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


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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) (Fe0.835Co0.06B0.08Nb0.015Ga0.01)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 Hc, 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) (Fe0.758Co0.03Nb0.05Ga0.015B0.08)5.5 and (Nd0.8Dy0.2) (Fe0.77Co0.13Nb0.03B0.08)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. hysteresis meter. 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

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 10^20 per cubic meter. 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 Nd1.4+Fe4B4 phase,
phase C is Nd-rich phase found mainly along grain boundaries of the matrix. Phase D was identified as Fe$_2$Nb, a Laves phase having a MgZn$_2$ type structure. These results are similar to those reported in [1].

In contrast to the observations in [1], a new phase E was found in sample 1 and 2. It has been identified as (Fe, Co)NbB with CoNbB type orthorhombic structure. It is possible that the presence of Co promotes the creation of (Fe, Co)NbB. The lattice parameters of phase E are: $a = 5.929$ Å, $b = 17.162$ Å, $c = 3.260$ Å. The chemical compositions of the different phases found in sample 1 are summarized in Table I. It can be seen that most Ga atoms dissolved in the Nd-rich phase which primarily spreads along the grain boundaries of the matrix. It is very interesting to note that dislocations were clearly discernible in the vicinity of Fe$_2$Nb phase in the matrix of the (Nb, Ga)-containing magnet (Fig. 5a). The electron diffraction pattern associated with phase D ([111] zone axis) is also shown in the same figure. Figure 5b shows EDAX analysis of Fe$_2$Nb phase. In contrast, dislocations were not found in sample 2. Table 1 shows there are small amount of Nd in the Fe$_2$Nb phase which will cause great lattice distortion due to the difference in atomic radii between Nd and Nb. On the other hand, trace of Ga decreases the yield stress of Fe-based materials [3]. Therefore, it is reasonable to assume that 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.

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

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<th>symbol</th>
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<th>Fe</th>
<th>Co</th>
<th>Nb</th>
<th>Ga</th>
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