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THE MAGNETIC SEPARATION OF Nd-Fe-B POWDERS

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Abstract-The magnetic separation of Nd-Fe-B powders prepared by melt-spun and HDDR processes was investigated. The experiments show that the ununiform melt-spun powders can be separated into various standards by means of magnetic separation method. The magnetic powders with higher properties were obtained by the use of suitable separating field. For example, the properties of ununiform melt-spun powders are Br=7.95 kG, iHc=9.93 kOe and (BH)max=10.2 MGOe before separating. Through separating in different magnetic fields, the powders obtained with a separating field of 780 Oe has the optimum properties of Br=7.7 kG, iHc=11.0 kOe and (BH)max=15.3 MGOe. The magnetic properties of the HDDR magnetic powder are hardly separated by the magnetic separation method.

I. INTRODUCTION

The Nd-Fe-B powders prepared by melt-spun and HDDR processes have been studied in detail[1,2]. The melt-spun powder produced in industrial scale presents frequently ununiformity in phase structure and magnetic properties due to inappropriate rapid solidification process or the deviation of alloy composition. The demagnetization curves of the ununiform powder appear the concave shape. The thermomagnetic curves are consistent with the presence of the matrix $Nd_2Fe_{14}B$ phase and soft magnetic α-Fe phase with an estimated volume fraction of about 20 wt%, therefore, the magnetic properties of the powder were decreased remarkably[3]. This paper describes our recent work on the magnetic separation of ununiform melt-spun powder in different separating fields. The aim is to divide the ununiform magnetic properties into different standards which may be suitable for different applications, and to provide some useful reference data, in order to show that the magnetic powder with optimum magnetic properties can be easily extracted from the ununiform magnetic melt-spun powders with low magnetic properties. Moreover, the HDDR magnetic powders were also separated and compared with melt-spun powders.

II. EXPERIMENTAL PROCEDURE

The ununiform melt-spun Nd-Fe-B powders with (BH)max=7.85 and 10.2 MGOe and HDDR magnetic powder with (BH)max=11.5 MGOe were separated in different magnetic fields by a special magnetic separation equipment. Ten kinds of the magnetic fields were selected to divide melt-spun powders and three kinds of magnetic fields for HDDR magnetic powder. Thermomagnetic curves of the ununiform melt-spun Nd-Fe-B powder and demagnetization curves of all the powders were measured by Faraday's magnetic balance and LDJ-9500 Vibrating Sample Magnetometer (VSM) respectively.

III. RESULTS AND DISCUSSION

The thermomagnetic curves of the ununiform melt-spun Nd-Fe-B powder with Br=7.38 kG, iHc=14.3 kOe and (BH)max=7.85 MGOe is shown in Fig.1. Two transition points, Tc_1 around 340 °C and Tc_2 around 770 °C, correspond to the Curie temperatures of the $Nd_2Fe_{14}B$ phase and α-Fe phase respectively, indicating that two phases exist in the ununiform powder. The volume of the α-Fe phase with soft magnetic characteristic is estimated to be more than about 20%. The demagnetization curves of the powder obtained with a separating field of

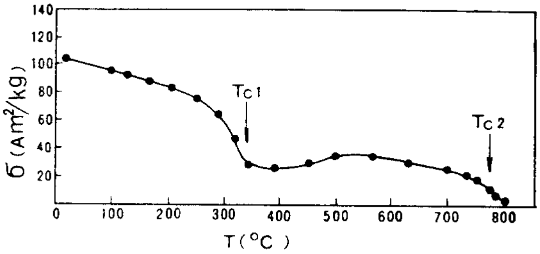

Fig.1 Thermomagnetic curves for the ununiform melt-spun Nd-Fe-B powder with Br=7.38 kG, iHc=14.3 kOe and (BH)max=7.85 MGOe.

514 Oe (b) and ununiform powder (a) are shown in Fig.2. Because of a large amount of α-Fe phase, the curve (a) appears the concave shape.

The variation of the magnetic properties and the weight percentage of the powder with different separating fields is shown in Fig.3. The remanence Br of the powder extracted from the ununiform powders has a slight change with increasing field intensity, but the coercivity iHc and the energy product (BH)max are increased remarkably with increasing field intensity. The shape of demagnetization curves of the powder is gradually improved. When the field intensity is raised to 514 Oe, the powder extracted from the ununiform powders exhibits higher magnetic properties: Br=7.9 kG, iHc=16.3 kOe and (BH)max=13.85 MGOe as shown in Fig.2b. In the case of the low separating fields , the low values of iHc and (BH)max are due to the high volume of α-Fe phase

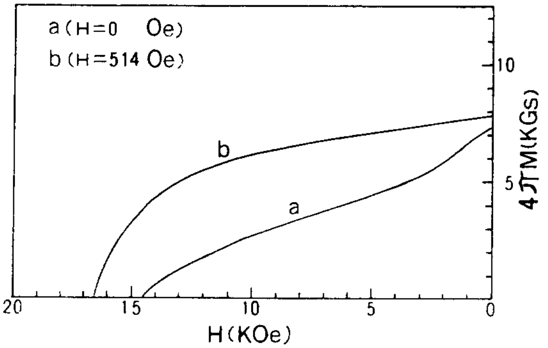

Fig.2 Demagnetization curves of melt-spun Nd-Fe-B powders (a) not divided powder (Br=7.39 kG, iHc=14.3 kOe (BH)max=7.85 MGOe; (b) the powder divided in the field intensity of 514 Oe.

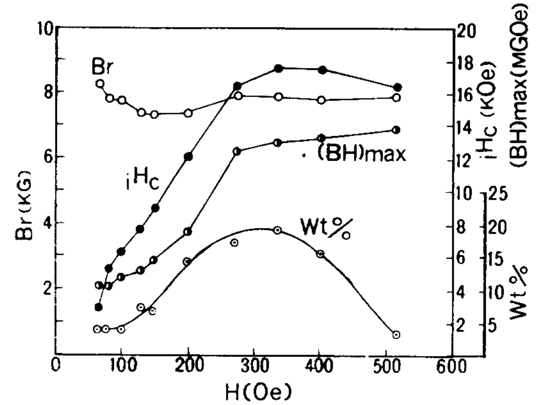

Fig.3 The variation of the magnetic properties and the weight percentage of the melt-spun powder with different separating fields; (the properties before separation: Br=7.38 kG, iHc=14.3 kOe and (BH)max=7.85 MGOe).

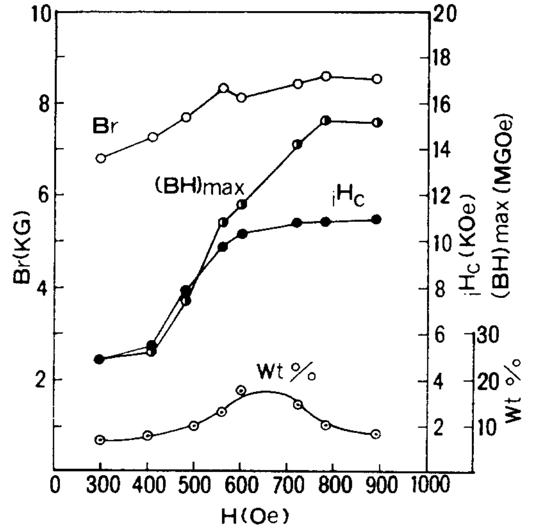

Fig.4 The Variation of magnetic properties and weight percent distribution of the powder extracted from the ununiform melt-spun powder of (BH)max=10.2 MGOe with different separating fields.

Table I The magnetic properties of the two sorts of melt-spun powders before and after separation.

<table>
<tr><th rowspan="2">No.</th><th colspan="4">before separation</th></tr>
<tr><th colspan="2">Br
(kGs)</th><th>iHc
(kOe)</th><th>(BH)max
(MGOe)</th></tr>
<tr><td>1</td><td colspan="2">7.38</td><td>14.3</td><td>7.85</td></tr>
<tr><td>2</td><td colspan="2">7.95</td><td>9.93</td><td>10.2</td></tr>
<tr><th rowspan="2">No.</th><th colspan="4">after separation</th></tr>
<tr><th>separation field H(kOe)</th><th>Br
(kGs)</th><th>iHc
(kOe)</th><th>(BH)max
(MGOe)</th></tr>
<tr><td>1</td><td>0.514</td><td>7.90</td><td>16.3</td><td>13.85</td></tr>
<tr><td>2</td><td>0.780</td><td>7.70</td><td>11.0</td><td>15.30</td></tr>
</table>

in the divided powder. Through ten kinds of magnetic separating fields, it is seen that the weight percentage of the divided powder presents a normal distribution. The peak value of this curve is about 20 wt% at H=320 Oe. in other words, a large amount of the powder with medium magnetic properties can be obtained from the ununiform powder using separating field of 320 Oe. But the peak value and the field intensity in correspondence with the value are different for the ununiform melt-spun powders with various properties. Fig.4 shows the variation of magnetic properties and the weight percent distribution of the powders extracted from the ununiform melt-spun powder of (BH)max=10.2 MGOe with different separating fields. It shows a similar trend to Fig.3, but the higher separating field. Table I illustrates the magnetic properties of the two sorts of melt-spun powders before and after separation under two given separating fields. It can be seen that the different magnetic field intensity should be used in relation to the ununiform powders with different properties in order to extract the powders with higher properties. The magnetic properties of the powder extracted with a separating field of 780 Oe (2) reaches the optimum values of Br=7.7 kG, iHc=11.0 kOe and (BH)max=15.3 MGOe. Table II illustrates the separating results of HDDR Nd-Fe-B magnetic powder with (BH)max=11.6 MGOe. It can be noted that the separating results of the powder has not any regularity. The magnetic properties of HDDR powder are hardly divided by the magnetic separation.

Table II The magnetic properties of HDDR Nd-Fe-B powders after magnetic separation.

magnetic separation field H(kOe)	magnetic properties Br (kGs)	iHc (kOe)	(BH)max (MGOe)
0	7.38	12.1	11.6
0.232	6.98	11.8	10.2
0.270	7.03	12.0	10.6
0.333	7.24	12.1	11.3

IV. CONCLUSIONS

The following conclusions may be drawn from this investigations:

1. Ununiform magnetic melt-spun powders can be separated into various standards with different magnetic performance by using suitable magnetic separation method.
2. The powders extracted from the ununiform melt-spun powders show much higher magnetic properties than those of the original ununiform magnetic melt-spun powders.
3. A high weight percentage of the powders (20-60 wt%) with medium magnetic properties can be extracted from the original ununiform powders which have low magnetic properties.
4. The optimum separating field (for best magnetic properties) depends on the original ununiform melt-spun powders.
5. HDDR powders are hardly separated by magnetic separation.

REFERENCES

[1] J.J. Croat, J.F. Herbst, R.W. Lee and F.E. Pinkerton, J. Appl. Phys. 55, 1984, 2078.

[2] I.R. Harris, 12th Int. Workshop on RE Magnets and their Appl. 1992, 347.

[3] S.X. Zhou, P.Johansson, S.J. Savage and Li-Ya Cui, IEEE Trans. Magn. Mag-26, 1990, 1739.

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Studies of the Rare-Earth Magnets

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Specimens of Nd-Fe-B magnets with various compositions have been prepared and their physical properties investigated. The basic composition of Nd-Fe-B system is $Nd_{15-x}Dy_xFe_{78.5}B_{6.5}$ with x varying between 0 and 2. Addition of Dy improves the coercivty significantly. The bulk density of the finished magnets is ~7.5 g/cc. A remanence of 12.5 kG, and an energy product of 42.1 MGOe have been observed with magnets containing x=1. Thermomagnetic measurements of the alloys showed that the starting alloys exhibited a Curie temperature of ~330 C.

I. INTRODUCTION

Studies for new permanent magnetic materials has been focussed near the composition $Nd_2Fe_{14}B$ which crystallize in the tetragonal structure. In order to enhance our understanding of the magnetic properties of these systems we have examined magnets of the composition $Nd_{15-x}Dy_xFe_{78.5}B_{6.5}$, with x varying between 0 and 2.0

In this paper, we discuss the magnetic properties such as Curie temperature of the starting alloys and the technical magnetic properties such as residual induction, coercivity and energy product of the finished magnets fabricated through the hydrogen decrepitation route. [1].

II. EXPERIMENT

$(NdDy_x)_{15}Fe_{78.5}B_{6.5}$ (X=0, 1.0, 1.5, 2.0) alloys were prepared by induction melting under flowing argon atmosphere. All ingots were wrapped in tantalum foils and were annealed in partial argon atmosphere at temperatures ranging form 900℃ to 1050℃ for 30 days and then quenched in water.

The specimens were hydrided in a reactor. The hydrided powder was further ball milled under argon atmosphere to obtain a much finer powder. The average particle size of the powder thus obtained ranged from 1 to 6 micrometers[2-7].

X-ray diffraction of the powder with Cr-Kα radiation was used to determine the phase purity and lattice parameters. The lattice parameters were utilized to calculate the volume of the unit cell.

Thermomagnetic analyses were performed from room temperature up to approximately 1000 K to determine the Curie temperatures of the magnetic phases and the relative amount of major and minor phases present. Magnetic moments were measured on loose powders, utilizing a vibrating sample magnetometer at room temperature.

The procedure for magnet fabrication is shown in Fig.1.

III. RESULTS AND DISCUSSION

1. X-ray diffraction

Fig.2 shows the x-ray diffraction pattern of one of the specimens, labeled C; other specimens exhibited similar patterns.

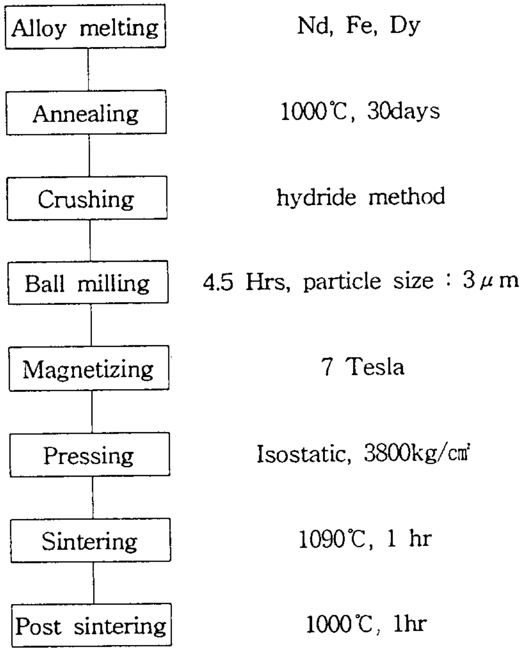

Fig.1 Flow diagram or in the processing technique

Lattice constants measured were a=8.805Å, c=12.205Å. Intensities of the (410), (411), (314), (214) lines were larger when the patterns were obtained on the slides exposed to magnetic fields, thus indicating that the easy direction of anisotropy of the specimen was along the c-axis.

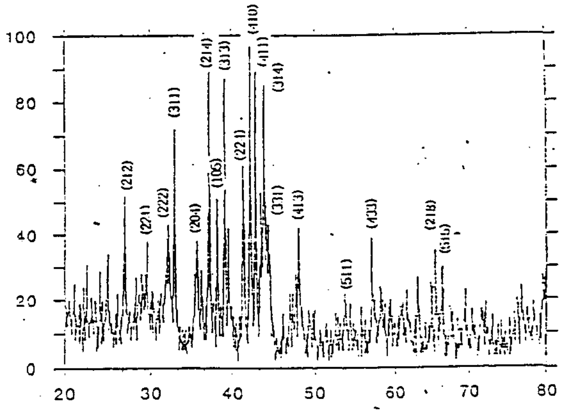

Fig.2 X-ray diffraction pattern of specimen C

2. Magnetic properties

We measured bulk density, Curie temperature, magnetic moment, residual induction, coercive force and energy product for the alloys and their magnets [1],[8].

Results of the magnetic properties obtained at 295 K are given in Table 1 and can be compared with the corresponding values of the commercially available Nd-Fe-B magnets (Br=13.6 kG, BH_{max}=36.2 MGOe, $_i$Hc=10.9 kOe) [9].

Magnetic properties of the Nd-Fe-B magnets, especially the coercivity, strongly depend on the annealing conditions [10].

We have annealed the starting alloys for 30 days at temperatures ranging from 950℃ to 1050℃. As shown in the Table, the highest intrinsic coercivity iHc=18.2 kOe is obtained for $Nd_{13}Dy_2Fe_{78.5}B_{6.5}$ with annealing temperatures from 950℃ to 1050℃ for 30 days.

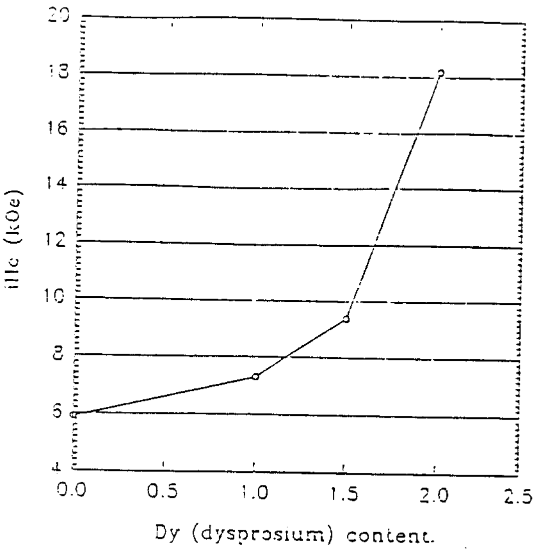

Fig.3 Coercive ferce vs Dy content of $Nd_{15-x}Dy_xFe_{78.5}B_{6.5}$

As shown in the Table, Tc was almost 330℃ and magnetic moment is 130 emu/g (applied field is 17 kOe), Br was 13 kG. Coercive forces range from 5.9 kOe to 18.2 kOe. As Dy contents were increased, coercive forces became larger. These results were shown in Fig.3.

IV. SUMMARY

Permanent magnets based on Nd-Fe-B were fabricated through hydrogen decrepitation route. Results indicate that addition of dysprosium, in combination with appropriate fabrication procedures, produce magnets with high coercivities and high energy products. In particular, we observed that the coercive forces were found to be larger as Dy content increased.

At a composition of $Nd_{13}Dy_2Fe_{78.5}B_{6.5}$ iHc was 18.2 kOe. At a composition of $Nd_{14}DyFe_{78.5}B_{6.5}$ $(BH)_{max}$ was 42.1MGOe.

Tc of the starting alloy was about 330℃ and the bulk density of the finished magnet was about 7.50g/㎤.

Table 1. Properties of $(NdDy_x)_{15}Fe_{78.5}B_{6.5}$ rare-earth magnets

specimen	oxygen content in the chamber (ppm)	conditions of hydride crushing	density (g/㎤)	T_c (℃)	M (emu/g)	B_r (kG)	iH_c (kOe)	$(BH)_{max}$ (MGOe)
A	below 30	500 PSI (800℃)	7.35	330	129.21 (17.4kOe)	11.29	5.9	33.43
B	below 30	500 PSI (800℃)	7.53	327	133.58 (17.4kOe)	12.50	7.3	42.07
C	below 30	500 PSI (800℃)	7.57	320	124.07 (17.6kOe)	12.95	9.4	37.68
D	below 30	500 PSI (800℃)	7.44	328	129.92 (17.0kOe)	11.25	18.2	40.42

REFERENCES

[1] S. F. Chang, Y. Xu, S. G. Sankar, W. E. Wallace, 11th International Workshop on Rare-earth Magnets, Pittsburgh, PA. USA, Ed. S.G. Sankar, vol. 2, pp. 400-411 (1990)

[2] Y. C. Yang, X. D. Zhang, L. S. Kong and Q. Pan, Appl. Phys. Lett. vol. 58, p.2042 (1991)

[3] Y. C. Yang, X. D. Xhang, S. L. Ge, Q. Pan, L. S. Kong, H. L. J. Yang, B. S. Zhang, Y. F. Ding, and C. T. Ye, J. Appl. Phys., vol. 70 p.6001 (1991)

[4] Y. Z. Wang and G. C. Hadjipanayis, J. Appl. Phys., vol. 70, p.6009 (1991)

[5] M. Anagostou, C. Christides, M. Pissas, and D. Niarchos, J. menu Appl. Phys., vol. 70, p.6012 (1991)

[6] M. Endoh, K. Nakamura, and H. Mikami, Digest of INTERMAG'92 Conf., DC-01, (1992. 4. St. Louis)

[7] Wei Gong and G. C. Hadjipanayis, Digest of INTERMAG'92 Conf., DC-02, (1992. 4. St. Louis)

[8] T. Sgawa, S. Fujimura, M. Yamamoto, Y. Matsura, K. Hiraka, IEEE Trans. Mag., MAG-20. p.1584 (1984)

[9] P. Pourarian, S. Sinizu, R. T. Obernyer, S. G. Sankar and W. E. Wallace, 11th International Workshop on Rare-earth Magnets, Pittsburgh. PA. USA, Ed. S.G. Sankar, vol. 1, pp.401-407 (1990)

[10] N. Tang, Y. Liu, J. F. Hu, X. Y. Yin, X. R. Xu, H. Zhao, G. L. YaO, 11th International Workshop on Rare-earth Magnets, Pittsburgh. PA. USA, Ed. S.G. Sankar, vol. 1, pp.408-417 (1990).