Structure and magnetic properties of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ ($0 \leq x \leq 0.8$) compounds

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Received 23 April 2004

Published 15 September 2004

Online at stacks.iop.org/JPhysD/37/2632

doi:10.1088/0022-3727/37/19/003

Abstract

The structure and magnetic properties of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds with $x = 0$ – 0.8 have been investigated using x-ray powder diffraction (XRD) and magnetic measurements. It has been found that all the compounds crystallize in a $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type structure. Substitution of Er for Nd leads to a contraction of the unit-cell volume. The Curie temperature, T_c , and the saturation magnetization, M_s , of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ decrease monotonically with increasing Er content. The easy magnetization direction (EMD) of $\text{Nd}_3\text{Fe}_{25}\text{Cr}_{4.0}$ at room temperature is close to the [040] direction but may be a little out of the basal plane. With increasing Er content, the EMD changes closer to the [40–2] direction and the tilt angle increases. Both the XRD patterns and ac susceptibility indicate the appearance of a spin reorientation for $x = 0$ – 0.4 as the temperature decreases from room temperature to 77 K. The spin reorientation temperature, T_{sr} , increases monotonically with increasing Er content from 158 K for $x = 0$ to 198 K for $x = 0.4$. A first order magnetization process (FOMP) occurs for all the compounds, and the critical field of the FOMP decreases with increasing Er content from 6.6 T for $x = 0$ to 2.0 T for $x = 0.7$.

1. Introduction

Collocott *et al* [1] found a new phase in the iron-rich corner in the Nd–Fe–Ti ternary phase diagram, which was later identified to be of $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type structure with monoclinic symmetry and space group $A_{2/m}$ [2]. Since then, much attention has been paid to the $\text{R}_3(\text{Fe,M})_{29}$ compounds, where R stands for rare-earth and M for stabilizing element. A series of novel $\text{R}_3(\text{Fe,M})_{29}$ compounds and their interstitial compounds have been synthesized for $\text{R} = \text{Sm, Ce, Pr, Nd, Y, Gd, Tb}$ and $\text{M} = \text{Mo, Ti, Cr, Mn, V}$, etc [3–8]. Yang *et al* [9] and Hu *et al* [10] have synthesized $\text{Sm}_3(\text{Fe,Ti})_{29}\text{N}_y$ and $\text{Sm}_3(\text{Fe,Ti})_{29}\text{C}_y$ compounds, respectively, which exhibit outstanding intrinsic magnetic properties. Subsequently, Li *et al* [11] have

synthesized $\text{Sm}_3\text{Fe}_{12.1}\text{Co}_{16}\text{Mo}_{0.9}$, which is a very promising candidate for novel rare-earth permanent magnetic materials. However, the exact nature of the magnetic anisotropy of these compounds is still not quite clear, and so it is of great value to study the structure and magnetic properties of the 3:29 series. It is well known that the $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type structure is an intermediate structure between the rhombohedral $\text{Th}_2\text{Zn}_{17}$ (2:17R) and tetragonal ThMn_{12} (1:12) structures with a ratio 1:1. However, so far $\text{R}_3(\text{Fe,M})_{29}$ compounds with $\text{Nd}_3(\text{Fe,Ti})_{29}$ type structures, R being a heavy rare-earth element heavier than Ho, have not been synthesized.

The magnetocrystalline anisotropy of the R–Fe inter-metallic compounds results from the contribution of both R- and Fe-sublattices. A study of the compound $\text{Y}_3(\text{Fe,Ti})_{29}$

Figure 1. XRD patterns of the random and magnetically aligned powder samples of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds.

has indicated that the magnetocrystalline anisotropy arising from the Fe sublattice favours the $[40-2]$ direction [12]. The anisotropy of the R sublattice in $\text{R}_3(\text{Fe},\text{M})_{29}$ arises from the crystal field interaction at the R sites. In the $\text{R}_3(\text{Fe},\text{Ti})_{29}$ -type structure, there are two different R sites with opposite signs of the second order crystal field parameter A_{20} . When A_{20} is negative at a site, a rare-earth with a positive second-order Stevens coefficient, α_J (Sm, Tm, Er, Yb), will contribute uniaxial anisotropy, while for positive A_{20} , a rare-earth with a negative α_J (Pr, Nd, Tb, Dy, Ho) will exhibit uniaxial anisotropy [13]. So it is expected that the substitution of one rare-earth for the other with different α_J will influence the magnetic properties, especially the magnetocrystalline anisotropy in $\text{R}_3(\text{Fe},\text{Ti})_{29}$.

In this paper, the effect of substitution of Er for Nd on the structure and magnetic properties, especially the magnetocrystalline anisotropy, of $\text{Nd}_3(\text{Fe},\text{Cr})_{29}$ is studied.

2. Experimental

A series of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds with $x = 0-1.0$ were prepared by arc melting the constituent elements in a high purity argon atmosphere. The purity of the starting elements was higher than 99.9%. All the ingots were remelted at least four times for homogenization. The ingots were wrapped in molybdenum foil and sealed in a quartz tube and then annealed at 1293 K for 72 h under the protection of an argon atmosphere, followed by water quenching. X-ray powder diffraction (XRD) with $\text{Cu-K}\alpha$ radiation was used to check the structure and to measure the lattice parameters of the compounds. Thermomagnetic curves (TMA) were measured in a vibrating-sample magnetometer (VSM) with a field of 0.05 T to check the single phase characteristic and to measure the Curie temperature (T_c). The Curie temperature was derived from the M^2-T curves by extrapolating M^2 to zero. The magnetization curves were measured using a superconducting quantum interference device (SQUID) magnetometer with an applied field of up to 7 T or using a Quantum Design PPMS-14 system with a field up to 14 T. The saturation magnetization (M_s) was determined from $M-(1/H)$ curves by extrapolating

$1/H$ to zero. Ac susceptibility measurements were carried out from 77 K to room temperature.

In order to examine the magnetocrystalline anisotropy at room temperature, finely powdered particles were mixed with epoxy resin and packed in a cylindrical plastic tube. For normal alignment, the plastic tube was put in a field of about 1 T with the cylinder axis parallel to the field direction, so that the cylindrical axis was the easy magnetization direction (EMD). For alignment of rotation, the tube was rotated around its cylindrical axis, which was perpendicular to the applied field, and so the cylindrical axis was the hard magnetization direction (HMD).

3. Results and discussion

Both XRD and TMA reveal that all the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds for $x = 0-0.8$ are substantially 3:29 single phase except for $\text{Nd}_{0.6}\text{Er}_{2.4}\text{Fe}_{25}\text{Cr}_{4.0}$, which contains a small amount of the 1:12 phase. As an example, figure 1(a) shows XRD patterns taken from randomly oriented powder samples of $\text{Nd}_3\text{Fe}_{25}\text{Cr}_{4.0}$, which can be well indexed based on the monoclinic $\text{Nd}_3(\text{Fe},\text{Ti})_{29}$ -type structure of space group $A_{2/m}$. The lattice parameters calculated from the XRD patterns are listed in table 1. It can be seen that the lattice constants a , b , and c and the unit cell volume, V , show a tendency to decrease with increasing Er content. This is due to the fact that Er has a smaller atomic radius than that of Nd. Single phase $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds with the $\text{Nd}_3(\text{Fe},\text{Ti})_{29}$ -type structure could not be synthesized for $x = 0.9$ and 1.0.

In order to examine room temperature magnetocrystalline anisotropy, XRD patterns of the normally aligned powder samples of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds were measured, as shown in figure 1(b)–(e). Compared with the randomly aligned powder sample with $x = 0$ (figure 1(a)), it can be seen from figure 1(b) that for the normally aligned sample, the intensity of (040) reflection increases obviously, while those of the (23-1), (40-2), and (30-4) reflections decrease, and other reflections disappear. It is well known that the 3:29-type structure can be derived by replacing a fraction of the Ca sites in the CaCu_5 -type structure by a pair of transition

Table 1. Structure and magnetic parameters of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds.

Composition	a (Å)	b (Å)	c (Å)	V (Å ³)	T_c (K)	T_{sr} (K)	M_s (emu g ⁻¹)	B_{cr} (T)
$x = 0$	10.607	8.556	9.717	875.5	418	158	113.9	6.6
$x = 0.1$	10.629	8.559	9.686	874.8	415	166	111.8	6.2
$x = 0.2$	10.620	8.554	9.683	873.3	410	173	—	—
$x = 0.3$	10.608	8.534	9.698	871.5	408	185	96.9	5.1
$x = 0.4$	10.594	8.509	9.691	867.0	406	198	—	—
$x = 0.5$	10.583	8.510	9.680	865.3	401	—	77.6	3.6
$x = 0.6$	10.571	8.498	9.672	862.5	399	—	—	—
$x = 0.7$	10.560	8.480	9.646	857.4	394	—	61.9	2.0
$x = 0.8$	10.556	8.486	9.655	858.6	380	—	—	—

metal atoms (dumbbell) along the hexagonal axis of the CaCu_5 structure. According to the transformation relationship between the 3 : 29-type and 1 : 5-type structures [12], it can be seen that the (040), (23-1), (40-2), and (30-4) reflections in the 3 : 29-type structure correspond to the (1-10), (140), (110), and (22-1) reflections in the 1 : 5-type structure, respectively. Among them, the [1-10], [140], and [110] directions are in the basal plane in the 1 : 5 type structure, whereas the [22-1] direction makes a tilt angle with respect to the basal plane [14]. This suggests that the EMD of the $\text{Nd}_3\text{Fe}_{25}\text{Cr}_{4.0}$ compound is very close to the [040] direction, but it is a little out of the basal plane of the 1 : 5 structure.

Figure 1(f) shows the XRD pattern of a rotation-alignment sample of the $\text{Nd}_3\text{Fe}_{25}\text{Cr}_{4.0}$ compound. It is clear that only the (204) reflection is retained and all the other reflections have almost disappeared. This further confirms that the HMD of the $\text{Nd}_3\text{Fe}_{25}\text{Cr}_{4.0}$ compound is in the [204] direction ([001] direction in the 1 : 5-type structure), and the HMDs of these compounds can be obtained by the rotation-alignment method.

As the Er content (x) increases (see figure 1(c)–(e)), the intensity of the (40-2) and (30-4) reflections increases significantly, and the intensity of the (040) reflection decreases, whereas the (23-1) reflection increases first with increasing Er content from 0 to 0.3, through a maximum at $x = 0.3$ and then decreases with further increasing x , which shows a complex change in the anisotropy of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds with Er substitution. It is worth noting that with increasing Er content, the EMD of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds is most likely to change from the direction close to the [040] direction to another close to the [40-2] direction, and make a larger tilt angle with respect to the basal plane of the 1 : 5 structure.

It is well accepted that in rare-earth (R)–transition metal (T) intermetallic compounds, in the first approximation, the rare-earth sublattice anisotropy constant, K_1^R , is determined by the product of the second order crystal field parameter, A_{20} , and the second order Stevens coefficient, α_1 . It can be expressed by

$$K_1^R = \frac{1}{2} \alpha_1 \langle r^2 \rangle \langle 3J^2 - J(J+1) \rangle A_{20}, \quad (1)$$

where the quantities in angular brackets denote the expectation values. It has been reported that, in $\text{R}_3(\text{Fe,Ti})_{29}$ compounds, the crystal field parameters A_{20} have opposite signs for the two R sites, namely $A_{20} < 0$ for the 4i sites (2 : 17-like) and $A_{20} > 0$ for the 2a sites (1 : 12-like) [13]. From the model of Courtois *et al* [12], one knows that the anisotropy resulting from Nd atoms with $\alpha_1 < 0$ favours the easy plane.

Figure 2. Ac susceptibility against temperature for $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds.

Considering that the radius of the Er atom is smaller and favours the 4i site, the anisotropy resulting from Er atoms with $\alpha_1 > 0$ favours the easy axis, namely the [204] direction [15]. Therefore, it is easy to understand that the tilt angle made by the EMD of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds with respect to the basal plane of the 1 : 5 structure increases with Er content.

The temperature dependence of the real component, χ' , of the ac susceptibility for the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds was measured in the temperature region from 77 K to room temperature and is shown in figure 2. It can be seen that for the samples with $x = 0$ –0.4, an obvious anomaly can be observed in the curves at certain temperatures. Such an anomaly has also been observed in the $\text{Nd}_3(\text{Fe,Ti})_{29}$ compound and has been explained as a spin reorientation by Morellon *et al* [16]. The spin reorientation temperature, T_{sr} , at which the spin reorientation occurs was derived from the peak position. The value of T_{sr} increases with Er content from 158 K for $x = 0$ to 198 K for $x = 0.4$ as shown in figure 4(a). It is worth noting that the anomaly vanishes when the (040) reflection is surpassed by the (30-4) reflection in the XRD pattern of the aligned sample, namely when the tilt angle of the EMD with respect to the basal plane of 1 : 5 structure is big enough.

Figure 3 shows the TMA of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds measured in a temperature range from room temperature to above the Curie temperature. Curie temperatures T_c of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ are listed in table 1. Figure 4(a) shows the composition dependence of T_c . It can be seen that the Curie temperature, T_c , decreases monotonically

Figure 3. TMA of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds.

Figure 4. (a) Curie temperature, T_c , and spin reorientation temperature, T_{sr} ; (b) saturation magnetization, M_s ; and (c) critical field, B_{cr} , of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds as a function of the Er content.

with increasing Er content. It is well known that for the R–T intermetallic compounds the Curie temperature, T_c , is determined by the T–T, R–T, and R–R interactions. Among them, the T–T interaction is dominant, whereas the R–R interaction is of minor importance and can usually be neglected. Because the transition metal content is fixed in the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds and only the Nd and Er contents vary, the change in the Curie temperature is mainly determined by the R–T interaction. Based on the molecular field model, the Curie temperature, T_c , can be expressed as [17]

$$T_c = \frac{1}{2}[T_T + (T_T^2 + 4T_{RT}^2)^{1/2}]. \quad (2)$$

Here

$$T_T = n_{TT}N_T \left[\frac{4S^*(S^* + 1)\mu_B^2}{3k_B} \right], \quad (3)$$

$$T_{RT} = n_{RT}|\gamma|(N_T N_R)^{1/2} \times \left[\frac{2(S^*(S^* + 1))^{1/2} \times g_J(J_R(J_R + 1))^{1/2}\mu_B^2}{3k_B} \right], \quad (4)$$

where $\gamma = 2(g_J - 1)/g_J$. $2(S^*(S^* + 1))^{1/2}\mu_B$ is the effective moment of the iron atom in the paramagnetic state and N_T , N_R are the number of Fe and R atoms per unit volume. g_J and J_R are the Lande factor and the total angular moment of the rare-earth atoms, respectively. T_T and T_{RT} are the interaction coefficients of T–T and T–R, which stand for the contributions of the T–T and T–R interactions to T_c . In equation (2), the T_{RT}^2 is proportional to the de Gennes factor ($G_J(J)$) of the R ion, where $G_J(J) = (g_J - 1)^2 J_R(J_R + 1)$. It is well known that the value of $G_J(J)$ decreases as the atomic number in the series of heavy rare-earth elements increases, and the $G_J(J)$ value of Er is larger than that of Nd. Since the atomic radius of Er is smaller than that of Nd due to the lanthanide contraction, substitution of Er for Nd leads to a decrease of the unit cell volume and an increase in the values of N_T and N_R . On the other hand, n_{RT} decreases as the atomic number of the rare-earth elements rises. So the value of n_{RT} should decrease with Er substitution. The dependence of T_c on the composition may result from the competition of all these factors mentioned earlier.

The magnetization curves of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds with $x = 0$ – 0.7 were obtained at 5 K in a field up to 5 T, applied either parallel or perpendicular to the alignment direction, as shown in figure 5. Figure 6 shows the magnetization curves of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds measured at 5 K using PPMS with the applied field perpendicular to the alignment direction of the samples. It can be seen that the magnetization curves for the compounds from $x = 0$ to 0.7 show a jump-like increase at certain fields. This phenomenon has also been observed in $\text{Tb}_3(\text{Fe}, \text{V})_{29}$ [18] and has been explained as a first order magnetization process (FOMP), which arises from a magnetic field induced spin phase transition from one spin phase to the another. The critical field, B_{cr} , at which the FOMP occurs was derived from the maximum of the dM/dH versus H curve. The values of B_{cr} are listed in table 1. Figure 4(c) shows the value of B_{cr} as a function of Er content (x). The critical field of the FOMP decreases with increasing Er content from 6.6 T for $x = 0$ to 2.0 T for $x = 0.7$.

The values of the saturation magnetization, M_s , at 5 K are derived and listed in table 1. Figure 4(b) shows the composition dependence of M_s . It can be seen that the M_s value decreases monotonically with increasing Er content due to antiparallel coupling between the Er and Fe moments. Considering that the Er moment is antiparallel to the Fe and Nd moments and assuming that all the moments are collinear, the saturation moments, M_s , of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds can be expressed as

$$M_s = M_0 - 3x(\mu_{\text{Nd}} + \mu_{\text{Er}}), \quad (5)$$

where M_0 is the moment of $\text{Nd}_3\text{Fe}_{25}\text{Cr}_{4.0}$ and μ_{Nd} and μ_{Er} are the average magnetic moments of the Nd and Er atoms, respectively. Assuming that the μ_{Nd} and μ_{Er} have free atomic moments of $3.62\mu_B$ and $9.59\mu_B$, respectively, taking the value of M_0 to be $113.9 \text{ A m}^2 \text{ kg}^{-1}$, the values of M_s were calculated and are shown in figure 4(b) by the dashed line. It is seen that the calculated result is lower than the experimental one. This may be due to changes in the moments of the free Er and Nd ions as a result of the crystal field effect.

Figure 5. Magnetization curves of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds.

Figure 6. Magnetization curves of the HMD samples of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds in a field up to 13 T.

4. Conclusion

The structure and magnetic properties of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds with $x = 0-0.8$ have been investigated. It has

been found that all the investigated compounds crystallize in a $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type structure. Substitution of Er for Nd leads to a contraction of the unit-cell volume. The Curie temperature, T_c , and the saturation moments, M_s , of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ compounds decrease monotonically with increasing Er content. With increasing Er content the EMD changes from a direction close to the $[040]$ direction to one close to the $[40-2]$ direction but always makes a tilt angle with respect to the basal plane. Both the XRD patterns and ac susceptibility of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{25}\text{Cr}_{4.0}$ samples indicate a spin reorientation phenomenon for $x = 0-0.4$ as the temperature decreases from room temperature to 77 K. The spin reorientation temperature, T_{sr} , increases monotonically with increasing Er content from 158 K for $x = 0$ to 198 K for $x = 0.4$. The FOMP occurs in all the compounds, and the critical field of the FOMP decreases with increasing Er content from 6.6 T for $x = 0$ to 2.0 T for $x = 0.7$.

Acknowledgment

This work was supported by State Key Project of Fundamental Research Grant, China (No G2000067106).

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