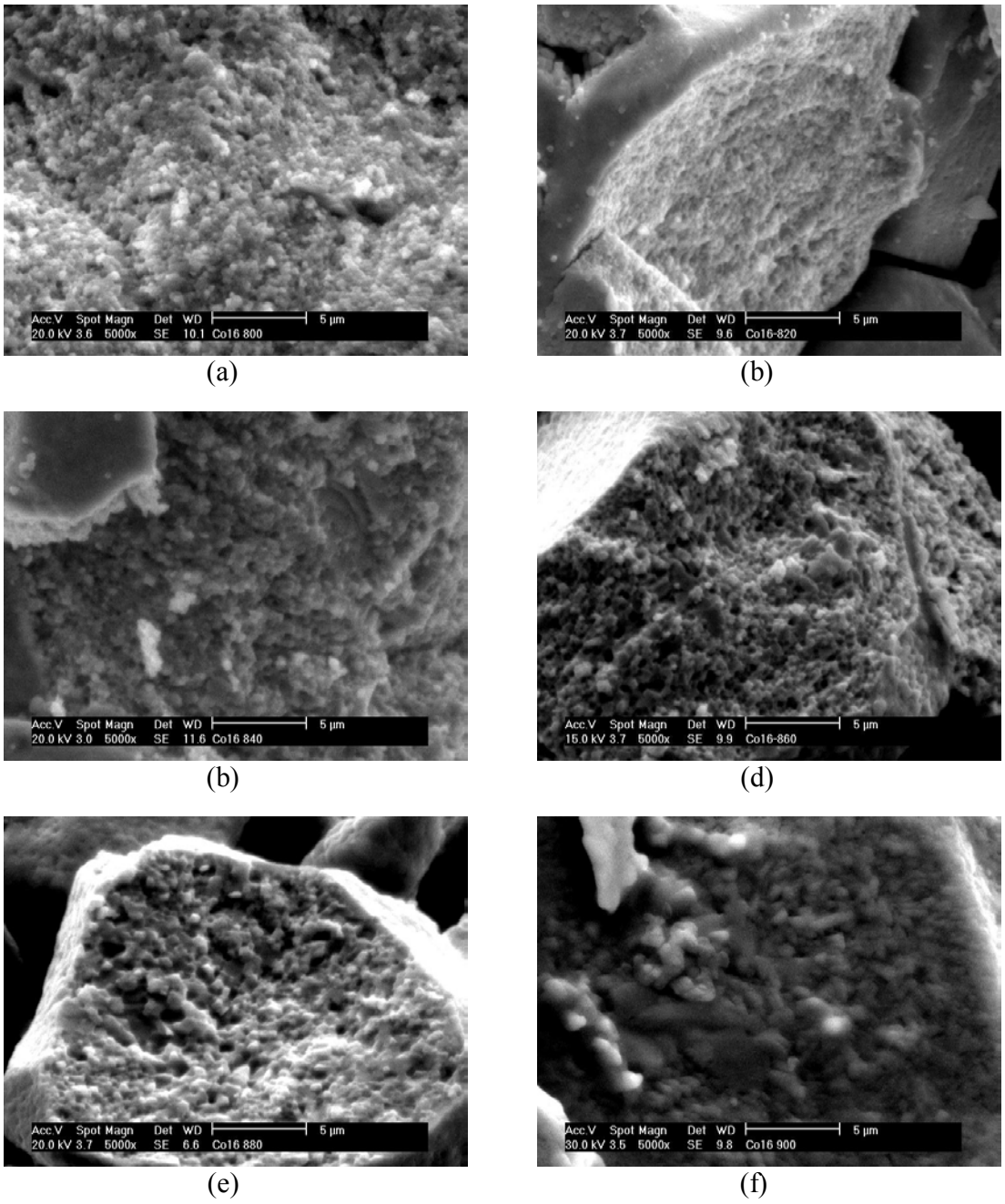


Figure 4 – Microstructures of the $Pr_{14}Fe_{bal}B_6Co_{16}Nb_{0.1}$ HDDR powders: (a) 800°C; (b) 820°C; (c) 840°C; (d) 860°C; (e) 880°C and (f) 900°C. (5000x).

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).

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