Research on Magnetic Property of Nd2Fe14B/α-Fe Nanocomposite Under Different Roller Speeds

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Abstract: Nano-composite permanent magnetic material is a new type permanent magnetic material, and it is the synthesis of soft and hard magnetic phase within nanoscale. On the basis of exchange coupling hard magnetizing theory, nano-composite permanent magnetic material can at the same time have high residual magnetization intensity of soft magnetic phase and high coercivity of hard magnetic phase, and is expected to develop into new generation high performance permanent magnetic material. Nevertheless, magnetic energy product of nano-composite permanent deriving from experiment differs greatly from theoretic value, and this is mainly due to fairly great difference between micro-structure of material and theoretical model. In this paper, the constituent is taken as (Nd, Pr, Dy)2(Fe, Nb)14B/α-Fe, the fusant rapid quenching method is adopted to research the impact of different roller speeds to magnetic property, through result of VSM, XRD and SPM, magnetic property, phase composition and micro structure of alloy are analyzed.

Keywords: Fusant quick quenching, magnetic property, nano-composite permanent magnetic material.

1. INTRODUCTION

Nd2Fe14B/α-Fe nano-composite permanent magnetic material has good magnetic property and very great development potential, and thus draws wide attention. Nevertheless, during long term research it is found that, this material also has some disadvantages¹, and partial magnetic property can be improved via mixing method. In this paper, Nd2Fe14B/α-Fe nano-composite material is adopted as the base to add the elements to optimize magnetic property. For selection of substitute element, Pr2Fe14B and Dy2Fe14B both have anisotropy field higher than Nd2Fe14B [1], and addition of Nb can refine the crystal grain and enhance exchange coupling effect. Nd is substituted by Pr and Dy to improve anisotropy field of composite magnet, small quantity of Nb substitutes Fe to refine the crystal grain structure, enhance exchange coupling effect [2] of hard magnetic phase Nd2Fe14B and soft magnetic phase α-Fe.

Due to above consideration, in this paper the constituent of set sample is (Nd, Pr, Dy)2(Fe, Nb)14B/α-Fe. Furthermore, according to documentation [3], content of α-Fe is designed as 25.9%, and detailed mix ratio is shown in Table 1 below.

2. EXPERIMENT

At first the raw material is prepared by 100 g via designed proportion, and then it is placed into water-cooling copper crucible of electric arc furnace. Prior to smelting, it is vacuumized to below 10 Pa via mechanical pump, and then the ingot casting is broken into 10-20 g small pieces, and placed into high temperature resistance quartz tube with 0.5 mm diameter small hole at lower end, the quartz tube is placed in the vacuum quick quenching chamber, it is vacuumized to nearly 4.5×10⁻⁴ Pa via molecular pump and mechanical pump. The sample is heated and molten by vortex current, the melting temperature is about 1300, the molten sample is sprayed to surface of cooling roller via argon pressure of 0.5-1.2 atm, and speed of cooling roller is set as 10-45 m/s. The thickness of thin strip is about 20-40 µm. For sample made at high quick quenching speed, since it is amorphous structure, amorphous phase inside crystallization shall be of appropriate heat treatment, so that (Nd, Pr, Dy)2(Fe, Nb)14B/α-Fe nano-composite structure shall be formed.

The sample thin strip is placed in the sample tray made from thin iron piece, and then placed in the tube furnace with...
pass-through of argon current, to conduct crystallization treatment to sample via controlling temperature rise rate, heating temperature and heating time. After crystallization, the sample is taken away from heating furnace, cooling can be speeded up by water cooling, to derive the series thin strip sample of ours.

3. PHASE STRUCTURE ANALYSIS

Fig. (1) is XRD spectrum of sample with 25.9% α-Fe constituent at different roller speeds, and it shows that at any speed, existence of α-Fe phase and Nd2Fe14B phase can be observed, and this presents that thin strip is double phase composite material. With increasing of roller speed, thin strip sample is gradually changed to amorphous structure from crystal structure. At 10 m/s, the sample has very strong crystal characteristic, diffraction peak is sharp, according to Scherrer Equation [4], crystal size at this status is fairly great. The strength of (004) and (008) diffraction peak is fairly great, and this means that at inside of sample, the hard magnetic RE2Fe14B phase grows along the direction parallel with thin strip face, and the easy magnetization axis is vertical with thin strip face. With increase of roller speed, strength of (004) and (008) diffraction peak is gradually decreased, strength of (214) and (410) diffraction peak is increased, other 2:14:1 phase characteristic peak occurs at circumference of (214) diffraction peak, and this means that crystal grain of 2:14:1 phase grows by approaching to the direction parallel with thin strip face, easy magnetization axis is changed to the direction parallel with thin strip face. At 30 m/s, diffraction peak is widened, increase of half width means that crystal grain size inside the material is decreased, exchange coupling effect is increased, furthermore there are many diffraction peaks, this means that internal texture is enhanced in favor of improving magnetic property. With increase of quick quenching speed, amorphous package starts to occur, and the sample enters into over-quick quenching status.

Research of Zhang [5], etc deems that, at fairly low quick quenching speed, easy magnetization axis of thin strip is gradually changed to direction parallel with thin strip plane. Because: on one hand, when the alloy of molten status is sprayed on the roller face, since there is a great difference of temperature for roller-fit face and free face, inside of thin strip shall incur temperature gradient vertical with roller face, i.e., the heat current direction is vertical with roller face. According to the theory on the basis of temperature gradient, quick quenching thin strip grows on the moving roller face, due to tensile stress along roller tangential direction, crystal grain has slippage, and is rotated by anisotropy of strain energy. At joint effect of tensile stress and strain energy easy magnetization axis of RE2Fe14B can be easily distributed at the direction vertical with thin strip. On the other hand, coring and growth of double phase nano-composite permanent magnetic material rely on diffusion extent of atom, during quick quenching process with fairly high roller speed, coring and growth of RE2Fe14B phase is fairly difficult, nevertheless crystallization temperature of α-Fe phase is fairly low, and can be firstly separated out during this process to form (110) texture. Thus α-Fe can provide crystallization core during growth process of RE2Fe14B phase, so that crystal face of RE2Fe14B crystal grain can have the priority of growth at induction of (110) texture of α-Fe, and easy magnetization direction of crystal grain of RE2Fe14B phase is parallel with thin strip plane, and built-up magnet has anisotropy.

4. SPM ANALYSIS

Fig. (2) is atomic force microphotograph (AFM) at quick quenching speed of 20 m/s, 25 m/s, scan range is 2 µm × 2 µm. It shows that, the crystal grain size belongs to nanometer range and is all less than 100 nm. With increase of roller speed, mostly of them are 60~90 nm, and there is a great difference of size among crystal grains, distribution evenness thereof is fairly bad; at 25 m/s, crystal grain distribution is at 40~60 nm and is relatively concentrated, magnetic property is greatly improved. According to existing theory, fine size and even distribution of crystal grain is in favor of improving exchange coupling effect, fairly strong exchange coupling effect can enable the magnetic moment of crystal grain rotate toward the same direction to improve residual magnetism of nano-composite magnet; furthermore, fine and even crystal grains are of comprehensively coupling in favor of improving effective anisotropy of soft magnetic phase of double phase composite material, so as to improve coercivity of magnet. Magnetic energy product of magnet is determined by product of residual magnetism and coercivity, within

![Fig. (1). XRD spectrum of sample of 25.9% α-Fe content at different roller speeds.](image)
certain range, magnetic property of quick quenching thin strip shall be increased with increasing of rollerspeed.

Fig. (2). Comparison (2 μm × 2 μm) on AFM chart of sample of 25.9% α-Fe content at quick quenching speed of 20 m/s (left) and 25 m/s (right).

5. COMPARISON ON MAGNETIC PROPERTY

This part researches magnetic property of sample of 25.9% α-Fe content at different quick quenching speeds. External field applied by VSM is 1T.

Table 2 shows that, for nano-composite material with Nd4.2Pr3.6Dy0.7Fe85.5Nb1B5 constituent, the magnetic property related parameter is greatly impacted by quick quenching speed. At 10 m/s, thin strip is at crystallization status, according to XRD analysis, thick crystal grain size is not in favor of exchange coupling effect, magnetic property is not good, each magnetic property parameter is not high. With increasing of quick quenching speed, XRD chart presents that diffraction peak is widened, quantity is increased, internal texture is enhanced. Furthermore SPM chart presents that, crystal grain size gradually becomes small, exchange coupling effect is enhanced with occurrence of heteromorphism at one domain. At 30 m/s the magnetic property is optimal: Hcj=538.33 kA/m, Br=0.68 T, Mr/Ms = 0.73, (BH) max = 53.44 kJ/m3. After quick quenching speed is greater than 30 m/s, sample thin strip is at over-quick quenching status, XRD analysis shows that, thin strip has amorphous phase incurring decrease of magnetic property. Coercivity, remanence ratio, magnetic energy product reach max. value at 30 m/s, residual magnetism reaches max. value at 25 m/s, and is 0.7 T.

Table 2. Magnetic property parameters of thin strip at different quick quenching speeds.

<table>
<thead>
<tr>
<th>v (m/s)</th>
<th>Hcj (kA/m)</th>
<th>Br (T)</th>
<th>Mr/Ms</th>
<th>(BH) Max (kJ/m3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>141.6</td>
<td>0.47</td>
<td>0.51</td>
<td>10.53</td>
</tr>
<tr>
<td>15</td>
<td>177.7</td>
<td>0.49</td>
<td>0.56</td>
<td>14.9</td>
</tr>
<tr>
<td>20</td>
<td>206.5</td>
<td>0.56</td>
<td>0.58</td>
<td>16.64</td>
</tr>
<tr>
<td>25</td>
<td>409.7</td>
<td>0.7</td>
<td>0.69</td>
<td>41.05</td>
</tr>
<tr>
<td>30</td>
<td>538.3</td>
<td>0.68</td>
<td>0.73</td>
<td>53.44</td>
</tr>
<tr>
<td>35</td>
<td>517</td>
<td>0.45</td>
<td>0.73</td>
<td>27.19</td>
</tr>
<tr>
<td>45</td>
<td>488.4</td>
<td>0.43</td>
<td>0.71</td>
<td>13.67</td>
</tr>
</tbody>
</table>

Magnetic property has fairly great change with variation of quick quenching speed, because cooling speed of thin strip is different at setting process. When quick quenching speed is fairly low, cooling speed is fairly low at setting process of thin strip, super-cooling degree of fusant is relatively small, coring rate is fairly low, thus crystal grain size is fairly great incurring uneven distribution of crystal grain, during which time alloy thin strip is fairly thick. When quick quenching speed is 30 m/s, mean thickness of thin strip is fairly small and crystal grain is small, because the cooling speed and variation range along thickness direction is moderate, degree of super-cooling is increased in some extent, liquid alloy has very high coring rate and can just ensure setting of while thickness, so that the alloy can have a large number of even coring, and the growth thereof shall not go too far, so that the crystal grain size is small and texture is of even distribution. It is approaching to microstructure of one-dimensional theoretical model by assuming that maximum exchange coupling effect is acquired, so as to prove fairly good exchange coupling effect between soft and hard magnetic phase. When quick quenching speed is too high, super-cooling fusant temperature is very low, according to coring theory diffusion rate of atom in fusant and related energy fluctuation and concentration fluctuation shall apparently decrease, furthermore, with increase of setting cooling speed, coring time shall also be shortened, and these factors shall counteract the impact of increase of drive of thermodynamics and coring rate shall apparently decrease. When coring rate is approaching to 0, atom configuration in the fusant shall basically be invariable, i.e., during setting process it is “frozen” to form the long range and unordered amorphous status, and suppress the formation of crystal status.

According to traditional Stoner-Wohlfarth ferromagnetic theory model [7], theoretical maximum value of remanence ratio of isotropy permanent magnet of single easy axis is only 0.5 Ms. In our nano-composite permanent magnetic material, remanence ratio is Mr / Ms > 0.5, i.e., there is a residual magnetism enhancement effect. Because crystal grain size of hard magnetic phase and soft magnetic phase is in the range of nanometer, in case of effect of external magnetic field, at the magnetic exchange coupling effect, soft magnetic phase magnetic moment shall be changed to the average direction parallel with hard magnetic phase magnetic moment, i.e., there is a strong ferromagnetic exchange coupling effect between two phases. Therefore magnetization and back magnetization of the permanent magnet has single hard magnetic phase characteristic. The result shows that, there is a strong exchange coupling effect between hard magnetic crystal grain size and soft magnetic crystal grain size in the prepared sample.

6. BRIEF SUMMARY

Magnetic property of sample of constituent of Nd4.2Pr3.6Dy0.7Fe85.5Nb1B5 at different roller speeds is mainly analyzed and researched, and the result presents that:

1) XRD spectrum of thin strip at different quick quenching speeds is analyzed, phase constituent of composite material is determined subject to variation of each diffraction peak, to conclude the microstructure. At 30 m/s, there are many diffraction peaks with fairly great half peak width, it presents
that internal texture of magnet is fairly strong, crystal grain size is fairly small, and exchange coupling effect is fairly strong.

(2) AFM image of thin strip of 20 m/s, 25 m/s, 30 m/s is analyzed. Within scan range of 2 µm × 2 µm, crystal grain size gradually becomes small and even with increasing of quick quenching speed. The fine and even crystal grain size is in favor of enhancement of exchange coupling effect. MFM chart of 30 m/s shows that, there is a heteromorphism of one domain due to fairly strong exchange coupling effect.

(3) When 1T external magnetic field is applied, magnetic property of thin strip is increased with increasing of quick quenching speed, it is maximum at 30 m/s and then gradually decreased. The optimal magnetic property is: \( H_{cj} = 538.33 \) kA/m, \( B_r = 0.68 \) T, \( M_r/M_s = 0.73 \), \( (BH)_{max} = 53.44 \) kJ/m³.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflict of interest.

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