

# High-coercivity nanocomposite permanent magnet based on Nd–Fe–B–Ti–C with Cr addition for high-temperature applications

Rintaro Ishii\*, Toshio Miyoshi, Hirokazu Kanekiyo, Satoshi Hiroswawa

*R&D Division, Neomax Co., Ltd., 2-15-17 Egawa, Shimamoto-cho, Mishima-gun, Osaka-fu 618-0013, Japan*

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## Abstract

We have investigated influences of various elements (Ti, Nb, Mo, Ta, Hf, W, V, Cr) on magnetic properties of Fe–B/Nd<sub>2</sub>Fe<sub>14</sub>B-based Nd–Fe–B–Ti–C nanocomposite magnets in order to obtain larger coercivity required for high-temperature applications. As a result, addition of Cr was found to be most effective among additive elements investigated to enhance coercivity. Thermal flux losses of high-coercivity ( $H_{cJ} = 1609$  kA/m) Nd–Fe–B–Ti–C–Cr nanocomposite magnet at 200 °C are less than 2%.

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## 1. Introduction

Nanocomposite magnets based on Fe<sub>3</sub>B/Nd<sub>2</sub>Fe<sub>14</sub>B in the vicinity of Nd<sub>4.5</sub>Fe<sub>73</sub>Co<sub>2</sub>B<sub>18.5</sub>Cr<sub>2</sub> have large remanence values exceeding 1 T. However, their thermal resistances in terms of thermal flux losses are insufficient to be used in applications such as sensors and motors for automobile devices. This shortcoming of the Fe<sub>3</sub>B/Nd<sub>2</sub>Fe<sub>14</sub>B-based nanocomposite permanent magnets with Nd content around 4–5 at% has been largely improved by alloying Ti and C simultaneously, which led to a new series of Fe–B/Nd<sub>2</sub>Fe<sub>14</sub>B-based nanocomposite magnets with Nd content from 6 to 9 at% and B content from 10 to 15 at% [1,2]. Addition of a few at% of Ti suppresses formation and growth of the  $\alpha$ -Fe and the metastable Nd<sub>2</sub>Fe<sub>23</sub>B<sub>3</sub>, promoting formation of Nd<sub>2</sub>Fe<sub>14</sub>B during the rapid solidification process. As a result of increasing volume fraction of the Nd<sub>2</sub>Fe<sub>14</sub>B phase, high coercivity of  $H_{cJ}$  ranging from 460 to 1100 kA/m and large energy products ranging from 124 to 100 kJ/m<sup>3</sup> have been achieved. The role of a few at% of C has been found to refine the microstructure and increase coercivity through the formation of very fine intergranular precipitates of TiC [3].

In the (Nd<sub>0.95</sub>La<sub>0.05</sub>)<sub>9.5</sub>Fe<sub>78</sub>B<sub>10.5</sub>M<sub>2</sub> system, Ti, Nb, V and Cr suppress the formation of Nd<sub>2</sub>Fe<sub>23</sub>B<sub>3</sub>, and Ti and Cr refines grain sizes [4]. In order to further improve coercivity of the Nd–Fe–B–Ti(–C) system, effects of the addition of refractory metal elements need to be investigated. The only refractory element that has been investigated for the Nd–Fe–B–Ti system is Nb, which was found to increase  $H_{cJ}$  and decrease the dependence of magnetic properties on cooling rate during rapid solidification [5]. However, in the Nd–Fe–B–Ti–C system, effects of additive except for Nb has not been examined in detail. Therefore, we investigated influences of various elements on the magnetic properties of Nd–Fe–B–Ti–C type of magnets in order to obtain higher coercivity and a larger energy product. We report in this paper, the effects of Cr addition on magnetic properties, structural properties and thermal resistance of Fe–B/Nd<sub>2</sub>Fe<sub>14</sub>B-based nanocomposite magnets.

## 2. Experimental procedures

Rapidly solidified ribbons were prepared by the melt spinning technique in an argon atmosphere onto a copper wheel rotating at a surface speed of 5–20 m/s. Elemental metals of purity higher than 99.8% were used. The ribbons were then annealed between 873 and 1073 K for 0.36 ks in an argon atmosphere. Magnetic properties of the heat-treated

\*Corresponding author. Tel.: +81 75 961 3173; fax: +81 75 962 9690.

E-mail address: [ISHIIR@neomax.co.jp](mailto:ISHIIR@neomax.co.jp) (R. Ishii).

ribbons were measured with a vibrating sample magnetometer (VSM). Crystalline phases in the heat-treated ribbons were examined by means of X-ray diffraction (XRD) analysis. Structures of the heat-treated ribbons were examined with a transmission electron microscope (TEM).

Resin-bonded magnets were processed from pulverized powder of the optimally heat-treated ribbons by a compression molding process, using approximately 2 wt% of epoxy resin. The compression pressure was 10.1 MPa and the diameter of the specimen was 10 mm and the height was 7 mm. No coating was applied on the magnets. Magnetic properties of the bonded magnets were measured with a B–H tracer. Temperature dependence of thermal flux losses after exposure to 20–200 °C air for 3.6 ks was measured at room temperature with the extraction method.

### 3. Results

The Cr addition dramatically enhances coercivity of the Fe–B/Nd<sub>2</sub>Fe<sub>14</sub>B-based Nd–Fe–B–Ti–C nanocomposite magnets. In Fig. 1, the remanence (B<sub>r</sub>) of optimally solidified and heat-treated ribbons is plotted against the intrinsic coercivity (H<sub>cJ</sub>). Solid squares in Fig. 1 show magnetic properties of Nd<sub>9</sub>Fe<sub>73</sub>B<sub>12.6</sub>C<sub>1.4</sub>Ti<sub>3</sub>M<sub>1</sub> (M = Ti, Nb, Mo, Ta, Hf, W, V, Cr). All elements in this research, except for Ti, increased H<sub>cJ</sub>. In particular, H<sub>cJ</sub> increased up to 1470 kA/m by addition of Cr.

Compositional adjustment further enhances H<sub>cJ</sub> of Nd–Fe–B–Ti–C–Cr nanocomposite magnets. The open circle in Fig. 1 shows the magnetic property of the ribbon that has the largest H<sub>cJ</sub> value. H<sub>cJ</sub> increased with increasing Nd concentration. In addition, H<sub>cJ</sub> also increased with increasing the total amount of B and C. It was also

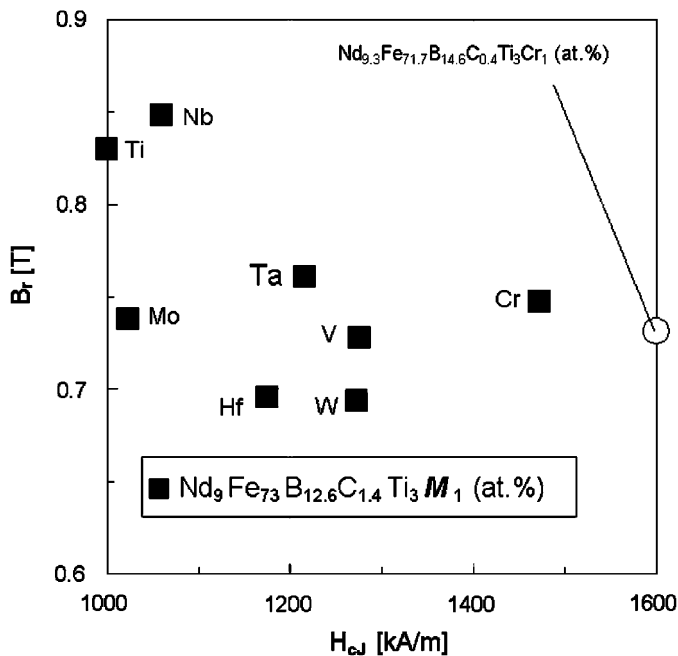


Fig. 1. Magnetic properties of the optimized ribbons of Nd<sub>9</sub>Fe<sub>73</sub>B<sub>12.6</sub>C<sub>1.4</sub>Ti<sub>3</sub>M<sub>1</sub> (solid squares) and Nd<sub>9.3</sub>Fe<sub>71.7</sub>B<sub>14.6</sub>C<sub>0.4</sub>Ti<sub>3</sub>Cr<sub>1</sub> (open circle).

confirmed that decreasing the C/B ratio is effective to suppress the decrease of B<sub>r</sub>.

In Fig. 2, shown by open squares is the relationship between H<sub>cJ</sub> and Cr content. With increasing Cr content, H<sub>cJ</sub> increases. The relationship between Curie temperature (T<sub>c</sub>) of Nd<sub>2</sub>Fe<sub>14</sub>B phase and Cr content is shown by open squares in the figure. T<sub>c</sub> is almost constant regardless of Cr content, which is in contrast with the dependence of T<sub>c</sub> of Nd<sub>2</sub>(Fe–Cr)<sub>14</sub>B in which all Cr atoms are assumed to replace Fe atoms in the Nd<sub>2</sub>Fe<sub>14</sub>B phase [6].

Fig. 3 shows XRD patterns of the Cr-added Nd–Fe–B–Ti–C nanocomposite magnet and the Nb-added one. No definite differences between these two XRD patterns are seen, suggesting that constitution of phases in these specimens is same. Both XRD patterns have a relatively larger peak near 44° compared to that of Nd<sub>2</sub>Fe<sub>14</sub>B. This suggests the existence of Fe<sub>23</sub>B<sub>6</sub> as the Fe-boride phase.

As mentioned above, elaborate adjustment of concentrations of the constituent elements has yielded a series of higher performance nanocomposite magnets based on the Nd–Fe–B–Ti–C–Cr system. Structural properties, magnetic properties and thermal resistance of alloys made of the aforementioned three specimens (Nd<sub>9</sub>Fe<sub>73</sub>B<sub>12.6</sub>C<sub>1.4</sub>Ti<sub>3</sub>Nb<sub>1</sub>, Nd<sub>9</sub>Fe<sub>73</sub>B<sub>12.6</sub>C<sub>1.4</sub>Ti<sub>3</sub>Cr<sub>1</sub> and Nd<sub>9.3</sub>Fe<sub>71.7</sub>B<sub>14.6</sub>C<sub>0.4</sub>Ti<sub>3</sub>Cr<sub>1</sub>) are reported here. Fig. 4 shows the TEM bright images of (a) Nd<sub>9</sub>Fe<sub>73</sub>B<sub>12.6</sub>C<sub>1.4</sub>Ti<sub>3</sub>Nb<sub>1</sub>, (b) Nd<sub>9</sub>Fe<sub>73</sub>B<sub>12.6</sub>C<sub>1.4</sub>Ti<sub>3</sub>Cr<sub>1</sub>, and (c) Nd<sub>9.3</sub>Fe<sub>71.7</sub>B<sub>14.6</sub>C<sub>0.4</sub>Ti<sub>3</sub>Cr<sub>1</sub>. In terms of grain sizes, there is no definite difference between (a) and (b). Average grain sizes of (a) and (b) are around 60 nm. On the other hand, the specimen (c) consists of very fine grains. Average grain size of (c) is around 20 nm. Fig. 5 shows demagnetization curves of pulverized powders of these alloys. Magnetic properties in detail are shown in Table 1. Fig. 6 shows the temperature dependence of thermal flux losses of resin-bonded magnets made of these powders.

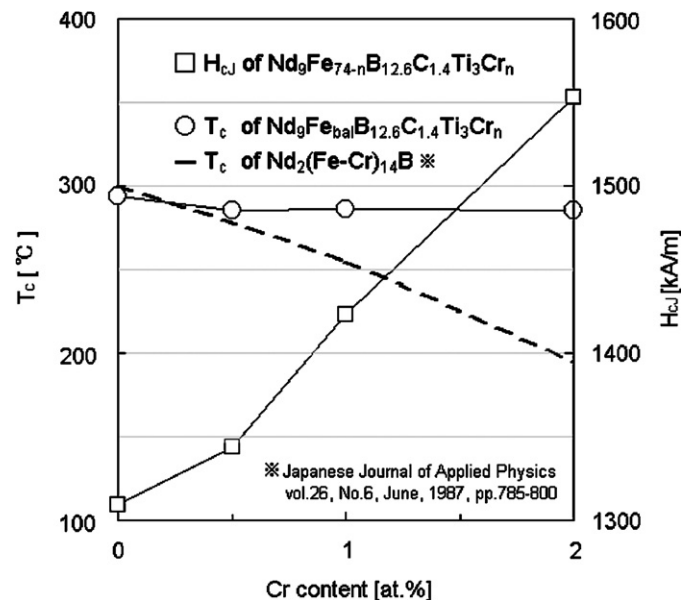


Fig. 2. Relationship among Cr content, T<sub>c</sub> and H<sub>cJ</sub>.

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