Critical exponents of amorphous $Gd_{0.70}Pd_{0.30}$

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The magnetization of liquid-quenched amorphous $Gd_{0.70}Pd_{0.30}$ was measured between temperatures of 77 and 300 K in fields up to 6 kOe. The ferromagnetic Curie temperature T_C and the critical exponents β, γ, and δ were found to be 131.77±0.20 K, 0.34±0.02, 1.010±0.003, and 3.95±0.10, respectively. These experimentally determined values of the critical exponents very clearly satisfy the scaling law $\gamma=\beta(\delta-1)$. Of special interest is the value of unity for γ in the neighborhood of the ferromagnetic Curie temperature, the usual Curie-Weiss plot of inverse susceptibility yielding a paramagnetic Curie temperature of 155.20±0.20 K. EPR measurements made over the temperature range of 135 to 100 K reveal an almost-free Gd^{3+}-ion response broadened and made asymmetric by conduction-electron effects, while at the same time exhibiting no significant directional dependence on the applied field. This result and the lack of hysteresis at 77 K support the idea that liquid-quenched $Gd_{0.70}Pd_{0.30}$ becomes an isotropic amorphous ferromagnet at T_C. Finally, examination of sections of a given sample (produced via the hammer-and-anvil technique) reveal that, even at the highest cooling rates ($\sim 10^7$ K s^{-1}), entirely amorphous material may be produced only toward the sample edges, in agreement with current models of amorphous cooling in liquid-quenched samples.

I. INTRODUCTION

Among the alloy systems that easily form metallic glasses are (1) a group of metal-metalloid alloys, and (2) some metal-metal alloys where one of the components is an early transition metal. One of the characteristics associated with the glass-forming tendency (GFT) is a low liquidus temperature T_m. Polk and Giessen[1] suggest that the GFT increases with the reduced melting-point depression $\Delta T_{mr}=(\bar{T}_m-T_m)/\bar{T}_m$, where $\bar{T}_m$ is the weighted average of the melting points of the components and T_m is the low liquidus temperature.

One of the earlier noted, easy glass-forming systems is Pd-Si, where T_m occurs as a deep eutectic in a narrow composition range.[2,3] The rare-earth—Pd phase diagrams are similar to the Pd-Si system,[4,5] with the exception that the lowest-temperature eutectics occur in the rare-earth-rich region, hence making the study of these systems inviting for their GFT as well as for their magnetic properties.

The value of ΔT_{mr} for Gd-Pd equal to 0.43 is 5% greater than the values associated with Sm-Pd and Dy-Pd and 10% greater than those of Ho-Pd and Er-Pd. It thus comes closest to the ΔT_{mr} of Pd-Si, which is 0.48. It was further felt that crystallite formation would be hindered by selecting a Gd concentration near the deep eutectic in the Gd-Pd phase diagram at which the alloy would have to traverse a number of equilibrium phases if cooled slowly from the melt temperature to room temperature. For this reason, a concentration of 70 at. % Gd was chosen.

The choice of Pd allows the investigation of a rare-earth alloy incorporating an element intermediate between an early member of the transition group with a local moment and a noble metal, both cases having been reported on by other authors.[6–8] At the concentrations used in this work, the *d*-band states of the Pd will lie well below the Fermi level of the alloy, and no complications are to be expected of a paramagnon nature. In addition, Gd being an *S*-state ion, the alloy chosen is free of any local-anisotropy-field interactions. The magnetization measurements bear this out, as do the EPR spectra, the latter being compatible with local Gd moments in the presence of conduction-electron effects.

A number of $Gd_{0.70}Pd_{0.30}$ alloys were prepared at various quench rates, as indicated by hammer speed, via the conventional hammer-and-anvil technique.[9,10] Samples, as-prepared, were analyzed by x-ray diffraction, and three specimens representative of distinctly different degrees of long-range order were chosen for magnetic study. These alloys were produced at hammer speeds of 332, 437, and 793 cm s^{-1}. The magnetization, as a function of field and temperature, of two sections of the sample prepared at the highest hammer speed (hereafter referred to as the "fast sample"), was examined in detail, with special emphasis being given to the portion taken from near the sample edge. The magnetic response of a section taken from near the center of the "fast sample" indicated the presence of gadolinium precipitates, in keeping with similar results reported earlier by Poon and Durand on Gd-Au.[7] For this reason, determinations of spontaneous magnetization and initial susceptibility were confined to the near-edge portion of the "fast sample."

The isothermal magnetization versus field was measured at steps of approximately 1 K both above and below the transition temperature T_C within the range $|T-T_C|\leq 8$ K. Appropriate Arrott-Kouvel plots[11]

were made of the data to determine the initial susceptibility at seven temperatures above T_C, ranging from 132.93 to 139.69 K, and the spontaneous magnetization at seven temperatures below T_C, ranging from 123.48 to 130.16 K.

II. SAMPLE PREPARATION AND EXPERIMENTAL PROCEDURES

Samples in the form of circular foils having a thickness of approximately 40 μm and an effective diameter of 1 cm were produced by quenching from the liquid state using the piston-and-anvil technique.[9,10] The composition chosen (70 at. % Gd, 30 at. % Pd) is close to a deep eutectic in the phase diagram of Gd-Pd, and cooling rates of up to 10^7 K s^{-1} were achieved. The structure of the samples was checked by x-ray scanning with Cu $K\alpha$ radiation using a standard Norelco diffractometer; traces for three samples prepared for this work appear in Fig. 1. The apparent progression to truly amorphous material with increasing hammer speed can be seen. In the case of the "fast sample," using the broad maximum centered at 32.0° having a full width at half maximum (FWHM) of 6°, application of the Scherrer formula[12] yields a microcrystal size of 13 Å, characteristic of glassy metals. By contrast, similar analysis of the sample prepared at a hammer speed of 332 cm s^{-1} indicates microcrystalline order extending over some 200 Å. The samples appeared stable and exhibited no observable annealing effects at room temperature over the experimental period of a few months.

All magnetization measurements were made by means of a Faraday balance utilizing a set of Lewis coils[13] which allowed rapid reversal of the field gradient (complete reversal being achieved for a given field value in a matter of seconds) and thereby eliminated any spurious dc bias in the signal by averaging the magnitude of the magnetic force at a given field and temperature over direct and reversed senses.

The sensitivity of this Faraday balance was determined to be $m\chi_m \geq 5.8\times10^{-9}$ cm^3, where m is the sample mass in grams and χ_m is the mass-specific susceptibility in emu. Single samples were suspended on a quartz stirrup (whose known diamagnetism was accounted for) and oriented such that their demagnetization factors were essentially zero. In addition, independent measurements on Nb—45 at. % Ir indicated that the magnetic force experienced by the balance had a *total* variation of no more than 1.5% over a 5 mm spread of the sample's vertical position within the central region of the dc magnetic field and gradient.

It is obviously important to guarantee that the previous magnetic history of the specimen will not unduly influence results, and thus, when conducting magnetization measurements in the vicinity of the transition temperature (both above and below T_C), care was always taken to warm the samples well into the paramagnetic regime ($T \geq 230$ K) before returning, at $H=0$, to a new temperature near T_C.

EPR spectra of the edge portion of the "fast sample" were examined both above and below T_C by standard means at the X band using a microwave frequency of 9.265 GHz.

III. RESULTS AND DISCUSSION

At all temperatures measured, the central portion of the "fast sample" gave a nonlinear magnetization response at the smallest measured values of applied field. This was in marked contrast to the edge portion, which exhibited linear behavior at all fields examined ($H \leq 5$ kOe) and for all temperatures sufficiently above T_C. By comparing these results with those of the edge portion of the *same* "fast sample" taken at identical temperatures (see example shown as Fig. 2), it was seen that the limiting slope of the magnetization at high fields (~ 5 kOe) of the central portion was the same as the constant slope measured at all fields for $M(H)$ of the edge portion.

The results of Fig. 2 are indicative of a saturated component contributing to the total magnetization of the central portion of the sample at high fields in addition to the

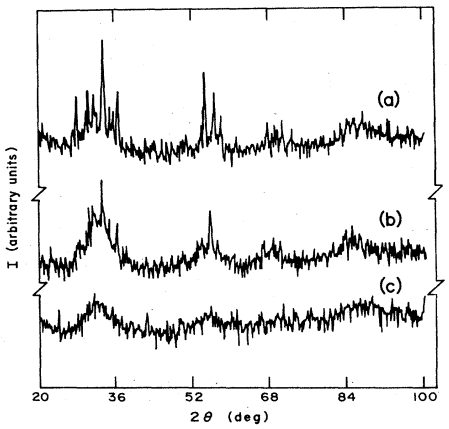

FIG. 1. X-ray intensity $I(\theta)$ vs scattering angle 2θ for $Gd_{0.70}Pd_{0.30}$ samples prepared at hammer speeds of (a) 3.32, (b) 4.37, and (c) 7.93 m s^{-1}.

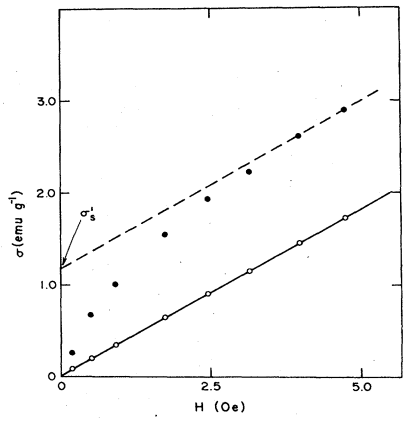

FIG. 2. Specific magnetization σ of the 7.93 m s^{-1} $Gd_{0.70}Pd_{0.30}$ sample vs applied magnetic field H at $T=272.2$ K. Solid circles represent data for "central" portion of the sample and open circles represent data for the "edge" portion. Intercept of dashed line is σ_s', the saturation magnetization associated with gadolinium precipitates in "central" portion.

Gd-Pd. Under this assumption (i.e., saturation of this component at about 5 kOe), an extrapolation of the limiting slope to $H=0$ yields σ_s', the saturation magnetization of the "impurity." Figure 3 shows a Debye-Weiss curve for $S=\frac{7}{2}$, $T_C=293$ K, and $H_0=5$ kOe in which the normalized saturation magnetization values of the central portion, determined as indicated for $T_C<T<293$ K, are shown as data. The agreement confirms that the "impurity" in the central portion of the sample appears to be microcrystalline inclusions of pure gadolinium. This effect has been noted by Poon and Durand[7] in their extensive work on Gd-Au, but apparently overlooked in previous studies by others. Our situation differs from theirs, however, in that while they had to work on a sample for which they took account of known Gd crystalline inclusions, the edge portion of our "fast sample" appears to be completely free of these complicating impurities.

An estimate of the concentration of Gd in the central portion of the "fast sample" was made under the assumption that the edge portion is free of all but the amorphous Gd-Pd contribution to the magnetization per unit mass, i.e., $\sigma_{\text{edge}}=\sigma_{\text{am}}$, and that the mass-specific magnetization of the gadolinium precipitates is about the same as that of as-cast Gd-Pd material at room temperature, i.e., $\sigma_{\text{Gd*}}\approx\sigma_{\text{as-cast}}$. Then, $\sigma_{\text{center}}\simeq f\sigma_{\text{Gd*}}+(1-f)\sigma_{\text{am}}$, and thus the fraction of Gd precipitates is given by

$$f=\frac{\sigma_{\text{center}}-\sigma_{\text{am}}}{\sigma_{\text{Gd*}}-\sigma_{\text{am}}},$$

which, upon utilizing data from room temperature, yields $f=0.016$, or about 2%. In contrast to most alloy systems, our $Gd_{0.70}Pd_{0.30}$ as-cast polycrystalline specimen possessed a specific magnetization (emu/g) at room temperature that was about 60 times greater than that of the liquid-quenched samples, thus providing a further indication of the ubiquity of extensive Gd precipitates in all but some portions of the most rapidly quenched samples.

One assumes that at the Curie temperature a second-order phase transition occurs which can be characterized by a set of three critical exponents. A result of the static scaling hypothesis is that these exponents, rather than satisfying the less restrictive inequality relationship imposed by thermodynamic considerations alone, must in fact satisfy the following equality, $\gamma=\beta(\delta-1)$.[14–17] As a result, it is necessary to have only two such parameters to adequately describe the system. In this paper, β, γ, and δ will have their usual meanings. The temperature dependence of the spontaneous magnetization just below T_C is given by $\sigma_0\sim|T-T_C|^{\beta}$, and that of the initial inverse susceptibility just above T_C by $\chi_0^{-1}\sim|T-T_C|^{\gamma}$, while the magnetization versus field at the critical isotherm ($T=T_C$) satisfies $\sigma\sim H^{1/\delta}$.

It was found here, as in previous work by others, that it is generally easier to determine the initial inverse susceptibility $\chi_0^{-1}\equiv(H/\sigma)_{M=0}$ than the spontaneous magnetization σ_0 because of large gradients in $\sigma^2(H/\sigma)$ at small values of the argument for $T\lesssim T_C$.[18] In this regard, it is reassuring to note that a least-squares fit to the data of $\chi_0^{-1}/(d\chi_0^{-1}/dT)$ versus T as shown in Fig. 4, while surprisingly yielding a value of $\gamma=1.01$, at the same time indicates a ferromagnetic Curie temperature of $T_C=131.77$ K since this latter value is well confirmed by the single $\sigma^2(H/\sigma)$ curve for $T=131.73$ K passing directly through the origin. The inset of Fig. 4 reveals the linearity of $\chi_0^{-1}(T)$ in the neighborhood of T_C, indicating that γ must be unity, and confirming that this value does not result from approximating the derivative in $\chi_0^{-1}/(d\chi_0^{-1}/dT)$ by a ratio of finite differences.

The critical exponent β was determined by examining plots of σ^4 as a function of H/σ for $123.48\leq T\leq 130.16$ K. Plotting the fourth power of the magnetization alleviated the problem of the exceedingly steep initial slopes associated with σ^2 versus H/σ, which makes accurate extrapolation to $H/\sigma=0$ extremely difficult. Figure 5

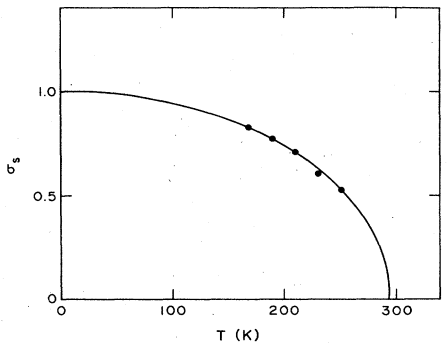

FIG. 3. Debye-Weiss curve for $S=\frac{7}{2}$ and $T_C=293$ K. ●, normalized saturation magnetization $\sigma_s(T)$ of the "central" portion of the 7.93-m s^{-1} $Gd_{0.70}Pd_{0.30}$ sample.

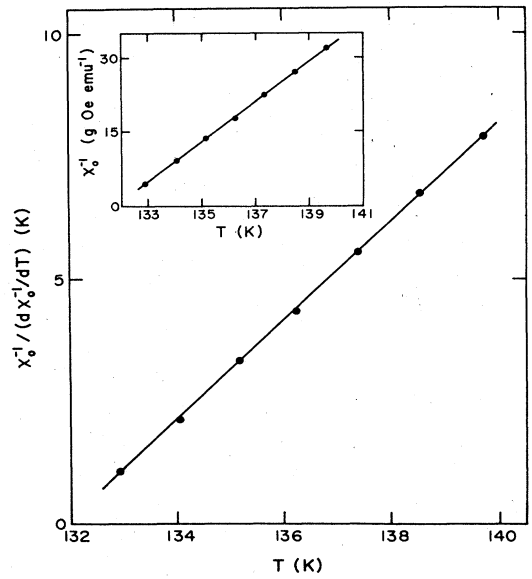

FIG. 4. $\chi_0^{-1}/(d\chi_0^{-1}/dT)$ vs T for the "edge" portion of the 7.93-m s^{-1} $Gd_{0.70}Pd_{0.30}$ sample in the vicinity of T_C, χ_0^{-1} being the initial inverse susceptibility. Least-mean-squares fit to data is $\chi_0^{-1}/(d\chi_0^{-1}/dT)=1.001T-131.90$, with $r^2=0.999\,79$, yielding $\gamma=1.01$ and $T_C=131.77$ K. Inset shows direct linear relation between χ_0^{-1} and T near T_C.

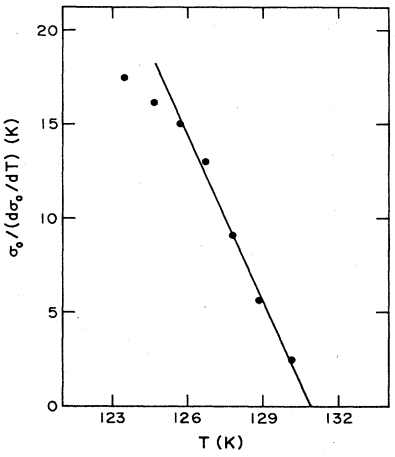

FIG. 5. $\sigma_0/(d\sigma_0/dT)$ vs T for the "edge" portion of the 7.93-m s^{-1} $Gd_{0.70}Pd_{0.30}$ sample in the vicinity of T_C. Least-mean-squares fit to the data of the five uppermost temperatures is $\sigma_0/(d\sigma_0/dT) = -2.930T + 383.77$, with $r^2 = 0.9934$, yielding $\beta = 0.341$ and $T_C = 130.98$ K.

shows $\sigma_0/(d\sigma_0/dT)$ as a function of T near T_C, from which a value of $\beta = 0.341$ is determined. These data are not as good as those from which γ is determined, and, in fact, it can be seen that extrapolation yields a T_C value of about 131.0 K. Considering the degree of difficulty associated with the extrapolation of $\sigma^4(H/\sigma)$ and the possibility for further inaccuracy when determining not only σ_0 but also $d\sigma_0/dT$ directly from the data, this difference in value of T_C does not seem unacceptably large (i.e., ~ 0.75 K). Because of these reasons, however, we have identified the ferromagnetic Curie temperature with the higher value determined from the γ data.

$\sigma(H)$ was determined at $T = 131.73$ K (essentially the critical isotherm), and Fig. 6 gives the data in double-logarithmic form, which yields a value for δ of 3.95. In the present case it is to be noted that the range of variation of σ is only about a factor of 2. For this reason, some further evidence was sought, confirming this value of δ. Near T_C the equation of state provides that $d\sigma^2/d(H/\sigma) \sim |T - T_C|^{\beta(3-\delta)}$, and Fig. 7 is a plot of $\Delta\sigma^2/\Delta(H/\sigma)$ versus $|T - T_C|^{\beta(3-\delta)}$, with $\beta = 0.34$ and $\delta = 3.95$. While, again, the range of ordinate is not great,

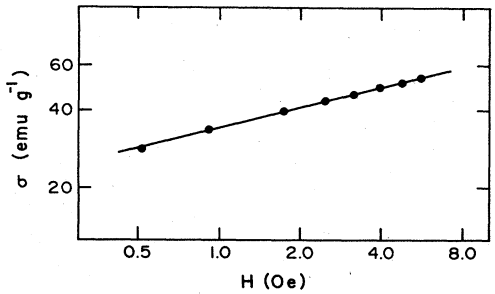

FIG. 6. Specific magnetization σ of the "edge" portion of the 7.93-m s^{-1} $Gd_{0.70}Pd_{0.30}$ sample vs applied magnetic field H at critical isotherm. $\sigma \sim H^{1/\delta}$ yields $\delta = 3.95$.

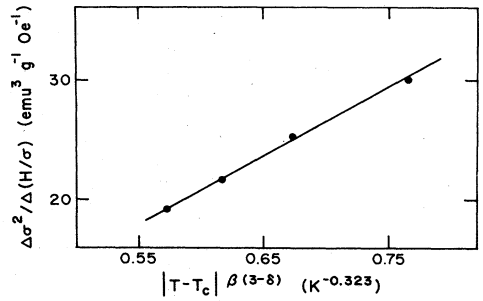

FIG. 7. Plot of $\Delta\sigma^2/\Delta(H/\sigma)$ as measured for the "edge" portion of the 7.93-m s^{-1} $Gd_{0.70}Pd_{0.30}$ sample vs $|T - T_C|^{\beta(3-\delta)}$ for values of $T_C = 131.77$ K, $\beta = 0.34$, and $\delta = 3.95$.

it should be noted that this is a set of data, separate from the critical isothermal magnetization, which is compatible with this value of δ.

Finally, the errors associated with fitting the β and δ data by straight lines give rise to the assignment of the following values: $\beta = 0.341 \pm 0.020$ and $\delta = 3.95 \pm 0.10$, with the experimentally measured value of $\gamma = 1.010 \pm 0.003$ lying well within the error range determined via $\gamma = \beta(\delta - 1)$ of 1.01 ± 0.09.

Figure 8 shows the inverse susceptibility as a function of temperature and reveals that the edge portion of the fast amorphous $Gd_{0.70}Pd_{0.30}$ sample satisfies the Curie-Weiss law with a paramagnetic Curie temperature $\Theta_p = 156.0$ K and a slope of 23.89 g cm^{-3} K^{-1}. Previous work on La-Pd compounds indicates that they are diamagnetic[19] and that the Pd does not, therefore, contribute a local moment. Thus, under the assumption that only the gadolinium ions contribute significantly to the overall magnetic moment, one can show that

$$g^2 = \frac{3k_B}{N_A \mu_B^2 J(J+1)(d\chi_m^{-1}/dT)} [M_{Gd} + (1/x - 1)M_{Pd}] ,$$

where the M_i are the respective gram-atomic weights and x is the atomic fraction of gadolinium. Inserting

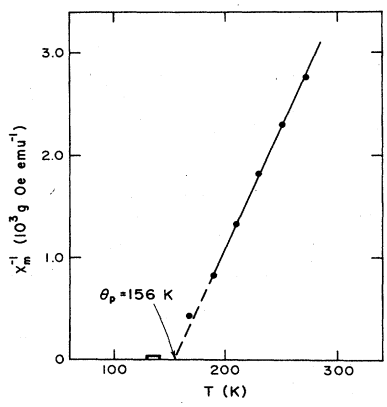

FIG. 8. Curie-Weiss plot of χ_m^{-1} vs T for "edge" portion of the 7.93-m s^{-1} $Gd_{0.70}Pd_{0.30}$ sample, yielding $\Theta_p = 156.0$ K. The rectangle indicates region examined in inset of Fig. 4.

$d\chi_m^{-1}/dT$ for our case yields $g=2.069$, corresponding to $\mu_{\text{eff}}=8.21\mu_B$. While it might at first seem likely that this enhancement over the free-Gd-ion value of $7.94\mu_B$ arises from the palladium, the even larger, known enhancement in $Gd_{0.80}Au_{0.20}$ ($\Delta\mu\sim 1\mu_B$) precludes any possibility of deducing a value for μ_{Pd} in our amorphous $Gd_{0.70}Pd_{0.30}$. The inset of Fig. 4 expands the scale of the region in the rectangular box near T_C and reveals initial susceptibilities about 2 orders of magnitude greater than the *paramagnetic* susceptibility at $T\gg\Theta_p$, most likely indicative of the onset of large local ordering fields.

Long-range effects might be expected to influence the critical fluctuations of the magnetization over the temperature range given by Kadanoff *et al.*,[20]

$$t_c\equiv 1-T/T_C=\left[\frac{g\mu_B S M_\infty}{k_B T_C}\right]^{1/\beta(\delta-1)},$$

which, for our Gd-Pd with $S=\frac{7}{2}$, $g\simeq 2$, $M_\infty\simeq 2000$ Oe, $T_C\simeq 132$ K, $\beta=0.34$, and $\delta=3.95$, yields a value of $t_c\simeq 0.007$. However, just as Poon and Durand[7] found in Gd-Au, we also were able to determine the critical parameters of Gd-Pd by analyzing isothermal magnetization data over a range of temperatures centered on T_C corresponding to a t_c of 0.06 both above and below T_C. Thus in Gd-Pd, as in Gd-Au, long-range forces appear to be operational over a wider temperature interval than that expected on the basis of the Kadanoff criterion, again indicating that the short mean free path in amorphous materials does not seem to guarantee a drastic reduction in such interactions as compared to their crystalline counterparts.

Finally, the EPR spectra taken at temperatures both above and below T_C reveal a broad asymmetric line centered at a field of 0.327 T. The response is representative of a system having $g=2.010$ (close to the free-Gd-ion value) in the presence of conduction-electron effects.[21–23] The spectra exhibited no significant orientation dependence either above or below T_C, which is consistent with the idea that the liquid-quenched $Gd_{0.70}Pd_{0.30}$ forms an isotropic ferromagnetic upon cooling below T_C. It was also noted that, at liquid-nitrogen temperatures (77 K), the specimen revealed no measurable magnetic hysteresis upon cycling between fields of 0 and 5 kOe, and in this sense behaved similarly to the $Gd_{0.75}Au_{0.25}$ glass investigated by Sellmyer *et al.*[24]

IV. SUMMARY

$Gd_{0.70}Pd_{0.30}$, upon rapid quenching from the melt, has been shown to form a metallic glass at least near the edges of the resulting sample. Our results, in this regard, are consistent with predictions of the mathematical "splat cooling" model developed by Miyazawa and Szekely,[25] as well as with the results of Atzmon and Johnson.[26] Ordinary x-ray-diffraction spectra are able to determine the lack of long-range order, but it will require an analysis by small-angle x-ray scattering or transmission electron microscopy to make a structural identification of precipitates in the alloy, assuming that the electronic contrast is sufficiently great for the level of such impurity concentration. In the present work, detailed high-temperature magnetization measurements indicated the presence of gadolinium precipitates in the "central" portion of the "fast sample," but only because gadolinium has such a high ferromagnetic Curie temperature. For rare-earth alloys, in general, however, such precipitate formations could exist and yet remain undetected by a monitoring of the magnetic response.

Critical exponents measured for the truly amorphous sample are found to obey Widom's scaling law $\gamma=\beta/(\delta-1)$, although the values as a group are not characteristic of any particular model. While $\gamma=1$ could be associated with mean-field theory, the value of 0.34 measured for β cannot.

γ values for Gd systems are among the smallest observed (i.e., closest to unity). In addition, many papers, including the present one, have noted the "extension" of the operative long-range interaction in these amorphous systems. Dunn and Poon[27,28] refer to a possible indirect d-f exchange interaction which, in our case, would involve the localized d electrons of Gd and the itinerant d electrons of Pd. Recently, Brierley and Griffin[29] have developed a magnetic equation of state for $Li(Tb_xY_{1-x})F_4$ on the basis of a randomly diluted dipolar coupled Ising ferromagnet, which, for x equal to 0.63 and 0.90, yields values for γ of 1.12 and 1.06, respectively. The resolution of just what is responsible for the observed long-range order behavior in amorphous systems obviously awaits a choice of alloy capable of providing suitable discrimination between possible contributing interactions.

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