The effect of Tb doping on the magnetic properties and microstructure of a TbNdFeCoB/Fe₇Co₃ nanocomposite permanent magnet

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Abstract
Melt-spun NdFeB-type nanocomposite magnetic materials are important in a number of applications, including electromagnetic sensors, magnetic resonance imaging, and voice coil motors. Herein we investigated the magnetic properties and microstructures of melt-spun TbₓNd₁₀₋ₓFe₈₀Co₄B₆/Fe₇Co₃ (x = 0.1, 0.2, and 0.3) ribbons by means of x-ray diffractometry, scanning electron microscopy, and magnetization experiments. Tb doping resulted in the formation of a 2:14:1 phase with a higher anisotropy field, which suppressed the separation of Fe₇Co₃ branch crystals and improved the magnetic properties of the material. Optimum magnetic properties that include a remanence (B_r) of 0.62 T, a coercivity (H_c) of 612.0 kA m⁻¹, and a maximum magnetic energy product (B(H)_max) of 94.9 kJ m⁻³ were obtained for the annealed Tb₀.₃Nd₉.₇Fe₈₀Co₄B₆/Fe₇Co₃ ribbon. Furthermore, the effective anisotropy constant, Kₑff, and the saturation magnetization, Mₛ, in the nanocomposite were determined by the LATS (law of approach to saturation) method. In this study, the values of Kₑff and Mₛ were found to lie between those of hard and soft magnetic phases, consistent with the exchange-coupling concept.

1. Introduction
Nanocomposite magnetic materials prepared by the melt spinning technique are predicted to possess excellent magnetic properties based on exchange-coupling interactions between adjacent grains [1–3]. Among these, NdFeB-type nanocomposites have been widely used for applications in a variety of fields, including electromagnetic sensors, magnetic resonance imaging, and voice coil motors due to their low rare-earth contents, excellent corrosion resistance, and temperature stabilities. However, the relatively low coercivity and poor thermal stability are the major practical obstacles for such applications. To improve the coercivity and thermal stability of magnets, researchers in recent years have focused more on modifying the processing conditions [4, 5] and/or doping alloy elements such as Nb, Ti, Pr and Dy [6–8]. Among these, added Tb, prefers to enter the 4f sites in the 2:14:1 phase to form the Tb₂Fe₁₄B intermetallic compound [9–13], which has a significantly higher anisotropy field (Hₐ) and Curie temperature than that of Nd₂Fe₁₄B, then changes the intrinsic characteristics of the permanent magnet, engenders ultra-high coercivity and improves the thermal stability of magnets. Furthermore, Tb-rich phases in the outer regions of the Nd₂Fe₁₄B grains also contribute to enhancing the coercivities of samples. On the other hand, melt spinning is one of the common methods for preparing NdFeB-type nanocomposites, in which, the quenching rate is an important factor that affects magnetic properties. Condensation was shown to decrease, and the nucleation rate was shown to increase with increasing melt-spinning rate, and uniform thin grains were easier to obtain through thermal annealing; these grains enhance exchange coupling between the soft and hard magnetic phases, and engender the material with excellent magnetic properties [14]. Lastly, Fe₇Co₃ which has the highest magnetic moment according to the
Slater-Pauling curve is considered to be the best soft magnetic phase. Based on this background, in this study, we over-quenched TbNdFeCoB/Fe7Co3 nanocomposite permanent magnets at a rate of 40 m s\(^{-1}\); subsequently, they were annealed at the appropriate temperature. Further, the effect of Tb content in the TbNdFeCoB/Fe7Co3 nanocomposite on the magnetic properties and microstructure was studied.

### 2. Experimental procedures

Alloy ingots with Tb\(_x\)Nd\(_{10-x}\)Fe\(_{80}\)Co\(_4\)B\(_6\)/Fe\(_7\)Co\(_3\) (\(x = 0.1, 0.2, \) and 0.3) chemical compositions were prepared by melting 99.9% pure Nd, Co, Fe, Cr, Tb-Fe (Tb% = 71.5%) alloy and Fe-B (B% = 19.6%) alloy, and then re-melted at least four times to ensure homogeneity. Melt-spun ribbons were prepared at a rate of 40 m s\(^{-1}\) in a high-purity argon environment at a chamber pressure of 0.6 \(\times\) 10\(^5\) Pa, and then hot annealed at 700 °C for 5 min. The thermal stability of the amorphous phase was investigated by differential scanning calorimetry (DSC) (TA Instruments SDT-Q600) over the 20 °C–1000 °C temperature range at 10 °C min\(^{-1}\) under N\(_2\). The microstructures and phases of the ribbons were examined by scanning electron microscopy (SEM, Hitachi S-4800) and x-ray diffractometry (XRD, Bruker D8 FOCUS) with Cu K\(_\alpha\) radiation over the 20°–90° 2\(\theta\) range. Magnetic properties were examined using a vibrating sample magnetometer (VSM, Lake Shore 7407) with the field direction in the ribbon plane; the maximum applied field was 1600 kA m\(^{-1}\).

### 3. Results and discussion

Figure 1 displays the x-ray diffraction patterns of the as-spun Tb\(_x\)Nd\(_{10-x}\)Fe\(_{80}\)Co\(_4\)B\(_6\)/Fe\(_7\)Co\(_3\) (\(x = 0.1, 0.2, \) and 0.3) ribbons. The diffraction patterns of these ribbons show only smooth broad maxima with no appreciable diffraction peaks that correspond to any crystalline phase, which is characteristic of an amorphous structure and indicates that doping with Tb increases the glass-forming ability of the material.

Figure 2 shows SEM images of the as-spun Tb\(_x\)Nd\(_{10-x}\)Fe\(_{80}\)Co\(_4\)B\(_6\)/Fe\(_7\)Co\(_3\) (\(x = 0.1, 0.2, \) and 0.3) ribbons. The ribbon surfaces appear smooth and to be single phase, which is consistent with the XRD results. When compared to the Tb-free alloy [15, 16], Tb addition promotes the formation of amorphous phases. Amorphous alloys devoid of atomic long range order and crystalline defects are characterized by excellent soft magnetic properties [17]. Table 1, which summarizes relevant magnetic data, reveals that the coercivities (H\(_{ci}\)) and the remanence ratios (\(J_r/J_s\)) of the alloys are very low; the maximum value of H\(_{ci}\) is only 26.3 kA m\(^{-1}\), which confirms the soft magnetic nature of these amorphous alloys.

Figure 3 displays DSC curves for the melt-spun Tb\(_x\)Nd\(_{10-x}\)Fe\(_{80}\)Co\(_4\)B\(_6\)/Fe\(_7\)Co\(_3\) (\(x = 0.1, 0.2, \) and 0.3) ribbons. There are two obvious exothermic peaks (\(x = 0.1 \) and 0.2) that correspond to the crystallization temperatures of the Fe\(_7\)Co\(_3\) and (Tb, Nd)\(_2\)Fe\(_{14}\)B phases, respectively. On the other hand, five exothermic peaks at about 473.2, 633.8, 644.9, 665.2, and 685.9 °C are observed when \(x = 0.3\). It should be noted that the 473.2 °C peak corresponds to the crystallization temperature of the Fe\(_7\)Co\(_3\) phase, while the other exothermic peaks correspond to the crystallization temperatures of the Tb\(_{2}\)Fe\(_{14}\)B, Tb\(_2\)Co\(_{14}\)B, Nd\(_2\)Fe\(_{14}\)B, and Nd\(_2\)Co\(_{14}\)B phases, respectively. Inspection of figure 3 reveals that the 2:14:1 phase initially exhibits only two exothermic peaks.
Figure 2. SEM images of $\text{Tb}_x\text{Nd}_{10-x}\text{Fe}_{80}\text{Co}_4\text{B}_6/\text{Fe}_7\text{Co}_3$ as-spun ribbons. (a) $x = 0.1$, (b) $x = 0.2$, and (c) $x = 0.3$.

Table 1. Magnetic data for $\text{Tb}_x\text{Nd}_{10-x}\text{Fe}_{80}\text{Co}_4\text{B}_6/\text{Fe}_7\text{Co}_3$ melt-spun ribbons ($x = 0.1, 0.2, \text{and } 0.3$).

| Alloy | $H_c (\text{kA m}^{-1})$ | $J_s(T)$ | $J_r(T)$ | $J_s/J_r$ |
|-------|-----------------|--------|--------|------------|
| Tb0.1 | 24.6            | 0.14   | 1.69   | 0.08       |
| Tb0.2 | 23.9            | 0.15   | 1.98   | 0.08       |
| Tb0.3 | 26.3            | 0.17   | 1.78   | 0.10       |

Figure 3. DSC curves of materials with different Tb content.
because of the low Tb content \((x = 0.1 \text{ and } 0.2)\); however, other exothermic peaks were observed with increasing Tb content \((x = 0.3)\). Among these, the Nd\(_2\)Fe\(_{14}\)B main phase was found to crystallize at 665.2 \(^\circ\)C. In addition, doping with Tb was observed to reduce the temperatures at which the two main phases crystallize, which can be explained by the free-volume model \([18]\). The addition of Tb changes the free volume of the melt-spun alloy available for the diffusion of atoms during crystallization; consequently, the short range order of the local ribbon structure changes, and diffusivity increases, resulting in a decrease in the crystallization temperature. Furthermore, the enthalpy of the phase transition is associated with the area under the peak, which reveals that more heat is released during the crystallization of the main phase. Due to glass-transformation temperatures of 400 \(^\circ\)C–700 \(^\circ\)C, we chose 700 \(^\circ\)C as the annealing temperature to ensure the full crystallization of all alloys.

The x-ray diffraction patterns of Tb\(_{x}\)Nd\(_{10-x}\)Fe\(_{80}\)Co\(_{4}\)B\(_6\)/Fe\(_7\)Co\(_3\) \((x = 0.1, 0.2, \text{ and } 0.3)\) ribbons annealed at 700 \(^\circ\)C for 5 min are shown in figure 4. According to previous reports \([19, 20]\), the peaks observed at 20 values of 44.7\(^\circ\), 65.1\(^\circ\), and 82.4\(^\circ\) correspond to the (110), (200), and (211) reflections of Fe\(_7\)Co\(_3\) respectively, with the remaining peaks ascribable to hard magnetic 2:14:1 phases. We also found that the relative intensities of the diffraction peaks that correspond to the Fe\(_7\)Co\(_3\) phase decreased distinctly with the introduction of Tb, which indicates that Tb doping inhibits the growth of Fe\(_7\)Co\(_3\) grains and increases the volume fraction of the hard magnetic phase. At the same time, the diffraction peaks that correspond to the Fe\(_7\)Co\(_3\) and the 2:14:1 phases in the Tb-doped ribbons are clearly broadened compared to those of the Tb-free ribbon, which indicates that Tb doping leads to a decrease in grain size. Indeed, especially for the Tb\(_{0.3}\)Nd\(_{9.7}\)Fe\(_{80}\)Co\(_{4}\)B\(_6\)/Fe\(_7\)Co\(_3\) ribbon, the considerable increases in peak intensities reflect a significant conversion to the 2:14:1 phase; hence, figure 5 only provides the microstructure of this sample. Particles of uniform size and regular spherical structure are clearly observable in figure 5. Furthermore, the mean grain size for the Fe\(_7\)Co\(_3\) and 2:14:1 phases were calculated by Scherrer’s equation to be about 35 nm and 67.1 nm, respectively; consequently, each particle includes approximately many grains.

Figure 6 shows the Curie temperatures of the two phases of the Tb\(_{x}\)Nd\(_{10-x}\)Fe\(_{80}\)Co\(_{4}\)B\(_6\)/Fe\(_7\)Co\(_3\) alloys ribbons annealed at 700 \(^\circ\)C as functions of Tb content \((x = 0.1, 0.2, \text{ and } 0.3)\). The Curie temperature of the soft magnetic phase was observed to increase a little, but basically remained stable with increasing Tb content; however, the Curie temperature of the hard magnetic phase clearly increased with increasing Tb content. These observations are ascribable to the higher Curie temperature \((620\text{ k})\) of the Tb\(_2\)Fe\(_{14}\)B intermetallic compound compared to that of Nd\(_2\)Fe\(_{14}\)B \((585\text{ k})\), which suggests the addition of Tb increases thermal stability and enables the more-expensive use of NdFeB nanocomposite magnets.

Figure 7 shows the magnetic properties of the annealed Tb\(_{x}\)Nd\(_{10-x}\)Fe\(_{80}\)Co\(_{4}\)B\(_6\)/Fe\(_7\)Co\(_3\) \((x = 0.1, 0.2, \text{ and } 0.3)\) ribbons, with the corresponding data listed in table 2. The three key properties of a permanent magnetic material, namely remanence, coercivity, and the maximum magnetic energy product, increase simultaneously with increasing Tb content, which is possibly ascribable to the added Tb entering the Nd\(_2\)Fe\(_{14}\)B-phase lattice to form a \((\text{Tb, Nd})_2(\text{Fe, Co})_4\)B phase with a higher anisotropy field, which suppresses the separation of Fe\(_7\)Co\(_3\) branch crystals and improves the properties of the magnet. However, due to anti-ferromagnetic coupling between elemental Tb and Fe, the saturation magnetic polarization reduces little with increasing Tb content. We conclude that the rather rich combination of magnetic properties of the 0.3 at% Tb alloy ribbon
Figure 5. SEM images of Tb$_{0.3}$Nd$_{9.7}$Fe$_{80}$Co$_4$B$_6$/Fe$_7$Co$_3$ ribbons annealed at 700 °C. (a) ×10k and (b) ×50k.

Figure 6. Curie temperatures of the soft (red) and hard (black) magnetic phases of annealed Tb$_x$Nd$_{10-x}$Fe$_{80}$Co$_4$B$_6$/Fe$_7$Co$_3$ ribbons as functions of Tb content.

Figure 7. Magnetic properties of Tb$_x$Nd$_{10-x}$Fe$_{80}$Co$_4$B$_6$/Fe$_7$Co$_3$ ribbons annealed at 700 °C ($x = 0.1, 0.2, \text{and } 0.3$).
Table 2. Magnetic data of Tb$_{0.1}$Nd$_{0.9}$Fe$_{80}$Co$_{4}$B$_{6}$/Fe$_{7}$Co$_{3}$ (x = 0.1, 0.2, 0.3) annealed ribbons.

| alloy   | $H_{c}$ (kA m$^{-1}$) | $J_{c}$ (T) | $J_{s}$ (T) | $(BH)_{max}$ (kJ m$^{-3}$) |
|---------|-----------------------|-------------|-------------|-----------------------------|
| Tb0.1   | 326.9                 | 0.46        | 1.27        | 47.9                        |
| Tb0.2   | 455.9                 | 0.49        | 1.12        | 66.8                        |
| Tb0.3   | 612.0                 | 0.62        | 1.07        | 94.9                        |

$(H_{c})_{a} = 612.0$ kA m$^{-1}$, $J_{c} = 0.62$ T, and $(BH)_{max} = 94.9$ kJ m$^{-3}$ is a consequence of its fine microstructure, which significantly enhances the exchange coupling between hard and soft magnetic grains and engenders the material with excellent magnetic properties. The initial magnetic curve for the optimally annealed Tb$_{0.3}$Nd$_{0.7}$Fe$_{80}$Co$_{4}$B$_{6}$/Fe$_{7}$Co$_{3}$ ribbon is also shown in figure 7. At first, the initial magnetic polarization displays a moderately rising trend, and then rises quickly as the field becomes comparable to its intrinsic coercivity, which means that magnetization reversal is governed mainly by a coercive mechanism involving magnetic domain-wall pinning [21, 22], with grain boundaries likely to act as domain-wall-pinning centers. In addition, the effective anisotropy constant, $K_{eff}$, and the saturation magnetization, $M_{s}$, of a nanocomposite can be determined by the LATS (law of approach to saturation) method [23, 24], as described by the following formula:

$$M = M_{s}(1 - a/H - b/H^2 - c/H^3 - ...) + \chi_{p}H,$$

where $\chi_{p}$ is the paramagnetic susceptibility, and $\chi_{p}H$ can be ignored when the magnetic field is insufficiently large; a, b, c, and $\chi_{p}$ are coefficients related to the magnetic properties, $M_{s}$ is the saturation magnetization, and $H$ is the magnetic field. First, the saturation magnetization $M_{s}$ and coefficient $a$ were respectively determined to be about $1.78 \times 10^6$ A m$^{-1}$ and $4.13 \times 10^5$ A m$^{-1}$ on the basis of the linear relationship between $M$ and 1/$H$ at the higher magnetic field. The relationship between $M + M_{s} \times a/H$ and 1/$H^2$ is linear in the near-saturation stage; hence, the saturation magnetization $M_{s}$ and coefficient $b$ were determined to be about $1.67 \times 10^6$ A m$^{-1}$ and $4.47 \times 10^{10}$ (A/m)$^2$, respectively. For a tetragonal multi-crystal material, coefficient $b$ is given by the relationship:

$$b = 4K_{eff}^2/15\mu_{0}^{2}M_{s}^{2},$$

where $K_{eff}$, the effective anisotropy constant, was calculated to be about $0.86 \times 10^6$ J m$^{-3}$. Compared to the theoretical values for a 2:14:1 hard magnetic phase ($M_{s} = 1.28 \times 10^6$ A m$^{-1}$, $K_{1} = 4.9 \times 10^7$ J m$^{-3}$), and a Fe$_{7}$Co$_{3}$ soft magnetic phase ($M_{s} = 1.99 \times 10^6$ A m$^{-1}$, $K_{1} = 2 \times 10^4$ J m$^{-3}$), the saturation magnetization $M_{s}$ and the effective anisotropy constant $K_{eff}$ for the Tb$_{0.3}$Nd$_{0.7}$Fe$_{80}$Co$_{4}$B$_{6}$/Fe$_{7}$Co$_{3}$ nanocomposite in this paper lie between those of the hard and the soft magnetic phases, consistent with the exchange-coupling concept.

4. Conclusion

Tb$_{x}$Nd$_{1-x}$Fe$_{80}$Co$_{4}$B$_{6}$/Fe$_{7}$Co$_{3}$ (x = 0.1, 0.2, and 0.3) ribbons were prepared by the melt spinning technique. After hot annealing, the ribbons were composed of Fe$_{7}$Co$_{3}$ and 2:14:1 phases. The mean grain size of the Fe$_{7}$Co$_{3}$ and 2:14:1 phases were determined from Scherrer’s equation to be about 35 nm and 67.1 nm, respectively. This indicates that Tb doping forms a (Tb, Nd)$_{2}$(Fe, Co)$_{14}$B phase with a higher anisotropy field, which suppresses the separation of Fe$_{7}$Co$_{3}$, increases the grain size, and improves the properties of the magnet. The best magnetic properties were observed when 0.3 at% Tb was added; the annealed Tb$_{0.3}$Nd$_{0.7}$Fe$_{80}$Co$_{4}$B$_{6}$/Fe$_{7}$Co$_{3}$ ribbon exhibited a remanence ($J_{r}$) of 0.62 T, coercivity ($H_{c}$) of 612.0 kA m$^{-1}$, and a maximum magnetic energy product $(BH)_{max}$ of 94.9 kJ m$^{-3}$. The coercive mechanism was confirmed to involve magnetic domain-wall pinning, as evidenced by the initial magnetic curve. Furthermore, we found that the values of $K_{eff}$ and $M_{s}$ determined by the LATS method lie between the values for the hard and the soft magnetic phases, in agreement with the exchange-coupling concept.

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