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Structure and magnetic properties of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ ($x = 0.0\text{--}0.8$) compounds

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Abstract

The structure and magnetic properties of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds with $x = 0.0\text{--}0.8$ have been investigated by means of x-ray diffraction and magnetic measurements. It is found that all the compounds are single phase and crystallize in the $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type structure with monoclinic symmetry and space group $A_{2/m}$. The lattice constants a , b and c and the unit-cell volume V show a tendency to decrease with increasing Er content, reflecting the lanthanide contraction. The Curie temperature decreases with increasing Er content. The spin-reorientation temperature T_{sr} increases with increasing Er content till $x = 0.2$, then tends to saturate upon further increase in Er content. The saturation magnetization at 5 K decreases with increasing Er content owing to the antiferromagnetic coupling between Er and Nd moments. A first-order magnetization process is observed for compounds with $x = 0, 0.2$ and 0.4 at critical field values of 8 T, 4.5 T and 3.5 T, respectively, applied along the hard magnetization direction.

1. Introduction

The successful synthesis of the $\text{Nd}_2(\text{Fe,Ti})_{19}$ compound by Collocott *et al* [1] at the Fe-rich corner of the R (= rare-earth)–Fe–Ti phase diagram, where Ti is used as a stabilizing element, has brought renewed interest in the search for new permanent-magnet materials. The structure of the $\text{Nd}_2(\text{Fe,Ti})_{19}$ compounds was later established to be a preferable $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type of structure with monoclinic symmetry and $A_{2/m}$ space group [2–4]. Up to now, $R_3(\text{Fe,T})_{29}$ compounds with $R = \text{Ce, Pr, Nd, Sm, Gd, Tb}$ and Y , and $T = \text{Ti, V, Cr, Nb, Mo, Mn}$ and Re have been synthesized [5–11]. Yang *et al* [12] have synthesized an interstitial $\text{Sm}_3(\text{Fe,Ti})_{29}\text{N}_y$ compound, which exhibits excellent permanent-magnet properties. In order to find 3 : 29 compounds with easy-axis anisotropy at room temperature, which is important for application in permanent magnets, Yang *et al* [13] and Wang *et al* [14] have investigated the structure and magnetic properties of $\text{Gd}_3(\text{Fe}_{1-x}\text{Co}_x)_{25}\text{Cr}_4$ compounds and have synthesized Co-based $R_3\text{Co}_{29-x}\text{Cr}_x$ compounds with $R = \text{Sm, Nd}$ and Gd . It was found that in the

$\text{Gd}_3(\text{Fe}_{1-x}\text{Co}_x)_{25}\text{Cr}_4$ compounds and $\text{Sm}_3(\text{Fe}_{1-x}\text{Co}_x)_{25}\text{Cr}_4$ compounds a uniaxial anisotropy can be obtained at room temperature for $x \geq 0.4$. Furthermore, substitution of Co for Fe in these compounds leads to a drastic increase of the Curie temperature. However, up to now 3 : 29 phase R – T intermetallics with R a heavy rare-earth element, such as Ho or Er, have not yet been synthesized. The 3 : 29 structure, like the ThMn_{12} (1 : 12) and $\text{Th}_2\text{Zn}_{17}$ (2 : 17) structures, can be derived from CaCu_5 (1 : 5) structure. In fact, the 3 : 29 structure consists of $R(\text{Fe,M})_{12}$ and $R_2(\text{Fe,M})_{17}$ in the ratio of 1 : 1. It is well known that there exist two structures for the $R_2\text{Fe}_{17}$ compounds: the $\text{Th}_2\text{Zn}_{17}$ -type of structure for the light- R elements and the $\text{Th}_2\text{Ni}_{17}$ -type structure for the heavy ones. The compounds with the moderately heavy R elements may crystallize in both structures [15]. All $R_3(\text{Fe,M})_{29}$ compounds with $R = \text{light rare-earth and Gd, Tb and Dy}$ [9] have the $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type structure.

In order to investigate the effect of Er on the structure and magnetic properties of the 3 : 29 compounds, we have selected the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{29-y-z}\text{Co}_y\text{Cr}_z$ system, because in the $R_3(\text{Fe,Cr})_{29}$ compounds, the necessary Cr content

decreases when R changes from the light to the heavy R [9], whereas in the $R_3(\text{Fe},\text{Co},\text{Cr})_{29}$ compounds with $R = \text{Gd}$ [13, 14], the necessary Cr content increases with increasing Co content. To keep the Cr content constant, we have fixed the Co content of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{29-y-z}\text{Co}_y\text{Cr}_z$ compounds, taking $z = 5$ and $y = 6$. In the present paper, a study of the effect of substitution of Er for Nd in $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds on the structure and magnetic properties is presented.

2. Experimental methods

Ingots with composition $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ were prepared by arc-melting the constituent elements with a purity of at least 99.9% in an argon atmosphere. All the ingots were remelted at least four times to ensure homogeneity. In order to compensate for the loss of R during the melting and annealing, 5% excess of R relative to the stoichiometric composition was added. The ingots were annealed in a sealed quartz tube under the protection of an argon atmosphere at 1363 K for 72 h to maximize the amount of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$. At the end of the annealing, the alloys were water-quenched. XRD with $\text{CuK}\alpha$ radiation and thermomagnetic analysis were employed to check the phase homogeneity. The thermomagnetic curves were measured in a superconducting quantum interference device (SQUID) magnetometer from 5 K to room temperature and in a vibrating-sample magnetometer (VSM) from room temperature to above the Curie temperature. The Curie temperatures were derived from the M^2 - T curves and by extrapolating M^2 to zero. Magnetization curves were recorded in a SQUID magnetometer at 5 K in external magnetic fields up to 5 T. The values of saturation magnetization M_s were obtained from M - $1/B$ plots by extrapolating $1/B$ to zero.

In order to determine the anisotropy field, fine-powdered particles were mixed with epoxy resin and packed in a plastic tube of cylindrical shape. In the usual magnetic alignment, the epoxy is allowed to harden while the plastic tube is positioned in an applied magnetic field of about 1 T with the cylinder axis parallel to the field direction, so that the cylinder axis becomes the easy magnetization direction (EMD). In the case of rotation alignment, the epoxy hardened while the plastic tube rotated around its cylinder axis in a magnetic field that was applied perpendicular to the axis, so that the cylinder axis will correspond to the hard magnetization direction.

3. Results and discussion

The phase homogeneity of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds has been checked by means of both XRD and thermomagnetic analysis. It was found that the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds are single phase for $x = 0.0$ – 0.8 , crystallizing in the $\text{Nd}_3(\text{Fe},\text{Ti})_{29}$ -type of structure with monoclinic symmetry and space group $A_{2/m}$. As an example, figure 1(a) shows the XRD pattern of randomly oriented powder of $(\text{Nd}_{0.6}\text{Er}_{0.4})_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ which could very well be indexed based on the $\text{Nd}_3(\text{Fe},\text{Ti})_{29}$ -type of structure. As mentioned above, the 3 : 29 structure consists of the 1 : 12 and 2 : 17 structures at a ratio 1 : 1. Since there are two different structures of the 2 : 17 phase—the $\text{Th}_2\text{Zn}_{17}$ -type of structure for the light R elements and the $\text{Th}_2\text{Ni}_{17}$ -type of structure for

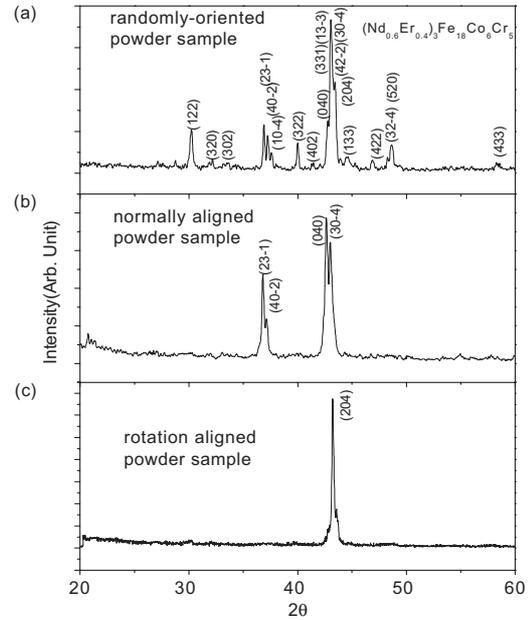


Figure 1. XRD patterns of (a) randomly-oriented, (b) normally-aligned and (c) rotation-aligned powder samples of $(\text{Nd}_{0.6}\text{Er}_{0.4})_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$.

Table 1. The unit-cell parameters of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds.

x	a (Å)	b (Å)	c (Å)	β (deg)	V (Å ³)
0	10.556	8.539	9.708	96.607	869.16
0.2	10.515	8.482	9.654	96.777	855.03
0.4	10.509	8.465	9.641	96.745	851.77
0.6	10.516	8.454	9.624	96.841	849.45
0.8	10.51	8.455	9.623	96.868	848.99

the heavy ones—there should also be two different structures of the 3 : 29 phase when the R changes from light to heavy. However, the experimental results indicate that no structural transition occurs up to $x = 0.8$ in the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ system. The lattice constants a , b , c and the unit-cell volume V have been derived from the XRD patterns and are listed in table 1. It can be seen that V decreases with increasing Er content due to the smaller radius of the Er atom compared with that of Nd.

In order to examine the magnetocrystalline anisotropy, XRD patterns of magnetically-aligned-powder samples were measured. As an example, figure 1(b) shows the XRD pattern of magnetically-aligned powder of the $(\text{Nd}_{0.6}\text{Er}_{0.4})_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compound. It can be seen that the (040) reflection is strongly enhanced, whereas the (23-1), (40-2) and (30-4) reflections remain unchanged, or have weakened, and other peaks have almost disappeared, compared with the XRD pattern of the randomly-oriented powder shown in figure 1(a). This indicates that the $(\text{Nd}_{0.6}\text{Er}_{0.4})_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ ($x = 0.0$ – 0.8) compounds possess easy-plane-type anisotropy at room temperature. Figure 1(c) shows the XRD pattern of rotation-aligned powder of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds. It can be seen that the [204] reflection is strongly enhanced and that the other reflections have disappeared, which further demonstrates that the EMD is in the plane perpendicular to the [204] direction.

According to the transformation relationship between the 1:5 and the 3:29 structure, the (040), (23-1), (40-2) and (30-4) reflections in the 3:29 structure correspond to the (1-10), (100), (110) and (111) reflections in the 1:5 structure. Based on the study of the magnetic anisotropy of $\text{Y}_3(\text{Fe,Ti})_{29}$ compounds [16] and the model of Courtois *et al* [16], the contribution of the Fe sublattice to the magnetic anisotropy in $\text{Y}_3(\text{Fe,Ti})_{29}$ compounds is along the [110] direction of the 1:5 structure and the Nd ($\alpha_J < 0$) and Er ($\alpha_J > 0$) sublattices prefer the [1-10] and [110] directions of the 1:5 structure, respectively. It can be seen in figure 1(b) that the EMD of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds is in a direction intermediate between the [1-10] direction favoured by the Nd sublattice and the [110] direction favoured by both the Fe and Er sublattices.

The temperature dependences of the magnetization of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds measured in a low field of 0.05 T are shown in figure 2(a) from room temperature to above the Curie temperature and in figure 2(b) from 5 K to room temperature. The derived Curie temperatures are shown in figure 3 and also listed in table 2, where it can be seen that the Curie temperature decreases with increasing Er content. In the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds, the Curie temperature is dominated by T - T interactions with a small contribution from the R - T interaction as evidenced by the slight variation in T_c that accompanies the addition of Er. In a molecular-field approximation, the magnetic-ordering temperature of R - T intermetallic compounds can be expressed as follows [17]:

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

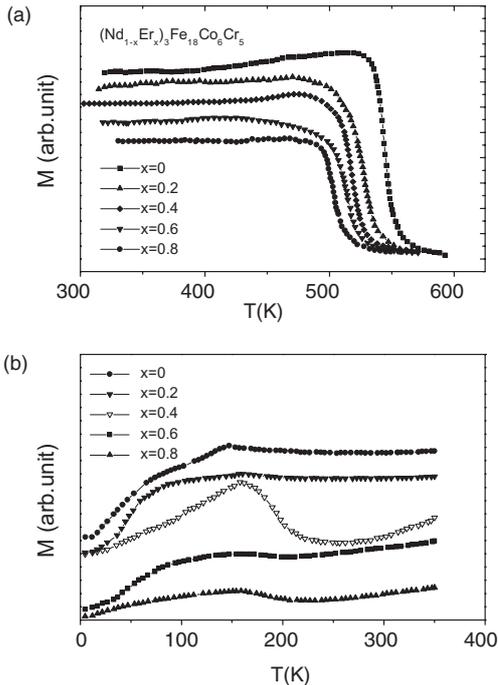


Figure 2. Thermomagnetic curves of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds measured in a field of 0.05 T in the temperature range from (a) room temperature to above the Curie temperature and (b) 5 K to room temperature.

where

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

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

with $\gamma = 2(g_J - 1)/g_J$, $2(S^*(S^* + 1))^{1/2} \mu_B$ the effective moment of Fe in the paramagnetic state, and N_T and N_R are the number of Fe and R atoms per unit volume, respectively. g_J and J_R are the Landé factor and the total angular moment of the R atoms, respectively. T_T and T_{RT} stand for the contributions to T_c from the T - T and R - T interactions, respectively. It can be seen that T_{RT}^2 is proportional to the de Gennes factor of the R ion, $G(J) = (g_J - 1)^2 J(J + 1)$, which is 1.84 for Nd and 2.55 for Er. Moreover, due to the lanthanide contraction, N_T and N_R should increase upon substitution of Er for Nd in $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds. On the other hand, n_{R-T} decreases with increasing atomic number of the R element [18]. Therefore, the dependence of T_c on the Er concentration is the result of the competition of $G_J(J)$, N_T , N_R and n_{R-T} .

It can be seen in figure 2(b) that compared with the Er-rich compounds, the magnetization increases obviously with increasing temperature for the Nd-rich compounds, as observed in the $\text{Nd}_3\text{Fe}_{24.5}\text{Cr}_{4.5}$ compound [9], which was considered as magnetohistory effect and was described based on the presence of narrow Bloch walls [19]. With further increasing temperature, all the compounds show a peak at a certain temperature in the thermomagnetic curves, which correspond to spin orientation of the EMD [20]. Such spin-reorientation transition of the EMD was also observed for $\text{Nd}_3\text{Fe}_{24.5}\text{Cr}_{4.5}$ compounds [9]. The spin-reorientation temperature T_{sr} at which the spin reorientation occurs has been

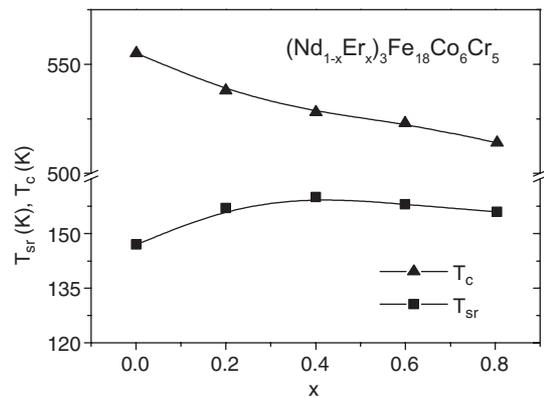


Figure 3. Curie temperature T_c (▲) and spin-reorientation temperature T_{sr} (■) as functions of Er content for $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds.

Table 2. Magnetic parameters of $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds.

x	T_c (K)	T_{sr} (K)	M_{5K} ($\mu_B/f.u.$)
0	555	147	39.5
0.2	538	157	29.8
0.4	528	160	26.3
0.6	523	158	18.9
0.8	514	156	17.8

derived for all the investigated compounds and is shown in figure 3. It can be seen that the spin-reorientation temperature increases with increasing Er content up to $x = 0.2$ and tends towards saturation for further increasing Er contents. The spin reorientation is a result of the competition of the Nd-, Er-, Fe- and Co-sublattice anisotropies that have different types of anisotropy and show different temperature dependences.

Figure 4 shows the magnetization curves at 5 K measured on the magnetically aligned samples of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds in fields applied parallel or perpendicular to the aligned direction. The values of the saturation magnetization are shown in figure 5 where it can be seen that the saturation magnetization decreases with increasing Er content due to the antiferromagnetic coupling between the Er and Nd moments. Based on a simple dilute

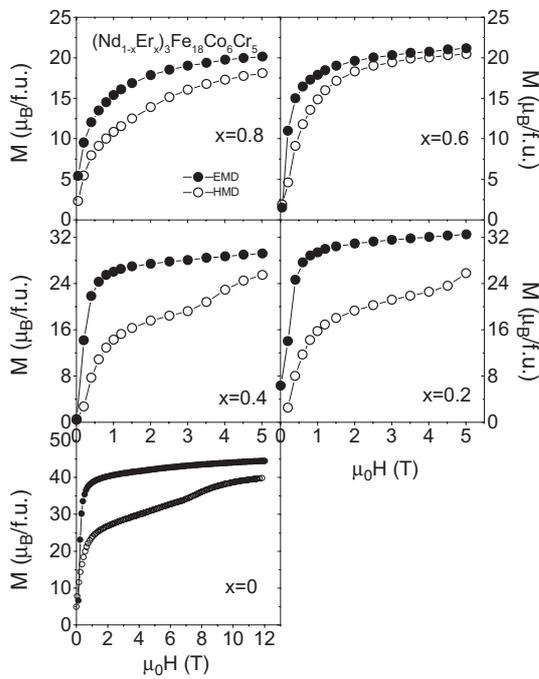


Figure 4. Isotherms at 5 K for $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds with the external field applied either parallel (●) or perpendicular (○) to the alignment direction of the powder samples.

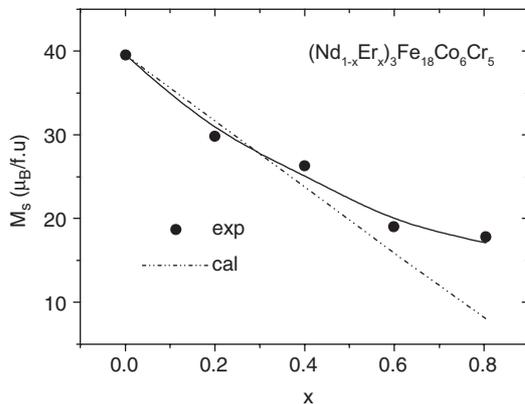


Figure 5. Experimental (●) and calculated (---) saturation magnetization as functions of Er content for $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds.

model and supposing that the magnetic moments of Er are antiparallel to those of Nd, Fe and Co moments, the saturation magnetization M_s of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds at 5 K is expressed by

$$M_s = M_0 - 3x(\mu_{\text{Nd}} + \mu_{\text{Er}}) \quad (2)$$

where M_0 , μ_{Nd} and μ_{Er} represent the saturation magnetization of $\text{Nd}_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ and the average free-ion magnetic moment of Nd and Er, respectively. The calculated results are also presented in figure 5 as a dashed line. The difference between the experimental and calculated values may be due to deviation of the R moments in the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds from the free-ion moments.

In figure 4 anomalies can be seen in the hard magnetization curves of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds with $x = 0, 0.2$ and 0.4 . These anomalies correspond to a first-order magnetization process (FOMP) [21, 22], which indicates a magnetic-field-induced spin phase transition from one spin phase to another. This transition process can be understood as follows: when the field is applied parallel to the hard magnetization direction of the samples, the direction of magnetization of the samples may first turn to the *a*- or *c*-axis and then turn to the *b*-axis, the hard magnetization direction. The critical fields for the FOMP were determined to be 8 T, 4.5 T and 3.5 T for $x = 0, 0.2$ and 0.4 , respectively.

4. Conclusion

All $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds with $x = 0.0-0.8$ are of single phase and crystallize in $\text{Nd}_3(\text{Fe,Ti})_{29}$ -type of structure with monoclinic symmetry and space group $A_{2/m}$. Substitution of Er for Nd in $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds leads to a decrease of the unit-cell volume due to the lanthanide contraction. All $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds possess planar anisotropy at room temperature. The Curie temperature shows a tendency to decrease with increasing Er content. A spin-reorientation transition is observed in all compounds. The spin-reorientation temperature T_{sr} increases with increasing Er content up to $x = 0.2$ and tends towards saturation with further increasing Er content. The saturation magnetization at 5 K of the $(\text{Nd}_{1-x}\text{Er}_x)_3\text{Fe}_{18}\text{Co}_6\text{Cr}_5$ compounds decreases with increasing Er content. A FOMP is observed for $x = 0, 0.2$ and 0.4 in the hard-direction magnetization curves, in the field range used.

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