PHYSICAL REVIEW B VOLUME 50, NUMBER 17 1 NOVEMBER 1994-I

Magnetic phase diagram of $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ alloys

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(Received 9 May 1994)

The magnetic phase diagram of the pseudoternary $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ compounds has been experimentally and theoretically investigated. The thermal dependence of the low-field ac susceptibility on powdered samples exhibits a sharp peak, due to an axial-conical spin reorientation (SR) transition. The SR temperature (T_{SR}) decreases with increasing Gd content. The angular dependence of magnetization on magnetically aligned powdered samples has been measured in a steady applied magnetic field, in the 4.2–300 K temperature range. From these measurements we have determined the angle θ between the spontaneous magnetization and the tetragonal c axis. Onset temperatures for magnetization canting agree with the T_{SR} obtained from susceptibility data within 10 K. Both the T_{SR} vs x and the θ vs T dependences have been calculated using a single-ion, mean-field model for the rare-earth and transition-metal interactions. Very good agreement between theory and experiment was obtained.

I. INTRODUCTION

Intermetallic compounds of the general formula $R\mathrm{Fe}_{12-x}M_x$ (R =rare-earth element), with the ThMn_{12} structure, have attracted much attention in the last few years.[1] Some members of this class of compounds qualify as candidates for permanent-magnet materials due to their strong uniaxial anisotropy,[2] which can also be induced for other members by interstitial nitrogen addition.[3] Other compounds exhibit a variety of spin reorientation phenomena, which are of interest in themselves. The cause of such phenomena is, essentially, the strong temperature dependence of the magnetic anisotropy at the rare-earth site, combined with an Fe sublattice anisotropy of opposite sign. The former can be satisfactorily described with a model involving such quantities as a mean exchange field and several crystalline electric-field (CEF) parameters. No reliable first-principles computation method for CEF parameters is available at present, so they are usually obtained from fits to experimental data. In this context, the $R\mathrm{Fe}_{12-x}M_x$ compounds are a favorable case because the rare-earth ion occupies a single site in the ThMn_{12} structure.

Among the 1:12 compounds, the magnetic properties of the $R\mathrm{Fe}_{11}\mathrm{Ti}$ series have been most extensively investigated.[4–8] For $\mathrm{DyFe}_{11}\mathrm{Ti}$, in particular, experiments have also been carried out on single crystals.[9–11] Most compounds of this series are of the easy-axis type (magnetization parallel to the tetragonal c axis) at and above room temperature.[12] This magnetic structure is favored by the Fe sublattice anisotropy, and it remains stable down to the lowest temperatures for R =Y, Sm, Gd, Ho, Tm, and Lu. $\mathrm{DyFe}_{11}\mathrm{Ti}$, on the other hand, was found[9] to be easy plane (**M** parallel to [100]) from 4.2 K to $T_1 = 58$ K, then to become conical with a canting angle θ smoothly varying form 40° (at T_1) to 0° at $T_2 = 200$ K, and finally to remain axial from T_2 upwards. Susceptibility measurements, later carried out[11] on the same monocrystalline $\mathrm{DyFe}_{11}\mathrm{Ti}$ sample, have yielded transition temperatures $T_1 = 69$ K and $T_2 = 187$ K, respectively. These values are probably more accurate due to the better temperature resolution of the χ vs T measurements. In the same work, the magnetization direction below T_1 was reexamined and found to be [110], in agreement with an earlier determination[10] made on a different monocrystalline sample.

From a theoretical analysis of their single-crystal magnetization curves, Hu *et al.*[9] determined a set of CEF parameters for the Dy^{3+} ion in $\mathrm{DyFe}_{11}\mathrm{Ti}$. A full description of the CEF in this system requires five parameters: B_{20}, B_{40}, B_{44}, B_{60}, and B_{64}. Once a set of B_{nm} is known for a given R ion, those for other R' ions in the same structure can be obtained by scaling with appropriate atomic parameters, so that the magnetic properties of the corresponding $R'\mathrm{Fe}_{11}\mathrm{Ti}$ compound can be calculated; the temperature-dependent magnetic structures thus obtained for this series are in reasonable agreement with experiment.[9]

Mixed systems of the $(R_{1-x}R'_x)\mathrm{Fe}_{11}\mathrm{Ti}$ type, in which R and R' are randomly distributed over the rare-earth sites, are of great interest since they offer an opportunity of varying the magnetic anisotropy in a continuous manner. Spin reorientation temperatures will vary accordingly, and thus more experimental data can be generated, which will provide a more severe test for models containing quantities to be empirically determined. Under rather mild conditions to be discussed below, exchange and CEF constants for R and R' are proportional to each other, so no new adjustable parameters will be introduced by the presence of a second rare-earth element.

In this paper we report on magnetic measurements on the mixed system $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$. Gd was chosen as

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a substitute for Dy because the S-state ion Gd^{3+} does not contribute to the CEF-induced magnetic anisotropy, so its main expected effect is to reduce the basal anisotropy due to Dy^{3+} by simple dilution. In addition, Gd is sufficiently close to Dy in the periodic table that its substitution can be expected not to introduce appreciable changes in interatomic distances, exchange interactions, and lattice CEF coefficients.

Two experimental techniques were used in this work: low-field ac susceptibility and angular-dependent magnetization. In the latter experiments, magnetically aligned powder samples were rotated in a fixed applied field and the parallel and perpendicular components of the magnetization were measured. Thus, in addition to determining spin reorientation temperatures, we could also measure magnetization canting angles with reasonable accuracy. These measurements were performed in the 4.2–300 K temperature range. The magnetic phase diagram was theoretically calculated and compared to our experimental data.

II. THEORETICAL MODEL

In order to calculate the spontaneous magnetization direction we use the well-known model based on a single-ion Hamiltonian for the rare earth R:[14]

$$\mathcal{H}_{\mathbf{R}}=\sum \mathbf{B}_{nm}\mathcal{O}_{nm}-g_J\mu_B\mathbf{J}\cdot(\mathbf{B}_{\mathrm{ex}}+\mathbf{B}_{\mathrm{app}}) . \tag{1}$$

In Eq. (1) the B_{nm} are CEF parameters and the $\mathcal{O}_{nm}$ are operator equivalents.[15] The molecular field at the R site is given by

$$\mathbf{B}_{\mathrm{ex}}=\frac{2(g_J-1)}{g_J}n^*\mathbf{M}_{\mathrm{Fe}} , \tag{2}$$

where $\mathbf{M}_{\mathrm{Fe}}$ is the Fe sublattice magnetization and n^* is an effective exchange coupling constant between the R and Fe spins. We neglect the much weaker R-R interaction.

For a given direction (θ,φ) of the $\mathbf{M}_{\mathrm{Fe}}$ vector, the eigenvalues of (1) are calculated and the angular-dependent free energy per R ion is obtained:

$$F_R=-kT\ln\{\sum \exp(-E_n/kT)\} . \tag{3}$$

In order to account for the full magnetocrystalline anisotropy, the Fe sublattice anisotropy energy, most simply expressed as a uniaxial term $E_a=K_1^{\mathrm{Fe}}\sin^2\theta$, is added to the above expression. Minimization of the resultant free energy with respect to θ and φ then yields the equilibrium magnetization direction.[16] The SR temperatures are defined as those at which the free-energy minimum starts deviating from $\theta=0°$ (axis-cone transition) or jumps to $\theta=90°$ (cone-plane transition).

For a mixed compound such as $(R_{1-x}R'_x)\mathrm{Fe}_{11}\mathrm{Ti}$, the free energy is

$$F(\theta,\varphi)=(1-x)F_R+xF_{R'}+K_1^{\mathrm{Fe}}\sin^2\theta . \tag{4}$$

The CEF parameters are given by

$$B_{nm}=\theta_n\langle r^n\rangle_{4f}A_{nm} , \tag{5}$$

where θ_n (denoted as $\alpha_J, \beta_J, \gamma_J$ for $n=2,4,6$, respectively) are Stevens' coefficients,[15] and the radial averages $\langle r^n\rangle_{4f}$ are tabulated in the literature.[17] The A_{nm} are lattice terms which can be assumed to be independent of the rare earth in a series of isostructural compounds: this has been shown to be true, e.g., for A_{20} in $R_2\mathrm{Fe}_{14}\mathrm{B}$, except at the ends of the lanthanide series.[18] Thus, corresponding B_{nm}'s are in a fixed ratio for a given pair of R ions. For Gd, in particular, the B_{nm} vanish.

The exchange field depends on R through Eq. (3), where n^* is often assumed to be R independent in a series of compounds. This assumption has been criticized[19] on empirical grounds: a drop by a factor of 2 from Pr to Tm is indeed observed in various systems, and a theoretical evaluation[20] of the $4f$-$5d$ exchange interaction confirms this trend. It appears, however, that the n^*vs R dependence is strongest on the light-rare-earth side. For $R\mathrm{Fe}_{11}\mathrm{Ti}$, in particular, it was found[5] that n^* is practically constant from Tm through Tb, is a little larger for Gd, then increases steeply for Sm and Nd. For the materials of this study, therefore, a constant n^* seems to be a reasonable approximation.

The input data used in our calculations were taken from the papers by Hu *et al.*[5,9] To account for the temperature dependence of M_{Fe} and K_1^{Fe}, we used the corresponding experimental curves[5] for $\mathrm{YFe}_{11}\mathrm{Ti}$, scaled with the appropriate T_C.

III. EXPERIMENTAL DETAILS

Polycrystalline $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ ($x=1$, 0.8, 0.6, 0.4, 0.2) samples were prepared by arc melting appropriate amounts of the pure metals in argon atmosphere. A 10% excess of the rare-earth metals was added to allow for evaporation losses. The melted buttons were wrapped in Ta foil, sealed in quartz tubes with argon, annealed at 850 °C for two weeks, and quenched in water. X-ray-diffraction diagrams showed the annealed samples to be mostly single phase with the tetragonal ThMn_{12} structure, a few samples only exhibiting a small amount of TiFe_2 impurity. The Curie temperature was measured by the kink-point method with a vibrating-sample magnetometer. As shown in Table I, the T_C varies linearly with x for the pseudoternary series, indicating that deviations from the nominal $R:R'$ ratio were not significant.

The susceptibility of powdered samples was measured with a modified mutual inductance Hartshorne bridge operating at 15 Hz. Only the real part χ' was measured. The sample temperature was varied with help of a liquid-helium flow cryostat. Preliminary experiments were carried out in the 80–300 K temperature range[21] on a similar, liquid-nitrogen-cooled setup. Magnetization measurements were carried out on magnetically oriented powders by means of an extraction magnetometer. Magnetic orientation was achieved by fixing the powdered sample in epoxy resin in a field of 0.5 T. In magnetic polar plot experiments, the oriented sample was rotated in a constant field of 0.5 T provided by an electromagnet, and both parallel and perpendicular components of magnetization with respect to the applied magnetic field were measured as a function of the rotation angle, by means of two mutually perpendicular sets of pickup coils.[22]

IV. RESULTS AND DISCUSSION

The temperature dependence of the initial susceptibility of the $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ alloys is displayed in Fig. 1. A sharp peak is observed for all compositions, at decreasing peak temperatures for increasing Gd concentrations.

Although spin reorientation transitions (especially second-order ones such as the uniaxial-conical transition) are usually reflected in a well-defined $\chi(T)$ peak, the experimental definition of the transition temperature T_{SR} is somewhat controversial. It would seem quite natural to place T_{SR} at the χ vs T maximum, and this assignment is indeed supported by model calculations[23] appropriate to the $T=130$ K transition in $\mathrm{Nd_2Fe_{14}B}$, which yield a sharp cusp at T_{SR}. On the other hand, some authors[8,24] assign T_{SR} to the inflection point (i.e., minimum of $d\chi/dT$), slightly above the peak maximum. We adopt the former view, associating T_{SR} with the $\chi(T)$ maximum.

The SR temperatures thus obtained are given in Table I. It is worth mentioning that our T_{SR} for $\mathrm{DyFe_{11}Ti}$ agrees closely with former determinations, and particularly so with the peak maximum in Ref. 8; the latter authors, however, find $T_{\mathrm{SR}}=214$ K in accordance with their inflection-point criterion.

Figure 2 displays the concentration dependence of T_{SR}, together with the corresponding theoretical curve. The agreement is seen to be excellent. The CEF parameters used in the calculation were those given in Ref. 9. Calculations were done with $B_{\mathrm{app}}=0$, which is appropriate for comparison with low-field susceptibility data.

A more detailed understanding of the reorientation phenomena can be gained from magnetization polar plots. An example is given in Fig. 3, showing the angular dependence of the parallel $(M_{\parallel})$ and perpendicular $(M_{\perp})$ components of magnetization for $(\mathrm{Dy}_{0.8}\mathrm{Gd}_{0.2})\mathrm{Fe}_{11}\mathrm{Ti}$ at

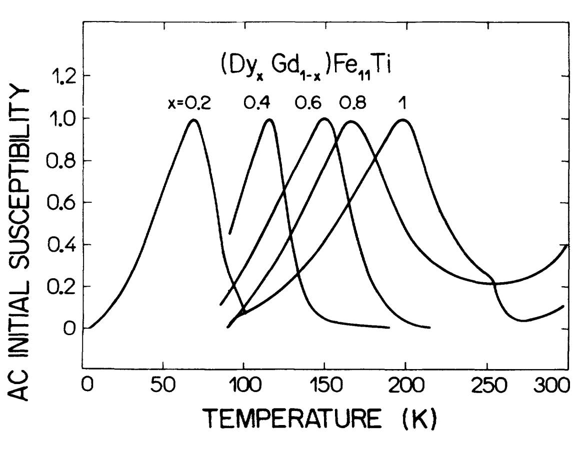

FIG. 1. Temperature dependence of ac susceptibility (in arbitrary units) of $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ unoriented powder samples. Data were arbitrarily normalized for display.

TABLE I. Curie temperatures (T_C) and axis-cone spin reorientation temperatures (T_{SR}) of $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ alloys.

x	0.2	0.4	0.6	0.8	1.0
T_C (K)	587	574	565	545	534
T_{SR} (K)	69	116	147	167	196

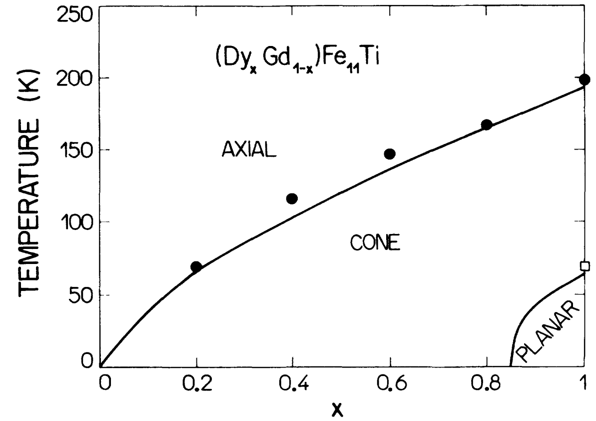

FIG. 2. Concentration dependence of spin reorientation temperature of $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ alloys, determined form susceptibility vs temperature maxima (black circles). The cone-planar transition temperature for $\mathrm{DyFe_{11}Ti}$ (white square) is from Ref. 11. Full curves: theoretical phase boundaries, calculated with model described in text.

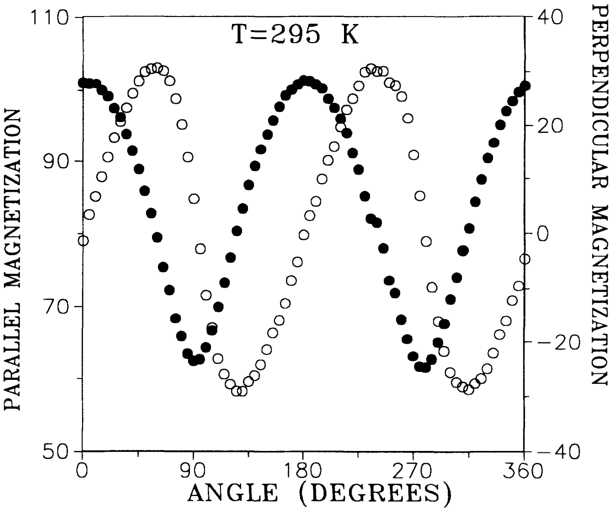

FIG. 3. Room-temperature magnetic polar plot for $(\mathrm{Dy}_{0.8}\mathrm{Gd}_{0.2})\mathrm{Fe}_{11}\mathrm{Ti}$. $M_{\parallel}$ (black circles) and $M_{\perp}$ (white circles) magnetization components with respect to applied field ($B_{\mathrm{app}}=0.5$ T) are shown (in arbitrary units) as functions of rotation angle. Angles 0° or 180° correspond to magnetic alignment direction of powder sample.

room temperature. $M_\parallel$ is a maximum and $M_\perp$ vanishes for a field direction coinciding with the easy axis of the aligned powder ($\alpha=0°$ or $180°$). The data shown are very similar to the polar plots obtained on a monocrystalline $\mathrm{DyFe_{11}Ti}$ sample,[11] demonstrating that our oriented powders have acquired a substantial uniaxial anisotropy at room temperature.

Figure 4 shows some $M_\parallel$ plots of $(\mathrm{Dy_{0.8}Gd_{0.2}})\mathrm{Fe_{11}Ti}$ at lower temperatures. The maximum around $\alpha=180°$ is seen to progressively flatten and to eventually split into two symmetrical maxima as the easy magnetization direction deviates from the initial alignment direction. For all samples, the onset of these changes in polar plot shape coincides, within at most 10 K, with the T_{SR} obtained from susceptibility data. The observed decrease in amplitude of the $M_\parallel$ variation with α reflects the smaller anisotropy in the spin reorientation temperature range. Some similar data for $(\mathrm{Dy_{0.6}Gd_{0.4}})\mathrm{Fe_{11}Ti}$ are shown in Fig. 5.

The angular separation between split maxima corresponds to twice the canting angle θ between magnetization direction and the c axis, which can thus be determined. Even where the maxima are poorly resolved, owing either to a small θ or to imperfect alignment of the powder samples, θ can be visually evaluated within ±10°. The canting angles thus determined are summarized in Fig. 6, with the exception of $\mathrm{DyFe_{11}Ti}$ which has been discussed in detail elsewhere.[11] This figure also displays the theoretically calculated temperature dependence of the canting angle θ. The agreement with experiment is found to be quite satisfactory.

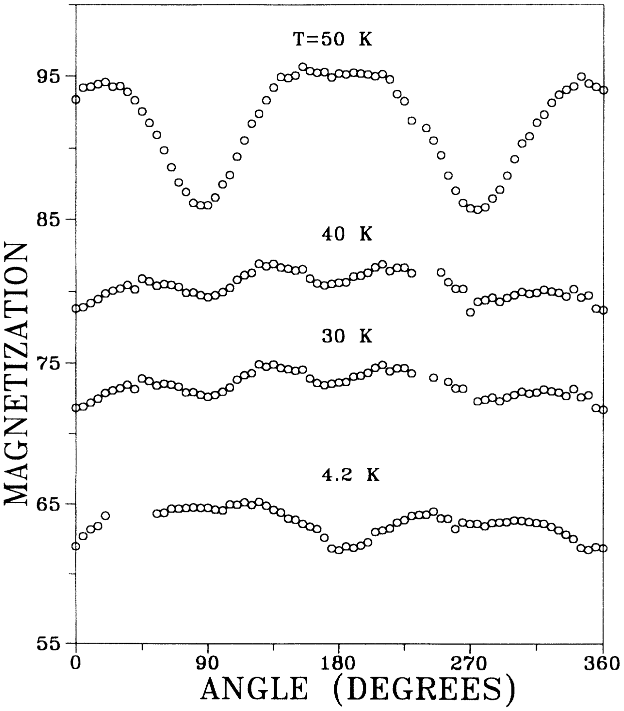

FIG. 4. Magnetic polar plots ($M_\parallel$ only in arbitrary units) of $(\mathrm{Dy_{0.8}Gd_{0.2}})\mathrm{Fe_{11}Ti}$ at indicated temperatures.

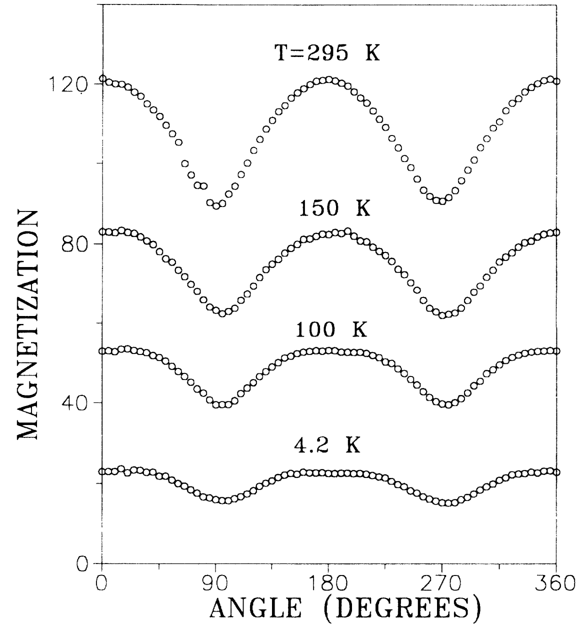

FIG. 5. Magnetic polar plots ($M_\parallel$ only in arbitrary units) of $(\mathrm{Dy_{0.6}Gd_{0.4}})\mathrm{Fe_{11}Ti}$ at indicated temperatures.

One interesting result is that the conical-planar transition, that occurs for $\mathrm{DyFe_{11}Ti}$ at 69 K, is suppressed by Gd substitution at relatively small concentrations. The absence of this transition for $(\mathrm{Dy_{0.6}Gd_{0.4}})\mathrm{Fe_{11}Ti}$ can be noticed on the polar plots of Fig. 5, which show well-defined minima at $\alpha=90°$ and 270° at all temperatures, including 4.2 K. Our calculations corroborate this result, as shown in Fig. 6. The dashed curve in this figure is the theoretical θ vs T for $(\mathrm{Dy_{0.86}Gd_{0.14}})\mathrm{Fe_{11}Ti}$, the highest Gd concentration to support a planar ground state, according to calculations (see the conical-planar boundary in Fig. 2). The $(\mathrm{Dy_{0.8}Gd_{0.2}})\mathrm{Fe_{11}Ti}$ alloy should therefore be expected to remain conical at low temperatures, with $\theta \leq 40°$. The experimental result for this composition is not totally clear, however: the $T=4.2$ K polar plot in Fig. 4 exhibits two blurred maxima, from which, at best, an average $\theta \approx 60°$ can be estimated. A full $\theta=90°$ reorientation, on the other hand, would not be consistent with these data points. The low-temperature spin structure of this alloy is most likely to be inhomogeneous, due to the proximity of the critical concentration for stability of a planar state.

Some remarks are pertinent on the possible effect on T_{SR} of the applied magnetic field during the polar-plot experiments. Intuitively, an applied field parallel to the c axis adds stability to the axial magnetization, thereby depressing the SR temperature. For $\mathrm{DyFe_{11}Ti}$, a calculation has shown[8] that a longitudinal field of 0.3 T should decrease the axis-cone and the cone-plane transition temperatures by 16 and 37 K, respectively. In an angular-rotation experiment, however, a field component in the basal plane is also present, the effect of which will further depend on the particular orientation of each crystallite if a powder sample is considered. We did not attempt such

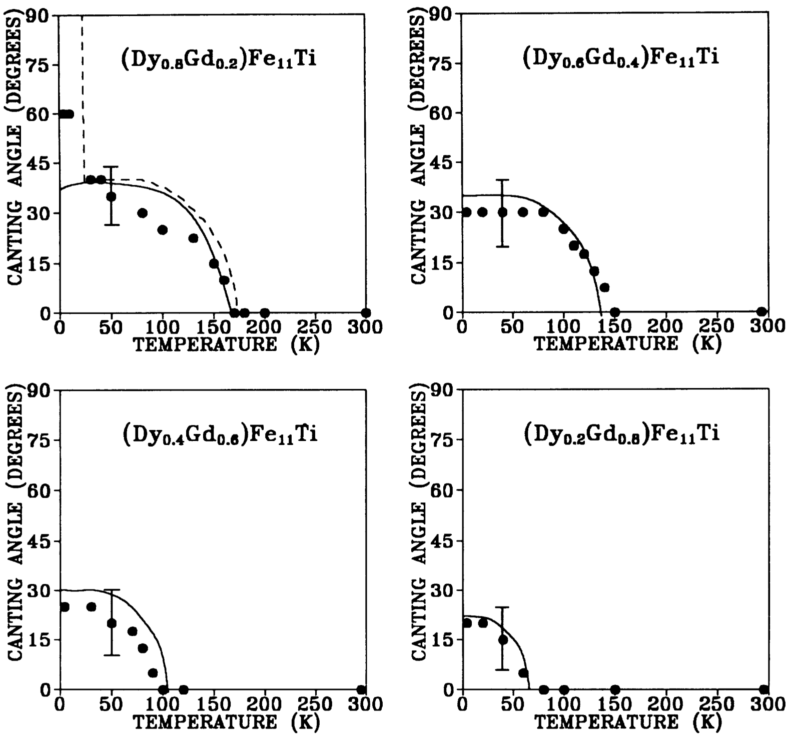

FIG. 6. Black circles: canting angle θ of magnetization with respect to c axis, as determined from magnetic polar plots. Full curves: calculated θ vs T for indicated alloy concentrations. Dashed curve: calculated θ vs T for $(\mathrm{Dy}_{0.86}\mathrm{Gd}_{0.14})\mathrm{Fe}_{11}\mathrm{Ti}$, showing cone-plane transition.

a calculation, but it is reasonable to expect that the field effect on T_{SR} should be reduced by averaging, as compared to the single-crystal, axial-field case. In the present study, the observed agreement between the susceptibility peak T_{SR} and the reorientation onset in polar plots is an indication that this effect is indeed small. This may not always be the case, e.g., the spin reorientations in $(\mathrm{Tb}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ (Refs. 8 and 13) do exhibit a pronounced field sensitivity.

V. SUMMARY AND CONCLUSIONS

We have investigated the spin reorientation in pseudoternary $R\mathrm{Fe}_{11}\mathrm{Ti}$ compounds, with Dy and Gd randomly occupying the R sites. The axis-cone SR temperatures could be accurately determined from ac susceptibility measurements, since this transition gives rise to a sharp peak in the χ vs T plots. We have also measured the magnetization angular dependence of oriented powders in a fixed applied field. As the resulting polar plots are quite sensitive to deviations of the easy magnetization direction from the initial alignment axis, the canting angle θ could be determined. The SR temperatures obtained from both methods were consistent with each other. Theoretical calculations for both the T_{SR} vs x and the θ vs T dependences have been performed with a single-ion model for the rare earth, using crystal-field parameters previously obtained by Hu *et al.* by fitting single-crystalline $\mathrm{DyFe}_{11}\mathrm{Ti}$ magnetization data. Very good agreement between theory and experiment was obtained. No evidence for a cone-plane reorientation transition, similar to that occurring for $\mathrm{DyFe}_{11}\mathrm{Ti}$ at low temperatures, has been found for any of our alloys. This result is also in agreement with model calculations which predict the suppression of this transition for Gd concentrations as low as 0.15.

In conclusion, we have shown that the magnetic properties of $(\mathrm{Dy}_x\mathrm{Gd}_{1-x})\mathrm{Fe}_{11}\mathrm{Ti}$ mixed compounds can be quantitatively accounted for with the single-ion model appropriate for $\mathrm{DyFe}_{11}\mathrm{Ti}$, without changing any parameters or introducing new ones.

ACKNOWLEDGMENTS

This work was partially supported by Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP) and by Comisión de Investigaciones Científica y Tecnológica (CICYT) under Grant No. PB92-0095. L.C.C.M.N. acknowledges financial support from Conselho de Desenvolvimento Científico e Tecnológico (CNPq) and a traveling grant from Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES).

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