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Magnetic Properties of Nd-Dy-Fe-B-Nb Sintered Magnets Produced Using the Hydrogen Decrepitation Process

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Abstract

Sintered permanent magnets with a nominal composition $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb$ have been prepared using hydrogen decrepitation process. A study on the relationship between processing and magnetic properties has been carried out. For a particular processing condition, the intrinsic coercivity reached the maximum value of 15.6 kOe. The energy product of the best Nb-containing sintered magnet achieved approximately 36 MGOe.

Introduction

Niobium addition to Nd-Fe-B magnets results in an improvement in coercivity, squareness factor and thermal stability[1-4]. The substitution of Dy for Nd in Nd-Fe-B sintered magnets also results in an increase in the anisotropic field and coercivity[5,6]. Previous investigation of these Nb containing Nd-Dy-Fe-B magnets used conventional powder metallurgy process[7]. In the present work, sintered Nb-containing Nd-Dy-Fe-B magnets have been prepared via the hydrogen decrepitation (HD) process[8]. In order to establish the optimum processing conditions[9], a study of the relationship between the magnetic properties of the sintered magnets and the milling time has been carried out for the Nd-Dy-Fe-B-Nb magnets.

Experimental

Alloy ingots were prepared by induction melting the pure constituents under argon atmosphere. The alloy was poured into rectangular water cooled copper mould. The as-cast microstructure of the starting alloy with the familiar columnar grain structure is shown in Fig. 1.

In order to produce the magnets via the HD process small pieces of the bulk ingot were placed in a hydrogenation vessel which was evacuated to backing-pump pressure and hydrogen was then introduced to a pressure of 2 bar. The HD material was then transferred to a roller ball-mill and milled using cyclohexane as the milling medium. The resultant fine powder decrepitated was then dried and transferred to a small cylindrical rubber tube, pulsed in a magnetic field of 60 kOe and isostatically pressed at 200 MPa. The green compacts were vacuum sintered at 1050°C for 1 hour and furnace cooled (~3.5°C/min.). The sintered magnets were then magnetized and their demagnetization curves determined.

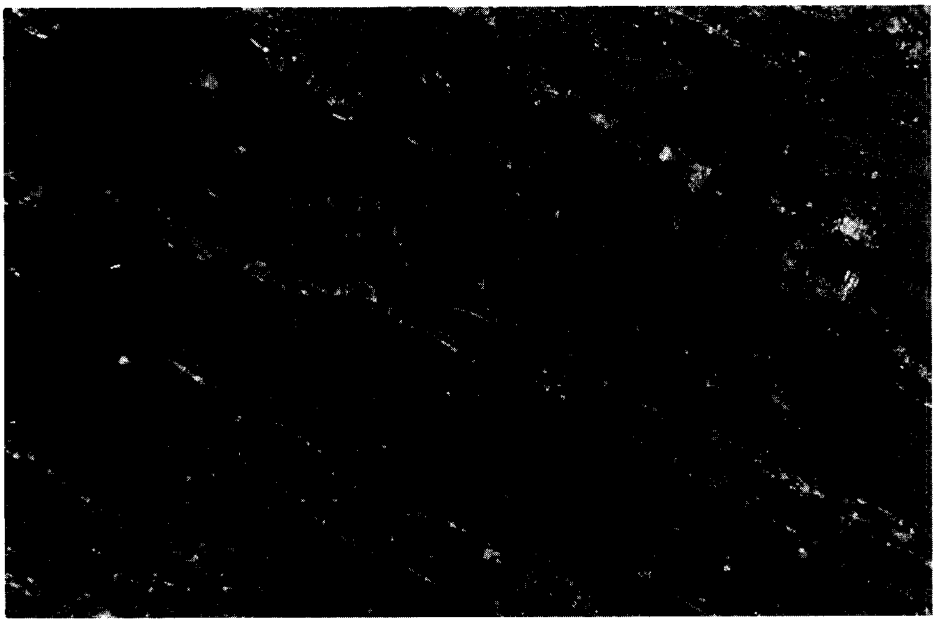

Fig. 1. Microstructure of the $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb$ ingot.

Results and Discussion

Figs. 2 and 3 show how the coercivity and remanence are affected by the milling time. An initial increase in both properties is observed, with material milled for 12 hours having a iHc greater than 15 kOe. The intrinsic coercivity of these Nb-containing magnets passes through a maximum and then decreases dramatically. The remanence, unlike iHc, goes through a flat maximum and then decreases drastically. Total deterioration of the magnetic properties is observed when the milling time reaches 60 hours.

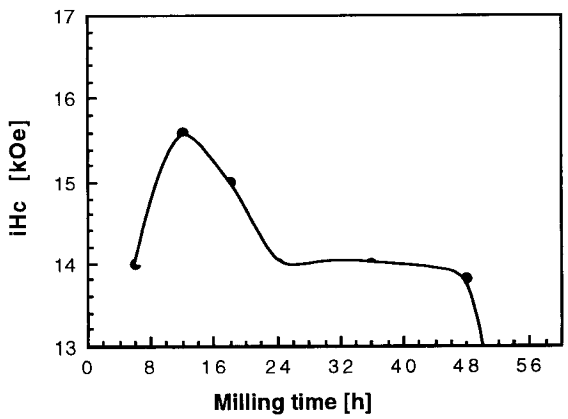

Fig.2. Variation of iHc with the milling time.

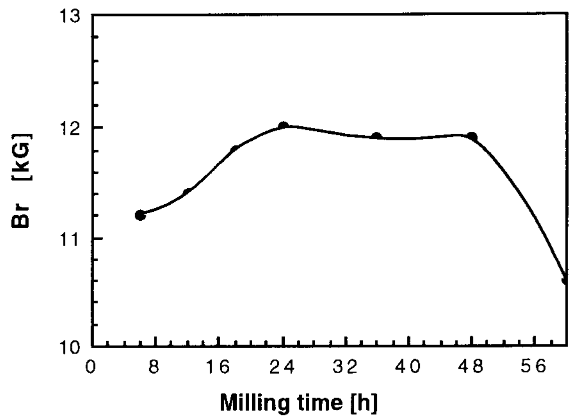

Fig.3. Variation of Br with the milling time .

The effects of milling time on the energy product and squareness factor are shown in Figs.4 and 5, respectively. In the same way, there is an initial enhancement of the energy product, reaching a maximum value (35.8 MGOe) for the material milled for 24 hours, followed by a decrease.

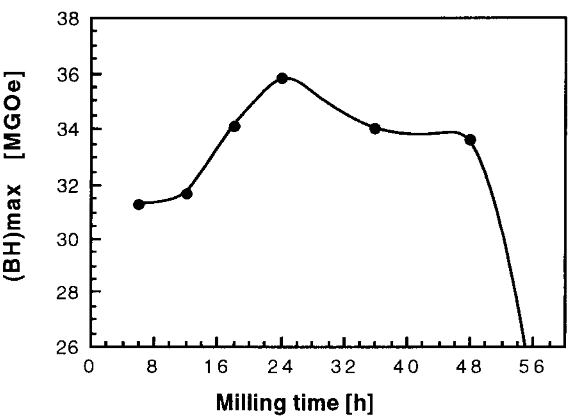

Fig.4. Variation of (BH)max with the milling time.

The preparation of sintered magnets from RE-Fe-B powders becomes difficult when powders with fine particle size are to be sintered. Christodoulou et al[10] reported a dramatic decrease in remanence and intrinsic coercivity of the sintered magnet when the particle size passes below a critical value. This behaviour was attributed to the oxidation of the fine powder. It has also been reported[11] that overmilling reduces the intrinsic coercivity by causing damage to the particle surface.

The second quadrant demagnetization curve for a $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb$ HD sintered magnet is shown in Fig. 6 (best iHc). A very nice loop shape is obtained in the magnet prepared with the powder milled for 12 hours (SF=0.91). A summary of magnetic properties is given in Table 2. Microstructural studies are underway in an attempt to explain the coercivity behaviour observed in this work.

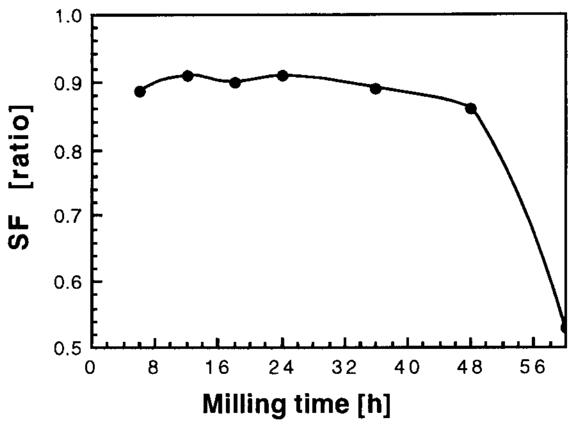

Fig.5. Variation of squareness factor with milling time.

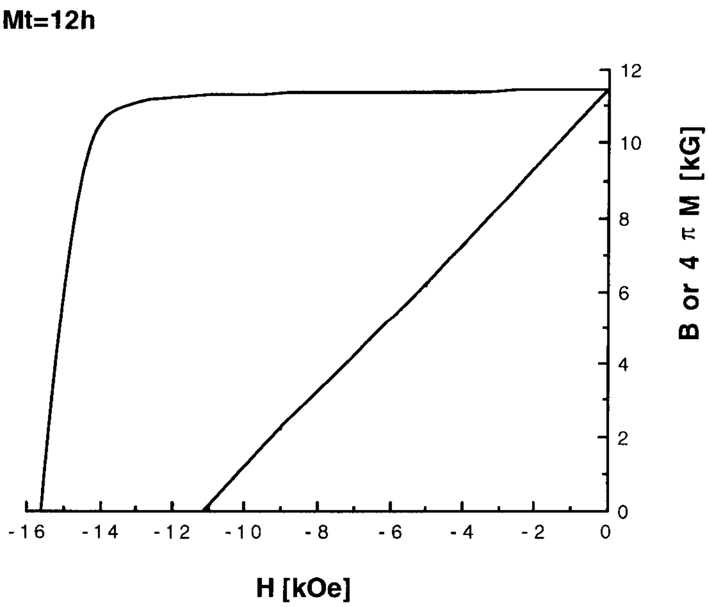

Fig.6. Demagnetization curve for a $Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb$ magnet prepared using the HD powder milled for 12 hours.

Table I. Magnetic properties of the Nd-Dy-Fe-B-Nb HD magnets.

Milling Time [Hours]	Br [kG]	iHc [kOe]	(BH)max [MGOe]	bHc [kOe]	SF ratio
6	11.2	14.0	31.3	11.1	0.89
12	11.4	15.6	31.7	11.1	0.91
18	11.8	15.0	34.1	11.5	0.90
24	12.0	14.0	35.8	11.8	0.91
36	11.9	14.0	34.0	11.6	0.89
48	11.9	13.8	33.6	11.4	0.86
60	10.6	5.6	14.0	5.4	0.53

Average error: Br±0.5 , iHc±0.5 , (BH)max:±0.9

Conclusions

$Nd_{14.5}Dy_{1.5}Fe_{76}B_7Nb$ HD magnets prepared from the ingot material exhibit a maximum in iHc when the HD powder is milled for 12 hours. Good overall magnetic properties have been achieved for the sintered magnet prepared from the hydrided powder milled for 24 hours (Br = 12 kG, iHc = 14 kOe, bHc = 11.8 kOe, (BH)max = 35.8 MGOe and SF = 0.91). There was a total degradation in all magnetic properties when the hydride powder is milled for 60 hours.

Acknowledgments

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