Magnetic Structure of E rFeO₃ below 4.5 K

G. Gorodetsky~

Department of Physics, University of the Negev, Beer-Sheva Department of Electronics, The Weizmann Institute of Science, Rehovot, Israel

R. M. Hornreich^{*} and I. Yaeger^{*} Department of Electronics, The Weizmann Institute of Science, Rehovot, Israel

H. Pinto, G. Shachar, and H. Shaked

Department of Physics, Nuclear Research Center-Negev, Beer-Sheva, P.O.B. 9001, Israel (Received 6 April 1973)

Using long-wavelength ($\lambda \approx 2.4$ Å) neutrons, diffraction patterns of ErFeO₃ were taken at 140, 4.2, and 1.5°K. Two possible structures for the $Fe³⁺$ spin system at 1.5°K were found to be consistent with the neutron data. Both of the possible structures belong to the antiferromagnetic G configuration. In the first, the Fe^{3+} antiferromagnetic axis is in the orthorhombic a b crystallographic plane at an angle of $33^\circ \pm 4^\circ$ to the b axis. In the second, the Fe³⁺ antiferromagnetic axis is in the b c plane at an angle of $51^{\circ} \pm 8^{\circ}$ to the b axis. Magnetization and torque measurements showed that the spontaneous moment is along the a axis for all temperatures below that of liquid nitrogen. It exhibits a sharp peak at $T \sim 4.5$ K, thereafter dropping sharply with decreasing temperature. This result is consistent only with the latter of the two possible structures. Thus, for $T < 4.5$ K, the Fe³⁺ spins are in essentially a mixed G_{yx} mode rather than the G_{xy} mode suggested previously. It is concluded that a

reorientation of the Fe³⁺ antiferromagnetic axis from the c axis (at 4.5°K) toward the b axis takes place as the temperature is decreased below 4.5 K. Possible mechanisms responsible for this low-temperature spin reorientation are reviewed.

I. INTRODUCTION

The rare-earth orthoferrites, $RFeO₃$, crystallize in an orthorhombically distorted perovskite structure.¹ This structure belongs to the space group D_{2h}^{16} -Pbnm with R in the (4c) and Fe in the (4b) positions as shown in Fig. 1. The possible magnetic configurations with symmetry isomorphic² to D_{2h}^{16} were defined by Wollan and Koehler³ and by Bertaut⁴ and are listed in Table I. The possible magnetic structures with symmetry isomorphic⁵ to D_{2h}^{16} have also been classified by Bertaut according to their magnetic symmetry, and this classification is given in Table II.

Immediately below the ordering temperature the $Fe³⁺$ magnetic structure in the rare-earth (RE) orthoferrites is that of ^a "canted antiferromagnet. " More specifically, they exhibit an essentially antiferromagnetic structure G_x (i.e., a \bar{G} configuration with its spin axis parallel to the a crystallographic axis) with an accompanying weak ferromagnetic structure F_{μ} , e^{-8} As seen in Table II, G_x and F_{μ} belong to the magnetic space group $Pb'n'm$. This means that the product $F_{\mu}G_{\mu}$ is invariant under the operations of the symmetry elements of the paramagnetic space group $1'Pbnm$.

As the temperature is lowered, the $Fe³⁺$ spins in some of the RE orthoferrites exhibit a spin reorientation. That is, they undergo a transition to a different magnetic structure, which is usually the canted antiferromagnetic structure $G_{\boldsymbol{\epsilon}}F_{\boldsymbol{\epsilon}^*}^{\boldsymbol{6},7}$ $G_{\boldsymbol{\epsilon}}$ and F_x belong to $Pbn'm'$ and the product G_xF_x is therefore also $1'Pbnm$ invariant.

Turning to the case of $ErfeO₃$, the weak ferromagnetism of this compound was studied by Bozorth et $al.$ $\mathbf{^6}$ and its antiferromagnetic structure was first investigated by Koehler et $al.^9$ These and later

FIG. 1. Orthorhombic unit cell of $RFeO₃$. Only the R^{3+} and Fe^{3+} ions are shown.

 $\overline{\mathbf{8}}$

TABLE I. Definition of magnetic configurations (Refs. 3 and 4). $\overline{3}$ and $\overline{4}$).

Configuration	site				range (°F	
designation					-100 62 _C $\overline{4}$ 90	
F					$4.5 -$ $\mathbf{1}$	
G						
А	┿			᠇		
C					In det	

studies by other workers^{7,8} led to the conclusions on the magnetic structure of $ErfeO₃$ summarized in Table III. A comparison with Table II shows that the magnetic structure above $4.5 \text{ }^{\circ}\text{K}$ is in complete accord with the considerations outlined above. However, the situation below $4.5\,^{\circ}\text{K}$ deserves special consideration: The components $C_*(\text{Er}^{3*})$ and $G_{\rm y}({\rm Fe}^{3+})$ belong to *Pbnm*, $G_{\rm x}({\rm Fe}^{3+})$ to $Pb'n'm$, and $F_x(\text{Fe}^{3*})$ to Pbn'm'. The coexistence of these components thus implies a decrease from orthorhombic to monoclinic symmetry (Table III). Furthermore, it is difficult to see how an antiferromagnetic component G_x and a ferromagnetic component F_x will coexist along the common a crystallographic axis. In view of these difficulties, we have decided to reexamine the magnetic structure of ErFeO, below 4.5 $\mathrm{^{\circ}K}$.

II. MAGNETIC STRUCTURE AT 1.5'K—NEUTRON DIFFRACTION

Neutron $(\lambda \sim 2.4 \text{ Å})$ diffraction patterns of a polycrystalline sample of $ErfeO₃$ were taken at 140, 4.2, and 1.5 K . The resulting patterns are shown in Fig. 2. The relatively long neutron wavelength was used in order to obtain good angular resolution. Oriented $(\pm 3.5^{\circ})$ graphite, 50 mm thick, was placed in the beam between the monochromator and the sample in order to eliminate any high-order contamination in the beam.¹⁰ The observed integrated intensities of these pattern are listed in Table IV.

TABLE II. Possible magnetic structures in the (4b) and (4c) positions in D_{2h}^{16} with symmetry isomorphic (Ref. 5) with D_{2h}^{16} . (G_{α} with $\alpha = x$, y, z designates a G configuration with the spin axis along a, b, c , respectively, etc.)

Space group	(4b)	(4c)	
Pbnm	$A_x G_y C_z$	\cdots \cdots C_{z}	
Pbn'm'	$F_x C_y G_z$	$Fx Cy$	
Pb'nm'	$C_x F_y A_z$	$C_x F_v$	
Pb'n'm'	$G_x A_y F_z$	\cdots \cdots F_{σ}	
Pb'n'm'		$G_{\mathbf{r}}A_{\mathbf{v}}$	
Pb'nm		$\cdots \cdots A_{n}$	
Pbn'm		$\cdots \cdots G_{n}$	
Pbnm'		$A_x G_v \cdots$	

TABLE III. Published magnetic structures of ErFeO3.

Temperature range $(^{\circ}K)$	Fe(4b) sublattice	Er(4c) sublattice	Space group	Ref.
-100 62 _f	$\cdots F_{n}$ G.		Pb'n'm	$6 - 10$
-4.5 90	$\cdots G$. F_{\star}		Pbn'm'	$6 - 10$
$4.5 - 1.25$	$G_x F_x G_y \cdots$	$\cdots \cdots c_{n}$	$P2_1/m$	$6 - 9$

In determining the magnetic structure at $1.5\textdegree K$ it was assumed that (i) the magnetic structure of the Er lattice is C_{s} , as was found by Koehler et al.⁹ This identification is evident from the appearance of the $\{010\}$ and $\{100\}$ diffraction peaks in Fig. 2. (ii) The magnetic configuration of the Fe lattice is essentially G , as was reported by Koehler et al.⁹ This identification is evident from the appearance of the $\{011\}$ and $\{101\}$ diffraction peaks in Fig. 2. (iii) The antiferromagnetic spin axis of the Fe lattice is perpendicular to at least one of the crystal axes a , b or c . The symmetry of this class of structures is higher than that corresponding to structures with an arbitrary direction of the antiferromagnetic spin axis. The spin reorientation at ferromagnetic spin axis. The spin reorientation and $\sim 100 \text{ °K}$ in ErFeO₃ satisfies this restriction, ¹¹ and as we shall show, spin structures satisfying this restriction do exist in our case; hence, structures having a lower symmetry were not considered.

With these assumptions, the structure determination reduces to a search for that direction of the antiferromagnetic axis of the $Fe³⁺$ spin system with-

FIG. 2. Neutron $(\lambda \sim 2.4 \text{ Å})$ diffraction patterns of $ErfeO₃$ at 140, 4.2, and 1.5 °K.

in the crystallographic planes ab , ac , and bc which will best fit the observed integrated intensities. In determining the quality of a given fit, we used the following three criteria.

a. The discrepancy factor R . We defined a discrepancy factor

$$
R = 100 \sum \left| \frac{I_{\text{obs}}}{\sum I_{\text{obs}}} - \frac{I_{\text{calc}}}{\sum I_{\text{calc}}} \right| , \qquad (1)
$$

where the summations were carried out over the ${\rm integrated~intensities^{12}}$ of the four lines listed in megrated intensities of the four lines instead in Table IV at $1.5 \, \textdegree K$. Since these four lines do not contain (practically) any nuclear contribution, it was possible to calculate R as a function of two parameters: $\mu_{\texttt{Er}}/\mu_{\texttt{Fe}}$ and the angle $\vec{\mu}_{\texttt{Fe}}$ makes with one of the crystal axes. Here μ_M is the ionic magnetic moment on an M ($M = Er$, Fe) site. R was subsequently calculated for $\mu_{\texttt{Fe}}$ in the ab , ac , and bc planes for $1.2 < \mu_{\text{Er}} / \mu_{\text{Fe}} < 1.8$. The results of these calculations for the positive quadrants of the $ab, ac,$ and bc planes are shown in Fig. 3. We see that there are rather well-defined regions of low $($ < 2%) R within each of the three quadrants. These $\text{low-}R$ regions were next examined against the other criteria.

b. The temperature dependence of the intensit of the $\{101\}$ line. The $\{101\}$ line is essentially magnetic in origin, with the magnetic contribution coming only from the Fe^{3*} spin system. At 1.5°K the spin configuration is of the G type. Let α , β γ be the direction cosines of the Fe³⁺ spin ax then follows¹² that

$$
I_G^{101} \simeq 1 - \frac{\alpha^2/a^2 + \gamma^2/c^2}{1/a^2 + 1/c^2} \,, \tag{2}
$$

where I_G^{101} is the calculated integrated intensity of 101} (for a *G*-type configuration) and a , c are unitthat at 140 \degree K the structure is G_x . Hence, at 140 \degree K cell dimensions. Now, it is well known⁷ (Table III) we have

$$
I_{G_x}^{101} \simeq \frac{a^2}{a^2 + c^2} \tag{3}
$$

The experimental ratio (see Table IV)

$$
\frac{I_{obs}^{101} (1.5 \text{ °K})}{1.018 I_{obs}^{101} (140 \text{ °K})} = 2.47 \pm 0.13
$$
 (4)

is now set equal to $I_G^{101}/I_{G_r}^{101}$. The coefficient in the denominator is introduced in order to account for the nonsaturation of I_{obs}^{101} at 140 °K. For c^2/a^2 $= 2.08¹³$ we then obtain

$$
2.08\alpha^2 + \gamma^2 = 0.61 \pm 0.13
$$
 (5)

The direction cosines must also satisfy

$$
\alpha^2 + \beta^2 + \gamma^2 = 1. \tag{6}
$$

We considered, in accordance with our earlier assumptions, only those solutions of Eqs. (5) and (6) in which the spin axis was perpendicular to one of Here the spin axis is in the ab plane making an ancrystal axes. (i) For $\gamma = 0$, $\beta = \pm (0.84)$ gle of \pm (33° \pm 4°) with the b direction. (ii) For α $= 0$, $\beta = \pm (0.62 \pm 0.11)$. Here the spin axis is in the bc plane making an angle of $\pm (51^\circ \pm 8^\circ)$ with the b direction. (iii) For $\beta = 0$, no solution. Thus the criterion has therefore eliminated any possibl spin axis can not be in the ac plane. Our second lution in the ac plane. The regions allowed by the solutions (i) and (ii) are marked in Fig. 3 by pairs of dashed lines centered about the angles 33° (in the a b plane) and 51° (in the bc plane).

c. The ratio μ_{Er}/μ_{Fe} . We have calculated the ratio $\mu_{\text{Er}}/\mu_{\text{Fe}}$ from the lines $\{011\}$, $\{101\}$ (140 °K) and $\{010\}$, $\{100\}$ (1.5 °K). These lines are due to magnetic contributions from the Fe^{3+} and Er^{3+} spin

FIG. 3. Discrepancy factor, R , contours for variou $\mu_{\text{Er}}/\mu_{\text{Fe}}$ in the positive quadrants of the *ab*, *ac*, and *bc* planes. The vertical and horizontal dashed lines marl the bounds for structures satisfying cri spectively. The shaded areas mark the regions simulated by a strategies of the regions of the regions

TABLE V. Calculation of $\langle C\mu_{\mathbf{M}}^2 \rangle$.

^aWith $C\mu_H^2=I_{\text{obs}}(T)/I'_{\text{calc}}$ with $T=1.5$ and 140°K for the Er and the Fe sublattice, respectively.

^bAfter subtraction of a nuclear contribution of 2% to $I_{\rm obs}$ (140 °K).

cAfter adding a 1.8% correction to account for the nonsaturation of I_{obs} (140°K).

systems, respectively. Let us define the intensity per unit magnetic moment

$$
I'_{\text{calc}} = I_{\text{calc}} / \mu_M^2
$$

where

$$
M = \begin{cases} \text{Fe for } \{011\}, & \{101\} \\ \text{Er for } \{010\}, & \{100\} \end{cases}, \tag{7}
$$

where I_{calc} is the calculated integrated intensity.¹² We then have

$$
I_{\text{obs}} = C \mu_{\text{M}}^2 I_{\text{calc}}', \tag{8}
$$

where C is an experimental factor fixed for all lines. The numerical values of I_{obs}/I'_{calc} are given in Table V, When averaged over the respective pairs, they yield $\langle C\mu_{\texttt{Fr}}^2 \rangle = 144 \pm 3$ and $\langle C\mu_{\texttt{Fe}}^2 \rangle = 64$ $±2$, hence

$$
\mu_{\rm Er}/\mu_{\rm Fe} = (144 \pm 3/64 \pm 2)^{1/2} = 1.50 \pm 0.04. \qquad (9)
$$

The region allowed by this solution is marked in

Fig. 3 by the pair of dashed lines centered about $\mu_{Er}/\mu_{Fe} = 1.5$.

In Fig. 3, in which R is mapped for the spin axis in the positive quadrants of the $a\,b$, $b\,c$, and $a\,c$ planes, we have marked (shaded area) the two regions simultaneously satisfying criteria ² and 3. One region is in the bc plane with $\angle (\overline{\mu}, \overline{b}) = 51^{\circ} \pm 8^{\circ}$ and the other is in the a b plane with $\angle (\overline{\mu}, \overline{b}) = 33^{\circ}$ $\pm 4^{\circ}$. As for criterion 1 (low R), both of the shaded regions in Fig. 3 seem to be acceptable with the former being somewhat more favorable (lower R). Although not shown, R values were also calculated for the other six quadrants¹⁴ which contain b , i.e., $\bar{a}b$, $a\bar{b}$, $\bar{a}\bar{b}$, $\bar{b}c$, $b\bar{c}$, and $\bar{b}\bar{c}$. These calculations showed that none of the low- R regions in these quadrants satisfy criteria 2 and 3. As for the quadrants in the ac plane, an R calculation was not necessary as criterion 2 is not satisfied in the entire ac plane (no solution for $\beta=0$). Thus we conclude that there are only two structures (with $\mu_{\mathbf{r}_e}$ perpendicular to at least one of the crystal axes) that give a satisfactory fit to the diffraction data. The first, with the spin axis in the *a b* plane and $\phi_1 = \tilde{\phi}$ ($\tilde{\mu}$, \tilde{b}) $\simeq 33^\circ$, is shown in Fig. 4(a). This is essentially the structure proposed by Koehler $et al.^9$ The second somewhat more favorable structure (lower R), with the spins in the bc plane and $\phi_2 = \langle (\mu, b) \rangle \approx 51^\circ$, is shown in Fig. 4(b).

III. WEAK FERROMAGNETISM

In order to further clarify the nature of the lowtemperature spin structure, magnetization data at temperatures down to 1.9 °K and in applied magnetic fields up to 50 kG were recorded using a vibrating-sample magnetometer. In Fig. 5 we show the measured temperature dependence of the spontane-

FIG. 4. Two possible magnetic structures consistent with the 1.5°K neutron diffraction data. (a) Structure 1 is similar to that found by Koehler et al. (b) Structure 2, on the other hand, is somewhat more favorable (lower R) according to the present analysis.

FIG. 5. Temperature dependence of the spontaneous magnetic moment of $ErfeO₃$ in the a direction.

ous magnetization in the $\left[100 \right]$ direction. 15 In order to avoid a multidomain state in the crystal, a low magnetic field (50 G) was applied along the ferromagnetic axis while the measurements shown in Fig. 5 were carried out.

The observed temperature dependence is attributed 7,16 to a negative coupling coefficient between the spins of the rare-earth ions and the Fe3' cantedspin structure; i.e., the net polarizations of the $Er³⁺$ spin system is antiparallel to the weak ferromagnetic moment of the $Fe³⁺$ spins. Below the first spin reorientation region (i.e., 100-90 °K), the contribution of the Er^{3*} spins to the total magnetic moment increases at a higher rate than that of the $Fe³⁺$ spins as the temperature is decreased. As a result of this a compensation point occurs at T \simeq 45 °K where the contributions of the two spin systems to the total moment are equal and opposite. Upon further cooling, the direction of the magnetic moment reverses and its magnitude increases monotonically until a temperature of T \simeq 4.5 $^{\circ}$ K is reached. At this point, the temperature dependence of the magnetic moment changes sign and decreases quite sharply as the temperature is further decreased to 1.9 'K. However, torque measurements carried out in the 1.9 to 4.5 $\mathrm{^{\circ}K}$ temperature region do not show any angular deviation of the spontaneous magnetization away from the a crystallographic axis. Thus we conclude that the antiferromagnetic axis of the Fe³⁺ spin system is confined to the bc plane since, as is clear from Table II, there would otherwise appear a ferromagnetic moment along the c axis,

The field dependence of the magnetization at 4.2 and 2 'K, as measured along the orthorhombic axes, is shown in Fig. 6. By extrapolating the $4.2 \degree K$ set of curves to $H = 0$, one observes that the spontaneous magnetic moment lies along the a axis. At 2 'K, a metamagneticlike transition is observed in

the c direction. This effect indicates an ordering the c direction. This effect indicates an ordering of the Er^{3*} spin system along the c axis, in agreement with the neutron diffraction results of Sec. II. Note further that at 2^oK no spontaneous moment is observed along the c axis, thereby supporting our conclusion that the weak ferromagnetic moment of $ErfeO₃$ remains along the a crystallographic axis for $1.9 < T < 4.5$ °K.

IV. DISCUSSION

We have found, in Sec. II, that there are two possible structures for the Fe^{3+} spin system at 1.5 $^{\circ} \mathrm{K}$ that are in satisfactory agreement with the neutron diffraction data. In the first [structure 1, Fig. $(4(a))$, the spins lie in the ab plane with the antiferromagnetic axis at an angle of 33' to the crystallographic b axis. This is the structure first suggested by Koehler *et al*, δ and, more recently, by Pataud and Sivardiere, ¹⁷ on the basis of their respective neutron diffraction studies. However, we have seen that this structure assignment is not an unambiguous one. The alternate structure [structure 2, Fig. 4(b), in which the spins lie in the bc plane at an angle of 51° to the b axis, is also an acceptable interpretation of the diffraction data.

To distinguish between the two possible structures, we must turn to the magnetization data pretures, we must turn to the magnetization data provided in Sec. III. In structure 1, the Fe³⁺ spins are essentially in a mixed G_{xy} mode. Owing to the antisymmetric exchange interaction'6 between the $Fe³⁺$ spins, there must be a weak ferromagnetic moment in the c crystallographic direction (F_{ϵ}) associated with such a structure. On the other hand, in structure 2, the Fe^{3+} spins are essentially in a

FIG. 6. Field dependence of the magnetization of ErFe03 for fields applied along the crystallographic axes at 4. ² and 2'K.

mixed G_{vs} mode and the antisymmetric exchange will result in a weak ferromagnetic moment parallel to the *a* crystallographic axis (F_x) . Since the spontaneous magnetization is in fact along the a axis in the 1.9 to 4.5 $\,^{\circ}$ K temperature range, it follows that the low-temperature phase of $ErfeO₃$ is described by the mixed spin mode $G_{yx}F_x$ (structure 2). Thus we conclude that, as the temperature is lowered below $4.5\,^{\circ}\text{K}$, the Fe³⁺ antiferromagnetic spin axis turns gradually from the c toward the b crystallographic axis and, at $1.5\,^{\circ}\text{K}$, makes an angle of approximately 51° with the latter axis.

This rotation of the $Fe³⁺$ spins is a consequence This rotation of the $Fe³⁺$ spins is a consequence of the exchange coupling between the $Fe³⁺$ and $Er³⁺$ magnetic moments. Both Mossbauer¹⁸ and optical spectroscopy¹⁹ studies have shown that the groundstate splitting of the $Er³⁺$ ion increases sharply as the temperature is reduced below $4.5\,^{\circ}\text{K}$. Further, the Er^{3+} spin system is known, from all the neutron diffraction studies, to be in essentially a C_{κ} mode in this temperature range. In other words, the Er^{3*} spin changes from a $C_{\boldsymbol{\ell}}F_{\boldsymbol{\ell}}$ to an essentially $C_{\boldsymbol{\ell}}$ mode in a relatively narrow temperature region in the neighborhood of 4. ⁵ 'K. This should result in an observable anomaly in the specific heat and such a peak has been reported at approximately ⁴ 'K by Pataud and Sivardiere.²⁰

Finally, we note that the mechanism underlying this low-temperature spin reorientation is still not completely clear. One possible mechanism is an Er-Er interaction leading to a cooperative ordering of the Er^{3*} spin in a C_{ϵ} mode at $T_{N}(Er^{3*}) \approx 4.5 \degree K_{\epsilon}$. This mode could then couple to the G_y mode of the This mode could then couple to the G_y mode of the Fe^{3*} moments, ⁴ leading to a gradual $G_z - G_y$ rotation of the Fe³⁺ spin system as the temperature is further lowered below $T_N(\text{Er}^{3+})$ and the magnitude of the $Er³⁺$ moments increases. Faulhaber et al.¹⁹ and others^{18,21} have suggested that this is indeed the case, with the Er-Er interaction beginning because of the dipolar field of the Er^{3*} moments. The dipole-dipole interaction does not however appear to be strong enough to induce such a cooperative ordering in $ErfeO₃$ at 4.5°K. The isomorphic compound $ErA1O₃$ is known to order cooperatively in a C_{ϵ} mode only at 0.6°K.²² Thus, unless the Er³⁺ magnetic moment in $ErAIO₃$ is much less than that in $ErfeO₃$, it is unlikely that a cooperative ordering of the Er^{3+} spin system underlies the low-temperature spin reorientation.

An alternate possibility is that the reorientation is induced primarily by the Fe-Er exchange interaction. Wood et al.²³ have shown that the g factors of the low-lying Kramer's doublet of $ErfeO₃$, in their local principal axis system, are $g_t = 1.2$, g_n = 4. 5, and g_{ξ} = 5.76. Here ξ and η lie in the a b plane and ζ is along the c axis. For temperatures somewhat above $5^{\circ}K$, the Fe³⁺ exchange field is in the $a b$ plane and the $Er³⁺$ moments are essentially along the local η axis.²¹ If, however, this same exchange field were to lie along the c axis rather than in the $a b$ plane it is clear that that part of the system free energy due to the Fe-Er interaction would be lowered, since $g_{\boldsymbol{\ell}} > g_{y\boldsymbol{\ell}}$. Now a c-direction would be lowered, since g_{ℓ} , g_{y} , Now a c-direction effective field will exist if the Fe³⁺ spin system, in whole or in part, is in a G_y configuration. Thus we see that the Fe-Er interaction can also, in principle, lead to a rotation of the Fe³⁺ spin axis. Pataud and Sivardiere 20 and also Belakhovsky ${\it et \ al.}^{24}$ hav ${\it e}$ suggested that this is the mechanism responsible for the spin reorientation.

In summary, we had found from neutron diffraction and magnetization studies that the low-temperature $(T<4.5\text{ °K})$ magnetic structure of the Fe³⁺ spin system in ErFeO₃ is $G_{yx}F_x$. The antiferromagnetic axis of the $Fe³⁺$ spin system rotates from the c toward the b crystallographic axis as T is lowered below 4.5 K , making an angle of $51^{\circ} \pm 8^{\circ}$ to b at 1.⁵ 'K. Either an Er-Er or the Fe-Er interaction (or a combination of both) could be the mechanism responsible for the spin reorientation. Further studies will be required to make a final determination.

ACKNOWLEDGMENT

We are grateful to Professor S. Shtrikman for many helpful discussions and suggestions throughout the course of this work.

- «Supported in part by the Air Force Materials Laboratory (AFSC) through the European Office of Aerospace Research under Grant No. AFOSR 72-2327.
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- are, in general, different than those calculated for a b.
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