

High-temperature critical susceptibility of gadolinium

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Low-field ac critical magnetic susceptibility measurements on a variety of toroidal samples of polycrystalline gadolinium are reported. The use of transient enhancement and temperature modulation of the ac susceptibility in bulk studies of critical magnetic behavior is discussed. Consistent values of $\gamma = 1.24 \pm 0.02$ and $\chi_0 = 0.0105 \pm 0.0005$ (relative susceptibility) are obtained for samples of varying purities and using different analytical techniques. The observation of a well-defined temperature range of ~ 7 K for critical behavior in ~ 99 -wt. % pure samples and in low (< 130 A m⁻¹) magnetic fields is consistent with recent theoretical studies of the effects upon critical magnetic susceptibility of a distribution in ordering temperature and demagnetizing fields.

I. INTRODUCTION

In the experimental determination of the high-temperature critical exponent γ of ferromagnetic materials, fits of the temperature dependence of the critical susceptibility χ are usually made to the equation of state

$$\chi = \chi_0 \epsilon^{-\gamma}, \tag{1}$$

where $\epsilon = (T - T_c)/T_c$, and χ_0 is the critical susceptibility amplitude. A measure of the difficulties of these determinations is indicated by the spreads of published values of γ for the elemental ferromagnets Fe, Co, Ni, and Gd (Table I). Important aspects of such determinations include whether or not the applied magnetic field is low enough to avoid field-induced effects and over what temperature range the intrinsic critical behavior is observed.

TABLE I. Survey of the critical susceptibility exponent γ values for Fe, Co, Ni, and Gd.

Element	γ	T_c (K)	H	Reference
Fe	1.37 ± 0.04		≥ 2000 A m ⁻¹	Noakes and Arrott (1964) (Ref. 1)
Fe	1.33 ± 0.03	1042	high ^a	Develey (1965) (Ref. 2)
Fe	1.333 ± 0.015	1043	≥ 2000 A m ⁻¹	Noakes <i>et al.</i> (1966) (Ref. 3)
Fe	1.30 ± 0.06		~ 0	Collins <i>et al.</i> (1969) (Ref. 4)
Co	1.21 ± 0.04	1388 ± 2	~ 14.5 kA m ⁻¹	Colvin and Araj's (1964) (Ref. 5)
Co	1.25			Lange <i>et al.</i> (1969) (Ref. 6)
Co	1.23 ± 0.05		~ 0	Glinka and Minkiewicz (1974) (Ref. 7)
Ni	1.35 ± 0.02	627.2	high	Kouvel and Fisher (1964) (Ref. 8)
Ni	1.28 ± 0.03	626.9	high	Araj's (1965) (Ref. 9)
	1.30 ± 0.03	626.2	high	
Ni	1.32 ± 0.02	631	high	Develey (1965) (Ref. 2)
Ni	1.30 ± 0.05	627.2	high	Kouvel and Rodbell (1967) (Ref. 10)
Ni	1.31 ± 0.01	626.94 ± 0.01	high	Arrott and Noakes (1967) (Ref. 11)
Ni	1.34 ± 0.01	627.4	high	Kouvel and Comly (1968) (Ref. 12)
Ni	1.33 ± 0.01	$625.39 \pm .03$	≥ 700 A m ⁻¹	Araj's <i>et al.</i> (1970) (Ref. 13)
Ni	1.31 ± 0.01	632.1	≥ 1600 A m ⁻¹	Kachnowski <i>et al.</i> (1976) (Ref. 14)
Ni	1.0	644	~ 13 MA m ⁻¹	Hatta and Chikazumi (1976) (Ref. 15)
		628	~ 1.2 MA m ⁻¹	
Ni	1.58 ± 0.15		~ 0	Minkiewicz <i>et al.</i> (1969) (Ref. 16)
Ni	1.44 ± 0.1		~ 0	Stump (1967) (Ref. 17)
Ni	1.41 ± 0.02	632.28 ± 0.02	0.4 A m ⁻¹	Herzum <i>et al.</i> (1974) (Ref. 18)
	1.41 ± 0.04	629.584 ± 0.008		
Gd	4/3	292.5 ± 0.05	high	Graham (1965) (Ref. 19)
Gd	1.17 ± 0.01	292.51	high	Develey (1965) (Ref. 2)
	1.16 ± 0.02	293.0		
Gd	1.196 ± 0.003	293.3 ± 0.1	high	Deschizeaux and Develey (1971) (Ref. 20)
Gd	1.24 ± 0.03	291.1 ± 0.1	< 130 A m ⁻¹	Wantenaar <i>et al.</i> (1980) (Ref. 21)

^aHigh generally refers to measuring fields in the range 40–2000 kA m⁻¹.

The effects of sample impurities, internal demagnetizing fields, and a spread of domain nucleation temperatures must also be considered. In a previous paper²² we considered theoretically the effects of broadening and internal demagnetizing fields on the determination of values of γ . In this paper we report an experimental study of gadolinium with emphasis on the use of low magnetic fields and techniques which allow the temperature range over which critical behavior occurs to be determined.

Gadolinium was chosen because it has the practical advantage of a room-temperature Curie point and because previous determinations of γ for gadolinium have mainly been restricted to high-magnetic-field (greater than 40 kA m⁻¹) measurements.^{2,19,20} Also, as the magnetocrystalline anisotropy persists above the ordering temperature,^{23,24} the critical behavior may differ from that expected of an isotropic Heisenberg system. Difficulties in producing high-purity rare-earth metals would suggest that gadolinium is not an ideal choice for critical-exponent studies because of the sample dependence of the critical temperature and the effects of rounding upon the susceptibility. One purpose of this study was to test whether critical fluctuations could be observed using bulk techniques on relatively impure samples.

II. EXPERIMENTAL

A. Samples

Two toroids of approximate dimensions 21.2 mm i.d. and 23.9 mm o.d. and a mass of approximately 3 g, were machined from nominally 99.9 wt. % pure ingots from Lunex Corporation (Gd1) and Rare-Earth Products (Gd2). Samples were examined in the as-machined (unannealed) state and then wrapped in gadolinium foil and annealed (Gd1A, Gd2A) in a low-pressure helium atmosphere at 850°C for 24 h and allowed to cool over 12 h. A larger sample (Gd2LA) of the Gd 2 supply (27.1 mm i.d. 33.2 mm o.d.) was annealed at 1000°C in a purified stream of argon and, in comparison with Gd1A and Gd2A, showed a marked reduction in the broadening of its magnetic transition. The resistivity ratios, $\rho(300 \text{ K})/\rho(4.2 \text{ K})$, of the Gd1 and Gd2 samples were approximately 34 and 13, respectively.

B. ac magnetic measurements

The toroidal samples formed the cores of transformers with primary and secondary coils wound in a single uniform layer giving a sample filling factor of unity. The complex susceptibility $\chi = \chi' - j\chi''$ was determined using standard phase-sensitive techniques described previously.²⁵ The relative susceptibility χ/μ_0 , where μ_0 is the permeability of free space, was obtained by determining the contribution from free space to the signal. Three methods were used to determine the free-space contribution: (i) calculation from the coil configuration, (ii) direct measurement at temperatures well above T_c , and (iii) least-squares-fitting during data analysis.

In the temperature-modulation studies²¹ the sample temperature was modulated at $f_m \leq 1$ Hz with amplitude $\Delta T \leq 10$ mK. This thermal wave was produced via a

heating coil wound uniformly and noninductively between the turns of the sample induction coils, and the resulting small-amplitude modulation in the voltage from the secondary was detected synchronously. This modulation signal S is an analog of the temperature derivative of susceptibility; full experimental and theoretical details have been published elsewhere.²⁵

In transient enhancement experiments,²⁶ the effect of an additional time-varying bias field upon the initial ac susceptibility is monitored. This technique permits the sensitive detection of small concentrations of domain walls²⁶ and is particularly useful in studies of the high-temperature critical exponent γ as it indicates the temperature range over which domain nucleation occurs above the average ordering temperature \bar{T}_c .

III. EXPERIMENTAL RESULTS

The behavior typical of the initial ac magnetic susceptibility for gadolinium about its ordering temperature can be seen in Fig. 3 of Ref. 26. Domain nucleation and broadening of the ferromagnetic transition can be examined in detail by plotting the difference between the enhanced χ''_{en} and unenhanced χ'' loss components versus temperature. The quantity $\chi''_{en} - \chi''$ defines the onset and region of thermal domain nucleation and gives an indication of the distribution and mean value of the Curie temperature. In the case of Gd2A, domain nucleation commences at about 293 K and the value $\bar{T}_c \sim 291.5$ K is determined as the temperature at which 50% of domain nucleation has occurred with the halfwidth at half-maximum (HWHM) of the distribution of Curie temperatures being ~ 0.5 K. Determination of the temperature of onset of domain nucleation is important in the analysis of critical susceptibility as it sets the lower-temperature limit for reliable data analysis; for the Gd2A sample this limit corresponds to $\epsilon_{min} \approx 5 \times 10^{-3}$. Similar studies of the other samples indicated a variation in the values of \bar{T}_c of about 1 K, with reductions in the broadening for the annealed samples at \bar{T}_c of a few tenths of a degree.

IV. KOUVEL-FISHER ANALYSIS

A. Analysis of susceptibility data

With Kouvel-Fisher analysis⁸ the experimental susceptibility data are tested against the critical equation of state (1) using the equation

$$T^* \equiv -\chi \left(\frac{d\chi}{dT} \right)^{-1} = \frac{T - T_c}{\gamma}, \quad (2)$$

by plotting T^* against T . This eliminates χ_0 from the analysis and, by examining the linearity of T^* vs T , an indication of the critical temperature region may be obtained. The inverse slope and the extrapolated intercept with the abscissa lead to values for γ and \bar{T}_c , respectively. A preliminary account of this type of data analysis has been published previously²¹; in the present paper further analyses with an emphasis on delineating the regions of critical behavior are given.

The consistency of γ and \bar{T}_c obtained from the previous

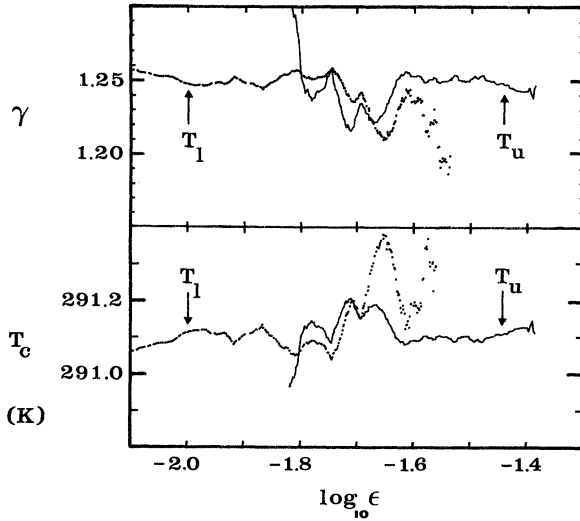


FIG. 1. Values of γ and \bar{T}_c obtained by least-squares-fitting the Kouvel-Fisher plot of the χ data of Ref. 21 over varying temperature ranges. Solid line shows the behavior of γ and \bar{T}_c as the fitted ranges are reduced towards T_l ; dotted line shows the behavior of γ and \bar{T}_c as the fitted ranges are reduced towards T_u .

paper²¹ was tested by linear least-squares-fitting the Kouvel-Fisher plots over restricted temperature ranges. This test of the deduced values of γ and \bar{T}_c on the temperature range of fitting was carried out by fixing one temperature extremity while progressively discarding data from the opposite end. These upper and lower limits, T_u and T_l , respectively, were fixed within temperature regions where deduced values of γ and \bar{T}_c did not depend on the actual choice of T_l or T_u (Fig. 1). Figure 1 shows the results of this fitting; essentially invariant behavior is observed for both γ and \bar{T}_c although large deviations occur when the temperature range of fitting becomes too narrow. Each γ or \bar{T}_c value is plotted at the ϵ value corresponding to the last point of the varied end (this also allows the range to be identified). From Fig. 1 we conclude that for the Gd2LA sample, $\gamma = 1.25 \pm 0.02$ and $\bar{T}_c = 291.1 \pm 0.1$ K.

A more detailed point-by-point study of the range over which the critical equation holds, was made for each sample by taking the midrange values of γ and \bar{T}_c obtained as above and then determining a value of χ_0 from every data point (χ_i, ϵ_i), using

$$\chi_{0i} = \chi_i \epsilon_i^\gamma \quad (\gamma, \bar{T}_c \text{ constant}). \quad (3)$$

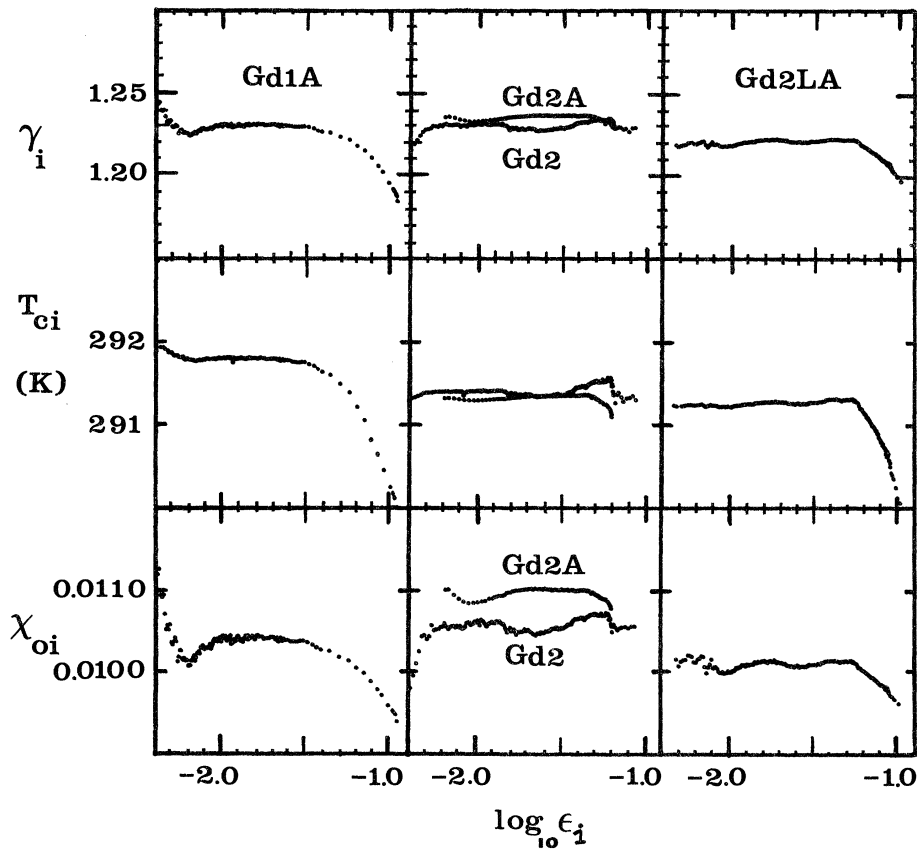


FIG. 2. Point-by-point calculations of γ_i , T_{ci} , and χ_{0i} from midrange values of γ and \bar{T}_c obtained from plots of the type illustrated in Fig. 1. Results are from low-field (up to approximately 15 A m^{-1}) experiments on the Gd1A, Gd2, Gd2A, and Gd2LA samples.

The plot of χ_{0i} against $\log_{10}\epsilon_i$ (Fig. 2) indicates an essentially temperature-independent critical range and leads to a midrange value of χ_0 . Similarly, deduced values of

$$\gamma_i = (\log_{10}\chi_0 - \log_{10}\chi_i) / \log_{10}\epsilon_i \quad (\chi_0, \bar{T}_c \text{ constant}), \quad (4)$$

and

$$\bar{T}_{ci} = T_i / [1 + (\chi_0/\chi_i)^{1/\gamma}] \quad (\chi_0, \gamma \text{ constant}), \quad (5)$$

for each point may be plotted against $\log_{10}\epsilon_i$. This technique, of determining two parameters from all the data, enables the consistency of values of the third parameter to be examined for every data point. Results for all samples studied (Fig. 2) show that for each sample there exists a well-defined temperature range of approximately 7 K over which the deduced parameters are temperature independent. Figure 2 also shows that, whereas there are distinct differences in the mean Curie temperatures of samples of differing purity, they have similar γ values. From Fig. 2 we conclude that $\gamma = 1.23 \pm 0.02$ and $\chi_0 = 0.0105 \pm 0.005$ (relative susceptibility). The depressed Curie temperatures of the Gd2 samples were attributed to trace quantities of yttrium observed by x-ray fluorescence.

B. Analysis of temperature-modulation data

The Kouvel-Fisher technique for analyzing χ data is also applicable to the temperature-modulation signal S using

$$T^{**} \equiv -S \left[\frac{dS}{dT} \right]^{-1} = \frac{T - T_c}{\gamma + 1}, \quad (6)$$

and a plot of T^{**} vs T is shown in Fig. 1 of Ref. 21. Kouvel-Fisher plots of the S data were also examined in detail using the same least-squares-fit analysis as carried out on the χ data (Fig. 1), and the results of this analysis

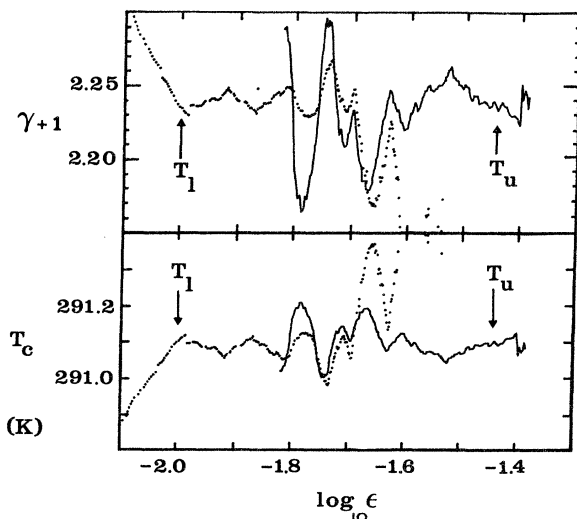


FIG. 3. Values of $\gamma + 1$ and \bar{T}_c obtained by least-squares-fitting the Kouvel-Fisher plot of S data (Ref. 21) over varying temperature ranges.

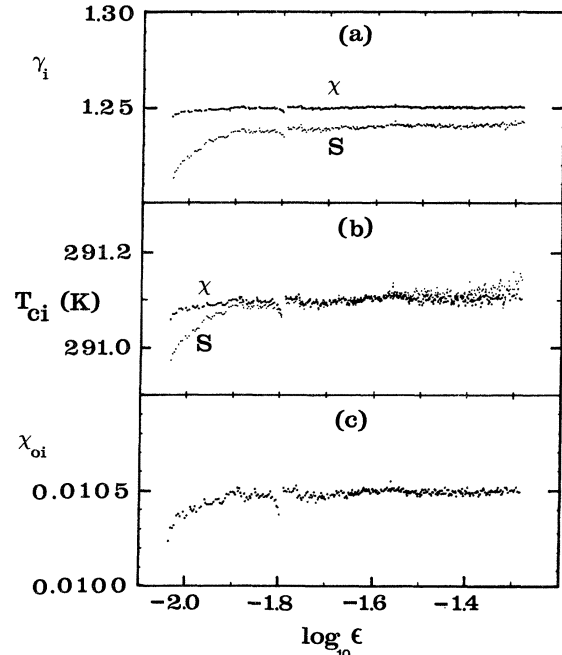


FIG. 4. Deduced values of (a) γ_i , (b) T_{ci} , and (c) χ_{0i} for the χ and S data of Ref. 21. Values were calculated by the point-by-point method described in the text.

on the S data of Ref. 21 are shown in Fig. 3. As expected, the values of \bar{T}_c and γ deduced from analysis of χ and S data are in agreement. Although the S data, which have been differentiated numerically, are noisier than those of the χ data, the approach based on Eq. (6) has the important advantage that it does not involve the free-space signal Q_0 (analysis of the χ data showed that a 1% error in Q_0 produced a 2% error in the deduced value of γ).

In a similar manner to that for the χ data, the S data were examined in detail with point-by-point calculations of $\gamma + 1$ and \bar{T}_c by holding two parameters constant at the midband values. The resulting plots for Gd2LA, together with corresponding result for simultaneous susceptibility measurements, are shown in Fig. 4. As expected, this analysis leads to the same value of \bar{T}_c as determined from the χ data.

V. DISCUSSION

A. Value of γ

Detailed analyses of susceptibility data (Figs. 1 and 2) based on the Kouvel-Fisher method⁸ yield the critical parameters $\gamma = 1.25 \pm 0.02$ and $\chi_0 = 0.015 \pm 0.0005$ (relative susceptibility) for the largest toroidal sample studied (Gd2LA). Similar analyses of the S data (Figs. 3 and 4) result in the critical exponent value $\gamma + 1 = 2.24 \pm 0.02$ with analyses of both χ and S data yielding the same value, $\bar{T}_c = 291.1 \pm 0.1$ K, for the mean ordering temperature of this sample. Kouvel-Fisher analysis of the χ data for the other samples gives values in the range $\gamma = 1.23 \pm 0.02$. The spread in the γ values obtained on analysis of χ data on the various samples, and the spread

of $\Delta\gamma = \pm 0.02$ in values obtained by various techniques on Gd₂LA are considered to be due to the errors ($\sim 1\%$) in the values of the free-space signal used to calculate the relative susceptibilities. These errors are not present in analysis of the S data and, in attaching more significance to these results, we conclude that $\gamma = 1.24 \pm 0.02$ and $\chi = 0.0105 \pm 0.0005$.

This value of γ is similar to that obtained for the case of the infinite-spin Heisenberg model subject to even the slightest amount of prolate anisotropy^{27,28} ($\gamma = 1.23$ corresponding to Ising-type behavior). It is possible that gadolinium, with its highly localized and large spin ($\frac{7}{2}$), and persistence of magnetocrystalline anisotropy above T_c ,²³ can be described by this model, although it is surprising that such anisotropic behavior is observed at the large experimental values of $\epsilon \sim 10^{-2}$. Rather, on the basis of calculations of the critical behavior of anisotropic²⁹⁻³¹ or dipolar³²⁻³⁴ magnetic systems, it is expected that isotropic behavior would be observed for $\epsilon \geq 10^{-3}$ with crossover effects taking place below that reduced temperature value. The value of $\gamma \sim 1.37$ expected for the isotropic Heisenberg model with or without dipolar effects (for example, Ref. 32) is significantly different from the present value of $\gamma = 1.24$, suggesting that gadolinium cannot be described as an isotropic magnetic system.

B. Effects of high applied fields

The majority of data summarized in Table I stems from magnetization measurements made in high applied magnetic fields. In such experiments there is the likelihood of field-induced effects disturbing the critical fluctuations. Herzum *et al.*¹⁸ have noted a systematic trend in the critical exponent values for nickel with lower values of γ being obtained for large applied fields. In the limiting case for sufficiently high fields the value $\gamma = 1$ is expected, corresponding to an infinite-range interaction. This has already been demonstrated by Hatta and Chikazumi¹⁵ who obtained $\gamma = 1$ for nickel in very high magnetic fields (up to approximately 13 MA m^{-1}).

The criterion for nonperturbative low-field measurements will vary from material to material and will be most stringent in high-purity samples, where there is little broadening and the magnetic susceptibility increases rapidly as the ordering temperature is approached. With the high sensitivity of ac techniques the field dependence of susceptibility is readily tested at all temperatures and very low measuring fields can be used. In the present studies of $\sim 99 \text{ wt. } \%$ ($\sim 96 \text{ at. } \%$) pure gadolinium, the susceptibility was found to be field independent for $10^{-2} \leq H \leq 10^3 \text{ A m}^{-1}$ at all temperatures ($\epsilon \geq 10^{-2}$) used to determine γ .

C. Demagnetizing fields

The bulk demagnetizing factors of single or polycrystalline samples may be fixed and the effects of the bulk

demagnetizing fields accommodated by grinding them into regular shapes, ellipsoids, or spheres, which have a unique demagnetizing factor. In ac studies such as reported here, the use of toroidal samples removes such bulk demagnetizing fields.

In a recent theoretical paper²² we reported the effects of internal demagnetizing fields arising from the same sample inhomogeneities which lead to broadening associated with the spread of T_c values. These internal fields influence the broadening so as to greatly reduce its effect upon the temperature dependence of χ in the critical region. In the present study of samples of fractional width in the distribution of ordering temperatures of $\sigma/\bar{T}_c \sim 1.7 \times 10^{-3}$, in the absence of demagnetizing fields, there would be a deviation of slightly greater than 1% expected over the experimental range $10^{-2} \leq \epsilon \leq 3.2 \times 10^{-2}$ ($294 \lesssim T \lesssim 300 \text{ K}$), whereas with an effective average value of the demagnetizing factor $D = 0.1$, the deviation from Eq. (1) would be reduced to slightly less than 0.4% over the same temperature range. This favorable circumstance acts to extend the temperature region over which critical behavior can be monitored.

VI. CONCLUSIONS

Kouvel-Fisher analysis of both susceptibility and temperature-modulation data for a variety of samples of polycrystalline gadolinium with differing purities and mean Curie temperatures leads to a consistent value $\gamma = 1.24 \pm 0.02$; these two techniques are independent with one involving χ alone and the other involving the response of χ to thermal oscillations. The analysis of the temperature-modulation data has the important advantage of not requiring the free-space susceptibility signal. The consistency between the results of susceptibility and temperature-modulation experiments indicates that the critical fluctuations remain in thermal equilibrium throughout the oscillations of the thermal wave. These techniques permit the accurate measurement of the low-field initial susceptibility. The results show that simple critical behavior holds over a well-defined range of temperatures for these relatively impure samples. This is consistent with theoretical studies which include the effects of distributions of ordering temperatures and internal demagnetizing fields. From the experimental value of $\gamma = 1.24$ we conclude that the critical susceptibility of gadolinium around $\epsilon \sim 10^{-2}$ cannot be described in terms of an isotropic Heisenberg system.

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