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Effect of Al on the structural and magnetic properties of $Er_2Co_{17-x}Al_x$ compounds

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Abstract

Powder x-ray diffraction and magnetization measurements were employed to investigate the effect of Al on the structural and magnetic properties of $Er_2Co_{17-x}Al_x$ compounds with $0 \leqslant x \leqslant 4$. Al substitution leads to a monotonic increase in the lattice parameters and an approximately linear decrease in the saturation magnetization. The Curie temperature is also found to decrease with Al concentration. Large thermomagnetic irreversibility seen in these compounds has been attributed to the domain wall pinning effect. The temperature variation of magnetization shows compensation points in the compounds with $x = 3$ and 4. The field-cooled magnetizations of these two compounds in low fields are found to be negative at temperature below the compensation temperatures, which has been explained on the basis of the temperature variations of the exchange coupling strengths of Er and Co sublattices. The easy magnetization direction at room temperature is found to be along the c-axis in all the compounds. The first order anisotropy constant at room temperature is found to increase with Al concentration, which has been attributed to the increase in the axial anisotropy of both the Er and Co sublattices as a result of Al substitution.

1. Introduction

During the past few decades R_2T_{17} (R = rare earth, T = Fe, Co) compounds have been extensively studied concerning their structure and intrinsic magnetic properties, especially the magnetocrystalline anisotropy ([1–4] and references therein). Several studies have been reported on the effects of the various elemental substitutions (such as Al, Ga, Si) for Fe in R_2Fe_{17} compounds on the formation, structure and magnetic properties ([5–7] and references

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therein). The results indicate that these non-magnetic atoms play an important role in determining the Curie temperatures (T_C) and the easy magnetization direction (EMD) of these compounds. Compared to the R_2Fe_{17} system, R_2Co_{17} compounds have the advantage that their Curie temperatures are quite high. Therefore, enhancing the uniaxial magnetocrystalline anisotropy is the main concern in this latter series of compounds. Magnetocrystalline anisotropy is of great importance from the point of view of applied research in the case of permanent magnet materials. A strong uniaxial anisotropy is a pre-requisite for achieving good hard magnetic properties. Substitution of nonmagnetic elements for Co has been found to have a profound influence on the magnetic properties in general and magnetic anisotropy in particular, in various R_2Co_{17} compounds. Recently, there have been reports on the substitution of Si and Ga in the Er_2Co_{17} compound [8, 9]. In order to gain further insight into the fundamental aspects of the magnetism in these compounds and also to search for novel hard magnetic materials, we have studied the effect of Al substitution for Co in Er_2Co_{17}. In this paper we report the structural and magnetic properties of $Er_2Co_{17-x}Al_x$ compounds with $0 \leqslant x \leqslant 4$.

2. Experimental details

All the compounds ($x = 0, 1, 2, 3, 4$) were prepared by arc melting the constituent elements of at least 99.9% purity in argon atmosphere. The ingots were melted several times to ensure homogeneity. Subsequently, they were annealed in high purity argon atmosphere at 900 °C for a week. Lattice parameters were determined from the x-ray diffraction (XRD) patterns taken on powder samples using Cu Kα radiation. Samples for magnetically aligned XRD measurements were prepared by mixing the powder with an epoxy resin and allowing it to harden in a magnetic field (H) of 10 kOe, applied parallel to the surface of the sample holder. Powder x-ray diffraction patterns of magnetically aligned samples were used to determine the EMD of the compounds. Magnetization measurements were carried out using a vibrating sample magnetometer/SQUID magnetometer in the temperature range 5–950 K, up to a maximum field of 60 kOe. Saturation magnetization (M_S) was obtained by plotting M against $1/H$ in the high field part of the magnetization curves and extrapolating the plot to the $1/H$ axis. Thermo magnetic analysis was performed from 5 to 300 K, both under zero-field-cooled (ZFC) and field-cooled (FC) modes. In the former case, the samples were cooled in the absence of a field and the magnetization was measured during warming by applying a nominal field such as 500 Oe. In the FC mode, the sample was cooled in the presence of a field and the magnetization was measured during warming, under the same field. The anisotropy field was estimated by measuring the magnetization on magnetically oriented cylindrical samples. The samples were oriented in a field of 10 kOe and the magnetization was measured by applying the field in directions parallel and perpendicular to the aligning field.

3. Results and discussion

Powder x-ray diffraction patterns show that the compounds with $x = 0, 1, 2$ are single phase and crystallize in the hexagonal Th_2Ni_{17} type structure. The compounds with $x = 3$ and 4 show coexistence of both hexagonal Th_2Ni_{17} and rhombohedral Th_2Zn_{17} type structures. From the Rietveld refinement (using the Fullprof Suite program) of the powder XRD data, it is found that the volume fractions of the rhombohedral phase for $x = 3$ and 4 are 24.8% and 24.9% respectively. Compounds with $x > 4$ always showed other impurity phases, in addition to the 2:17 phases, and therefore were not used for the present study. In this context, it is of interest to note that $Er_2Co_{17-x}Si_x$ compounds have a preference to crystallize in the Th_2Ni_{17}-type structure [8] while the $Er_2Co_{17-x}Ga_x$ compounds crystallize in the Th_2Zn_{17}-type

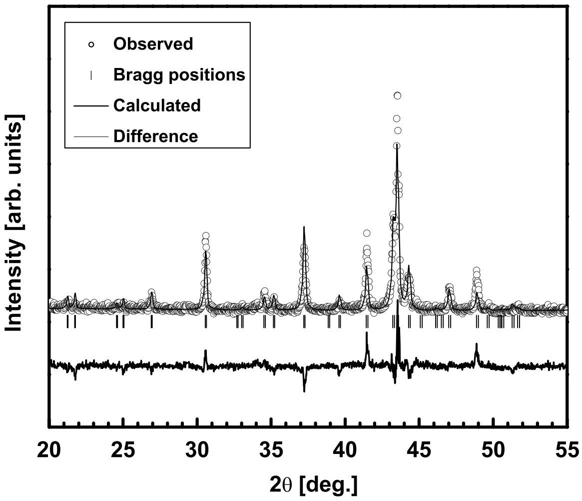

Figure 1. Rietveld fitted XRD pattern of $Er_2Co_{15}Al_2$. Both the observed data and the calculated plots are shown. The plot at the bottom corresponds to the difference between the observed and the calculated intensities.

Table 1. Lattice parameters (a, b and c), unit cell volume (V), saturation magnetization (M_S) and Curie temperature (T_C) in $Er_2Co_{17-x}Al_x$ compounds.

x	$a = b$ (Å)	c (Å)	V (Å^3)	M_S (at 5 K) (emu g^{-1})	M_S (at 300 K) (emu g^{-1})	T_C (K)
0	8.328	8.126	488.06	34	86	1199[a]
1	8.335	8.146	490.08	27	79	—
2	8.362	8.173	494.90	22	70	903
3	8.393	8.180	499.00	16	56	743
4	8.456	8.184	506.77	—	43	681

[a] Taken from [9].

structure for $x < 8$ [9]. These variations may be due to the differences in the ionic radii of the substituents.

Figure 1 shows the Rietveld refined XRD pattern of $Er_2Co_{15}Al_2$ taken at room temperature (RT). Similar plots were obtained for other compounds as well. The lattice parameters and the unit cell volumes calculated using these plots are listed in table 1. As can be seen, there is an increase in the lattice parameters and the unit cell volume with increase in Al concentration.

Magnetization measurements show that all the compounds are ferrimagnetic, as revealed by the fact that the saturation magnetization at 5 K is less than that at 300 K. From table 1, it can be seen that the M_S values decrease with Al substitution, both at 5 K as well as at 300 K. A similar variation has been reported in $Er_2Co_{17-x}Ga_x$ compounds [9]. This is due to the magnetic dilution effect as well as the charge transfer effect involving the Al and the 3d band of Co [10].

The M–H isotherms (figure 2) show that all the compounds are magnetically ordered at room temperature, which means that the Curie temperatures are all above RT. Since the upper limit of temperature in our high temperature magnetization set-up is 950 K, we could not get the T_C of the compounds with $x = 0$ and 1, whose T_C values are above 950 K. The values obtained

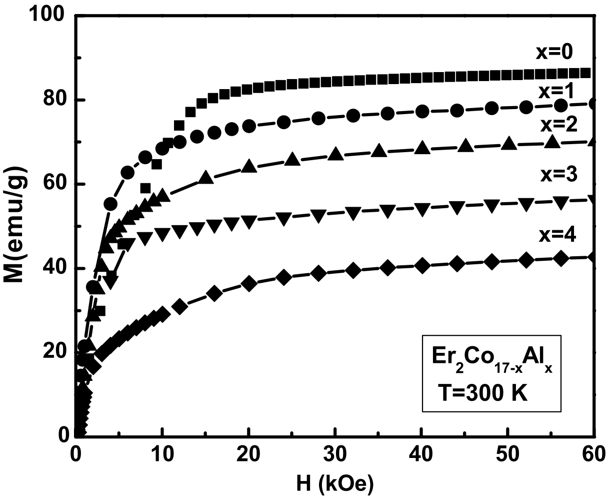

Figure 2. M–H isotherms of $Er_2Co_{17-x}Al_x$ compounds at RT as a function of Al concentration (x).

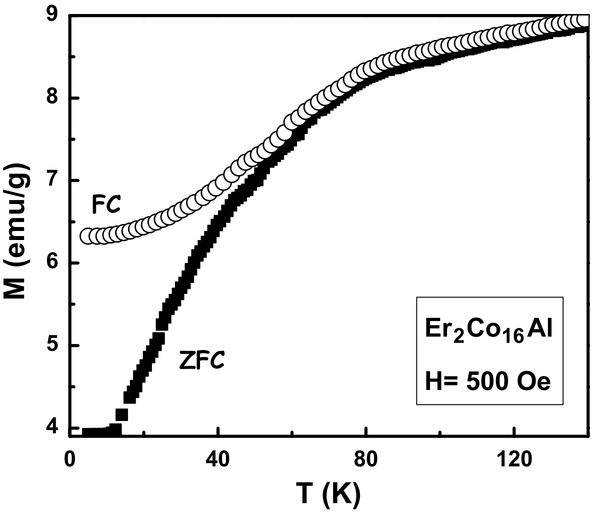

Figure 3. Temperature variation of magnetization under FC and ZFC modes in $Er_2Co_{16}Al$ in a field of 500 Oe.

for the compounds with $x = 2$, 3 and 4 are given in table 1. For comparison, we have taken the T_C of Er_2Co_{17} from the literature [9]. It can be seen that the T_C decreases with increase in Al concentration, which is mainly due to the weakening of the Co–Co exchange interaction. A similar reduction has been reported in Si and Ga substituted compounds as well [8, 9].

The temperature variations of magnetization both under ZFC and FC modes are shown for the compound with $x = 1$ in figure 3, for an applied field of 500 Oe. A large difference between the ZFC and FC magnetization, known as thermomagnetic irreversibility, is observed in both these compounds. A similar observation has been made in the compounds with $x = 0$ and 2 as well. Though the thermomagnetic irreversibility in general is observed in magnetically frustrated systems, it can also arise due to the domain wall pinning effects.

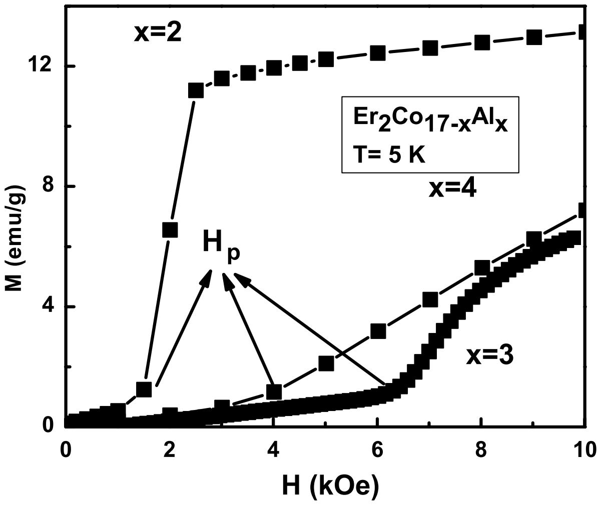

Figure 4. M versus H plots showing the domain wall pinning effect in $Er_2Co_{17-x}Al_x$ compounds at 5 K.

Since it is quite unreasonable to think of magnetic frustration in these compounds, we attribute the thermomagnetic irreversibility purely to the domain wall pinning [11]. Thermomagnetic irreversibility would arise in an anisotropic system if the domain walls encounter an impediment or barrier energy. Since the domain wall width is inversely proportional to the anisotropy constant, hard magnetic materials will have generally narrow domain walls which are more susceptible for the pinning. In the present case, an additional contribution to the pinning arises from the nonmagnetic Al ions, which act as pinning centres. Domain wall pinning can lead to large intrinsic coercivity, which is a very important parameter for permanent magnet materials [12, 13].

The domain wall pinning effect is also manifested in the M–H isotherms at low temperatures, as shown in figure 4. As is evident from this figure, up to a critical field known as the propagation field (H_p), the magnetization remains very small, and only at applied fields above H_p does it increase considerably. We attribute this behaviour to the domain wall pinning, which also causes considerable remanence in the M–H isotherms of these compounds.

Another interesting feature can be seen in figure 5, which shows the temperature variation of ZFC and FC magnetization data of the $Er_2Co_{14}Al_3$ in a field of 500 Oe. A similar plot has been obtained for $Er_2Co_{13}Al_4$ as well. While the ZFC magnetization remains positive throughout, the FC magnetization becomes negative for temperatures below 36 K for $x = 3$ and below 49 K for $x = 4$. However, when the samples are cooled in higher fields (20 and 50 kOe), as given in the inset of figure 5, the FC magnetization does not become negative, but shows a minimum at about 40 and 28 K in the compounds with $x = 3$ and 4, respectively. No other compound of this series showed a negative trend or a minimum in their M–T data down to 5 K. Bing *et al* [9] have made similar observations in the magnetization behaviour of $Er_2Co_{17-x}Ga_x$ compounds with $x > 3$.

The unusual behaviour seen in the M–T plots of these two compounds ($x = 3$ and 4) can be explained as follows. Since Er is a heavy rare earth, the coupling between Er and Co sublattice magnetic moments is antiparallel. It is also a well established fact that generally R sublattice anisotropy is dominant at low temperatures and decreases rather rapidly with temperature. On the other hand, the Co-sublattice anisotropy is quite strong even at room

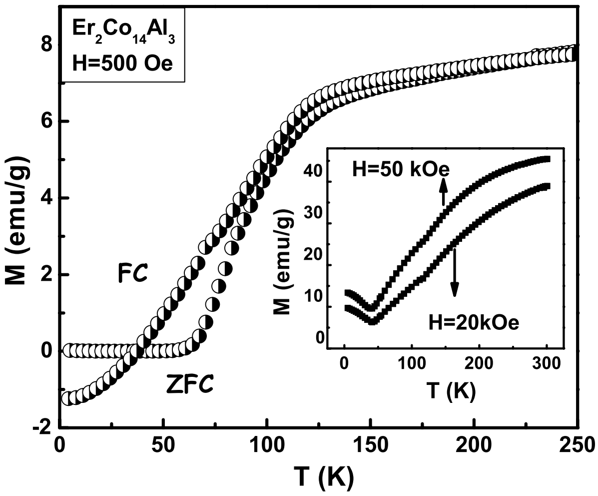

Figure 5. Temperature dependence of FC and ZFC magnetization in $Er_2Co_{14}Al_3$ in a field of 500 Oe. Insets show the variation of FC magnetization in fields of 20 and 50 kOe.

temperature and is fairly constant over a wide temperature range, because of the stronger Co–Co exchange. At 300 K, when the field is first applied to the sample in the FC mode, due to the stronger exchange coupling of the Co sublattice, the Co moments align parallel to the applied field. As the temperature is reduced, the antiparallel coupling between the Er and Co moments becomes stronger and the net magnetic moment will be given by the difference between the two sublattice contributions. The net magnetic moment can be written as

$$\mu_{\text{total}} = 17\mu_{\text{Co}} - 2\mu_{\text{Er}}. \tag{1}$$

Since the Co-sublattice contribution is greater, the net magnetic moment will be parallel to the applied field and hence positive. However, on diluting the Co sublattice with nonmagnetic Al, the magnetic moment of the Co sublattice decreases and reaches a stage where the two sublattice contributions become nearly equal, resulting in the compensation point. On further reduction of the Co-sublattice magnetic moment, the net magnetic moment will be dictated by the Er sublattice and hence the sign of the net magnetization becomes negative. This is true only if the effective field seen by the Er sublattice is less than the anisotropy field associated with the Er sublattice. In the present case, the Er and Co-sublattice magnetic moments become nearly equal only in two compounds, namely the ones with $x = 3$ and 4. This is the reason for observing the negative magnetization at low fields in these two compounds. The negative magnetization is retained up to 36 K in $x = 3$ and up to 50 K in $x = 4$, as the temperature is increased. This trend is expected because the dilution of the Co sublattice becomes larger at $x = 4$ and hence the Er-sublattice magnetic moment would dominate over a larger temperature range. In contrast, when the samples are cooled in a higher field, i.e., 20 or 50 kOe, we see that the magnetization does not become negative, but shows a minimum (inset of figure 5), which corresponds to the compensation point. This is due to the fact that when the applied field approaches the anisotropy field the Er sublattice tries to align in the direction of the applied field and consequently the magnetization remains positive throughout the temperature range of 5–300 K, but only shows the compensation point. It is also observed that the compensation point moves to lower temperatures when the applied field is increased from 20 to 50 kOe, in both the compounds. In addition to the substituted 2:17 compounds, the observation of negative magnetization has been reported in other materials as well [14–16].

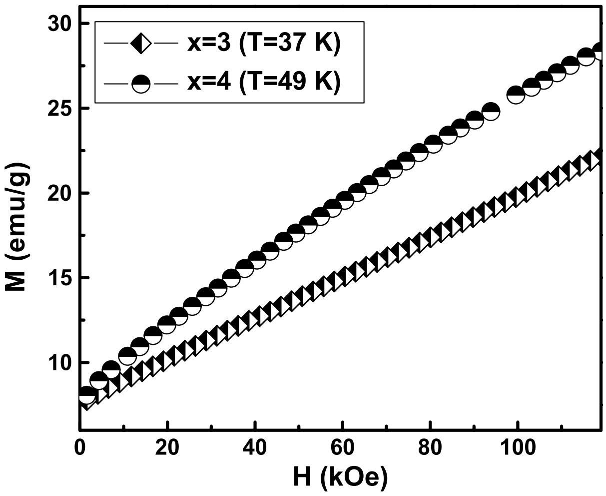

Figure 6. M–H isotherms of $Er_2Co_{17-x}Al_x$ compounds with $x = 3$ and 4 near their compensation temperatures.

Table 2. Intersublattice-molecular-field coefficient (n_{RT}), exchange coupling constant (J_{RT}), anisotropy field ($\mu_0 H_A$) and anisotropy constant (K_1) in $Er_2Co_{17-x}Al_x$ compounds.

x	n_{Er-Co} (T.f.u./μ_B)	J_{Er-Co} ($\times 10^{-23}$ J)	$\mu_0 H_A(T)$ (at RT)	K_1 (K/f.u.) (at RT)
0	—	—	1.6	9.7
1	—	—	1.8	10.0
2	—	—	2.6	12.5
3	3.7	18.4	3.6	13.5
4	2.6	12.8	—	—

From the above discussion, we note that the Er-sublattice magnetic moment remains antiparallel to that of the Co sublattice and that the magnetization of the compound changes considerably only when the applied field exceeds a critical field $\mu_0 H_{cr} = n_{RT}|m_T - m_R|$. Here n_{RT} represents the intersublattice-molecular-field coefficient and m_T and m_R represent the T- and R-sublattice moments, respectively. The intersublattice-molecular-field coefficient can be obtained from the reciprocal of the slope of the M–H plots obtained in high fields. Since our measurements were limited to a maximum field of 100 kOe, we could not calculate n_{RT} using this method. However, we could calculate n_{RT} for the compounds with $x = 3$ and 4, which show compensation points in their M–T data. This is based on a recent model [17, 18], which suggests that at the compensation temperature the slope of the M–H isotherms measured even in modest fields (~100 kOe) gives a reasonable estimate of n_{RT}. Figure 6 shows the M–H plots of the compounds with $x = 3$ and 4 measured at temperatures close to their compensation temperatures. Using these plots, we have calculated the n_{RT} (i.e. n_{Er-Co}) values and these are shown in table 2. The intersublattice exchange coupling constant $J_{RT}(J_{Er-Co})$ is related to n_{RT} as

$$J_{ErCo} = -N_{Co}\mu_B^2 g_{Er} n_{ErCo}/Z_{ErCo}(g_{Er} - 1) \qquad (2)$$

where Z_{Er-Co} is the number of Co neighbours of an Er atom, N_{Co} is the number of Co atoms per formula unit and g_{Er} is the Landé factor. Supposing that the Co and Al atoms are statistically distributed over the T sublattice, and taking $N_{Co} = 17 - x$, $Z_{ErCo} = 19 \times (17 - x)/17$ and

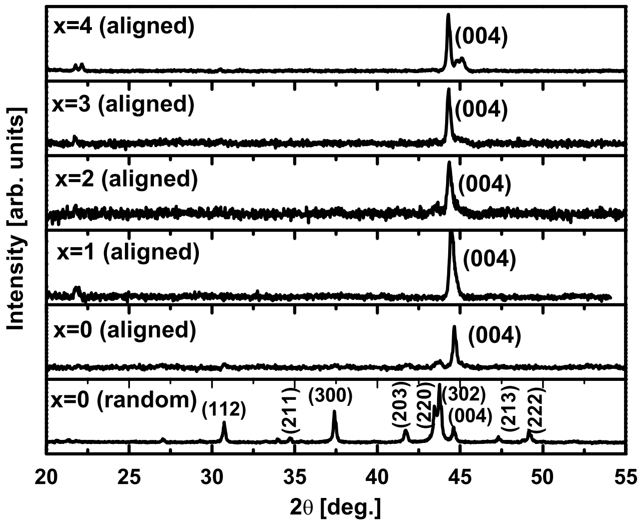

Figure 7. XRD patterns of magnetically aligned samples of $Er_2Co_{17-x}Al_x$ at room temperature.

$g_{Er} = 6/5$ we have calculated J_{Er-Co}, which is also listed in table 2. The decrease in n_{RT} and J_{RT} with x reflects the magnetic dilution of the Co sublattice by Al.

In order to study the variation of anisotropy in $Er_2Co_{17-x}Al_x$ compounds as a function of Al concentration, we have taken the powder x-ray diffractograms of the magnetically aligned samples at RT (figure 7). By comparing these diffractograms with those of the random powders (shown at the bottom of figure 7), we find that there is a considerable enhancement in the intensity of the (004) peak and a complete suppression of other peaks, in all the compounds. This implies that the easy magnetization direction lies along the c-axis both in the parent compound as well as in the Al-substituted compounds.

In R–T intermetallic compounds, both the R sublattice and the T sublattice contribute to the total anisotropy. The anisotropy of the R sublattice is induced by the crystalline electric field (CEF), and in the lowest order approximation it can be expressed as

$$K_1(R) = -\left(\frac{3}{2}\right)\alpha_J \langle r_{4f}^2\rangle \langle 3J_z^2 - J(J+1)\rangle A_2^0 \tag{3}$$

where α_J is the second order Stevens factor, r_{4f} is the mean radius of the 4f electron cloud, J is the total angular momentum and A_2^0 is the second order crystal field parameter. α_J is determined by the degree of asphericity of the 4f electron charge density whereas A_2^0 is mainly determined by the charge density around the 4f ion [19]. In general K_1^R is strongly temperature dependent. On the other hand, the anisotropy of the T sublattice originates from the incompletely quenched angular momentum of the 3d electrons. In Er_2Co_{17} compounds, the Er sublattice has a positive anisotropy constant and favours easy-axis anisotropy, due to the positive value α_J (of the Er^{3+} ion) and the negative value of A_2^0 for the investigated 2:17 structure. Zhang *et al* have reported that the EMD of Y_2Co_{17} is planar at RT, which implies that the Co-sublattice anisotropy constant is negative [20]. Thus it is reasonable to assume that K_1^{Co} is negative for other R_2Co_{17} compounds as well, at room temperature [21–24]. The fact that the EMD of Er_2Co_{17} is along the c-axis at RT implies that the Er-sublattice anisotropy is stronger than the Co-sublattice anisotropy. Since the EMD is axial in all the Al-substituted compounds also, it may be inferred that the total anisotropy constant remains positive, even after Al substitution.

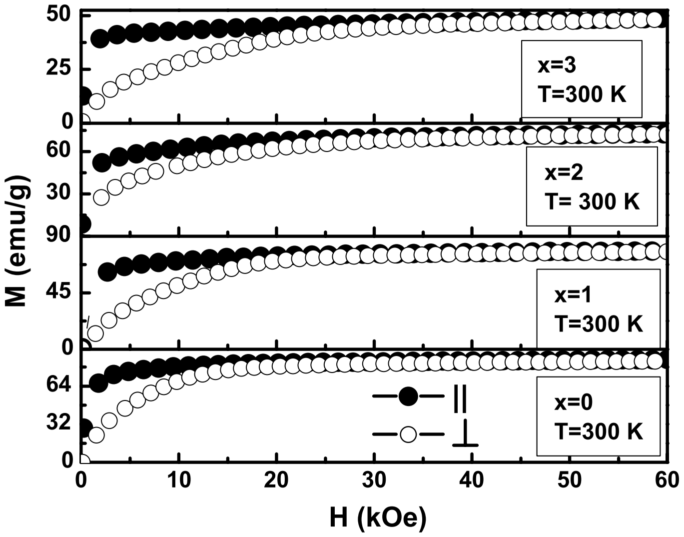

Figure 8. Magnetization plots of magnetically aligned $Er_2Co_{17-x}Al_x$ compounds with $x = 0$, 1, 2 and 3 for the fields applied parallel and perpendicular to the alignment direction, at room temperature.

In order to study the variation of anisotropy as a function of Al concentration, we have also calculated the anisotropy field (H_a) and the anisotropy constant (K_1). This has been done using the magnetization data collected on magnetically aligned samples, with the field applied both parallel and perpendicular to the alignment direction, at RT. Figure 8 shows the M–H plots of the aligned samples with $x = 0$, 1, 2 and 3. An estimate of the anisotropy field has been obtained from the point of intersection of the parallel and perpendicular plots. The anisotropy constant has been calculated using the relation $H_a = 2K_1/M_S$. The variation of H_a and K_1 with Al concentration is also shown in table 2. The H_a value obtained for the parent compound Er_2Co_{17} matches the value obtained by Merches *et al* [25]. It can be seen that both H_a and K_1 increase with Al concentration, which suggests that Al substitution has indeed resulted in an increase in the total axial anisotropy of these compounds. This increase may be due to a reduction in the planar anisotropy of the Co sublattice and/or an increase in the axial anisotropy of the Er sublattice. Anisotropy studies on $Y_2Co_{17-x}Al_x$ compounds have shown that the anisotropy of the Co sublattice has indeed changed from planar to axial upon Al substitution [20]. Therefore, it is reasonable to assume that a similar situation exists for the Co sublattice in the present case as well. The increase in the Co anisotropy may be qualitatively explained on the basis of the changes in the 3d band dispersion [26, 27], as explained below.

It is well known that in 2:17 compounds the dumb-bell sites of Co along the c-axis, whose bond lengths are smaller than those in the basal plane, are responsible for the planar anisotropy [28]. However, with Al substitution, some of these dumb-bell sites are occupied by Al [28]. Furthermore, there is an increase in the c-lattice parameter, which will cause an increase in the axial bond lengths. Another effect of Al substitution is the increase in the population of the 3d band of Co. All the above factors may result in a modification in the 3d band dispersion, thereby changing the anisotropy. In general, it has been observed that when the axial bond lengths are greater the band dispersion is such that the T-sublattice anisotropy prefers to be axial, as seen in the case of YCo_5 [26, 27]. Therefore, we feel that there should be some contribution from the Co sublattice towards the total axial anisotropy in the Al-substituted compounds.

Another factor to be considered in this context is the contribution from the Er sublattice to the enhancement of the total anisotropy. The change in the rare earth sublattice anisotropy upon Al substitution mainly arises due to the changes in the sign and/or magnitude of the crystal field parameter A_2^0. An increase in the magnitude of A_2^0 without causing a change in its sign is observed in $R_2T_{17-x}Al_x$ compounds [20, 29]. Increased A_2^0 values resulting from the Al substitution would lead to an increase in the positive value of $K_1(R)$. Therefore, based on our studies, we attribute the increase in the total axial anisotropy of $Er_2Co_{17-x}Al_x$ compounds to the enhancement of the axial anisotropy of both Er and Co sublattices.

In summary, Al-substituted Er_2Co_{17} compounds are found to exhibit interesting hard magnetic properties, with a maximum anisotropy field of 3.6 T. The anisotropy field obtained in the present case is more than the value of 2.6 T [8] obtained in Si-substituted Er_2Co_{17} compounds. Though the EMD has been shown to be along the *c*-axis in $Er_2Co_{17-x}Ga_x$ compounds, the anisotropy fields and the anisotropy constants are not reported. Given the fact that the saturation magnetization and T_C values are comparable in all these three different substitutions, it is clear that Al substitution leads to a considerable enhancement in the hard magnetic properties of $Er_2Co_{17-x}Al_x$ compounds. A significant domain wall pinning effect seen in these compounds may also be responsible for the enhanced hard magnetic properties. Since there are no reports on the domain wall pinning effect in the Si- and Ga-substituted compounds, it is not possible to compare the contribution of this effect to the overall hard magnetic behaviour of these compounds.

4. Conclusion

Substitution of Al gives rise to some interesting magnetic properties in $Er_2Co_{17-x}Al_x$ compounds. While the saturation magnetization and the Curie temperature decrease with Al concentration, the uniaxial anisotropy increases considerably. The domain wall pinning effect seems to be crucial in determining the magnetization process. Negative magnetization accompanied by compensation points has been observed in the compounds with $x = 3$ and 4. Considering the various magnetic properties, it seems that the present series of compounds is promising from the points of view of both fundamental importance as well as applied research in the area of permanent magnet materials.

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