High Coercive Force $\alpha$-Fe/Nd$_2$Fe$_{14}$B-type Nanocomposites

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α, were determined by placing a fully magnetized ribbon with a size of about 4 mm x 2.5 mm x 50 μm in a VSM without an applied magnetic field, and cycled from 25-180 °C. Wohlfarth's remanence analysis was employed to determine the strength of exchange-coupled interactions of the materials obtained.

3. Results and discussion

3.1 Effects of Cr and La/Cr substitutions

Nd₀.₅Fe₈₀₅B₁₀, Nd₀.₅Fe₇₅Cr₂B₁₀ and (Nd₀.₉₅La₀.₀₅)₀.₅Fe₇₅Cr₂B₁₀ were selected to study the effect of Cr and La/Cr substitutions in high boron and rare earth lean NdFeB ribbons. Shown in Figs. 1(a), 1(b) and 1(c) are the Thermo Magnetic Analysis (TMA) of three alloy ribbons processed to obtain their highest \( H_r \) and (BH)\(_{\text{max}}\). Four magnetic phases, namely, Nd₂Fe₁₄B, Nd₃Fe₂B₁₃, Fe₂B and α-Fe, were detected in the Nd₀.₅Fe₈₀₅B₁₀ ribbons. With a dilute substitution of Cr for Fe, Nd₀.₅Fe₇₅Cr₂B₁₀, and combined substitutions of Cr for Fe and La for Nd, (Nd₀.₅La₀.₅)₀.₅Fe₇₅Cr₂B₁₀, the Nd₂Fe₁₄B and Fe₂B phases vanished as shown in Figs. 1(b) and 1(c), respectively. A slight decrease in the Tₐ of the 2:14:1 phase from 309 to 291 °C was also noticed suggesting that some of the Cr and La/Cr had entered the crystal structure forming Nd₀.₅Fe₇₅Cr₂B₁₀ and (Nd₀.₅La₀.₅)₀.₅Fe₇₅Cr₂B₁₀ phases, respectively. Because of the suppression of the undesired Nd₂Fe₁₄B and Fe₂B phases, the amount of the 2:14:1 and α-Fe phases increased slightly. This suggests that Cr may form precipitates with B, reduce the effective boron content and, therefore, minimize the formation of Nd₂Fe₁₄B and Fe₂B phases.

Listed in Table I are the Bₐ, \( H_r \), and (BH)\(_{\text{max}}\) of the above three alloy ribbons after optimum treatment. For Nd₀.₅Fe₈₀₅B₁₀, α-Fe, \( H_r \), and (BH)\(_{\text{max}}\) of 8.3 kG, 7.3 kOe and 10.0 MGOe, respectively, were obtained.

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Condition</th>
<th>( R_b ) (kG)</th>
<th>( H_r ) (kOe)</th>
<th>(BH)(_{\text{max}}) (MGOe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nd₀.₅Fe₈₀₅B₁₀</td>
<td>15 min</td>
<td>8.3</td>
<td>7.3</td>
<td>10.0</td>
</tr>
<tr>
<td>Nd₀.₅Fe₇₅Cr₂B₁₀</td>
<td>10 min</td>
<td>8.9</td>
<td>5.4</td>
<td>12.0</td>
</tr>
<tr>
<td>(Nd₀.₅La₀.₅)₀.₅Fe₇₅Cr₂B₁₀</td>
<td>15 min</td>
<td>8.6</td>
<td>9.5</td>
<td>12.6</td>
</tr>
</tbody>
</table>

7.3 kOe is comparable to that of previously investigated ternary α-Fe/Nd₂Fe₁₄B nanocomposites. For the Fe was partially replaced by Cr, Nd₀.₅Fe₇₅Cr₂B₁₀, both Bₐ and (BH)\(_{\text{max}}\) increased, but with a loss of \( H_r \) (5.4 kOe). For (Nd₀.₅La₀.₅)₀.₅Fe₇₅Cr₂B₁₀, increases in Bₐ, \( H_r \), and (BH)\(_{\text{max}}\) can be noticed suggesting that a microstructural change might have occurred. From transmission electron microscopy, as shown in Figs. 2(a), 2(b) and 2(c), it is seen that the overall grain size of the Nd₀.₅Fe₈₀₅B₁₀ (Fig. 2(a)) is quite fine (around 20-40 nm). For Nd₀.₅Fe₇₅Cr₂B₁₀ and (Nd₀.₅La₀.₅)₀.₅Fe₇₅Cr₂B₁₀ ribbons, the grain size became coarser (around 60-80 nm), as shown in Figs. 2(b) and 2(c). Moreover, the grain size of the latter is more homogeneous than that of the former, and a continuous Cr-rich phase was found to exist near the grain boundary regions, as evidenced by TEM-EDXA. This Cr-rich phase near grain boundary region is presumed to have a direct impact on the high \( H_r \) obtained.

Fig. 1. TMA scans of fully processed (a) Nd₀.₅Fe₈₀₅B₁₀, (b) Nd₀.₅Fe₇₅Cr₂B₁₀ and (c) (Nd₀.₅La₀.₅)₀.₅Fe₇₅Cr₂B₁₀ ribbons.

Fig. 2. Transmission electron microscopy (TEM) of fully processed (a) Nd₀.₅Fe₈₀₅B₁₀, (b) Nd₀.₅Fe₇₅Cr₂B₁₀ and (c) (Nd₀.₅La₀.₅)₀.₅Fe₇₅Cr₂B₁₀ ribbons.
3.2 Effects of boron concentration

Since La and Cr co-substitutions result in high B, and Hc of the rare earth lean (=9.5 at%) α-Fe/R2Fe14B nanocomposites, the effect of boron concentration, from 6 to 10.5 at%, was studied to determine its effect on phase transformation and the magnetic properties of the ribbons. Shown in Figs. 3 are the TMA scans of (Nd0.95La0.05)3Fe88.5Cr7.5Bx (x = 6 through 10.5) ribbons after optimum annealing treatments. Despite the boron content being varied from 6 to 10.5 at%, only two magnetic phases, namely, α-Fe and R2Fe14B, were detected. The metastable Fe3B and Nd2Fe23B3 phases that usually appear in the ternary boron rich Nd-Fe-B system were not detected. This suggests that La and Cr substitutions are also effective in suppressing the formation of Fe3B and R2Fe23B3 phases in ribbons at least in the range of boron contents studied. In general, the volume fraction of R2Fe14B in the ribbons with a higher boron content, x = 10, 10.2 and 10.5, is higher than those with a lower boron content, x = 6 and 9, judging from the relative peak heights of R2Fe14B/α-Fe of the TMA curves.

![TMA curves for crystallized (Nd0.95La0.05)3Fe88.5Cr7.5Bx ribbons](image)

**Fig. 3.** TMA curves for crystallized (Nd0.95La0.05)3Fe88.5Cr7.5Bx ribbons (a) x = 6, (b) x = 9, (c) x = 10, (d) x = 10.2 and (e) x = 10.5.

Shown in Fig. 4 are the variation of Bn, Hc, and (BH)max with boron content of (Nd0.95La0.05)3Fe88.5Cr7.5Bx (x = 6 to 10.5) ribbons after optimum annealing treatments. At x = 6, a Bn of 9.5 kG, a (BH)max of 15.5 MGOe and an Hc of 7.1 kOe were obtained. Both Bn and (BH)max decrease initially with increasing boron content, then increase slightly when the boron content (x) was increased above 9 at%. On the contrary, the Hc increased initially with increasing boron content, reached a maximum of 12.6 kOe at x = 10.2 then decreased drastically at x = 10.5 indicating a possible phase transformation or a change of phase mixture. A Bn of 9.6 kG, a Hc of 9.5 kOe and a (BH)max of 15.5 MGOe were obtained at x = 10.5. This suggests that a Bn of more than 9 kG, (BH)max of more than 15 MGOe and Hc of more than 10 kOe can simultaneously be obtained in (Nd0.95La0.05)3Fe88.5Cr7.5Bx ribbons if x is greater than 9. Some of the boron was presumed to react with Cr forming Cr-boride(s) within R2Fe14B and near the grain boundary region. The increase of Hc with x, presumably, arises from three factors: the increase in the volume fraction of 2:14:1 phase, the presence of Cr-boride(s), and the presence of a non-magnetic Cr-rich phase near brain boundary regions.

![Comparison on the magnetic properties of (Nd0.95La0.05)3Fe88.5Cr7.5Bx](image)

**Fig. 4.** Comparison on the magnetic properties of (Nd0.95La0.05)3Fe88.5Cr7.5Bx (x = 6 to 10.5) ribbons annealed at optimum condition.

3.3 Effects of rare earth concentration

In order to investigate the possibility to further improve the magnetic properties of the La and Cr co-substituted ribbons, the alloys with different rare earth content (from 7.5 to 11.5 at%) but with 10 at% boron were investigated. Shown in Figs. 5 are the TMA scans of (Nd0.95La0.05)3Fe88.5Cr7.5B10 (y = 7.5 through 11.5) ribbons after optimized crystallization treatments. Three magnetic phases, namely, (Nd,La)2(Fe,Fe)3B14B, R2Fe23B3 and α-Fe were found at y = 7.5 and 8.5. However, only two magnetic phases, (Nd,La)2(Fe,Fe)3B14B and α-Fe, were observed on y = 9.5 to 11.5.

Shown in Fig. 6 are the change of Bn, Hc and (BH)max obtained for (Nd0.95La0.05)3Fe88.5Cr7.5B10 (y = 7.5 through 11.5) ribbons after optimum crystallization treatments. The Bn and Hc of the ribbons with y = 7.5 and 8.5 are extremely low. However, these values are improved significantly when the rare earth content was increased from y = 9.5 to 11. Most importantly, an Hc as high as 13.2 kOe, a Bn of more than 9.5 kG and a (BH)max of 18 MGOe have been achieved on (Nd0.95La0.05)3Fe7Cr7B10. These high values have never been reported for either the α-Fe/Nd2Fe14B or the Fe3B/Nd2Fe14B nanocomposites.

From TEM micrographs of ribbons with y = 7.5 through 11 after optimum thermal treatments, three
distinct phases, namely, (Nd,La)\(_2\)(Fe,Cr)\(_4\)B, R\(_2\)Fe\(_{22}\)B\(_3\) and α-Fe were found in \(y = 7.5\). However, only two magnetic phases, (Nd,La)\(_2\)(Fe,Cr)\(_3\)B and α-Fe, were detected in \(y = 9\) and \(11\). These results are consistent with the TMA scans.

![Fig. 5. TMA curves for crystallized (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{88}\),Cr\(_3\)B\(_{10}\) ribbons (a) \(y = 7.5\), (b) \(y = 8.5\), (c) \(y = 9.5\), (d) \(y = 11\) and (e) \(y = 11.5\).]

![Fig. 6. Comparison on the magnetic properties of (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{88}\),Cr\(_3\)B\(_{10}\) (\(y = 7.5\) to 11.5) ribbons after optimum thermal treatments.]

Moreover, the grain sizes of both the (Nd,La)\(_2\)(Fe,Cr)\(_4\)B and the α-Fe phases in \(y = 11\), shown as Fig. 7, are somewhat finer than those for \(y = 9\), shown as Fig. 2(c). The sizes of α-Fe and (Nd,La)\(_2\)Fe\(_{14}\)B are estimated around 20 nm and 20-40 nm for \(y = 11\), and 25 nm and 70-80 nm for \(y = 9\), respectively. This suggests that the higher La concentration may inhibit grain growth during crystallization. A fine grain size is essential to induce a good exchange coupling between the 2:14:1 and α-Fe phases, resulting in a high \(B_s\). Grain refinement and the increase of the volume fraction of \(R_2\)Fe\(_{14}\)B-type phase (i.e. a decrease in the amount of magnetically soft phase and grain boundary phase) are assumed to be the main factors that contribute to the increase in \(H_c\) from 9.1 kOe of (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{78}\),Cr\(_3\)B\(_{10}\) to the 13.2 kOe of (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{78}\),Cr\(_3\)B\(_{10}\).

![Fig. 7. Transmission electron micrograph of crystallized (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{78}\),Cr\(_3\)B\(_{10}\) ribbons.]

The change of \(\Delta M (= m_s(H)-(1-2m_s(H)))\), where \(m_s\) is the reduced magnetization and \(m_s\) is the reduced remanent magnetization, with the applied magnetic field of the (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{88}\),Cr\(_3\)B\(_{10}\) (\(y = 7.5\) through 11.5) alloy ribbons were measured and plotted in Fig. 8. The positive peak height in these plots indicates the existence of an exchange-coupling between magnetically hard and soft phases. Despite the presence of the grain boundary phase detected by TEM analysis, the sample of \(y = 9.5\) still exhibits the positive peak height at the region of applying field suggesting that the presence of grain boundary layers in (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{78}\),Cr\(_3\)B\(_{10}\) does not seem to eliminate the exchange coupling between the hard and soft grains. The exchange-coupling interaction between the α-Fe and (Nd, La)\(_2\)(Fe, Cr)\(_4\)B, in conjunction with the high presence of \(R_2\)Fe\(_{14}\)B phase and the fine grain size, results in the enhancement of \(B_s\) from 8.6 kG for \(y = 9.5\) to 9.7 kG for \(y = 11\).

3.4 Effects of Co substitution

Shown in Table II are the comparison of optimum \(B_s\), \(H_c\) and \((BH)_{max}\) for thermal treatments conducted in this study, with the Co content in (Nd\(_{0.95}\)La\(_{0.05}\))\(_3\)Fe\(_{88}\),Co\(_2\)Cr\(_3\)B\(_{10}\) (\(z = 0, 2.5, 5, 7.5\) and 10) alloy series. Initially, both \(B_s\) and \((BH)_{max}\) remain almost the same values at low Co concentration, i.e. \(z = 2.5\) and 5, then increase drastically when \(z\) was increased above 7.5. A \(B_s\) of more than 9.1 kG and \((BH)_{max}\) of more than 15.8
MGOe were obtained on samples with $z$ of 7.5 and 10.

![Graph showing the variation of $\delta M$ with applied magnetic field of (Nd$_{0.95}$La$_{0.05}$)$_2$Fe$_{85.3}$Cr$_2$B$_{10}$ ($z = 7.5$ to 11.5) ribbons.]

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Fig. 8. The variation of $\delta M$ with applied magnetic field of (Nd$_{0.95}$La$_{0.05}$)$_2$Fe$_{85.3}$Cr$_2$B$_{10}$ ($z = 7.5$ to 11.5) ribbons.

In order to understand the mechanism causing the changes of $B_c$ and $(BH)_{\text{max}}$ with the amount of Co-substitution, the content of magnetic phase was examined as a function of the Co-content for temperature ranges from 25 to 900 °C. Shown in Figs. 9(a), (b), (c), (d) and (e) are the TMA scans of the optimally treated (Nd$_{0.95}$La$_{0.05}$)$_2$Fe$_{78.2}$Co$_{5.5}$Cr$_2$B$_{10.5}$ ribbons, where $z = 0$, $2.5$, $5.0$, $7.5$, and $10$, respectively. Only two magnetic phases, $R_3$Fe$_6$B and $\alpha$-Fe, were found in the samples with Co substitution. The $T_c$ of 2:14:1 phase was found to increase

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Table II. $B_c$ and $(BH)_{\text{max}}$ of (Nd$_{0.95}$La$_{0.05}$)$_2$Fe$_{78.2}$Co$_{5.5}$Cr$_2$B$_{10.5}$ ($z = 0$, $2.5$, $5.0$, $7.5$, $10$) ribbons after optimum thermal treatments.

<table>
<thead>
<tr>
<th>Co Content ($z$)</th>
<th>$B_c$ (kG)</th>
<th>$H_c$ (kOe)</th>
<th>$(BH)_{\text{max}}$ (MGOe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>8.4</td>
<td>10.3</td>
<td>14.0</td>
</tr>
<tr>
<td>2.5</td>
<td>8.4</td>
<td>10.2</td>
<td>14.1</td>
</tr>
<tr>
<td>5.0</td>
<td>8.5</td>
<td>10.2</td>
<td>14.1</td>
</tr>
<tr>
<td>7.5</td>
<td>9.1</td>
<td>10.3</td>
<td>15.8</td>
</tr>
<tr>
<td>10</td>
<td>10.4</td>
<td>9.5</td>
<td>19.8</td>
</tr>
</tbody>
</table>

Such high $B_c$ values suggest that a strong exchange coupling between the magnetically hard and soft phases might have occurred. Unlike theoretical prediction, substituting Co for Fe does not seem to impact the $H_c$ substantially; the $H_c$ ranges from 9.5 to 10.3 kOe within the compositions studied. A $B_c$ of 10.4 kG, $H_c$ of 9.5 kOe and $(BH)_{\text{max}}$ of 19.8 MGOe have been achieved in ribbons with $z = 10$. The high $H_c$ is contrary to the expectation that Co substitution for Fe may weaken the anisotropy constant of the hard magnetic phase and subsequently lead to a decrease in the $H_c$ obtained on nanocomposites. Microstructural changes of high Co-content alloy may play a critical role in explaining the high $H_c$ values obtained. Although the role of Co in this system is not fully understood, the Co addition, with the presence of Cr, may change the liquid characteristics of precursor alloy for melt spinning, and, thus, modify the microstructure of nanocomposites.

Fig. 9. TMA scans of the thermally treated (Nd$_{0.95}$La$_{0.05}$)$_2$Fe$_{78.2}$Co$_{5.5}$Cr$_2$B$_{10.5}$ ($z = 0$ to 10) (a) $z = 0$, (b) $z = 2.5$, (c) $z = 5$, (d) $z = 7.5$ and (e) $z = 10$.

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Fig. 10. TEM microstructures of (Nd$_{0.95}$La$_{0.05}$)$_2$Fe$_{78.2}$Co$_{5.5}$Cr$_2$B$_{10.5}$ ribbons with optimum magnetic properties, where (a) $z = 0$, (b) $z = 5$, and (c) $z = 10$.

from 289 to 393 °C when the Co content was increased.

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from z = 0 to 10. This suggests that Co does enter the crystal structure of the Nd₃(FeₓCoₓ)₁₋ₓB phase. The Tᵢ of α-Fe was also found to increase from 712 to 860 °C when z was increased from 0 to 10. Again, this change in Tᵢ also implies that Co forms a solid solution of α-(Fe-Co).

Shown in Figs. 10(a), (b) and (c) are TEM micrographs of (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Coₓ₀.₅Cr₂B₁₀ with z = 0, 5 and 10, respectively. It is obvious that somewhat more grain growth occurred in the 5 at% Co-containing alloy (see Figs. 10(a) and (b)). The difference in the average grain size becomes less pronounced when z was increased from 5 to 10 as shown in Figs. 10(b) and (c). It appears, however, that the grain boundary becomes less defined and even surrounded by a highly disordered secondary phase (not identified) when z was increased to 10. A similar trend was also observed in our previous study on the low boron Nd₀.₉₅(Fe₀.₅Co₀.₅)₀.₉₅Cr₂B₁₀ alloy series. This change in microstructure may explain why the Hᵢ is insensitive to the Co-content as discussed in previous paragraph.

Shown in Table III are the variations of the Hᵢ, irreversible loss of induction, and reversible temperature coefficients of induction, α, with Co concentration of the materials studied. For z = 0, the irreversible loss and α are -3.5% and -0.184 °C/°C, respectively. Co-substitution for Fe reduces α from -0.184 °C/°C to -0.105 °C/°C when z was varied from 0 to 10. The decrease in the magnitude of α may be directly related to the increase of Tᵢ as observed in sintered Nd(Fe-Co)B magnets. However, the irreversible loss seems to vary from -2.7 to -3.5% without a correlation to the Co-content within the compositions studied. For z = 10, an irreversible loss of -3.4% and an α of -0.105%/°C were obtained. These values are comparable to commercially available NdFeB powders (an irreversible loss of -4.5% and an α of -0.105%/°C) for the bonded magnet application.

Table III. Comparison of the Hᵢ, irreversible loss of induction and reversible temperature coefficient of induction, α, of the (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Coₓ₀.₅Cr₂B₁₀ (z=0-10) ribbons after optimum treatments.

<table>
<thead>
<tr>
<th>Co Content</th>
<th>Hᵢ (kOe)</th>
<th>Inv. Loss of Induction (%)</th>
<th>α (°C/°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>z</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>10.3</td>
<td>-3.5</td>
<td>-0.184</td>
</tr>
<tr>
<td>2.5</td>
<td>10.2</td>
<td>-2.7</td>
<td>-0.144</td>
</tr>
<tr>
<td>5.0</td>
<td>10.2</td>
<td>-3.0</td>
<td>-0.131</td>
</tr>
<tr>
<td>7.5</td>
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<td>-0.118</td>
</tr>
<tr>
<td>10.0</td>
<td>9.5</td>
<td>-3.4</td>
<td>-0.105</td>
</tr>
<tr>
<td>Control</td>
<td>9.2</td>
<td>-4.5</td>
<td>-0.105</td>
</tr>
<tr>
<td>(commercial)</td>
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</tr>
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</table>

4. Conclusions

The α-Fe/R₁FeₓB-type exchange-coupled NdFeB nanocomposites with a coercivity, Hᵢ, of 9.5-13.2 kOe and energy product, (BH)ₘₐₓ, of 15.5-18.0 MGOe have been successfully developed on melt spun rare earth lean and boron-rich (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Cr₂B₁₀ (x = 10.2 to 10.5) and (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Cr₂B₁₀ (y = 10.5 to 11) alloy powders. A slight substitution of La for Nd in the above ribbons may refine the grains, and a slight substitution of Cr for Fe suppresses the formation of the R₂FeₓB₂ and FeₓB phases during crystallization and results in the formation of an α-Fe/R₁FeₓB mixture. For a fixed rare earth content, i.e. 9.5 at%, increases in the boron concentration resulted in a higher volume fraction of the R₃FeₓB₇ phase, which leads to an increase in the intrinsic coercive force from 7.1 kOe for (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Cr₂B₁₀ to 12.6 kOe for rich (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Cr₂B₁₀. A B of 9.6 kG, Hᵢ of 9.5 kOe and (BH)ₘₐₓ of 15.5 MGOe have been obtained on (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Cr₂B₁₀. Increasing the total rare earth content in (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Cr₂B₁₀ ribbons was found to increase the remanence and the intrinsic coercive force of the ribbons simultaneously. A B of 9.5 kG, Hᵢ of 13.2 kOe and (BH)ₘₐₓ of 18.0 MGOe have been obtained on (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Cr₂B₁₀. Both the magnetic properties and the magnitude of the reversible temperature coefficient of induction, α, can be improved by a suitable Co substitution for Fe in (Nd₀.₉₅La₀.₀₅)₀.₉₅Feₓ₀.₅Coₓ₀.₅Cr₂B₁₀ (z = 7.5 and 10) ribbons.

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References