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The influence of process parameters and alloy structure on the magnetic properties of NdDyFeBNb HD sintered magnets

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Abstract

Sintered permanent magnets based on the compositions $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ and $Nd_{16}Fe_{76}B_8$ have been produced using the hydrogen decrepitation (HD) process. The effects of process parameters upon the magnetic properties of Nd-based magnets have been studied. It is shown that grain growth occurs in the sintered magnets produced from very fine powders. This work also shows that the influence of the alloy microstructure on the magnetic properties of Nd–Dy–Fe–B–Nb sintered permanent magnets is significant. Magnets produced from the fine grained alloy (near equiaxed grains) exhibited superior intrinsic coercivities to those produced from the as-cast ingot (large columnarlike grains).

Keywords: Sintered magnets; Hydrogen decrepitation; Nd–Dy–Fe–B–Nb alloys

1. Introduction

The microstructure of Nd–Fe–B as-cast ingots has been found to play a role in the magnetic properties of sintered magnets [1–3]. The magnetic properties of Pr-based hydrogen decrepitation (HD) magnets can also be affected by the alloy microstructure [4]. Establishing the optimum processing conditions for each alloy is essential for producing sintered magnets with excellent magnetic properties [5,6]. None of these investigations reported about the influence of the alloy microstructure on the magnetic properties of Nd–Dy–Fe–B–Nb HD sintered magnets. In this study, sintered magnets of compositions $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ and $Nd_{16}Fe_{76}B_8$ have been prepared via the HD process and the influence of the alloy microstructure on the magnetic properties of (Dy, Nb)-containing magnets was studied. In order to establish the optimum processing conditions, a study of the relationship between magnetic properties of the sintered magnets and the milling time was carried out for both alloys. The microstructures of these Nd-based alloys and permanent magnets were investigated using scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX).

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2. Experimental

Commercial $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ alloys in the form of conventional cast ingot and flakes were studied. The well-known $Nd_{16}Fe_{76}B_8$ ingot alloy (Neomax composition) was included in this work as a standard reference. In order to produce the magnets via the HD process [7,8], the following procedure was adopted. Small pieces of the bulk ingot or flakes were placed in a stainless steel hydrogenation vessel, which was then evacuated to backing-pump pressure. Hydrogen was introduced to a pressure of 1 bar, resulting in decrepitation of the bulk material. This material was then evacuated to backing-pump pressure for 30–40 min and transferred to a roller ball mill under a nitrogen atmosphere and milled in a cyclohexane medium. The resultant fine powder was then dried for 1 h and transferred to a small cylindrical rubber tube under a nitrogen atmosphere. The fine powder was aligned by pulsing three times to a 6 T magnetic field, pressed isostatically at a pressure of 200 MPa and then vacuum sintered for 1 h at 1050 °C, followed by cooling on the furnace. Magnetic characterization of the HD sintered Nd-based permanent magnets was carried out using a permeameter. The second quadrant curve used to describe the quality of the magnet included a few important parameters: remanence (B_r), intrinsic coercivity ($_iH_c$), inductive coercivity ($_bH_c$) and maximum energy product (BH_{max}). Squareness factor (SF) was used to determine the stability of the magnets while undergoing demagnetization and defined as the applied field at 90% of the remanence (H_k) divided by the intrinsic coercivity. Measurements were performed after saturation in a pulsed field of 6 T (∼10 ms).

The microstructural observations and microanalysis were carried out using a Phillips XL30 scanning electron microscope fitted with energy dispersive X-ray analysis (light elements boron and oxygen not included). Average EDX data were obtained from several independent measurements from each phase. Grain size measurements were carried out using the Saltykov method (mean of several regions) [9]. Samples for grain analysis were etched with Villela's reagent to reveal grain boundaries.

3. Results and discussion

The microstructures of the cast ingot and flakes of a $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ alloy are shown in Figs. 1 and 2, respectively. The former shows a typical columnar grain structure, whereas the latter exhibits a fine, almost equiaxed, grain structure. A comparison between these two microstructures also demonstrates that, rather dramatically, the production method can change the grain size of a (Dy, Nb)-containing alloy. A detailed microstructural study on the $Nd_{16}Fe_{76}B_8$ ingot alloy, which exhibits a columnar grain structure, can be found elsewhere [10,11]. $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ alloys are composed of the matrix phase (Nd, Dy)$_2$Fe$_{14}$B (Φ), the neodymium-rich phase in the grain boundaries, the boron rich phase $(Nd, Dy)_{1+\varepsilon}Fe_4B_4$ and minor Nb-containing phases (the chemical analysis is discussed later).

Fig. 3 shows the variation of intrinsic coercivity with milling time for $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ sintered magnets prepared using the as-cast ingot and flakes and for $Nd_{16}Fe_{76}B_8$ magnets prepared using the standard as-cast ingot. The highest coercivity (1313 ± 10 kA/m), associated with the shortest milling time (8 h), was exhibited by sintered magnets prepared from dysprosium-containing HD flakes. Slightly lower coercivity (1241 ± 10 kA/m) was achieved in the magnets prepared using the cast ingot (decrepitated) milled for 12 h. Thus, an alloy with a smaller grain size required a shorter milling time to achieve full coercivity. A $Nd_{16}Fe_{76}B_8$ HD magnet processed with a milling time of 27 h exhibited an intrinsic coercivity of 1052±10 kA/m. This behavior is consistent with previous studies [12,13], which have shown that, dysprosium and/or niobium additions to Nd–Fe–B alloys increase considerably intrinsic coercivity. Niobium is also known to refine the alloy grain structure, which would diminish the milling time necessary to obtain magnets with full coercivity, and this is consistent with the present observations for the Nb-containing magnets. Previous work [4] also showed that the influence of the initial microstrucutre on the intrinsic coercivity of Pr–Fe–B–Cu HD sintered permanent magnets diminishes rapidly with increasing milling time and is a minimum when the coercivity reaches the maximum value of about 1591 ± 10 kA/m.

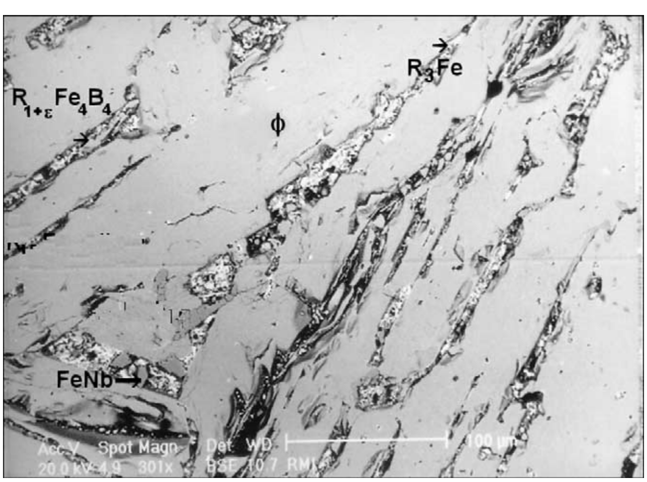

Fig. 1. Details of the microstructure of the $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ ingot (301X).

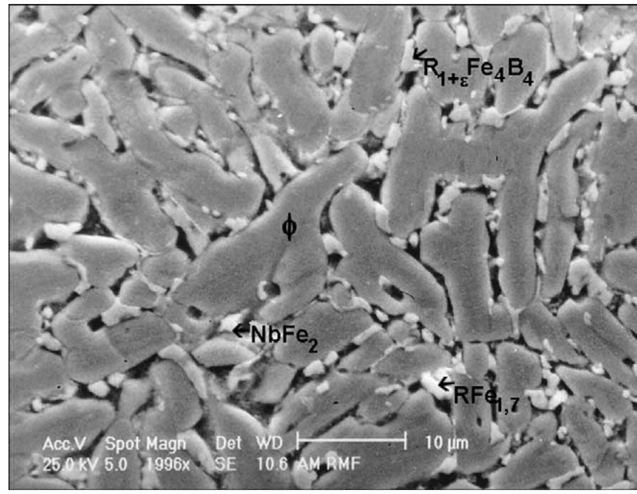

Fig. 2. Details of the microstructure of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ flakes (1996X).

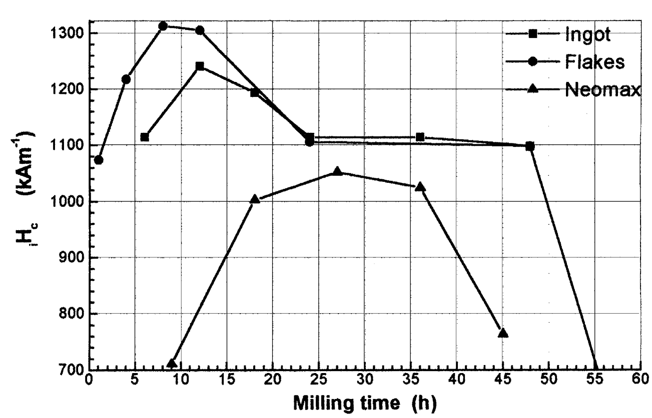

Fig. 3. Variation of coercivity with milling time for $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from ingot and flakes and for $Nd_{16}Fe_{76}B_8$ sintered magnets (Neomax).

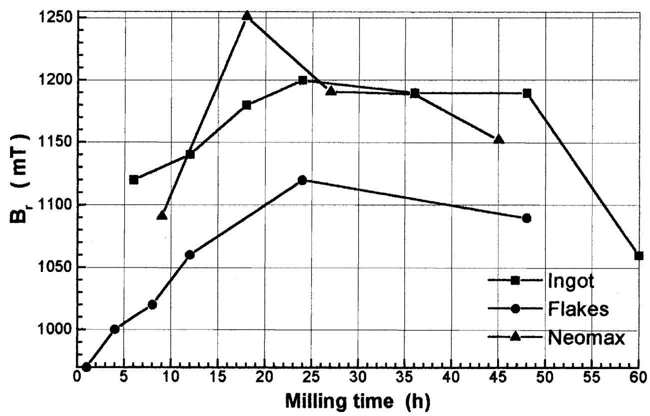

Fig. 4. Variation of remanence with milling time for $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from ingot and flakes and for $Nd_{16}Fe_{76}B_8$ sintered magnets (Neomax).

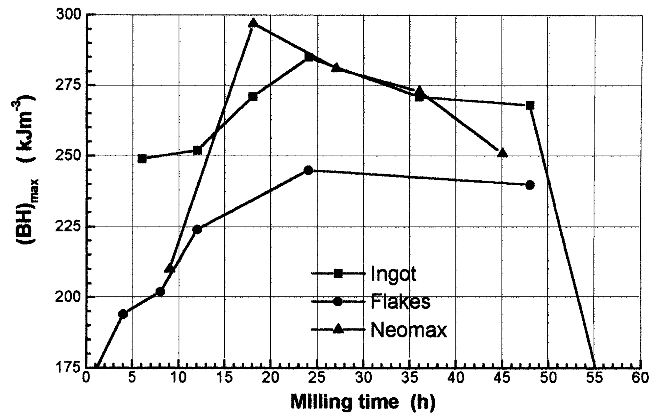

Fig. 5. Variation of energy product with milling time for $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from ingot and flakes and for $Nd_{16}Fe_{76}B_8$ magnets (Neomax).

Fig. 4 shows the variation of remanence with milling time for these HD sintered magnets. Conversely to the coercivity behavior, the $Nd_{16}Fe_{76}B_8$ HD sintered magnet (prepared with a milling time of 18 h) exhibited the best remanence (1251 ± 10 mT). The saturation polarization of $Nd_2Fe_{14}B$ (1.6 T) is considerably higher than that of $Dy_2Fe_{14}B$ (0.712 T) [14] and hence better remanence is expected in a magnet without dysprosium. A steady increase in remanence with milling time was observed in the magnets prepared using the $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ cast ingot, up to a maximum at 24 h and a decrease for longer milling times. Magnets prepared from $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ flakes exhibited a similar behavior but with considerably inferior values of remanence. Large single crystal Φ grains found in a standard cast ingot should, in general, produce sintered magnets with good values of remanence. Hence, the inferior values of remanence of the HD sintered magnets prepared from flakes are likely due to the more isotropic nature of the fine grains of the $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ alloy. Figs. 5 and 6 show the variation of energy product and density with milling time, respectively, and Fig. 7, the demagnetization curves of the samples with best overall magnetic properties. A summary of magnetic properties is given in Tables 1–3.

Figs. 8 and 9 show the Φ grain size distribution in the various HD sintered magnets prepared from the cast ingot an flakes, respectively. A narrow size distribution of matrix

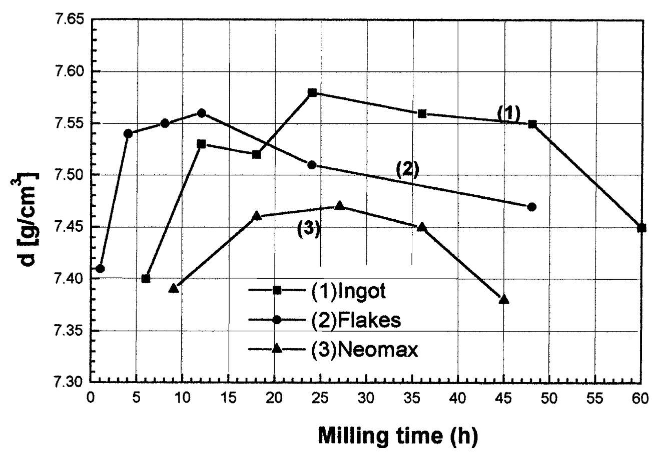

Fig. 6. Variation of density with milling time for $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from ingot and flakes and for $Nd_{16}Fe_{76}B_8$ sintered magnets (Neomax).

phase grains was observed in all HD magnets and, in the latter, the overall distribution ranged from 1 to 10 μm. Fig. 10 shows the variation of the average diameter of Φ grains with milling time for HD magnets prepared from ingot and flakes. In both cases, the diameter of the $(Nd, Dy)_2Fe_{14}B$ grains increased after a particular milling time. This behavior shows that grain growth of the matrix phase occurred in the sintered magnets prepared from very fine powders. The

Table 1
Magnetic properties of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ HD sintered magnets produced from cast ingot

Milling time (h)	B_r (±10) (mT)	${}_iH_c$ (±10) (kA/m)	${}_bH_c$ (±10) (kA/m)	H_k (±10) (kA/m)	SF = $H_k/{}_iH_c$ (ratio)	(BH_{max}) (±10) (kJ/m^3)	Density (±0.02) (g/cm^3)
6	1120	1114	883	991	0.89	249	7.40
12	1140	1241	883	1129	0.91	252	7.53
18	1180	1194	915	1075	0.90	271	7.52
24	1200	1114	939	1014	0.91	285	7.58
36	1190	1114	923	991	0.89	271	7.56
48	1190	1098	907	944	0.86	268	7.55
60	1060	445	430	236	0.53	111	7.45

Table 2
Magnetic properties of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ HD sintered magnets produced from flakes

Milling time (h)	B_r (±10) (mT)	${}_iH_c$ (±10) (kA/m)	${}_bH_c$ (±10) (kA/m)	H_k (±10) (kA/m)	SF = $H_k/{}_iH_c$ (ratio)	(BH_{max}) (±10) (kJ/m^3)	Density (±0.02) (g/cm^3)
1	970	1074	708	687	0.64	174	7.41
4	1000	1218	788	1011	0.83	194	7.54
8	1020	1313	796	1090	0.83	202	7.55
12	1060	1305	835	1122	0.86	224	7.56
24	1120	1106	883	984	0.89	245	7.51
48	1090	1098	875	1021	0.93	240	7.47

Table 3
Magnetic properties of $Nd_{16}Fe_{76}B_8$ HD sintered magnets produced from the cast ingot

Milling time (h)	B_r (±10) (mT)	${}_iH_c$ (±10) (kA/m)	${}_bH_c$ (±10) (kA/m)	H_k (±10) (kA/m)	SF = $H_k/{}_iH_c$ (ratio)	(BH_{max}) (±10) (kJ/m^3)	Density (±0.02) (g/cm^3)
9	1091	711	592	427	0.60	210	7.39
18	1251	1003	836	752	0.75	297	7.46
27	1191	1052	856	863	0.82	281	7.47
36	1189	1025	842	820	0.80	273	7.45
45	1152	764	688	649	0.85	251	7.38

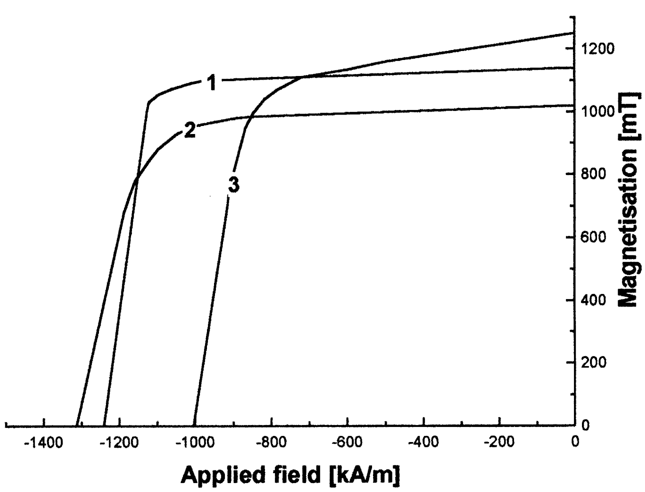

Fig. 7. Demagnetization curves of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from the ingot milled for 12 h (1), from flakes milled for 8 h (2) and from $Nd_{16}Fe_{76}B_8$ milled for 18 h (3).

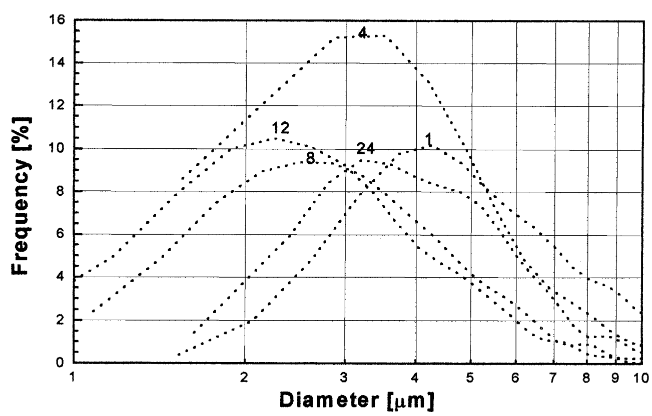

Fig. 9. Grain size distribution of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from flakes. Milling time is indicated for each curve.

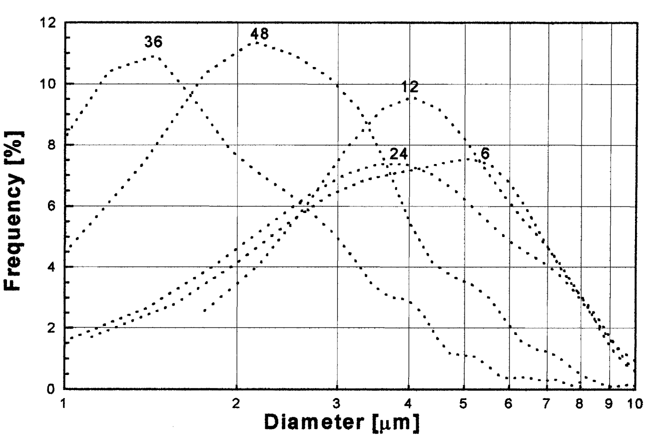

Fig. 8. Grain size distribution of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from ingot. Milling time is indicated for each curve.

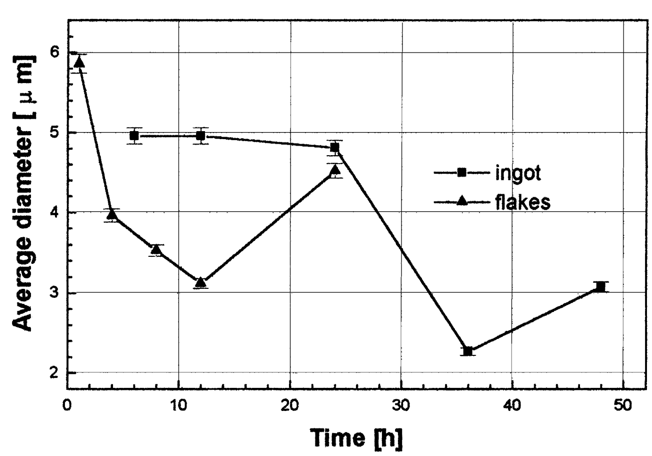

Fig. 10. Mean grain size of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ HD sintered magnets.

preparation of sintered magnets from rare earth based powders becomes difficult when powders with fine particle size are to be sintered. A dramatic decrease in remanence and coercivity of sintered magnets has been reported when the particle size decreases below a critical value [15]. This was attributed to oxidation of the fine powder. Furthermore, it has been reported that overmilling reduces the intrinsic coercivity by damaging the particle surface [16]. The present results show that, for a particular long milling time, grain growth could also be a contributory factor to decrease magnetic properties. The microstructures (general view) of the magnets prepared from as-cast ingot and flakes are shown in Figs. 11 and 12.

Chemical compositions of the phases determined by EDX on an SEM are listed in Table 4. The boron concentration cannot be detected by this means and hence it is the ratio of the metallic components concentrations that should be regarded as significant [17]. The matrix phase ($R_2Fe_{14}B$, R = Nd, Dy) showed a Fe:R atomic ratio of about 7.2, which is a reasonable value since boron was not analyzed. Another phase in these materials showed a Fe:R atomic ratio of about 1.7 and this might be a RFe_2 phase [18]. Laves phase $NbFe_2$

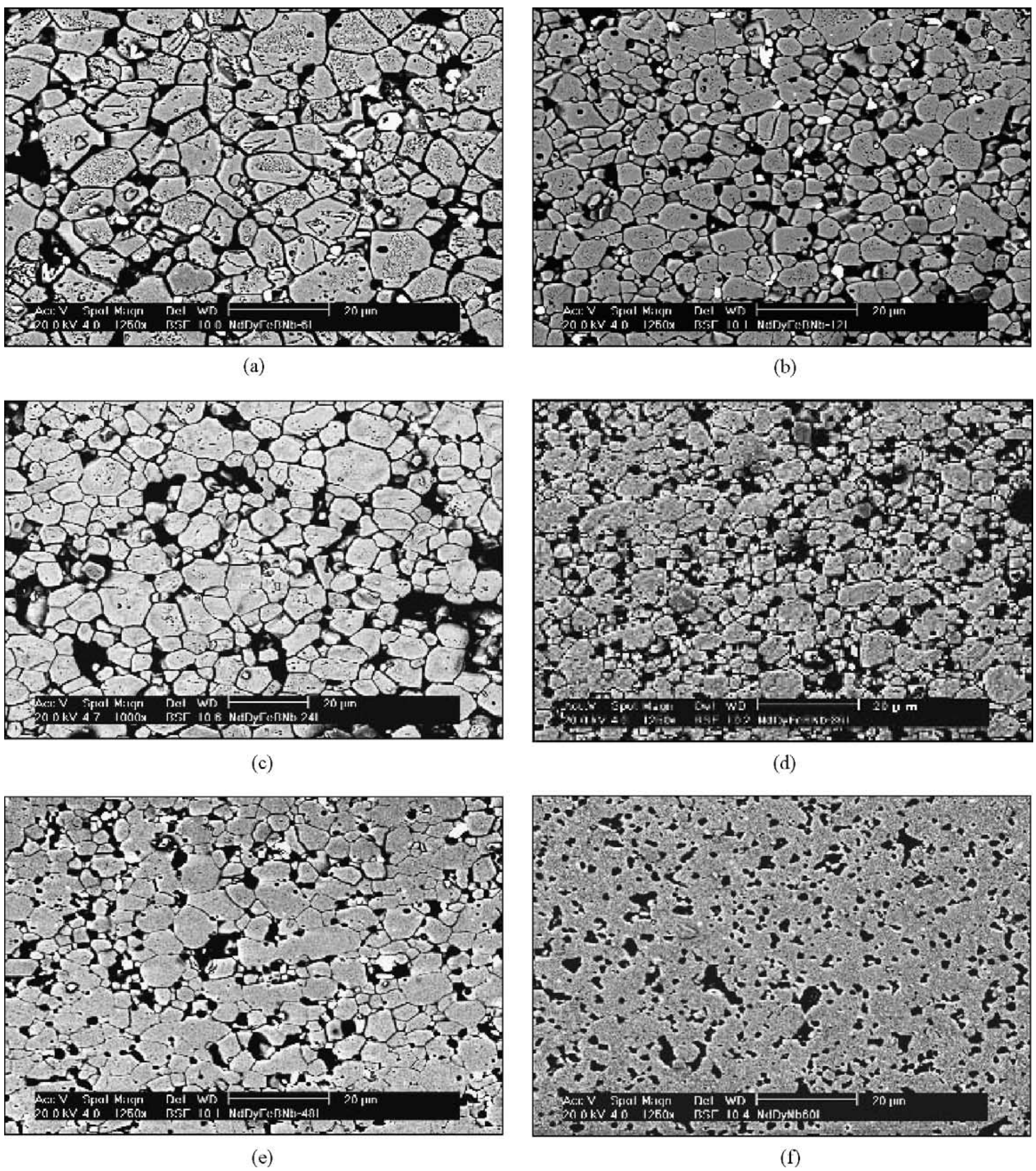

Fig. 11. General view of the microstructure of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from the decrepitated ingot milled for: (a) 6 h (b) 12 h (c) 24 h (d) 36 h (e) 48 h and (f) 60 h (etched with Villela's reagent).

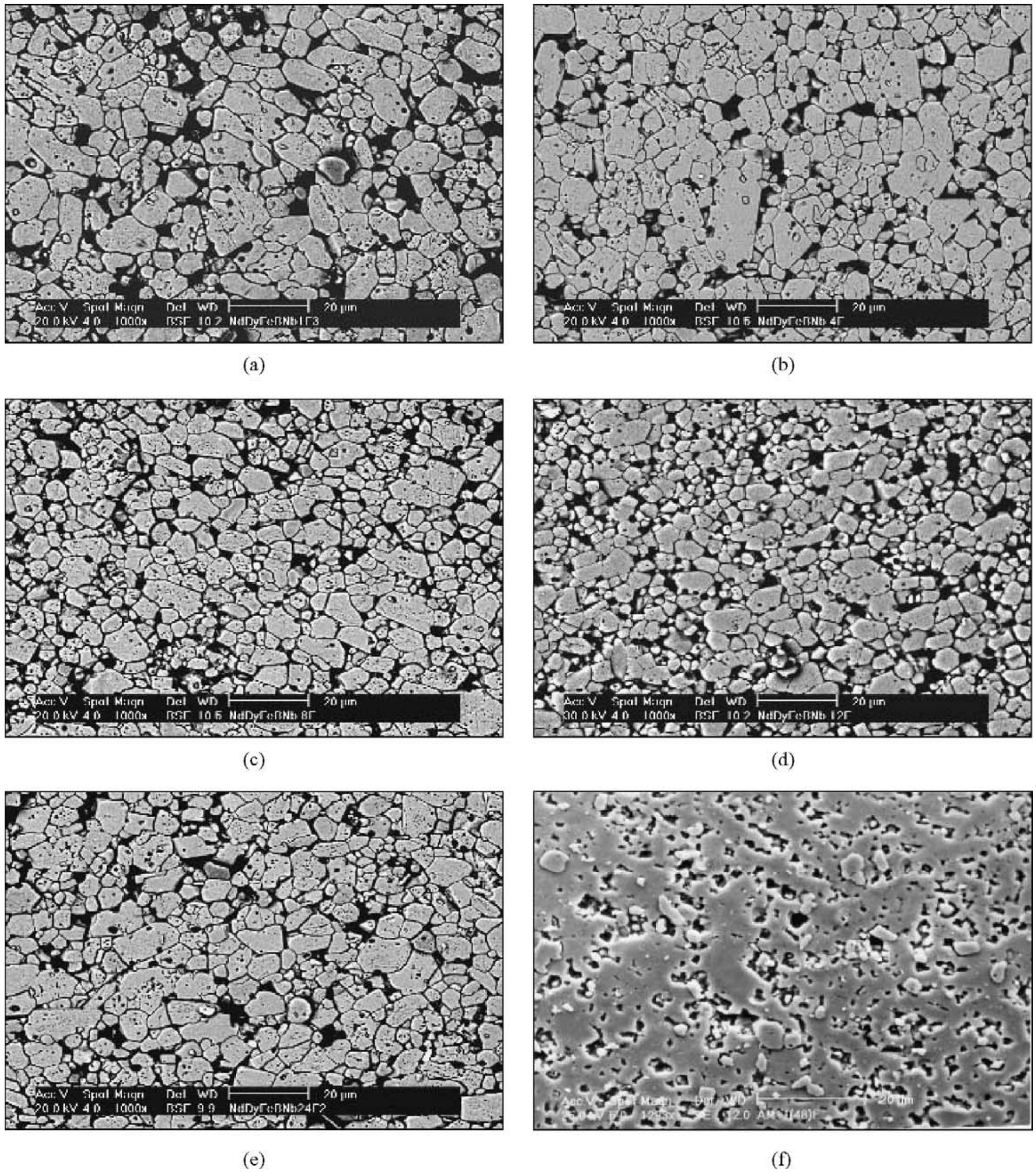

Fig. 12. General view of the microstructure of $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ magnets produced from decrepitated flakes milled for: (a) 1 h (b) 4 h (c) 8 h (d) 12 h (e) 24 h and (f) 48 h (etched with Villela's reagent).

Table 4
Composition determined by EDX at the centers of phases present in the $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ alloy (flake/ingot) and in the HD sintered magnets prepared from this material

Identified phase	R = (Nd, Dy) (at.%)	Fe (at.%)	Nb (at.%)	Appearance
$R_2Fe_{14}B$ (ϕ)	12	87	1	All
$R_{1+\varepsilon}Fe_4B_4$	19	80	1	Cast ingot/flake
$RFe_{1.7}$	37	62	1	Flake
FeNb	<1	51	48	Cast ingot
R_3Fe	74	26	1	Cast ingot/magnets prepared from ingot and flakes
$R_7Fe_{49}Nb_{44}$	7	49	44	Magnets prepared from ingot
$NbFe_2$	8	60	32	All

Results are average of several measurements Individual readings showed scatter of up ±3 at.%.

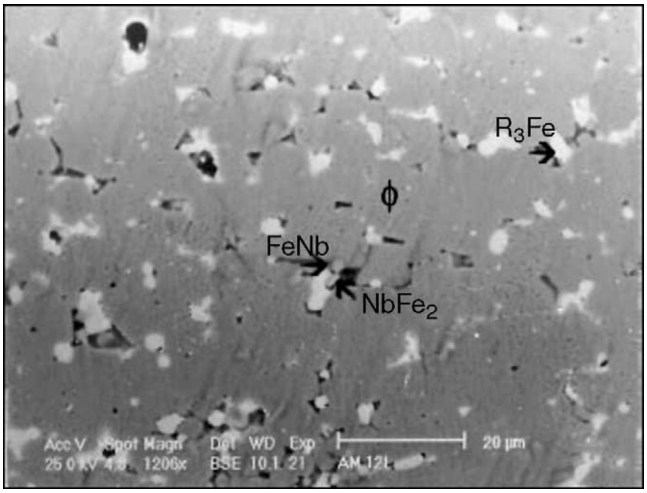

Fig. 13. Microstructure of a $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ HD sintered magnet prepared from the ingot (milled for 12 h).

has also been found in these materials [19–22]. The R_3Fe phase has already been identified previously [23] and also found in cobalt containing magnets on the form of Nd_3Co [22] or $Nd_3(CoFe)$ [17]. The $R_7Fe_{49}Nb_{44}$ phase was identified previously with the composition $Nd_7Fe_{48}Nb_{45}$ [24]. The FeNb phase could be NbFeB or Nb_3Fe_3B phases [25–27], according to boron content. Finally, a $R_8Fe_{60}Nb_{32}$ phase was found in the magnets prepared from the decrepitated cast ingot and flakes. The rare earth detected in the $R_8Fe_{60}Nb_{32}$ phase could also be due to the close proximity of Laves phases to R-rich phases. Boron rich phase (RFe_4B_4) has also been found in DyNb-containing magnets [27] and, recently, a niobium rich phase ($Nb_{87}Fe_{13}$) has been observed in Nd-FeBNb mixed powder sintered magnets [28]. Identification of the phases found in the present HD magnets can be seen in Figs. 13 and 14.

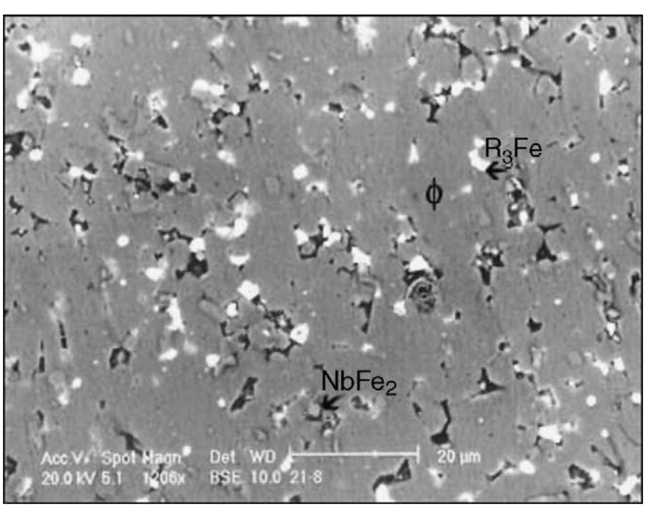

Fig. 14. Microstructure of a $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ HD sintered magnet prepared from flakes (milled for 8 h).

4. Conclusions

The different microstructures found in the start $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ alloy have a significant effect on the magnetic properties of the HD sintered magnets. Permanent magnets produced by the hydrogen decrepitation process from fine grained $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ flakes exhibit superior intrinsic coercivities to those produced from the as-cast ingot. Conversely, sintered magnets produced from the large grained $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb_1$ ingot alloy exhibit superior remanences and energy products to those produced from the flakes. This has been attributed to the more isotropic nature of this starting material. Diminished magnetic properties of sintered magnets produced with powders subjected to prolonged milling times have always been attributed to oxidation and particle surface damage but in this study it has been shown that this is also due to grain growth on sintering very fine particles. An excellent energy product was obtained in a $Nd_{16}Fe_{76}B_8$ HD sintered magnet produced using the conventional cast ingot alloy processed at a somewhat prolonged milling time. The Dy-containing sintered magnets produced from ingot exhibited good overall magnetic properties associated with a reasonable milling time. Processing time can be shortened with a refined grain alloy.

Acknowledgements

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