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2005 J. Phys.: Condens. Matter 17 1961

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J. Phys.: Condens. Matter **17** (2005) 1961–1973 doi:10.1088/0953-8984/17/12/018

A magnetization study of $ErMn_6Sn_{6-x}Ga_x$ single crystals ($0.11 \leqslant x \leqslant 1.20$)

F Canepa[1,4], M Napoletano[1], R Masini[2], C Lefèvre[3] and G Venturini[3]

[1] LAMIA-INFM and Dipartimento di Chimica e Chimica Industriale, Via Dodecaneso 31, 16146 Genova, Italy
[2] CNR-IMEM and Dipartimento di Fisica, Via Dodecaneso 33, 16146 Genova, Italy
[3] Laboratoire de Chimie du Solide Minéral, Université Henri Poincaré-Nancy I, associé au CNRS (UMR 7555), BP 239, 54506 Vandoeuvre les Nancy Cedex, France

E-mail: canepaf@chimica.unige.it

Received 19 January 2005, in final form 8 February 2005
Published 11 March 2005
Online at stacks.iop.org/JPhysCM/17/1961

Abstract

Dc and ac magnetic measurements have been performed on single crystals of $HfFe_6Ge_6$ type $ErMn_6Sn_{6-x}Ga_x$ compounds prepared by the flux method. At low temperature all the compounds display easy-axis magnetization and undergo a spin reorientation transition to easy-plane magnetization below room temperature. The complete ferrimagnetic order ($\mu_{sat} = 4\ \mu_B\ fu^{-1}$) is reached at 10 K only in the easy direction. From low field magnetization measurements at 10 K, a first order magnetic process (FOMP) has been detected for all the compounds and ascribed to the competition between the higher order magnetocrystalline terms. An additional transition detected from the imaginary part of the ac susceptibility for the $x = 0.85$ and 1.20 compositions was explained in terms of displacement of Bloch walls. The anisotropy constants, evaluated, following the Sucksmith and Thompson method, for some compositions at room temperature, decrease at increasing Ga content.

1. Introduction

The hexagonal $HfFe_6Ge_6$-type $RMn_6Sn_{6-x}Ga_x$ compounds (R = Y and rare earth elements) display interesting properties. In the non-magnetic R element series $YMn_6Sn_{6-x}Ga_x$, the replacement of tin by gallium reduces the antiferromagnetic character of the Mn sublattice, and with a sufficiently large amount of Ga the structure becomes ferromagnetic. With intermediate Ga content, metamagnetic behaviour is observed with concomitant giant magnetoresistance [1, 2]. As well as this effect on the exchange interactions, an unusual evolution of the magnetocrystalline anisotropy has been observed. Within the $TmMn_6Sn_{6-x}Ga_x$ series, the substitution of gallium for tin yields large coercive fields [3]

[4] Author to whom any correspondence should be addressed.

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while, within the $TbMn_6Sn_{6-x}Ga_x$ series, it leads to a considerable reduction of the magnetic hardness [4]. Further neutron diffraction measurements have evidenced changes in the easy magnetization directions: from easy plane to easy axis in thulium compounds and from easy axis to easy plane in terbium compounds [5, 6]. With thulium and terbium being characterized by the same sign of the β_j and γ_j Stevens coefficients and by different signs of the α_j coefficient, the relative behaviour of the corresponding compounds suggests that the substitution of gallium for tin yields a change in the second order CEF parameter. More quantitative results have been obtained using magnetization measurements of single crystalline $TmMn_6Sn_{6-x}Ga_x$ and $HoMn_6Sn_{6-x}Ga_x$ samples [7–9]. Finally, a study dealing with magnetically oriented $ErMn_6Sn_{6-x}Ga_x$ samples has been reported by Yao *et al* [10]. In order to get some information on any possible role of the sixth order CEF parameter, a magnetization study of $ErMn_6Sn_{6-x}Ga_x$ single crystals has been undertaken.

2. Experimental details

$ErMn_6Sn_{6-x}Ga_x$ crystals was obtained by the flux method related to the method previously reported for the crystallization of isotypic ternary stannides [11]. A mixture of $ErMn_6Sn_6$ alloy previously obtained by induction melting and elemental gallium was compacted into pellets and put in a silica tube with a large amount of tin metal, giving rise to the overall atomic ratio $ErMn_6Sn_{32-x}Ga_x$. A quartz wool stopper was introduced in the silica tube which was sealed under argon (200 mm Hg). The tube was placed in a furnace and quickly heated to 1273 K (50 K h^{-1}) where it remained for 24 h. The furnace was then slowly cooled down to 1223 K (6 K h^{-1}), heated again up to 1263 K at the same rate and finally slowly cooled down to 873 K for 65 h. The tube was quickly removed from the furnace, inverted and centrifuged manually using a David's sling device. After this treatment, the single crystals remained on the quartz wool stopper and the tin flux laid at the bottom of the tube. This method produces relatively large hexagonal prisms with masses up to 250 mg. Some of them were ground and analysed by x-ray diffraction (Guinier camera CuKα) with high purity silicon as internal standard ($a = 5.430\,82$ Å), and their composition checked using a SX50 electron probe.

The thermal variation of the magnetization was studied using a MANICS magneto-susceptometer in a field of 0.05 Tesla (T) and in the temperature range 5–450 K. The transition temperatures (T_c and T_{SR}) were taken at the maximum of the first derivative dM/dT. The field dependence of the magnetization in the 0–9 T field range (extraction method) was obtained using an Oxford Maglab^{2000}. Ac susceptibility measurements were carried out using Lake Shore 7221 equipment operating at a frequency of 333 Hz and in a rms field of 1 mT.

3. Results

3.1. Structural data

Examination of the Guinier patterns indicates that all the $ErMn_6Sn_{6-x}Ga_x$ ($x \leqslant 1.9$) single crystals obtained in this way are isotypic of $HfFe_6Ge_6$ (figure 1). The starting composition of the melt, cell parameters and results of the microprobe analysis are gathered in table 1. The plot of the final gallium content in the crystal as a function of the initial gallium concentration displays an almost linear dependence as previously observed for Tm and Ho isotypic crystals [8, 9] (figure 2(a)). The substitution of tin for gallium leads to a large decrease of the cell volume in good accordance with the previous results obtained on powder samples [10] (figures 2(b)–(d)). The contraction of the cell is isotropic since the c/a ratio does not significantly vary along the whole series (table 1). This feature should be related to the preferential occupation of the 2(c) site by the gallium atom as deduced from single crystal

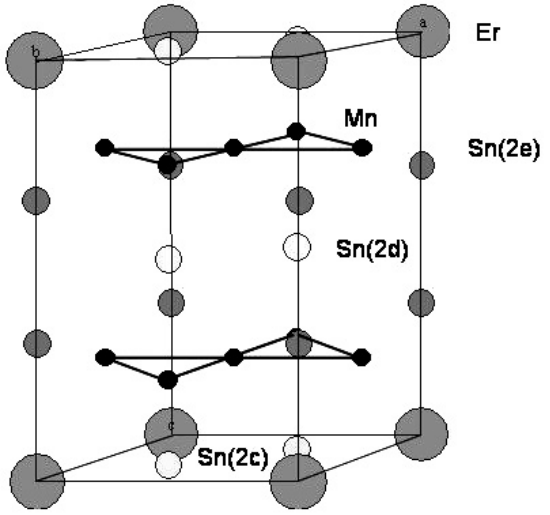

Figure 1. Crystal structure of $HfFe_6Ge_6$-type $ErMn_6Sn_{6-x}Ga_x$ compounds.

Table 1. Crystallographic and magnetic properties of $ErMn_6Sn_{6-x}Ga_x$ single crystals (T_c = Curie temperature; T_{SR} = reorientation temperature; M_s = maximum magnetization value for $H \parallel c$; M_{Mn} = moment per Mn atom assuming $M_{Mn} = (M_s + 9)/6$.)

Starting composition	a (Å)	c (Å)	V (Å^3)	c/a	x	T_c (K)	T_{SR} (K)	$M_s \parallel c$ (μ_B)	M_{Mn} (μ_B)
$ErMn_6Sn_{31.5}Ga_{0.5}$	5.508(2)	8.989(5)	236.1(3)	1.6320	0.11	372	35	4.0	2.17
$ErMn_6Sn_{31}Ga_1$	5.499(2)	8.976(5)	235.1(3)	1.6323	0.25	380	55	4.0	2.17
$ErMn_6Sn_{30.5}Ga_{1.5}$	5.493(2)	8.961(3)	234.2(2)	1.6312	0.42	381	81	3.8	2.13
$ErMn_6Sn_{30}Ga_2$	5.488(2)	8.954(3)	233.5(2)	1.6315	0.50	375	96	3.95	2.16
$ErMn_6Sn_{29}Ga_3$	5.473(2)	8.930(5)	231.7(3)	1.6316	0.85	371	156	3.85	2.14
$ErMn_6Sn_{28}Ga_4$	5.459(2)	8.907(4)	229.9(3)	1.6315	1.20	344	212	4.25	2.21

x-ray refinement of the structure of $TmMn_6Sn_{4.8}Ga_{1.2}$ [12]. The Ga 2(c) site is characterized by close Ga–Tm(Er) contacts within the (001) plane and by close Ga–Mn bonds mostly directed along the [001] direction (figure 1). The preservation of suitable Er–Ga and Mn–Ga distances needs a decrease of both a and c parameters. It is also worth noting that the replacement of the close Er–Sn by Er–Ga contacts are probably responsible of the modification of the CEF parameters related to the rare-earth site.

3.2. Thermomagnetic study

The thermomagnetic curves measured along the c axis and perpendicular to the c axis are depicted in figure 3. The six compounds order ferrimagnetically above the room temperature and undergo a spin reorientation transition at low temperature characterized by the vanishing of the magnetization measured perpendicular to the c axis and the simultaneous increase of the magnetization measured along the c axis. The Curie point slightly increases from 372 K for $x = 0.11$ to 381 K for $x = 0.42$ and then slightly decreases for larger Ga content. The relative values of the magnetization measured along the c axis and perpendicular to the c axis indicate that the moments are perpendicular to the [001] direction at high temperature

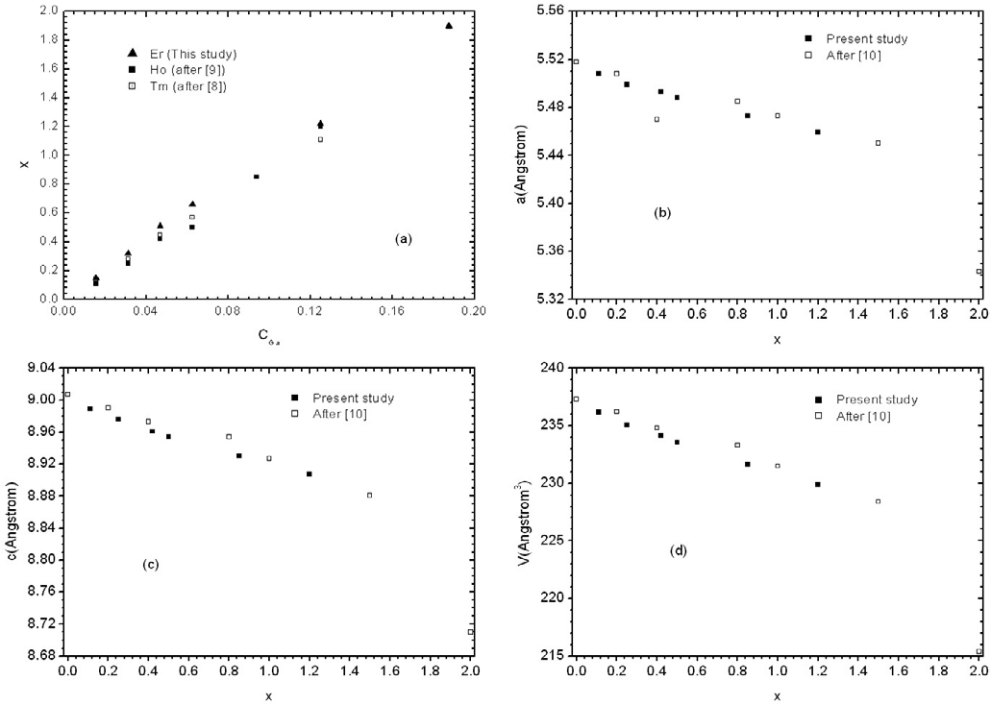

Figure 2. Crystal composition versus starting composition of the melt ($c_{Ga} = n$Ga/[nGa+nSn]) (a). x dependence on the cell parameters (b)–(d).

and aligned along the [001] direction at low temperature. The spin reorientation transition (T_{SR}) drastically increases with the Ga concentration. The plot of the transitions temperatures versus the Ga concentration is given in figure 4. There is a fair accordance with the previously reported results related to the polycrystalline samples [10].

The curve related to the compound $ErMn_6Sn_{5.89}Ga_{0.11}$ displays an additional anomaly in the 175–275 K temperature range. The small decrease of the magnetization measured perpendicular to the [001] direction and the absence of any anomaly measured along the [001] direction rules out any possible reorientation process. This behaviour should rather be related to the observations made for several $RMn_6Sn_{6-x}In_x$ compounds (R = Tm, Er) characterized by the so-called 're-entrant ferromagnetic behaviour' and by the stabilization of a helimagnetic structure in a narrow intermediate temperature range [13]. This behaviour results from a competition between an increasing antiferromagnetic character of the Mn sublattice upon cooling and a simultaneous increase of the Er(Tm)–Mn interaction, which favours a parallel alignment of the Mn sublattice. In the present case, the persistence of spontaneous magnetization indicates that the ferri- to helimagnetism process is not complete. What may be assumed is that the perfect ferrimagnetic arrangement is partly broken (canted or conical structure) in this temperature range.

3.3. Ac susceptibility measurements

Figure 5 shows representative results of ac susceptibility measurements for $ErMn_6Sn_{6-x}Ga_x$ single crystals with both H parallel to the c axis and H perpendicular to c in the range $0.11 \leqslant x \leqslant 1.20$. For clarity only the real part of the susceptibility is reported.

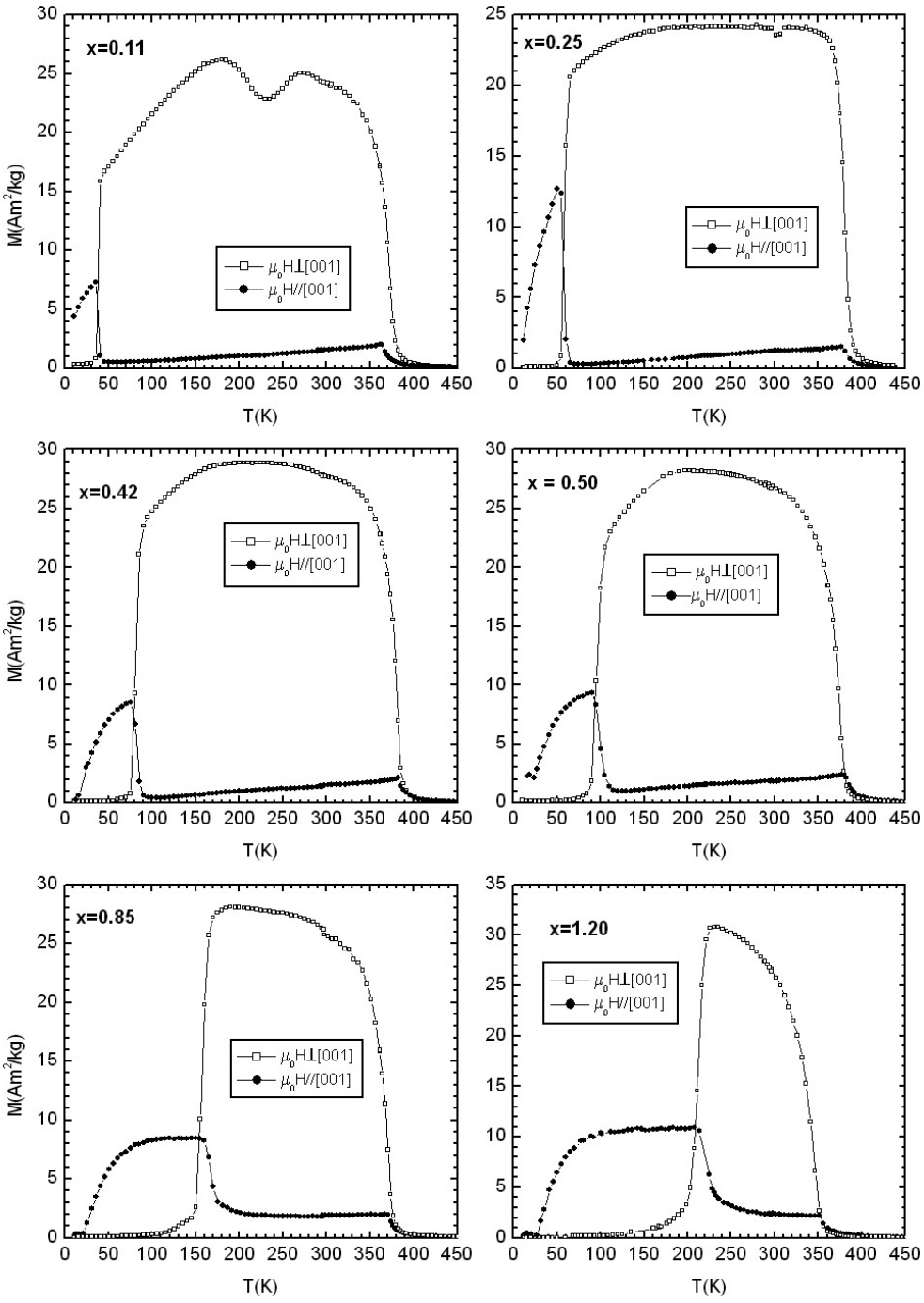

Figure 3. Thermomagnetic curves (ZFC) ($\mu_0 H_{DC} = 0.05$ T).

Samples with a low concentration of Ga ($x \leqslant 0.50$) display, in zero dc field and for both directions, a sharp transition at about 40 K, which shifts to higher temperatures (table 2)

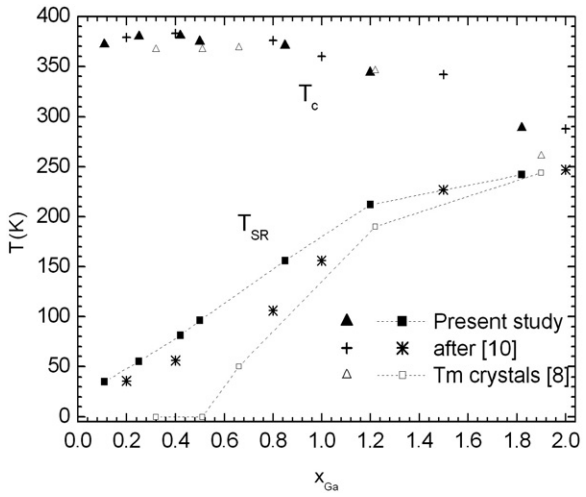

Figure 4. Transition temperatures as a function of the Ga contents in $ErMn_6Sn_{6-x}Ga_x$ and $TmMn_6Sn_{6-x}Ga_x$ compounds.

Table 2. Spin reorientation transition temperatures for the series $ErMn_6Sn_{6-x}Ga_x$ as detected from ac susceptibility.

Ga content x	T_{SR} with $H_{ac} \parallel c$	T_{SR} with $H_{ac} \perp c$
0.11	40	40
0.25	63	63
0.42	85	85
0.50	98	98
0.85	117, 170	158
1.20	127, 225	211

with increasing Ga content in agreement with the thermomagnetic data and indicating a spin reorientation transition. It should be pointed out that the difference in T_{SR} obtained from dc and ac magnetic measurements can be ascribed to the different methods used. Ac data are very accurate because the ac field has only a perturbing action on the magnetic structure; dc magnetization measurements, by contrast, affect to a greater extent the magnetic response of the compounds. At higher Ga content, the data with H perpendicular to the [001] direction have the same trend, i.e. a sharp transition which increases with Ga and slowly tails off at lower temperature; the data along the [001] direction, by contrast, show a very broad and double transition starting at a lower temperature.

A comparison of the ac susceptibility measurements for both directions shows that the susceptibility applied perpendicular to the c axis is always higher in the temperature range below 300 K. This behaviour may be due to the higher degree of freedom of the magnetic moments when the easy magnetization direction is in the plane where the moments can freely rotate.

3.4. Magnetization versus field

Figure 6 displays the magnetization data obtained at 10 K for all the six compounds, performed with the magnetic field applied both perpendicular and parallel to the c axis. The small jumps

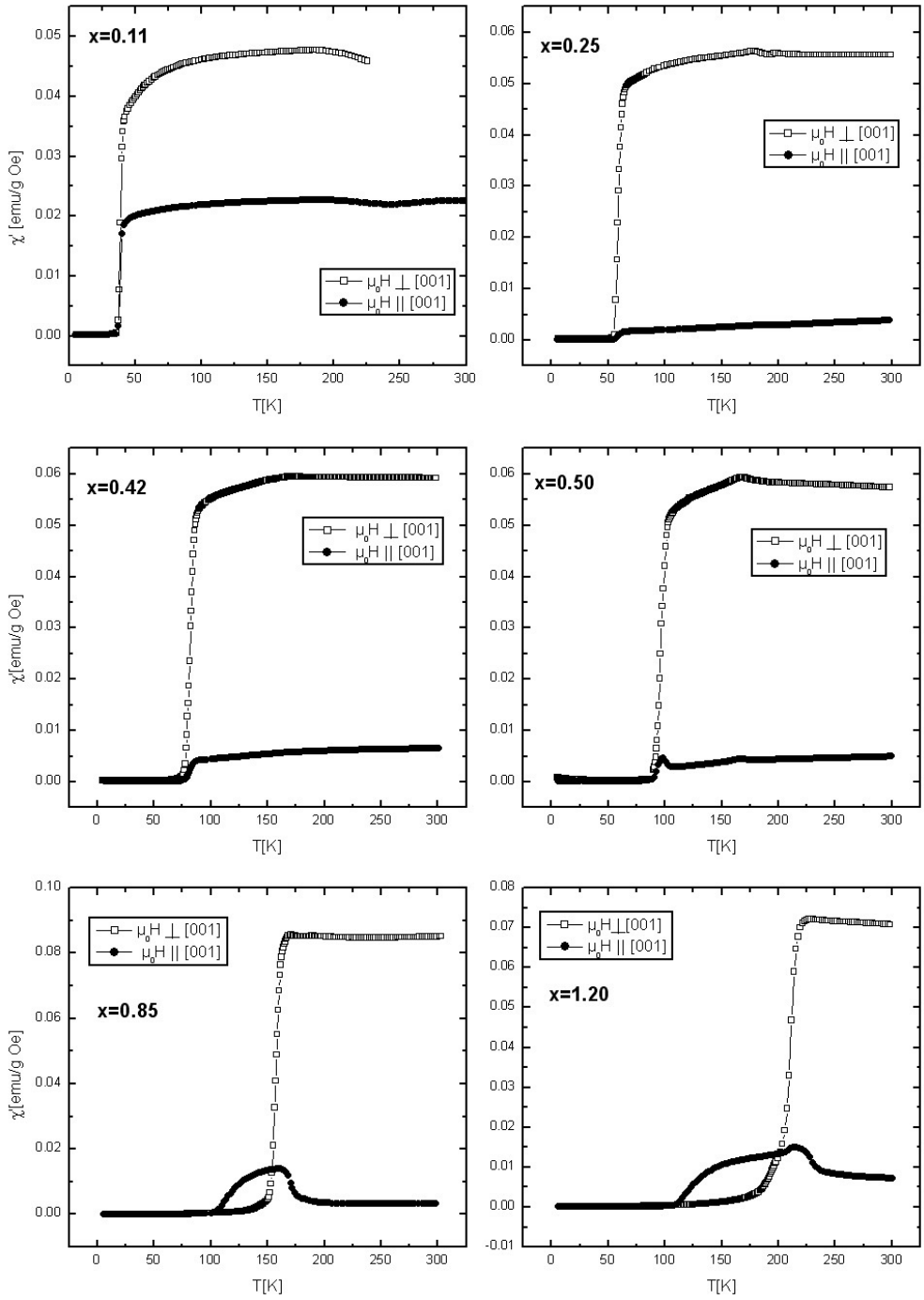

Figure 5. Ac curves versus temperature for $ErMn_6Sn_{6-x}Ga_x$ crystals.

detected along the hard directions can be ascribed to a small displacement of the samples from the original position; unfortunately any attempt to fix the single crystals in the hard direction

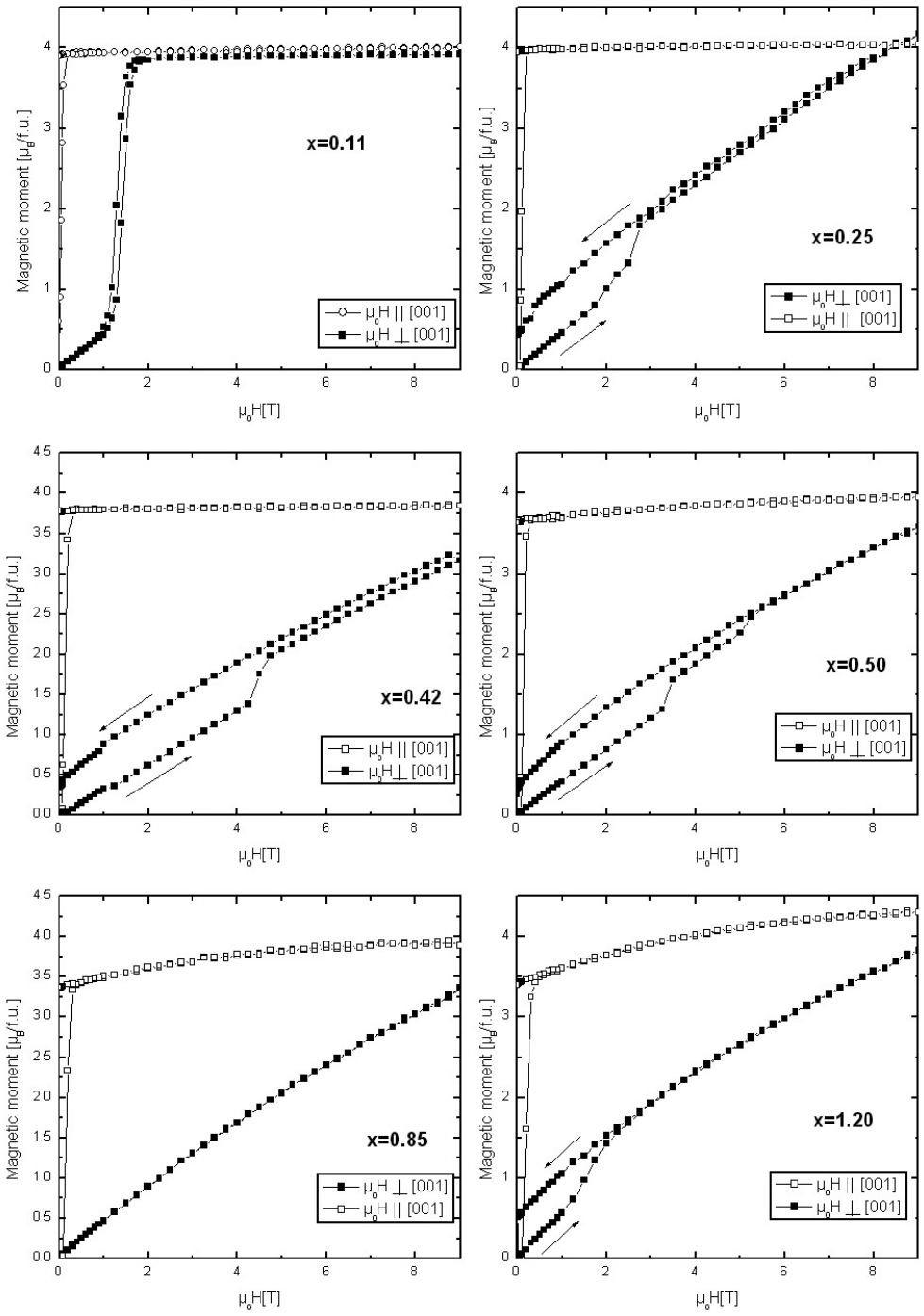

Figure 6. Magnetization curves at 10 K.

was unsuccessful so we decided, for this temperature, to provide just a qualitative analysis of the curves without evaluating the anisotropy constants.

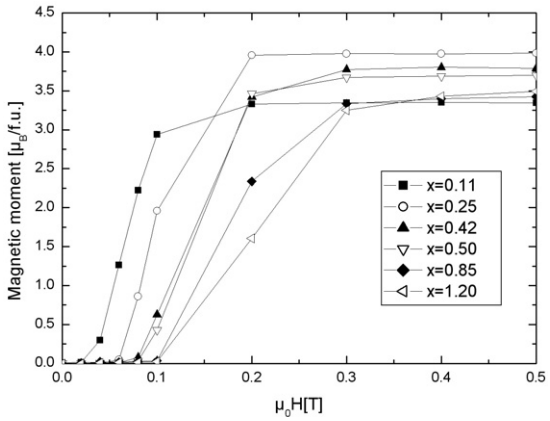

Figure 7. Low field part of the magnetization curves at 10 K.

The saturation values reach 4.0 μ_B fu^{-1}. Taking $\mu_{Er} = 9\ \mu_B$ and $\mu_{Mn} = 2.0$–$2.2\ \mu_B$, as already observed in other compounds of these series, the observed saturation values suggest a collinear antiferromagnetic interaction between the Mn atoms and the Er ones, resulting in a ferrimagnetic structure in good agreement with the data reported for the polycrystalline samples [10].

As is possible to observe at this temperature, all the compounds exhibit easy axis magnetization regardless of the Ga content, but except for the low doped compound saturation is not reached along the planar hard direction. The compound with the lowest Ga content $x = 0.11$ exhibits an anomalous behaviour: a spin reorientation transition may be observed with a threshold field of 1.5 T and with a hysteretic behaviour typical of a first order phase transition.

Another interesting feature is displayed by the easy axis low field magnetization curves reported in figure 7: at first the magnetic moment is almost undetectable, then at a critical field there is a sudden increase of magnetization, reaching saturation values. The threshold field of this transition increases with increasing Ga content.

In figure 8 the high temperature magnetization curves are reported for all the compounds with the magnetic field applied both perpendicular and parallel to the *c* axis. At this temperature all the compounds display easy plane magnetic anisotropy, and saturation is not reached even in the easy direction.

A field induced transition may be observed for intermediate compounds $x = 0.25$, 0.42, 0.50 with threshold fields of 0.5, 0.39, 0.37 T, respectively, and with hysteretic behaviour for $x = 0.42$ and 0.50.

Since in this case the compounds were fixed in the easy and hard directions without any problem, the anisotropy constants were easily evaluated at 290 K following the Sucksmith and Thompson method, but only for those compounds not affected by field induced transitions. The results are reported in table 3.

4. Discussion

The replacement of Sn with Ga in the $ErMn_6Sn_6$ compounds induces an abrupt change in the magnetic properties: as reported in [14] for the undoped compound, ferrimagnetic behaviour may be observed below 67 K where a helimagnetic to ferrimagnetic transition takes

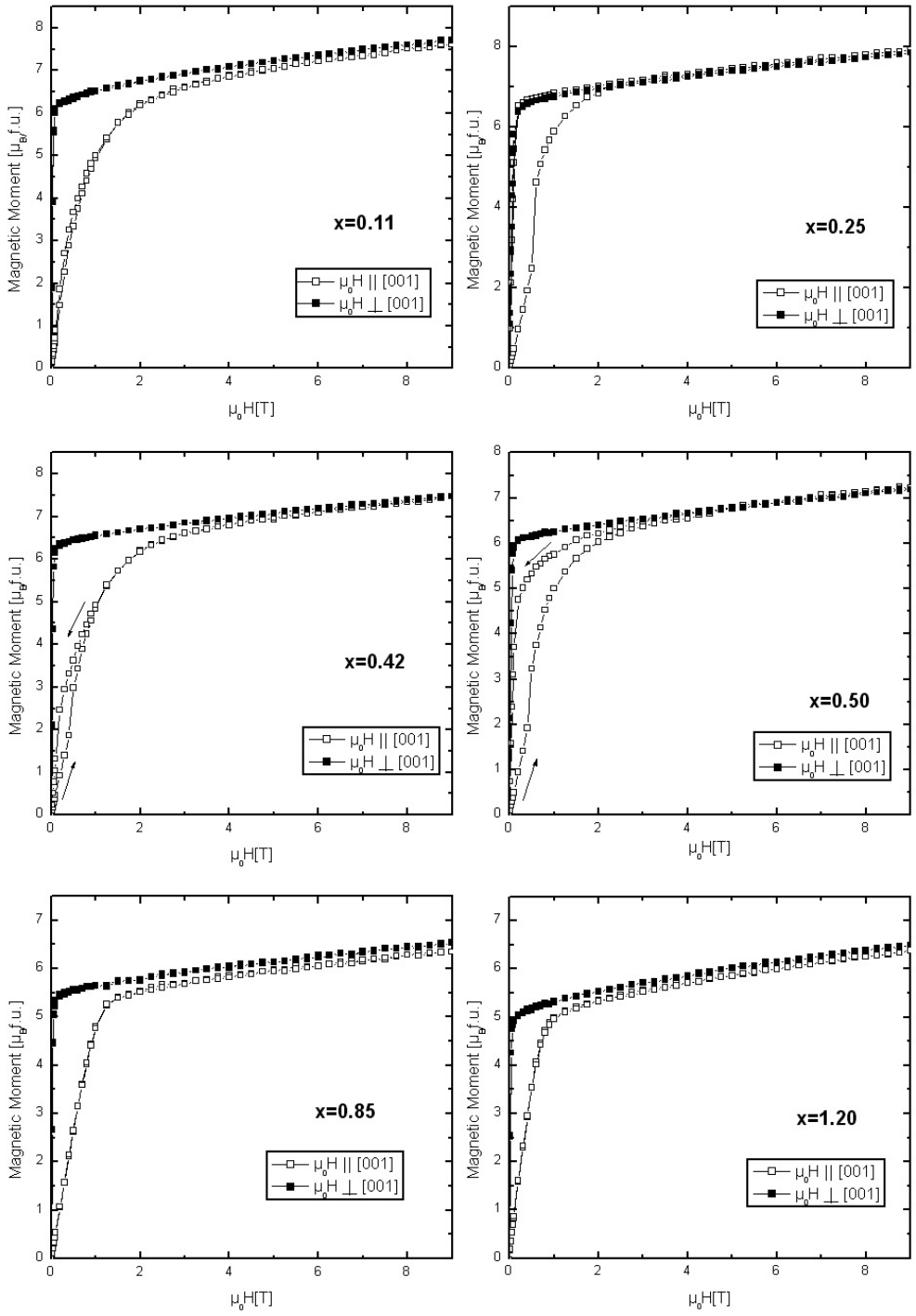

Figure 8. Magnetization curves at RT.

place followed by a final transition at 362 K from helimagnetism to paramagnetism. The introduction of Ga even in rather small quantities has as an immediate result the destruction of

Table 3. Room temperature ($T = 290$ K) anisotropy constants for some $ErMn_6Sn_{6-x}Ga_x$ compounds. For $x = 0.25$, 0.42 and 0.50 the field induced transition prevents us from evaluating the experimental data.

Ga content x	K_1 (J m^{-3}) $\times 10^5$	K_2 (J m^{-3}) $\times 10^5$
0.11	−4.49	2.02
0.25		
0.42		
0.5		
0.85	−1.64	0.415
1.20	−1.089	0.392

the helimagnetic region replaced by a ferrimagnetic structure in the whole ordered range except for the $ErMn_6Sn_{5.89}Ga_{0.11}$ sample characterized by a narrow temperature range where the perfect ferromagnetic arrangement is broken. This peculiar behaviour, probably restricted to a very small gallium concentration, needs to be checked through other investigations (Neutron diffraction, ^{119}Sn Mössbauer spectroscopy).

The Curie temperature displays a typical variation previously observed for all $RMn_6Sn_{6-x}Ga_x$ solid solutions: for small Ga content, the ordering temperature firstly slightly increases and then considerably decreases for large gallium concentration. This non-monotonous evolution probably arises from competing effects. The first increase should be related to the decrease of the interatomic contact between the magnetic species since such an effect is not observed in corresponding $RMn_6Sn_{6-x}In_x$ solid solutions characterized by an increase of the cell parameters. The decrease of T_c for large gallium content might be related to a modification of the valence electron concentration resulting in a variation of the long range magnetic interactions mediated by the conduction electrons.

At low temperature spin reorientation transitions from easy axis to easy plane take place, whose temperature increase on increasing Ga content as displayed in the ac susceptibility measurements, and in good agreement with the values reported in [10] for the polycrystalline samples. The enhancement of the temperature of the spin reorientation transition should be understood on the basis on increasing easy axis anisotropy of the erbium sublattice for increasing Ga content.

Particular attention has been devoted to the analysis of the low field behaviour of magnetization at 10 K displayed in figure 7: a linear increase of magnetization followed by a sharp increase up to saturation values is usually considered a clear indication of antiferromagnetic behaviour, but, in this case, this hypothesis had to be rejected because neutron diffraction data obtained for the $ErMn_6Sn_6$ compound clearly indicated a ferrimagnetic ordering below 67 K. Ga substitution cannot induce a change from a ferrimagnetic structure to a helimagnetic structure because in all the compounds of the series the main effect of the replacement of Sn or Ge with In or Ga is to enhance the overall ferromagnetic character of the manganese interactions [15].

A very low value of the initial magnetization as a function of the applied magnetic field can also be observed in the so-called pinning type magnets [16]: in this case the inhomogeneities act as pinning centres for wall motion. The wall displacements can occur only when the magnetic field is strong enough to overcome the pinning force. However, in this case we are dealing with single crystals where the inhomogeneities content is very low or even completely absent, as suggested by the profile analysis performed on $TmMn_6Sn_{6-x}Ga_x$ single crystals [17].

A third and more attractive hypothesis, suggested also by [10], is to ascribe this behaviour to a first order magnetic process (FOMP) due to the competition between the higher order magnetocrystalline terms [18].

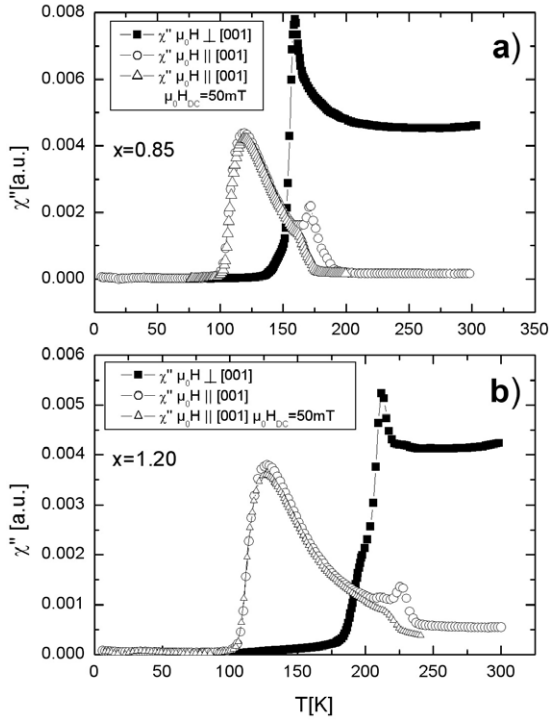

Figure 9. Imaginary part of the ac susceptibility of $ErMn_6Sn_{5.15}Ga_{0.85}$ (a) and $ErMn_6Sn_{4.80}Ga_{1.20}$ (b).

It should be emphasized that the high Ga content compounds display an additional transition easily detected in the imaginary part of the ac susceptibility obtained with the ac field applied parallel to the *c* direction at $T = 117$ and 127 K for $x = 0.85$ and 1.20, respectively, as reported in figure 9. At a first glimpse the dc thermomagnetic results of figure 3 at 0.05 T only display the spin reorientation transition at higher temperature. However, a careful analysis of the thermomagnetic curves along the *c* axis of the two compounds shows the existence of a plateau in the magnetization which starts at the transition temperature detected from ac data: this suggests that in this temperature region there are no more domains wall motions nor domain magnetization rotations. The change of the temperature dependence of the magnetization from the initial increase to the constant value of the plateau, related to the change in the dynamics of the domains, corresponds, for magnetically ordered materials with strong magnetocrystalline anisotropy, to a peak in the imaginary part of the ac susceptibility; this peak is a clear indication of energy losses in the displacements of Bloch walls [19]. Furthermore, the other four compounds do not display either the additional peak in the ac susceptibility or the plateau in the thermomagnetic data, probably because the spin reorientation transition occurs before the end of the domains dynamics. A superimposed dc field of 50 mT, reported in the same figure, does not affect either the transition temperature or the intensity of the effect, in agreement with the comments reported in [19] concerning domain wall dynamics.

5. Conclusions

The properties of $ErMn_6Sn_{6-x}Ga_x$ single crystals obtained in a tin–gallium flux are in fair agreement with previous results obtained from magnetically ordered powder samples [10]. As

in the $TmMn_6Sn_{6-x}Ga_x$ series [7, 8], it is observed that the replacement of tin by gallium leads to the stabilization of a ferrimagnetic structure in the whole ordered temperature range. However, it is observed that the spin reorientation transition takes place for smaller Ga content than in the corresponding thulium compounds (figure 4). This suggests that the initial in-plane magnetocrystalline anisotropy in the erbium stannide $ErMn_6Sn_6$ is much weaker than the anisotropy of the stannide $TmMn_6Sn_6$. This assumption should be related to the results of magnetization measurements performed on RMn_6Sn_6 (R = Tb, Ho, Er, Tm, Lu) [11] and $TmMn_6Sn_{6-x}Ga_x$ compounds [8] single crystals. According to these studies, an anisotropy field close to 1.5 T has been measured for the compound $ErMn_6Sn_6$ at 5 K, whereas no anisotropy field is observed for $TmMn_6Sn_{5.85}Ga_{0.15}$ up to the maximum applied field of 9 T [7]. Since both Er and Tm elements are characterized by the same sign of the α_j and β_j Stevens coefficients and by different signs of the γ_j coefficient, the quite different behaviour of Er and Tm compounds suggests the play of the sixth order CEF parameter. This assumption is corroborated by the thermal variation of the anisotropy fields in $ErMn_6Sn_6$ and $TmMn_6Sn_{5.68}Ga_{0.32}$, both the compounds being characterized by an easy plane magnetization direction. According to crystal field theory, the thermal averages of the highest order Stevens operator are correlated to high powers of reduced moments and therefore begin to play only at low temperature.

Accurate neutron diffraction experiments as a function of the temperature on these samples might contribute to understanding the evolution of the complex magnetic structure of the phases.

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