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Study on Nd-Fe-B Alloy Ingot Preparation and Its Microstructure

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The thick cast ingot, the thin cast ingot and the strip cast ingot were prepared separately with vacuum medium frequency induction melting furnace and band preparing furnace. The samples of Nd-Fe-B ingots were characterized by scanning electron microscopy and the features of organizational structure of type of ingot were also analyzed. The results show that, in thin sheet alloy ingot, grains are smaller and evenly distributed dispersion and its shape is columnar and Nd-rich phase is very thin. And there is no α-Fe segregation in the strip cast and so it is suitable for the manufacture of high performance sintered Nd-Fe-B magnets.

Keywords: Alloy ingots, Type of ingot, Nd-Fe-B, Organizational structure.

INTRODUCTION

Preparation of alloy ingots is the first part of the production process of sintering Nd-Fe-B magnet. Optimizing the organization of alloy ingots is one of the critical technologies of producing high-performance sintering Nd-Fe-B magnets. The quality of alloy ingots has a direct effect on the following powder preparation, magnetic tropism plus profiling and the sintering process of material base, it finally affects the organizational structure and magnetic property of Nd-Fe-B magnet, for example, it could result in the nonuniformity of magnetic microscopic constitution, big crystal grains and bad consistency in magnetic property[1-8]. From the perspective of making high-performance sintering Nd-Fe-B magnets, we hope that there isn't any bulky α-Fe dendrite in the ingot structure. α-Fe dendrite has a preferable plasticity, making the crushing performance of the cast ingot worse, making the powder-producing process difficult and resulting a longer sintering time to get an well-proportioned $Nd_2Fe_{14}B$. Although part or most of α-Fe dendrite in the cast ingot can be eliminated by homogenizing annealing, annealing will result in reunite and uneven distribution of Nd-rich phase. Agglomeration of Nd-rich phase in the cast ingot will affect the homogeneous distribution of it when sintering. The ideal microscopic constitution of the cast ingot should be without α-Fe and the crystal grain of the principal phase of $Nd_2Fe_{14}B$ is fine and Nd-rich phase is well-distributed[9-12].

By properly improving the structure of the water-cooling type matrix of the cast ingot and applying band preparing furnace to prepare flakey Nd-Fe-B alloy cast sheet, we analyze the effect of the tissue signature of different types of Nd-Fe-B alloy cast ingot and control of technological parameter in the preparing process of the cast ingot on the microscopic constitution of the cast ingot.

EXPERIMENTAL

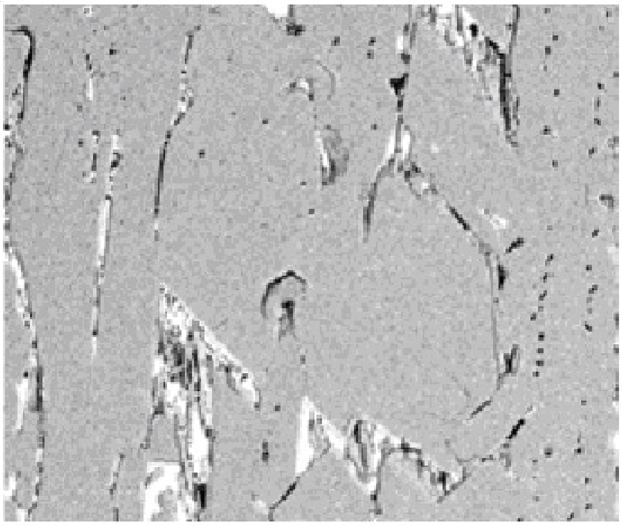
Most of the companies which make sinter Nd-Fe-B magnets in China apply vacuum medium frequency induction melting furnace to melt alloy and prepare alloy cast ingot with various thickness under the protection of the argon atmosphere. The dimension of slab Nd-Fe-B alloy cast ingot is 320 mm × 260 mm × 35 mm; while the dimension of lamellar Nd-Fe-B alloy cast ingot is 350 mm × 250 mm × 18 mm. The direction of the thickness of the cast ingot is the cooling direction after investment liquid pouring and it is simultaneous double-side cooling. The flake is the Nd-Fe-B alloy cast ingot prepared by the application of band preparing furnace. The thickness of flakey Nd-Fe-B alloy cast ingot ranges from 0.2 to 1 mm, usually about 0.3 mm. After the alloy cast ingot experiences hydrogen decrepitation and the crushing process, we use the jet mill to prepare the alloy powder with an average granularity of 4 μm. The alloy powder orientated in a magnetic field equal to or more than 2T, we use vertical die-stamping and isostatic cool pressing to take shape. The gas cools after being sintered by 4 h in the temperature of 1273-1373 K in the green compact with the vacuum conditions of less than 10^{-2} Pa. Then we adopt the drawing temperature for 3 to 5 h under the temperature of 1073-1173 and 773-873 K.

For slab and lamellar Nd-Fe-B alloy cast ingots, we just cut a part of the ingots to prepare the metallographical samples.

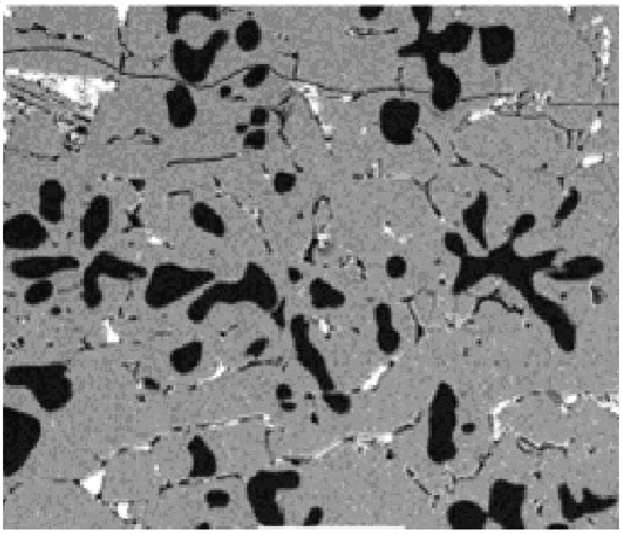
For flakey Nd-Fe-B alloy cast ingot, after picking parts of the flakes to embed, we prepare the metallographical sample. We apply scanning electron microscopy (SEM) to observe it organizational structure.

RESULTS AND DISCUSSION

Organizational structure of slab cast ingot: Fig. 1 is the SEM backscattered electron image of the nearby place (a) and center zone (b) of the surface of slab Nd-Fe-B alloy cast ingot. Fig. 1 (a) showed that in the nearby place of the surface of slab Nd-Fe-B alloy cast ingot, as the cooling speed is relatively fast, although no black α-Fe phase exists, white bulky lumpy Nd-rich phase exist. $Nd_2Fe_{14}B$ columnar crystals are bulky. From Fig. 1 (b), we observe that in the center zone of slab Nd-Fe-B alloy cast ingot, as the cooling speed is the smallest, not only many black α-Fe phase but also white bulky lumpy Nd-rich phase exist. $Nd_2Fe_{14}B$ columnar crystals are relatively bulky. The microscopic structure of slab Nd-Fe-B alloy cast ingot is quite different from that of ideal cast ingot.



(a)

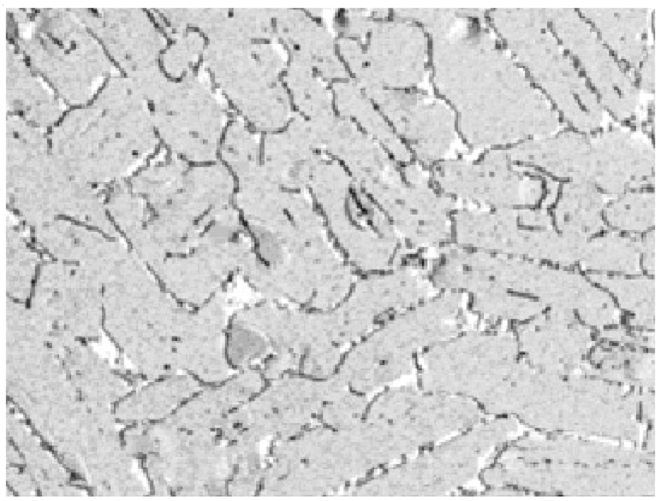


(b)

Fig. 1. SEM images of the slab Nd-Fe-B alloy cast ingot samples: (a) nearby place of the surface, (b) center zone of the surface

Organizational structure of lamellar cast ingot: Fig. 2 is the SEM backscattered electron image of the nearby place (a) and center zone (b) of the surface of lamellar Nd-Fe-B alloy cast ingot.

(a)

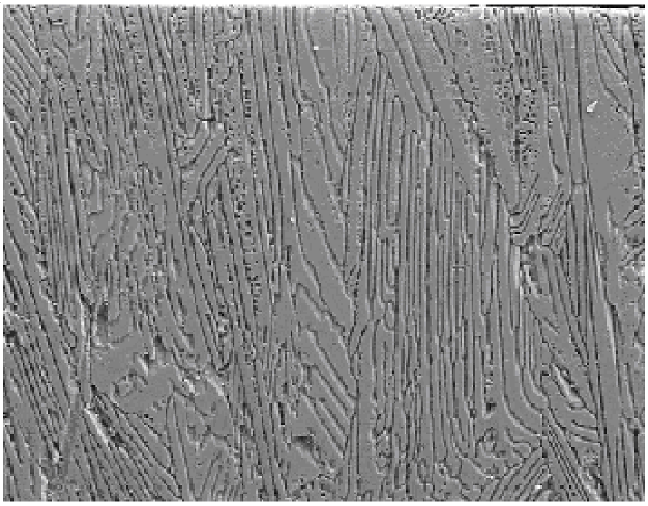


(b)

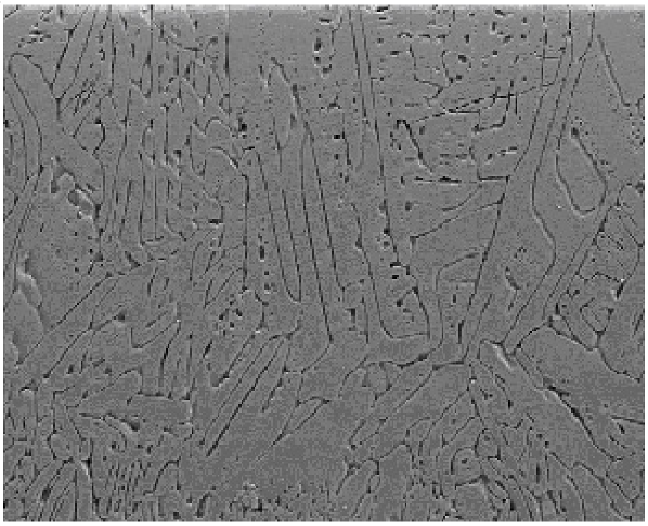
Fig. 2. SEM images of the lamellar Nd-Fe-B alloy cast ingot samples: (a) nearby place of the surface, (b) center zone of the surface

Fig. 2 (a) showed that in the nearby place of the surface of lamellar Nd-Fe-B alloy cast ingot, as the cooling speed is fast, no black α-Fe phase and white bulky lumpy Nd-rich phase exist. $Nd_2Fe_{14}B$ columnar crystals become obviously thinner. From Fig. 2 (b), we observe that in the center zone of the lamellar Nd-Fe-B alloy cast ingot, although the cooling speed is relatively small, no α-Fe is observed to exist. On comparison, it is inferred that $Nd_2Fe_{14}B$ columnar crystals are comparatively bulky, few white bulky lumpy Nd-rich phase exist. In Fig. 2 (a) and (b), Nd-rich phase is comparatively well-distributed. The microscopic structure of lamellar Nd-Fe-B alloy cast ingot is a little similar to the ideal structure of the cast ingot. In cooperation with the improvement of the technologies of the following production link, we can produce high-performance sinter Nd-Fe-B magnets.

Organizational structure of the flake: Fig. 3 shows the SEM backscattered electron image of the flakey Nd-Fe-B alloy plate with the thickness of 0.25 mm (a) and 0.5 m (b).



(a)



(b)

Fig. 3. SEM images of the flakey Nd-Fe-B alloy plate samples: (a) 0.25 mm, (b) 0.5 mm

For the flakey Nd-Fe-B alloy plate with the thickness of 0.25 mm, apart from few $Nd_2Fe_{14}B$ columnar crystals with a thickness of over 10 µm, the thickness of most $Nd_2Fe_{14}B$ columnar crystals is between 3 and 5 µm. The linear zone between $Nd_2Fe_{14}B$ columnar crystals is formed as Nd-rich phase fall off in the metallographical sample-preparing process by erosion, there isn't any α-Fe phase in it. Applying hydrogen decrepitation (HD) technology can certainly get high-quality alloy powder, providing a favorable precondition for the making of high-performance sinter Nd-Fe-B magnets. For flakey Nd-Fe-B alloy plate with the width of 0.5 mm, its columnar crystals are a bit larger with a thickness of about 8 µm.

Performance of sinter Nd-Fe-B magnets prepared by different types of cast ingots: The test results of the magnetic property of the sinter Nd-Fe-B magnets prepared by lamellar and slab Nd-Fe-B alloy cast ingot are separately listed in Tables 1 and 2. By comparing and analyzing Tables 1 and 2, we know that applying the same alloy punch and process control, the sinter Nd-Fe-B magnet prepared by lamellar Nd-Fe-B alloy cast ingot has a much higher remanence Br, coercivity magnetic induction H_{cb}, intrinsic coercive field H_{ci}. Its J-H demagnetization curve also has a bigger square degree, so that it can get a relatively high maximum magnetic energy product $(BH)_{max}$.

TABLE-1
MAGNETIC PROPERTY OF THE SINTER Nd-Fe-B MAGNETS PREPARED BY LAMELLAR Nd-Fe-B ALLOY CAST INGOT

Sample No.	Br	Hcb	Hci	$(BH)_{max}$	Hk
	KGs	KOe	KOe	MGOe	KOe
1	12.52	11.27	13.53	37.25	11.89
2	12.47	11.41	13.41	36.28	12.44
3	12.40	11.12	12.43	36.02	11.19
4	12.48	11.10	12.54	36.66	11.26

TABLE-2
MAGNETIC PROPERTY OF THE SINTER Nd-Fe-B MAGNETS PREPARED BY SLAB Nd-Fe-B ALLOY CAST INGOT

Sample No.	Br	Hcb	Hci	$(BH)_{max}$	Hk
	KGs	KOe	KOe	MGOe	KOe
1	12.41	10.25	11.99	31.38	9.37
2	12.42	10.41	11.58	31.29	8.67
3	12.20	10.26	11.47	29.64	9.00
4	12.37	10.29	11.36	29.88	9.33

Conclusion

• In the slab Nd-Fe-B alloy cast ingot, there is a great deal of α-Fe phase and white bulky lumpy Nd-rich phase. The crassitude of $Nd_2Fe_{14}B$ columnar crystals goes against the making of high-performance sinter Nd-Fe-B magnets.

• In the lamellar Nd-Fe-B alloy cast ingot, $Nd_2Fe_{14}B$ columnar crystals are relatively fine, there are little lumpy Nd-rich phase and no or quite little α-Fe. The microscopic constitution of lamellar Nd-Fe-B alloy cast ingot is comparatively similar to ideal that of the cast ingot. In cooperation with the improvement of the following technologies of production, it is possible to produce high-performance sinter Nd-Fe-B magnets.

• In the flakey Nd-Fe-B alloy cast piece, most of the $Nd_2Fe_{14}B$ columnar crystals are quite fine, Nd-rich phase are pretty well-distributed. There isn't any α-Fe phase. Applying Hydrogen decrepitation Technology (HD) can certainly produce high-quality alloy powder, making favorable conditions for the making of high-performance sinter Nd-Fe-B magnets.

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