Electron paramagnetic resonance in gadolinium near T_C †

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Electron paramagnetic resonance (EPR) in single crystals of gadolinium metal has been studied near its Curie temperature T_C ($\simeq$293 K). The EPR linewidth ΔH and the resonance field H_r are measured as a function of temperature between 280 and 330 K, for $\vec{H} \parallel c$ axis and $\vec{H} \perp c$ axis. Between 300 and 330 K the line shape is Dysonian and ΔH shows a crossover behavior: i.e., ΔH increases as $T \to T_C^+$, reaching a maximum at $T_m > T_C$ and then decreases for lower temperatures. This behavior of ΔH is similar to the observations in ferromagnets $CrBr_3$, EuO, and Ni. The derived values for the critical spin-spin relaxation rate in Gd are observed to scale as χ^ν, where χ is the uniform field susceptibility. The observed value of $\nu \simeq 0.84$ is about half the theoretical value, similar to the discrepancy observed in antiferromagnets.

I. INTRODUCTION

In the past few years, considerable understanding of the behavior of the electron paramagnetic resonance (EPR) in magnetic insulators near the magnetic ordering temperatures has been reached. Seehra and Huber[1] have reviewed the theoretical and experimental status of the phenomena up to about 1974. Briefly the status is as follows. In antiferromagnets, anomalies in the EPR linewidth ΔH near the Neél temperature T_N are strong functions of the anisotropy. For example, no significant anomaly in ΔH is observed in cubic antiferromagnets like $RbMnF_3$, whereas in anisotropic antiferromagnets like MnF_2 an order of magnitude increase in ΔH is observed as T approaches T_N. This is well understood.[2] Anomalies in ferromagnets depend less significantly on anisotropy and are considerably different from those observed in antiferromagnets because of the different role of the uniform field susceptibility χ in the two cases. In ferromagnets near T_C [e.g., $CrBr_3$ (Ref. 3) and EuO (Ref. 4)] ΔH increases as T approaches T_C until a temperature where $4\pi\chi \simeq 1$ (approximately equal to $\epsilon = (T - T_C)/T_C \simeq 0.1$ in EuO) and then ΔH decreases as T is lowered through T_c. This is consistent with the theoretical predictions.[1,5] A recent development has been the realization[6,7] that ΔH depends on the sample shape in the same way as the resonance field H_r as long as χ is independent of the applied field. Consequently the linewidth ΔH and the theoretically calculated linewidth $\Delta\omega$ in frequency units cannot be simply interchanged. Instead $\Delta H/H_r$ is proportional to $\Delta\omega$, rather than ΔH alone.[4] This has important implications in ferromagnets near T_C since H_r may vary significantly with temperature.

In this paper we report a careful study of the linewidth ΔH and the resonance field H_r in a single crystal of Gd near T_C. Gd is a metal which orders ferromagnetically at $T_C \simeq 293$ K.[8] Gd (with $4f^7 5d^2 6S^2$ electron configuration outside the xenon-atom configuration) contains paramagnetic ion cores $4f^7$ and conduction electrons $5d^1 6S^2$. In a magnetic resonance experiment, the conduction electrons are assumed to give the sample a microwave skin depth, yet they have negligible paramagnetic susceptibility on their own. According to our present understanding,[9] the electrons responsible for strong paramagnetism above T_C as well as for ferromagnetism below T_C are the $4f^7$ electrons which retain almost entirely their localized character in Gd metal. Consequently, in Gd the paramagnetic resonance absorption is expected to be due to the $4f^7$ electrons and not conduction electrons. It is also believed that the exchange interaction between the localized moments is carried via the $5d^1$ and $6S^2$ conduction electrons. Since the ground state of Gd^{3+} is $^8S_{7/2}$, the exchange interaction is primarily Heisenberg-like and the anisotropy near T_C is predominantly uniaxial.[9(b)]

Earlier measurements of the EPR linewidth in Gd have been done only on polycrystalline samples and only few data points are reported near T_C.[10,11] A very important reason for our choice of Gd for this study is the near Heisenberg-like character of the exchange interaction as noted above so that it would be of interest to see how well the predictions of the theory developed for Heisenberg insulators (with dipolar anisotropy) are valid in Gd. Another interesting aspect would be the comparison between the critical EPR dynamics in Gd *vis-a-vis* that observed in transition metal ferromagnets Fe and Ni. In Fe only a narrowing of ΔH has been observed as T_C is approached from above.[12] In earlier measurements similar observations were reported for Ni.[13] However, recent measurements by Sporel and Biller[14] have shown results very similar to those observed in [3]$CrBr_3$ and [4]EuO, viz. ΔH increases as T approaches T_C^+, reaching a maximum at $T_m > T_C (T_m = 365\,°C$, and $T_C \simeq 355\,°C$ for Ni) and then ΔH decreases as the temperature is lowered to T_C. It is very likely that the nonobservation of the critical broadening in Ni and Fe in earlier measurements was due to the fact that most of these measurements were done at microwave frequencies of 24 GHz or higher so that the critical effects were wiped out by the large resonance field necessary to observe the resonance. Our earlier measurements on $CrBr_3$ and EuO as well as the recent measurements of Sporel and Biller were done at 9 GHz. Similarly, the measurements reported here on Gd were also done at 9 GHz. In the following pages of this paper, we present the experimental details, the results and their discussion, and a summary of the major findings in that order. Comparison with the observations in other ferromagnets is made wherever appropriate.

II. EXPERIMENTAL PROCEDURE AND SAMPLE PREPARATION

The single crystals of Gd used in these studies were cut from a piece (99.9% purity) supplied by Materials Limited Corporation. The Curie temperature T_C of this sample was determined to be 292.7 ± 0.2 K, as discussed later. This agrees with the T_C (≃293 K) of high purity Gd,[8] suggesting that the quality of our samples is very good since lower quality commercial Gd samples usually have $T_C \approx 290$ K. For these studies two different samples with different geometries were used in order to minimize the demagnetizing effects. For the basal-plane measurement, a thin disc with the c axis normal to the disc was used. Measurements along the c axis were performed on a long rod with the c axis along the rod axis. In each case the axial ratio was about 6. The single crystal samples were oriented to within ±3° using Laue backscattering of x rays. The samples were cut with a wire saw and hand lapped to the final dimensions. Since the cutting and polishing operations result in a cold worked and possibly contaminated surface, the samples were electropolished prior to the experiments.

The EPR experiments were performed using a standard reflection spectrometer operating at X-band frequencies. The klystron was stablilized to the resonant cavity so that only the derivative of absorbed power is obtained as a function of the applied field. Sample sizes were kept as small as possible (~2 mg mass) in order to avoid cavity overloading and consequent spurious linebroadening.[15] Unlike ferromagnetic insulators, this was not a serious problem in the present case since the effective volume of the sample is considerably reduced due to skin effect.

Sample temperatures between 273 and 330 K were obtained using an ice-water bath and by heating the microwave cavity. The temperatures were easily stabilized to within ±0.03 K using a temperature controller (Artronix Model 5301 E) and a sensitive potentiometer (Leeds and Northrup Model K-5) in conjunction with a copper-constantan thermocouple.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Line shape analysis

The primary difficulty in determining the resonance parameters (ΔH and H_r) of Gd lies with its metallic nature. Using the dc conductivity as guide, the skin depth at 9 GHz in our samples is estimated to be less than 1% of the thickness of the samples. Therefore, the microwave field is not uniform within the sample and a nonsymmetric resonance line (dysonian lineshape) results. The measured A/B ratio of the resonance line (Fig. 1) for $T > 300$ K is equal to 2.3 ± 0.2, which corresponds to the limiting case of the Dysonian line shape when $T_D \gg T_2$ (T_D is the spin-diffusion time and T_2 is the spin-spin relaxation time).[16] This is consistent with the picture presented in Sec. I in that the resonance absorption is due to the S-state ions and not conduction electrons. Consequently diffusion of the spins is negligible in the present case.

The analysis of the line shape to obtain ΔH and H_r follows the procedure used by Peter *et al.*[17]

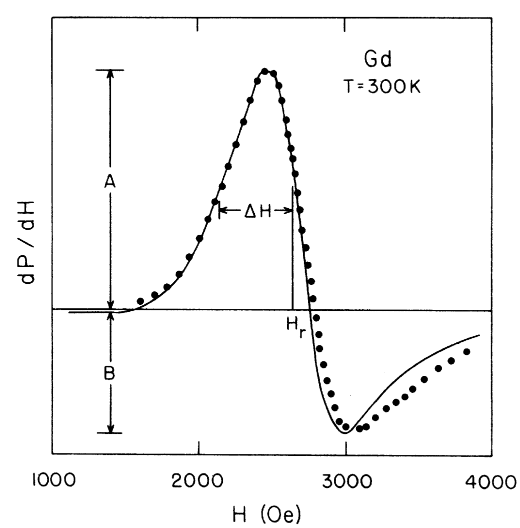

FIG. 1. Typical resonance line as observed in Gd. The solid line is the experimental data (field derivative of the power absorbed) while the dots are the fit discussed in the text. The fit shown was done using $dP/dH = (1 - 3.2x - x^2)/(1+x^2)^2$ with $\Delta H = 493$ and $H_r = 2660$ Oe. This data was obtained at $T = 300$ K with the field parallel to the basal plane direction. The parameters A and B used in the text are indicated.

Due to skin effect, the resonance line is comprised of both absorption and dispersion. The derivative of the line should then follow the function $F(x)$ given by

$$F(x) = (1 - Cx - x^2)/(1 + x^2)^2 , \quad (1)$$

where $x = (H - H_r)/\Delta H$, with H being the applied magnetic field and C is chosen to fit the observed line and determines the asymmetry parameter A/B (we note that for pure dispersion $C = 0$). From such a fit, shown in Fig. 1, the values of H_r and ΔH can be determined, as discussed in considerable detail by Peter *et al.*[17] The parameter C (or the ratio A/B) could have a temperature dependence, especially near T_C, where the microwave skin depth could change rapidly due to varying permeability of Gd. In Fig. 1, the fit to the observed line at 300 K is quite good, especially for fields less than H_r. The ratio $A/B = 2.3 \pm 0.2$ is constant within experimental errors for $330 > T > 300$ K. Below 300 K the ratio A/B decreases, reaching the value of 1.6 ± 0.1 at 290 K. For temperatures below 300 K, we can still fit the resonance lines to Eq. (1). The fit is good for $H < H_r$ but is in error by about 20% for the high-field tail where the experimental data tends to decrease more rapidly than Eq. (1).[18] In all cases, we estimate that the derived values of H_r and ΔH are reliable to ±20 Oe. However, below 300 K, the decreasing value of the ratio A/B means that the line shapes are no longer Dysonian. It is most likely that the changing permeability near and below T_C is affecting the lineshapes.

B. Resonance field and resonance linewidth

Using the procedure outlined above, the measured resonance field H_r and the linewidth ΔH were obtained as a function of temperature ($330 > T > 280$ K) for $\vec{H} \parallel c$ axis (the easy axis) and $\vec{H} \perp c$ axis. The results are shown in Figs. 2 and 3. The actual crystal direction within the c plane is left unspecified because experiments performed at various temperatures showed that any anisotropy in ΔH and H_r within the c plane is less than 3%, which is close to our experimental errors. As can be seen from Fig. 3, there is a considerable difference in the behavior of ΔH along the c axis and along the basal plane orientations, particularly near T_C. This directional anisotropy of ΔH near T_C is discussed in detail later. Above 325 K, there is an indication of the increase of linewidth with increasing temperatures. This is consistent with the work of Chiba and Nakamura,[11] who observed a linear increase in ΔH with temperature ($600 > T > 350$ K) in polycrystalline Gd. They attributed this increase to Korringa-type relaxation.

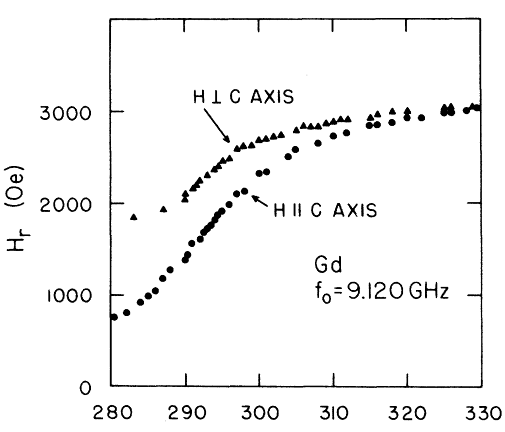

FIG. 2. Resonance field H_r of Gd plotted vs temperature.

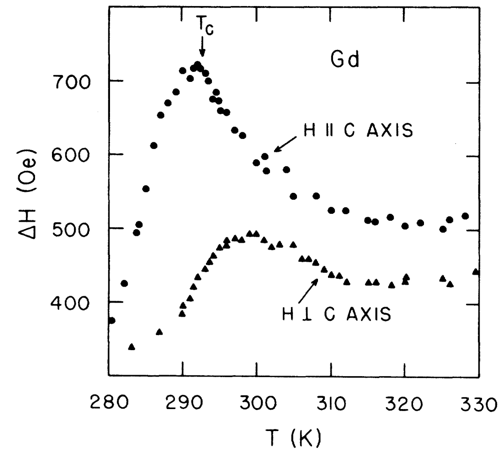

FIG. 3. Resonance line widths of Gd plotted vs temperature.

As noted in the introduction, $\Delta H/H_r$ is the quantity which is proportional to spin-spin relaxation rate $\Delta\omega$ rather than ΔH alone. Therefore we define $\Delta H^* \equiv (\Delta H/H_r)(H_r^\infty)$, where H_r^∞ is the resonance field away from T_c (at $T = 325$ K in the present case). Using the data of Figs. 2 and 3, the derived values of ΔH^* are shown in Fig. 4. Comparing the behavior of ΔH and ΔH^* in Figs. 3 and 4, it is quite evident that they have considerably different temperature dependences near T_C. It should be noted that the proportionality of ΔH^* to $\Delta\omega$ breaks down when χ depends on the applied magnetic field. For a ferromagnet like Gd, this is expected to happen close to T_C so that conclusions based on ΔH^* must be viewed with caution for temperatures where the sample magnetization M at resonance is different from χH. Magnetization measurements taken on our samples indicate that this begins to happen for

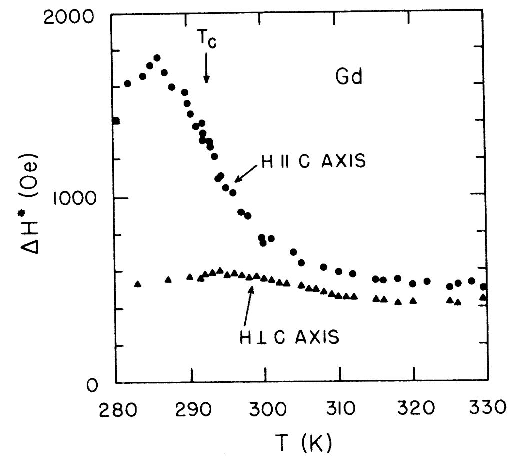

FIG. 4. Values of $(\Delta H)^* = (\Delta H/H_r)H_r^\infty$ of Gd plotted vs temperature. The values of ΔH and H_r are taken from Figs. 2 and 3, respectively, and H_r^∞ is the resonance field away from T_c, viz. 325 K.

temperatures below about 300 K. This is also the region where $4\pi\chi \geq 1$.

C. Critical behavior

From Figs. 3 and 4 it is evident that there is a considerable contribution to the linewidth from the critical fluctuations, the effect being more pronounced for the easy axis. In order to accurately assess the critical behavior we need to know the static magnetic properties of the particular samples used in these measurements. To this end we performed magnetization experiments using a Faraday balance. On the basis of an Arrott plot we find that for this particular sample $T_C = 292.7$ K, in good agreement with the values reported by other workers on high-purity material.[8] Because of the independent thermometry of the EPR and magnetization measurements, the value of T_C should be considered to have an error of ±0.2 K when applied to the EPR data.

By combining our data with the higher-temperature data of Chîba and Nakamura,[11] it is apparent that the EPR linewidth passes through a broad minimum at about 325 K which corresponds to $\epsilon \equiv (T - T_C)/T_C \simeq 0.11$. The minimum is followed by a maximum at a temperature T_m close to T_C. For $\vec{H} \parallel c$ axis, the maximum occurs almost at T_C whereas it occurs near 300 K for $\vec{H} \perp c$ axis. For $T < T_m$, obviously the linewidth decreases with decreasing temperatures (Fig. 3). Note that for ΔH^*, T_m is lowered by several degrees in both directions. However, analysis in terms of ΔH^* below 300 K is suspect since in this region the magnetization M is no longer linear with H. Instead of ΔH^*, ΔH may provide a better qualitative probe for spin relaxation below 300 K as long as the demagnetization corrections are not overwhelming.

The above observation of the linewidth crossover phenomenon in the critical region in Gd, a uniaxial metallic ferromagnet, is quite similar to the observations in insulating ferromagnets $CrBr_3$ and EuO. Thus, many of the predictions for a Heisenberg ferromagnet are found to be valid in Gd, as speculated in the introduction. Now we examine quantitatively the behavior of the EPR linewidth in Gd.

In the analysis of the linewidth in the critical region in $CrBr_3$, a uniaxial ferromagnet, it was shown that $(\Delta H)_\parallel^c/(\Delta H)_\perp^c \simeq 2$, where $(\Delta H)_\parallel^c$ and $(\Delta H)_\perp^c$ are the critical contributions to the linewidths for $\vec{H} \parallel c$ axis and $\vec{H} \perp c$-axis, respectively. We note that this result is mainly due to the uniaxial symmetry and holds for the critical contributions to the linewidth in the critical region ($\epsilon < 0.1$). In order to test this prediction, which was found to be valid in $CrBr_3$ as well as uniaxial antiferromagnets,[1] we must be careful to eliminate the effects of the noncritical contributions to the linewidth which are a significant constant contribution even in the critical region (Figs. 3 and 4). Of course $(\Delta H)^*$ rather than ΔH should be used for the calculations in order to take into account the anisotropy of the measured susceptibility. Hence we have

$$R \equiv (\Delta H)_\parallel^c/(\Delta H)_\perp^c = \frac{(\Delta H)_\parallel^* - (\Delta H)_{\parallel 325}^*}{(\Delta H)_\perp^* - (\Delta H)_{\perp 325}^*} \qquad (2)$$

In Fig. 5, we have plotted R, evaluated according to Eq. (2), as a function of temperature. As can be seen, the prediction of $R = 2$ is a good description of the data down to about 300 K, below which there is an apparent strong divergence of R as $T \to T_C^+$. The behavior below 300 K is discussed

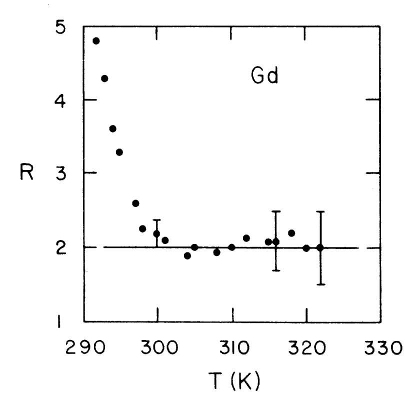

FIG. 5. Ratio R [defined in Eq. (2) of text] for Gd vs temperature as $T \to T_c^+$. $R = 2$ is the theoretical prediction for a uniaxial ferromagnet. The errors are estimates from uncertainties in the linewidth.

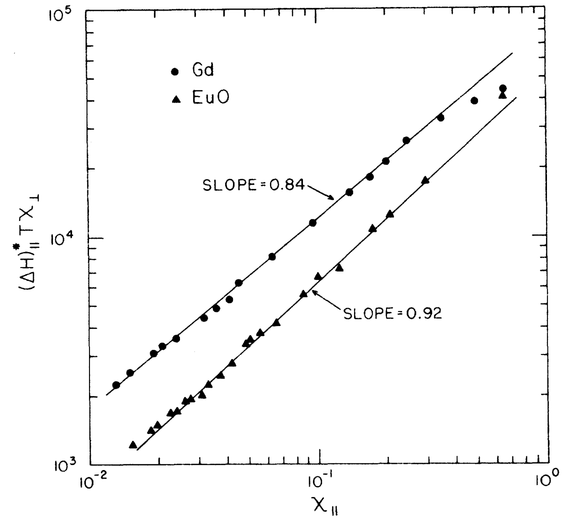

FIG. 6. $(\Delta H)^*_{\parallel} T\chi_{\perp}$ vs $\chi_{\parallel}$. $(\Delta H)^*_{\parallel}$ values are from Fig. 4 and $\vec{H} \parallel c$ axis and $\chi_{\parallel}$ and $\chi_{\perp}$ are the susceptibilities measured on our samples. Similar analysis is shown for EuO, using the data of Ref. 4. The straight lines are drawn using least squares fit. The apparent deviations for larger $\chi_{\parallel}$ are probably due to the crossover discussed in the text.

later in this section.

Now the temperature dependence of the linewidth is examined. The calculations for a uniaxial ferromagnet show[1,3] that $(\Delta H)^*_{\parallel} T\chi_{\perp}$ should vary as $\chi_{\parallel}^{\nu}$ with $\nu = \frac{7}{4}$. A log-log plot of $(\Delta H)^*_{\parallel} T\chi_{\perp}$ vs $\chi_{\parallel}$ is shown in Fig. 6, where we have used the values of $\chi_{\parallel}$ and $\chi_{\perp}$ measured with our samples. The observed $\nu = 0.84 \pm 0.02$ is nearly a factor of 2 smaller than the predicted value. It should be noted that in arriving at $\nu = \frac{7}{4}$, it was assumed that $\chi_{\parallel} \sim \kappa^{-2}$ and $\Lambda \sim \kappa^{1/2}$ (Λ = diffusion constant, κ^{-1} = correlation length), and any anisotropy in the two-spin correlation function was neglected. The latter assumption is definitely incorrect near T_C in a uniaxial magnet. As shown in[19] MnF_2, a uniaxial antiferromagnet, the relaxation rate near T_C is considerably lowered by properly taking into consideration the anisotropic temperature dependence of the parallel and perpendicular correlation functions. From the analysis of the anomalies in relaxation rates in antiferromagnets it is now established that the decoupling of the four-spin correlation function via random phase approximation severely overestimates the strength of the sigularity. Because of these reasons, the discrepancy in the value of ν is not unexpected.

Another significant aspect of the data in Fig. 6 is that the relaxation rate scales as χ^{ν}. This has also been established in[20]EuS, another Heisenberg ferromagnet. A reanalysis of the EPR data in[4]EuO also confirms similar scaling with $\nu = 0.92 \pm 0.01$, shown also in Fig. 6.

As noted earlier, the changing lineshape below 300 K might be due to the increasing permeability of Gd. This is further evident from Fig. 5 where the ratio R no longer follows the prediction for a uniaxial ferromagnet. In order to include the possibility that ΔH rather $(\Delta H)^*$ may be a better measure of the relaxation rate below 300 K in Gd, we also examined the temperature dependence of $(\Delta H)_{\parallel} T\chi_{\perp}$ vs $\chi_{\parallel}$. A fit as good as shown in Fig. 6 but with a slope of 0.70 in the same temperature region was obtained. This variation in the value of ν may be a good estimate of the limit of our confidence in this analysis.

D. Spin-spin relaxation

Here numerical values for the spin-spin relaxation time T_2 are obtained from $(\Delta H)^*_{\parallel}$. Using the theoretical analysis of Ref. 7, one can readily show that $T_2 = \hbar/[g^{\infty}\mu_B(\Delta H)^*_{\parallel}]$ where $h \equiv 2\pi\hbar$ is the Planck constant, g^{∞} is the high-temperature ($T = 325$ K here) g value and μ_B is the Bohr magneton. In deriving this expression for T_2, it was assumed that far away from T_C, g and χ are isotropic, an assumption in good agreement with our observations at 325 K. From our data we estimate that $g^{\infty} = 1.97 \pm 0.02$.[21] This value is in good agreement with the earlier data.[10] Substituting for g^{∞} and the constants, one gets $T_2 = 577 \times 10^{-10}/(\Delta H)^*_{\parallel}$ sec, with $(\Delta H)^*_{\parallel}$ in Oe. Using this expression, T_2 values are directly obtainable from Fig. 4. For example, at 325 K, $(\Delta H)^*_{\parallel} = 507$ Oe, yielding $T_2 = 1.14 \times 10^{-10}$ sec. Similarly, at 310 K with $(\Delta H)^*_{\parallel} = 572$ Oe, $T_2 = 1.01 \times 10^{-10}$ sec. These values of T_2 are in excellent agreement with those obtained by Alexandrakis *et al.*,[9] using transmission resonance at these temperatures. Similar transmission data below 310 K in the critical region is not available for additional comparison with the results of Fig. 4. This excellent agreement for the T_2 values obtained above from the independent absorption and transmission experiments provides additional confidence in the data and analysis presented in this paper.

IV. SUMMARY

The results reported in this paper show that in metallic Gd, the behavior of the EPR linewidth near T_C is similar to the observation in insulating ferromagnets described by Heisenberg exchange interaction (and weaker dipolar anisotropy). It is now evident that in such ferromagnets, the EPR linewidth shows a crossover behavior near T_C viz. an increase followed by a decrease as T approaches T_C^+ as long as the resonance field is considerably smaller than the exchange field. Furthermore, the quantity $(\Delta H)_{\parallel}^{*} T\chi_{\perp}$ is observed to scale as χ^{ν}. These observations are in agreement with the theoretical predictions. However, the observed value of ν is about half the theoretical prediction, similar to the observation in antiferromagnets. This discrepancy is believed to be due to an inadequate decoupling of the four-spin correlation function via the random phase approximation used in the theory.

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