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MICROSTRUCTURE AND COERCIVITY IN (Nd, Dy)-(Fe, Co)-B BASED SINTERED PERMANENT MAGNETS WITH MINOR Nb AND Ga ADDITIONS


Abstract. — The microstructure of both Nb- and (Nb, Ga)-containing (Nd, Dy)-(Fe, Co)-B based magnets was examined. A new phase (Fe, Co)NbB, having CoNbB-type orthorhombic structure, was found. Dislocations were visible in the vicinity of the Fe₂Nb precipitates in the (Nb, Ga)-containing magnet. The pinning of domain walls through Cottrell-atmosphere around the dislocations and the Ga-improved wettability of Nd-rich phase with respect to the matrix, as possible mechanisms, account for greater increase of coercivity in (Nb, Ga)-containing magnet than in that containing Nb alone.

1. Introduction

The application of magnets based on the hard magnetic phase Nd₂Fe₁₄B has been limited by the low Curie temperature and the high irreversible thermal loss. It is well known that an increase in coercivity is produced when Dy and Nb are added to the alloy. The microstructure of Nb-containing (Nd, Dy)-Fe-B sintered magnet has been investigated by Parker et al. [1]. A new magnet with the composition (Nd₀.₈Dy₀.₂) (Fe₀.₈₃₅Co₀.₀₆B₀.₀₆Nb₀.₀₁₅Ga₀.₀₁)₅.₅ has been reported by Tokunaga et al. [2]. It was found that the combined additions of Nb and Ga effectively increased intrinsic coercivity Hₑ, and made the irreversible thermal loss under 5 % after exposure at 520 K. The outstanding thermal stability of the magnet aroused our interest in studying the relationship between microstructure and coercivity in (Nd, Dy)-(Fe, Co)-B based magnets with minor Nb and Ga additions.

2. Experimental procedure

Two types of (Nd, Dy)-(Fe, Co)-B based magnet (Nd₀.₉Dy₀.₁) (Fe₀.₇₅₅Co₀.₀₆B₀.₀₆Nb₀.₀₁₅Ga₀.₀₁)₅.₅ and (Nd₀.₉Dy₀.₁) (Fe₀.₇₇₇₅Co₀.₁₂₃₅₇₉₈B₀.₆₈)₅.₅ designated as sample 1 and 2, respectively, have been prepared by the conventional powder metallurgy method. Permanent magnetic properties were measured with a D.C. hysteresismeter. X-ray diffraction analysis of sintered magnet powder was carried out using Cu-Kα radiation with a graphite crystal monochromater. For TEM investigation, thin slices perpendicular to the alignment direction (A.D.) were cut from the demagnetized magnets. After ion thinning, they were examined in the Philips EM420 electron microscope equipped with EDAX. Optical microscopic observations and EPMA examinations of demagnetized magnets were performed on the polished surface perpendicular to the A.D.

3. Results and discussion

The results of magnetic measurements for the samples 1 and 2 are shown in figure 1. It is apparent that the coercivity of (Nb, Ga)-containing magnet is significantly higher than that of the magnet containing Nb merely. Although the results in figure 1 are not optimum magnetic properties, they give a reasonable comparison of the effects of Nb- and (Nb, Ga)-additions to the (Nd, Dy)-(Fe, Co)-B magnet on the coercivity.

Fig. 1. — Demagnetization curves of magnet 1 and magnet 2.

The results of optical microscopic and X-ray diffraction examinations are somewhat similar for sample 1 and 2. Typical microstructure and X-ray diffraction diagram for sample 1 are illustrated in figures 2 and 3 respectively. Phase A is a hard magnetic phase with R₂Fe₁₄B tetragonal structure. In most grains of this phase, fine precipitated particles with a diameter of 300-700 Å are found. The number of particles is about 5 x 10²⁰ per cubicimeter. TEM observation and EDAX analysis have shown that these particles are richer in Nb than the matrix, as is illustrated in figures 4a and 4b. Phase B is the tetragonal Nd₁₊ₓFe₄B₄ phase, and

Fig. 2. — Optical micrograph showing five phases A, B, C, D, E in sintered magnet 1.
phase C is Nd-rich phase found mainly along grain boundaries of the matrix. Phase D was identified as Fe₂Nb, a Laves phase having a MgZn₂ type structure. These results are similar to those reported in [1].

Table I. – Chemical composition of different phases in magnet 1.

<table>
<thead>
<tr>
<th>symbol</th>
<th>Nd</th>
<th>Dy</th>
<th>Fe</th>
<th>Co</th>
<th>D</th>
<th>Nb</th>
<th>Ga</th>
<th>phase identified</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>10.03</td>
<td>1.52</td>
<td>73.59</td>
<td>4.13</td>
<td>4.87</td>
<td>0.48</td>
<td>0.99</td>
<td>Fe₂(Nb,X)B</td>
</tr>
<tr>
<td>B</td>
<td>11.09</td>
<td>0.21</td>
<td>33.76</td>
<td>8.84</td>
<td>37.00</td>
<td>4.45</td>
<td>2.82</td>
<td>Fe₁₋₀.₉₃D₄⁺</td>
</tr>
<tr>
<td>C</td>
<td>57.32</td>
<td>0.03</td>
<td>3.21</td>
<td>20.09</td>
<td>5.72</td>
<td>1.34</td>
<td>14.05</td>
<td>Nb – rich</td>
</tr>
<tr>
<td>D</td>
<td>2.09</td>
<td>0</td>
<td>57.86</td>
<td>7.45</td>
<td>0</td>
<td>31.76</td>
<td>0.27</td>
<td>Fe₂Nb</td>
</tr>
<tr>
<td>E</td>
<td>0.02</td>
<td>0</td>
<td>25.94</td>
<td>2.82</td>
<td>32.90</td>
<td>34.28</td>
<td>0</td>
<td>(Fe₆, Co)NbB</td>
</tr>
</tbody>
</table>

Dislocations may be easier to form in the (Nb, Ga)-containing magnets than in that containing Nb only. It is well known that dislocation makes a negligible contribution to the coercivity in the high hard magnets [4]. However, dislocation can lead to formation of Cottrell-atmosphere in a doped lattice [5]. As a possible mechanism Cottrell-atmosphere should produce substantial pinning of domain walls.

In addition, the fact that most Ga atoms dissolve in Nd-rich phase supports the assumption that Ga improves the wettability of Nd-rich phase with respect to the matrix. It will decrease the density of defects in the Nd-rich phase and become more effective in inhibiting grain growth during liquid phase sintering. As another possible mechanism, these should also contribute to increase in coercivity. The fact that the average diameter of grain in sample 1 is smaller than that in sample 2 is a strong support to this assumption.