# Magnetic properties of oxygen-reduced, rare-earth based $R_1Ba_2Cu_3O_{7-x}$ compounds with $R$ = Gd, Dy, and Ho

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The bulk magnetic properties of tetragonal, oxygen-depleted, polycrystalline samples of sintered $R_1Ba_2Cu_3O_{7-x}$ compounds with $x=0.95$ have been investigated. Materials containing the rare-earth (RE) ions Gd, Dy, and Ho were studied. In each case, the magnetic susceptibility $\chi$ was described by a Curie-Weiss (CW) dependence with effective moments that were very nearly equal to the free-ion values—behavior that is very similar to the superconducting counterparts of these materials where the oxygen has not been depleted. The field dependence of the magnetization $M(H,T)$ was found to be substantially modified by crystalline electric field effects and by the influence of RE-RE interactions.

## I. INTRODUCTION

A magnetization study has been conducted on several polycrystalline rare-earth-based materials that were derived from members of the 90-K family of superconducting mixed oxides.[1-4] These superconductors have the generic composition $R_1Ba_2Cu_3O_{7-x}$, with $x\approx 0$ and where the rare-earth (RE) species can be Y or any of the $4f$ series except Ce or Pr.[5,6] As is now well established, these materials are orthorhombic with a structure that is derived from the perovskites.[7] By a careful and controlled removal of oxygen, they can be rendered nonsuperconducting while maintaining a perovskite-related tetragonal structure that has very nearly the same lattice parameters as their superconducting counterparts.[8] The tetragonal compounds are stable down to an oxygen stoichiometry of six, i.e., $x=1$. Several studies have shown that the superconducting transition temperature $T_c$ of the superconducting phase is not significantly affected by the substitution of magnetic rare-earth ions for the nonmagnetic ion $Y^{3+}$.[5,6] Furthermore, we have previously presented evidence demonstrating that the superconducting and RE magnetic properties are independent to a high degree.[9,10] Consequently, it is of interest to investigate nonsuperconducting, oxygen-depleted forms of these same materials in order to compare their properties with those of stoichiometric samples. In this way, useful magnetization and susceptibility measurements can be obtained without the presence of hysteretic signals arising from superconducting currents.

An additional reason for investigating the magnetic properties of the oxygen-depleted RE-based compounds arises from the observation[11] of antiferromagnetism (AFM) in the related tetragonal compound $La_2CuO_4$. In this case, the magnetism originates from the Cu—O complexes that are thought to be intimately associated with the high-$T_c$ values in both the 35- and 90-K families of superconducting materials. Furthermore, long-range AFM ordering has recently been observed in neutron scattering studies[12] of oxygen-depleted Y 1:2:3 and Nd 1:2:3 materials and in $Y_1Ba_2Cu_3O_{7-x}$ with $x=1.0$ and 0.85.[13] Since the possible existence of a relationship between magnetic order and the relatively high-$T_c$ values has important theoretical implications, it is obviously of interest to investigate further the magnetic properties, including Cu-O based antiferromagnetism, in the 1:2:3-type materials as well. In fact, Dunlap *et al.*[14] have reported studies of the magnetic susceptibility $\chi$ of oxygen-deficient Gd 1:2:3 derived materials where an excess contribution to $\chi$ was observed. A substantial deviation from the Curie-Weiss (CW) susceptibility of Gd was found that peaked at a temperature of 18 K. This result was interpreted as an

 

antiferromagnetic transition in the Cu–O subsystem. Because of its important theoretical implications, we have independently investigated the Gd-based, oxygen-deficient compound as well as the related Dy- and Ho-based materials. Several interesting comparisons with the related superconductors were found. We do not, however, observe a magnetic anomaly in Gd 1:2:3 of the type reported by Dunlap *et al.* Studies of the Dy- and Ho-based compounds reveal a substantial influence of crystalline electric field (CEF) effects on the magnetic properties.

## II. EXPERIMENTAL ASPECTS

The materials used in these investigations were first prepared (using standard techniques[7,9]) as superconducting samples, with $T_c$ values between 90 and 95 K and large superconducting-state diamagnetism. In order to remove oxygen in a highly controlled and reproducible manner, the samples were heated (at 10 K/min) in He gas in a thermogravimetric apparatus so that the sample mass could be monitored continuously. When the mass loss corresponded to the desired oxygen stoichiometry, the sample was quickly cooled to room temperature with no additional weight change.[15] The overall mass loss was verified by external measurements as well. The oxygen defect parameter $x$ was determined to within an accuracy of about $\pm 0.05$. No phases other than the desired tetragonal phase were detected by x-ray diffraction. For measurements of the magnetic properties, a vibrating sample magnetometer was employed using applied magnetic fields up to 78 kOe. The field was applied parallel to the long axis of flat platelike samples, resulting in small demagnetization corrections to the susceptibility. Molar magnetic quantities refer to Avagadro's number $N_A$ of $R_1Ba_2Cu_3O_{7-x}$ formula units or equivalently to 1 mol of RE ions. The sample temperature was measured using a calibrated Si diode that was checked at several fixed points and a magnetic field-insensitive carbon glass resistor.

## III. EXPERIMENTAL RESULTS AND DISCUSSION

### A. Gd 1:2:3

Gd-based $R_1Ba_2Cu_3O_{7-x}$ was prepared with $x=0.95$, i.e., an oxygen content of 6.05. The susceptibility $\chi$ was determined by measurement of the magnetic moment in a fixed applied field of 414 Oe. The results are shown in Fig. 1 as a plot of reciprocal susceptibility versus temperature. The linear relationship shows that $\chi$ follows a Curie-Weiss dependence,

$$\chi=\chi_0+C/(T+\Theta) , \quad (1)$$

where $C(=N_A\mu_B^2p^2/3k_B)$ is the (molar) Curie constant, $T$ is the temperature, $\Theta$ is the Curie-Weiss temperature, and $\mu_B$ is the Bohr magneton. The temperature-independent term $\chi_0$ arises from core diamagnetism and, in some instances, may be dominated by van Vleck contributions (or other contributions such as Pauli paramagnetism

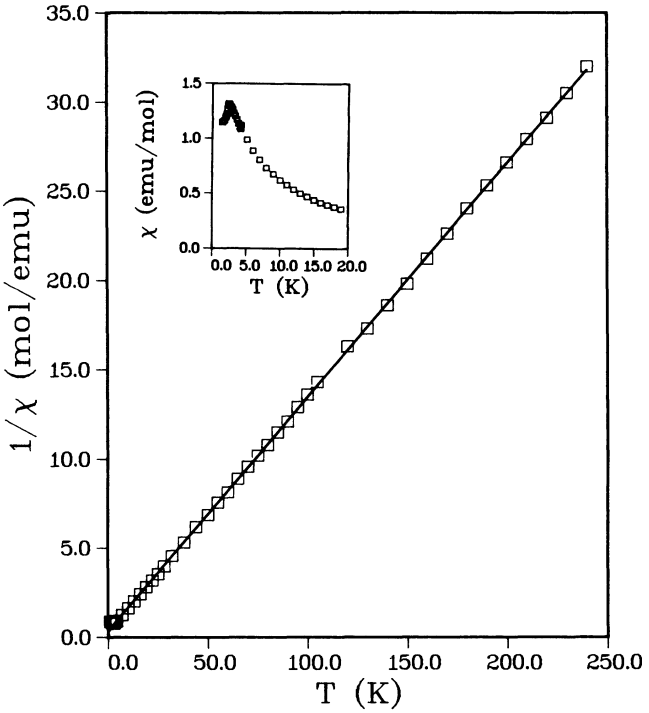


FIG. 1. The reciprocal of molar susceptibility $\chi$ of $Gd_1Ba_2Cu_3O_{6.05}$ vs temperature $T$. Inset: $\chi$ vs $T$, showing the antiferromagnetic transition of the Gd system.

in the case of the superconducting compounds). For the RE 1:2:3 compounds investigated here, the temperature-dependent RE paramagnetism completely dominates the constant term $\chi_0$. Fitting the data obtained above $T=50$ K with Eq. (1), we obtain a slope that corresponds to an effective moment per Gd ion, $p$, of 7.82; this determination is in good agreement with the Gd free-ion value of 7.92. The fitted value for the CW temperature $\Theta=+2.7$ K is quite close to the antiferromagnetic transition temperature $T_N$. The AFM transition is evident as a peak in the plot of $\chi$ vs $T$ (Fig. 1, inset); the peak temperature 2.4 K is close to the values found for specific-heat anomalies.[16–18] In addition, neutron scattering studies have revealed the onset of long-range magnetic order at temperature $T_N$ of 2.3 K.[19] Furthermore, these studies showed that the ordering is three dimensional (3D) in nature with the magnetic moments perpendicular to the basal plane. These properties cannot be accounted for by the magnetic dipolar interaction alone. Incorporation of indirect RE-RE interactions via virtual exchange of electrons away from the Fermi surface, however, provides a possible mechanism for the observed 3D ordering of moments.[20] This accounts for the fact that both the superconducting and semiconducting, oxygen-depleted RE 1:2:3 compounds exhibit similar magnetic properties. In fact, we have previously shown[9,10] that the Curie-Weiss behavior of superconducting Ho and Gd 1:2:3 observed above $T_c$ continues to temperatures well below $T_c$—demonstrating that there is substantial independence of superconductivity and RE magnetism in these materials.

In earlier work, Dunlap *et al.*[14] reported a significant

anomaly in the susceptibility of oxygen-depleted Gd 1:2:3 material. This anomaly was interpreted as AFM ordering of the Cu-O subsystem. In order to detect the presence of an added component in $\chi$, we show in Fig. 2 the deviation of the experimental data from the same Curie-Weiss law, extrapolated to lower temperatures. It is evident from these results that, within experimental error, the data follow Eq. (1) with decreasing temperature $T$ until deviations finally develop from the approaching *gadolinium* AFM transition. The earlier work of Dunlap *et al.* reported[14] a maximum deviation in $\chi$ at $T=18$ K of 0.18 $cm^3/mol$ of Gd. This deviation is well outside of our experimental uncertainties in the present work. We note that the data of van den Berg *et al.*[21] which extend up to 20 K also exhibit no visible anomaly in $\chi$ other than the AFM transition at 2.3 K. We can only conclude that the samples employed here and in the work of Dunlap *et al.* differ due to different processing conditions, porosity, etc. Our results have shown, for example, that the quantity of oxygen removed from various RE 1:2:3 materials depends on the RE species as well as the microstructure of the individual sample.[15] It is therefore highly desirable to make either an *in situ* measurement or careful subsequent analysis of oxygen content or both.

At low temperature, the Gd magnetization $M$ became a nonlinear function of the applied field $H$ and tended to saturate in large fields. This effect is illustrated in Fig. 3, a plot of $M$ vs $H$ at temperatures of 4.5, 10.0, and 20.0 K. In order to incorporate the influence of Gd-Gd antiferromagnetic interactions, we include an additive thermal factor $\Theta_M$ in the argument of the model relation and obtain the result

$$M(H,T)=M_{\rm sat}B_J(g\mu_B JH/k_B(T+\Theta_M)) \ , \qquad (2)$$

where $B_J$ is the Brillouin function for angular momentum $J$ and $M_{\rm sat}$ is the saturation magnetization; for free Gd ions with $S=J=\frac{7}{2}$ and $g=2$, its molar value is $3.91\times10^4$ $G cm^3/mol$. The broken curves in Fig. 3 were drawn using $\Theta_M=+2.7$ K and free-ion parameters for the other quantities. The fitted value of $\Theta_M$ coincides with that obtained from the susceptibility, $\Theta$, which may be somewhat fortuitous, although the values are expected to be comparable in magnitude for the $S$-state $Gd^{3+}$ ion. In other studies,

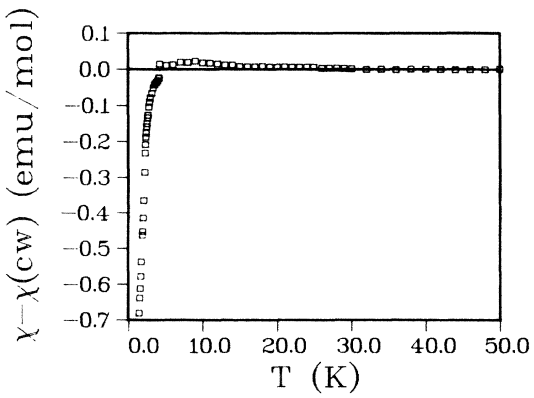


FIG. 2. The deviation of the data in Fig. 1 from the high-temperature ($T>50$ K) Curie-Weiss dependence.

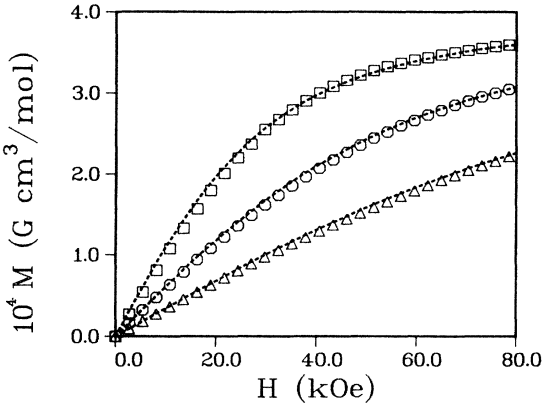


FIG. 3. The magnetization per mole of oxygen-depleted Gd 1:2:3 compound vs applied magnetic field $H$, at temperatures of 4.5 K (□); 10.0 K (○); and 20.0 K (△). The broken lines are model fits using Eq. (2).

Huang *et al.*[22] have shown that the AFM transition is itself suppressed in the presence of a large magnetic field. The model relation given in Eq. (2) has been shown to describe the magnetization $M(H,T)$ of the superconducting Gd 1:2:3 compound[10] as well as the 4.2-K magnetization of semiconducting Gd 1:2:3 materials.[23]

### B. Dy 1:2:3

We now consider the magnetic properties of the Dy-based compound $Dy_1Ba_2Cu_3O_{7-x}$ with $x=0.95$, as above. In order to measure the susceptibility, a fixed field of 280 Oe was applied to the sample. The results are presented in Fig. 4 as a CW plot. Fitting the experimental data above 100 K with Eq. (1), which describes the measurements quite well, yields a value of $p=10.77$ for the effective moment of the Dy ion. This value is slightly larger than the free-ion value of 10.64 and our result[24] obtained for superconducting Dy 1:2:3. For the CW temper-

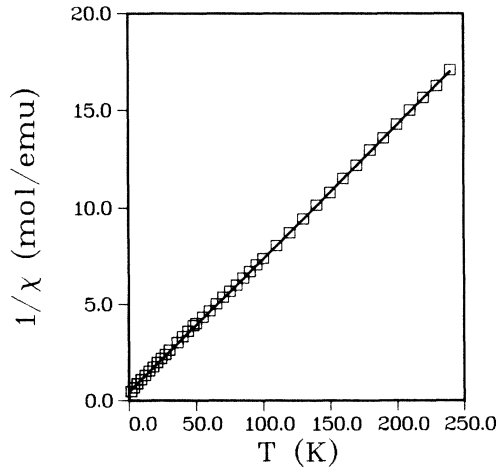


FIG. 4. The reciprocal molar susceptibility of $Dy_1Ba_2Cu_3O_{6.05}$ vs temperature.

ature $\Theta$, we obtained a value of +6.8 K. Examination of the deviations (not shown) from the high-temperature ($T > 100$ K) CW law, $\chi - \chi_{CW}$, reveals a small (negative) peak near 16 K. This coincides in temperature with a peak in the specific-heat capacity[17] that has been identified as a Schottky anomaly arising from a 40-K splitting of the Dy levels. From entropy and other considerations, both the ground state and first excited state were identified as doublets. Being a Kramers ion with half-integral spin, the Dy ion will retain at least a twofold degeneracy of its levels in the tetragonal electrostatic field of the crystal. At sufficiently low temperatures, only the ground-state doublet will be appreciably populated, and this may account for the larger deviations from a CW dependence at temperatures below about 4 K.

Unlike the case of a free $Gd^{3+}$ ion which has orbital angular momentum $L=0$, the free $Dy^{3+}$ ion has $L=5$ in an atomic configuration $^6H_{15/2}$. Its coupling to the crystal field produces a splitting of levels which is evident in the specific heat. Again, we attribute the measured CW temperature $\Theta$ to a combination of the influence of crystal-field effects and rare-earth dipole-dipole interactions. Due to the complexity of the system and a lack of knowledge of the quantum-mechanical states comprising the various crystal-field levels, however, we have not attempted to calculate the temperature-dependent susceptibility $\chi(T)$.

The isothermal magnetization results can provide complementary information on the properties of the Dy 1:2:3 material. In Fig. 5 a set of magnetization curves $M(H)$ is shown for temperatures $T$ of 1.5, 4.2, 10, 20, and 30 K. The first striking feature of these results is that the observed magnetization is considerably reduced from the saturation value for the free ion which is $10.0\mu_B$/Dy ion or equivalent $5.58\times10^4$ G cm$^3$/mol. This condition persists even in an applied field of 80 kOe at 1.5 K, and this is due to the admixture of angular momentum $J$ states and their splitting by the crystal field. Considering the high-field portions of the $M(H)$ curves at 1.5 and 4.2 K, one finds that $M$ increases approximately linearly with $H$. This suggests that the magnetization has two components: a saturating component due to the ground-state doublet and a second component roughly proportional to $H$. Qualitatively, the latter term can arise from a van Vleck susceptibility $\chi_{vv}$ associated with higher-energy crystal-field levels; alternately, this behavior may originate from an anisotropic magnetic response.

To model the magnetization, we include a linear term in the expression

$$M(H,T) = M_{sat} B_J(g\mu_B JH/k_B(T+\Theta_M)) + \chi_{vv} H \ . \quad (3)$$

The quantum number $J$ has been set equal to $\frac{1}{2}$ corresponding to a spin degeneracy of two in the ground-state doublet. With one exception, the broken curves in Fig. 5 have all been drawn using a single consistent set of parameters: $M_{sat} = 3.0\times10^4$ G cm$^3$/mol, $g=9$, $\Theta_M = 4.0$ K, and $\chi_{vv} = 0.11$ cm$^3$/mol. For the curve at $T=30$ K, a slightly reduced value of 0.09 cm$^3$/mol was used for $\chi_{vv}$, which is justified considering that this temperature is a large fraction of the 40-K splitting to the first excited doublet.[17] The resulting modeling is satisfactory, given the complexity of the energy levels and states. The values of the fitted parameters are reasonable; the value of $\Theta_M$ is comparable with both the CW temperature 6.8 K and the magnetic dipolar interaction energy. The value of $\chi_{vv}$ corresponds to a matrix element for the magnetic moment in the first excited state, $\langle | L+2S | \rangle$, of a few Bohr magnetons. Finally, the saturation magnetization corresponds to a moment of $5.5\mu_B$/Dy ion in the ground-state doublet. This is smaller than, but roughly comparable to, the value of $7.2\mu_B$ found by neutron scattering using antiferromagnetic, superconducting Dy 1:2:3 material.[25] In addition, a moment of $\approx 6\mu_B$ was estimated from the hyperfine component of the specific heat.[17] Overall, the modeling fits the experimental measurements adequately, and the derived parameters are reasonable with the correct order of magnitude. This procedure, however, has averaged over many details of the exact configuration of the $Dy^{3+}$ ion. Accordingly, the current results on bulk, polycrystalline samples are no substitute for information obtainable only from single-crystal measurements and microscopic studies.

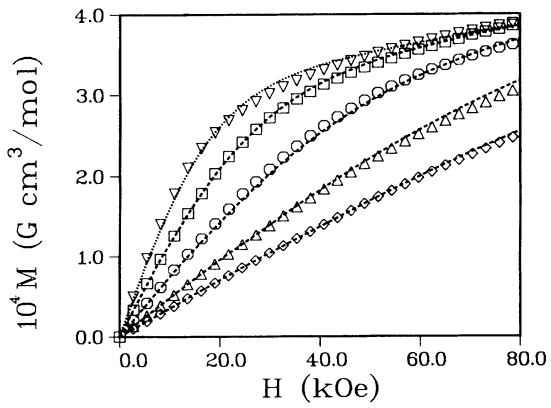


FIG. 5. The molar magnetization of oxygen-depleted Dy 1:2:3 vs field $H$ at 1.5 K (▽); 4.2 K (□); 10.0 K (○); 20.0 K (△); and 30.0 K (◇). The broken lines are calculated using Eq. (3) in the text.

### C. Ho 1:2:3

Oxygen-depleted $Ho_1Ba_2Cu_3O_{6.05}$ was prepared and its susceptibility was measured in an applied field of 300 Oe. Figure 6 shows that $\chi$ again follows a Curie-Weiss law over a rather broad temperature range. The line was fit to the data between 50 and 240 K, yielding a CW $\Theta$ of +9.5 K. Attributing the temperature dependence solely to the Ho ions, one obtains an effective moment $p$ of $10.78\mu_B$/Ho ion, compared with the value of 10.60 for the free ion. These results differ only slightly from those obtained for superconducting Ho 1:2:3 material. As shown in the inset of Fig. 6, the susceptibility curve flattens considerably below about 4 K, which can be understood qualitatively in terms of a distribution of low-lying CEF energy levels. In particular, specific-heat studies[17,18] have shown that Ho 1:2:3 has a large excess heat capacity that is spread over a broad temperature range above 1 K and that arises from a sum of Schottky anomalies. Thus, the observed flattening

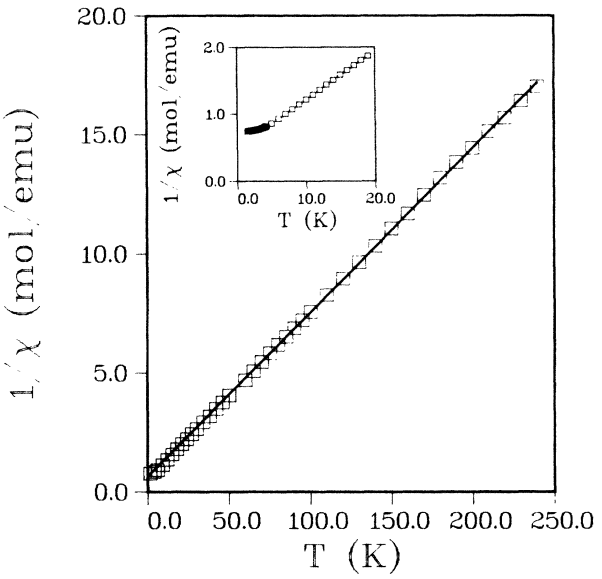


FIG. 6. The inverse molar susceptibility of $Ho_1Ba_2Cu_3O_{6.05}$. Inset: $1/\chi$ vs $T$ at low temperature.

of the $\chi$ curve is due to a thermal depopulation of these low-lying levels with eventual occupation of only the singlet ground state of the non-Kramers $Ho^{3+}$ ion.

A set of isothermal magnetization curves for Ho 1:2:3 is shown in Fig. 7 for temperatures of 4.5, 10, 20, and 30 K. These data are very similar in appearance to the corresponding results for Dy 1:2:3, despite the differences in energy-level schemes deduced from specific-heat studies. This may arise from a mixing of levels due to the large applied magnetic field, combined with the existing admixture of angular momentum $J$ states in individual CEF levels. Mindful of this considerable complexity, we have nonetheless applied the same model relation [Eq. (3)] to the Ho results that was used for Dy 1:2:3 so that the bulk magnetic properties can be compared. The broken curves shown in Fig. 7 are the result of this procedure (again using $J=\frac{1}{2}$). The values of the fitted parameters are

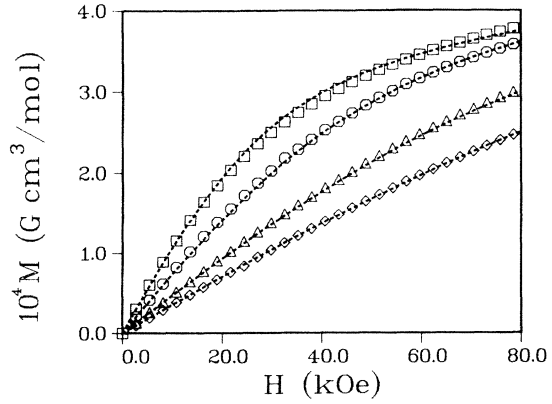


FIG. 7. The molar magnetization of oxygen-depleted Ho 1:2:3 at 4.5 K (□); 10 K (○); 20.0 K (△); and 30 K (◇). Broken lines are model fits (see text).

$M_{sat}=2.9\times10^4$ Gcm$^3$/mol, which is considerably smaller than the free-Ho-ion value of $5.58\times10^4$ Gcm$^3$/mol; $g=10$; and $\Theta_M=5.3$ K. For the linear term $\chi_{vv}$, these values, in units of cm$^3$/mol, were used: 0.11 at $T=4.5$ and 10 K, 0.09 at $T=20$ K, and 0.08 at $T=30$ K. The modeling follows the experimental results presented in Fig. 7 fairly well and yields parameters that are comparable to those obtained for Dy 1:2:3. It bears repeating, however, that this procedure averages over considerable detail on the microscopic level and, therefore, the results must be interpreted in this light.

## IV. DISCUSSION AND CONCLUSIONS

It is evident that the magnetic properties of the oxygen-depleted, semiconducting, Gd-, Dy-, and Ho-based RE 1:2:3 materials investigated here are very similar to those of their superconducting counterparts. The explanation of these observations is based on the fact that the crystal structures and lattice spacings are nearly identical; in addition, the RE's have very limited (if any) interaction with the conduction electrons at the Fermi surface,[26] so that the electrical conductivity has little influence.

An interesting contrast for both Dy and Ho 1:2:3 is the presence of a CW susceptibility that extends to relatively low temperatures. In seeming contradiction, both the magnetization and published specific-heat studies clearly show the influence of the CEF in these compounds. Additional evidence for CEF effects comes from inelastic neutron scattering studies in the case of Er 1:2:3 that demonstrated the existence of substantial level splitting in that system.[27] This seemingly contradictory set of circumstances may arise when relatively low-lying levels contain high angular momentum (and large magnetic moment) $J_z$ states. In several aspects, the $R\mathrm{Rh_4B_4}$ series is similar to the RE 1:2:3 compounds. For example, $\mathrm{DyRh_4B_4}$ has been shown[28] to follow a CW law down to $\approx 50$ K, and yet specific-heat studies indicate substantial level splitting from the CEF. Very similar results have been reported[29] for $\mathrm{TmRh_4B_4}$ in which the magnetic species is a non-Kramers ion, similar to Ho. Later studies[30] on a single crystal of $\mathrm{ErRh_4B_4}$ were used to establish a set of CEF parameters for this material, from which the magnetic susceptibility tensor was calculated. Although the calculated components of $\chi$ differed considerably, a polycrystalline average of $\chi$ was found to follow a CW dependence as observed experimentally. Similar theoretical results were found for other $R\mathrm{Rh_4B_4}$ compounds, using scaled CEF parameters. Furthermore, experimental studies of the hexagonal actinide compound californium trichloride have shown[31] that, while the measured susceptibility is anisotropic, a polycrystalline average of $\chi$ results in a CW dependence. It is likely that an analogous situation prevails in the case of the RE 1:2:3 materials. In future studies, it would be very desirable to complement the current work with single-crystal and inelastic neutron studies in order to arrive at a detailed understanding of the magnetic properties of the RE 1:2:3 compounds.

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