Ferromagnetism in reentrant $Pd\mathrm{Fe}_x\mathrm{Mn}_y$

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Muon spin relaxation in zero applied field has been used to study the ferromagnetism of the reentrant ternary alloy $Pd\mathrm{Fe}_{0.0035}\mathrm{Mn}_{0.05}$. The static linewidth below T_C shows that Mn participates in the ferromagnetic ordering at T_C. The temperature dependence of the dynamic depolarization demonstrates that relatively slow fluctuations ($\tau_{\mathrm{eff}} \approx 10^{-10}$–$10^{-11}$ s) persist in the region between T_C and the spin-glass temperature T_g. The transition at T_C is marked by critical fluctuations of the Mn spins, but the glass transition at T_g is not. A detailed analysis of the form of the dynamic depolarization function indicates that the muon does not occupy the interstitial sites nearest the Mn impurities.

I. INTRODUCTION

One of the unsolved problems in spin-glass research is the nature of the double transition seen in some materials. These systems exhibit a transition to a ferromagnetic state at a Curie temperature T_C, and a transition to a spin-glass-like state at a lower temperature T_g. The wide variety of materials exhibiting a reentrant phase diagram suggests that the double transition is a common feature of magnetic systems with competing interactions.[1]

Early measurements[2] on *Pd*Mn disclosed a narrow concentration regime between ferromagnetic and spin-glass phases that suggested reentrance, but later ac susceptibility data were interpreted as evidence against a double transition.[3] Verbeek *et al.*[4] discovered that the addition of small amounts of Fe to *Pd*Mn increases the ferromagnetic transition temperature while leaving the spin-glass transition line of the phase diagram unchanged, and thereby produces a system with a clear double transition. They interpreted their results within the mean-field Sherrington-Kirkpatrick (SK) model,[5] which predicts a reentrant system for certain parameter values. An alternative model, in which only the Fe spins develop a spontaneous magnetization at T_C, has also been put forward.[6,7]

In the present paper, we present zero-field muon-spin-relaxation (μSR) results on one alloy, $Pd\mathrm{Fe}_{0.0035}\mathrm{Mn}_{0.05}$, for which $T_C \approx 8$ K and $T_g \approx 2$ K. Our data allow us to conclude that the Mn participates in the spin freezing at T_C, that low-frequency fluctuations persist in the interval from T_C to T_g, and that critical fluctuations are clearly present near T_C but are not observed near T_g. A detailed analysis of the μSR relaxation function also leads us to suggest that the muon is physically excluded from the interstitial sites closest to the Mn sites.

The μSR experiments were carried out at the Los Alamos National Laboratory, Clinton P. Anderson Meson Physics Facility in a standard longitudinal configuration μSR spectrometer.[8] This experimental arrangement allows us to measure the dynamic contributions to the muon relaxation rate at all temperatures. Below the ordering temperature we can independently estimate the quasistatic and dynamic contributions to the relaxation. Sample temperatures above 2.5 K were obtained in a continuous-transfer cold-finger cryostat. A dilution refrigerator, described elsewhere,[9] was used to attain temperatures from 0.5 to 4.0 K. The sample was prepared as in Ref. 4, with an additional anneal done a few days before the measurement to avoid impurity segregation due to aging.

The determination of the quasistatic local-field distribution width, which demonstrates the Mn-spin freezing, is discussed in Sec. II, while the dynamic spin-lattice relaxation data are presented and discussed in Sec. III. We state our conclusions in Sec. IV.

II. STATIC FIELD DISTRIBUTIONS

Below T_C the freezing of some or all of the local moments will produce a distribution of quasistatic local

fields at the muon stopping sites. The distribution function of the local field can be calculated in a dilute random alloy for any distribution of the orientations of the local moments.[10] In the absence of muon spin-lattice relaxation, the zero-field muon depolarization function takes on the simple form[10,11]

$$P(t)=\tfrac{1}{3}+\tfrac{2}{3}[1-a_s(T)t]e^{-a_s(T)t}, \quad (1)$$

where $a_s(T)$ is a temperature-dependent static linewidth. When weak muon spin-lattice relaxation is present, the long-time part of the depolarization function becomes more complicated,[11–13] but the short-time decay is unaffected. We therefore use the parameter a_s defined by Eq. (1) to characterize the quasistatic field distribution.

For two dilute atomic species the calculation of $a_s(T)$ separates into a sum of contributions from the two species. In the present case we can explicitly write for $Pd\mathrm{Fe}_x\mathrm{Mn}_y$

$$a_s(T)=a_s(Pd\mathrm{Fe}_x,T)+a_s(Pd\mathrm{Mn}_y,T) . \quad (2)$$

The separate contributions from Fe and Mn can be calculated as in Ref. 10. An upper limit to $a_s(T)$, assuming ferromagnetic spin alignment and magnetic dipole coupling to the muon, can be found by setting the thermal average of the impurity spin equal to the saturation value, $\langle S_z\rangle=S$. Denoting this limit by a_{s0}, we find $a_{s0}(Pd\mathrm{Fe}_{0.0035})=3.3\ \mu\mathrm{s}^{-1}$ and $a_{s0}(Pd\mathrm{Mn}_{0.05})=68\ \mu\mathrm{s}^{-1}$, for a total linewidth of $a_{s0}=71\ \mu\mathrm{s}^{-1}$. In computing these numbers we have used the bare Fe moment of $(3.5\pm0.4)\mu_B$ from neutron scattering[14] and $S=1.75$. Free-ion values were used for Mn. The temperature dependence $a_s(T)$ expected in two different models will be discussed below.

The occurrence of an extended polarization cloud ("giant moment") around the Fe or Mn impurities could, of course, increase a_{s0} substantially. Such an effect is seen in the transverse-field μSR linewidth of very dilute *Pd*Fe alloys.[15] For $c\leq0.28$ at.% the observed linewidth is much larger than that estimated[16] from dipolar coupling to the bare Fe moment. At higher concentrations of magnetic impurity the effects of host polarization on the muon spin should be reduced for at least two reasons. First, the overlap of the giant moment clouds for $c\geq0.3$ at.% rapidly reduces the magnitude of the average moment per impurity at higher concentrations.[17] Second, the overlapping polarization clouds produce an approximately uniform magnetization density in the host. This magnetization density will shift the muon resonance frequency, but it contributes only weakly to the linewidth.[18] These conjectures seem to be borne out by the available μSR data in the paramagnetic phase of more concentrated *Pd*Fe and *Pd*Mn alloys. In $Pd\mathrm{Fe}_{0.03}$ the measured[19] linewidth is consistent with the value calculated from dipolar coupling to the bare Fe moment. In $Pd\mathrm{Mn}_{0.02}$ and $Pd\mathrm{Mn}_{0.07}$ the observed linewidth is actually slightly less than that estimated for the bare Mn moments.[18,20] On this basis we conclude that use of the bare impurity moments will give a sufficiently accurate estimate of a_{s0} in *Pd*FeMn.

Experimental data on our *Pd*FeMn specimen were obtained at several temperatures near and below T_C. At $T=7.5$ K the data can be fitted to Eq. (1) with $a_s(T)=(8.7\pm1.5)\ \mu\mathrm{s}^{-1}$. This point is shown in Fig. 1. Below 7.5 K $a_s(T)$ becomes larger, and we are unable to observe enough of the initial decay to obtain a reliable fit. The known spectrometer dead time of ≈30 ns allows us to set a lower limit $a_s(6\ \mathrm{K})\geq30\ \mu\mathrm{s}^{-1}$ which is also noted in Fig. 1. Closer to the transition the functional form of $P(t)$ is observed to change due to the increasingly significant dynamic effects, and a separate static linewidth is no longer visible. We can obtain an upper limit to the static linewidth by fitting the early part of $P(t)$, and attributing all the observed decay to quasistatic local fields. The results imply $a_s(T)\leq0.5\ \mu\mathrm{s}^{-1}$ from T_C to 10 K. Rates this small are essentially coincident with the baseline in Fig. 1, indicating that the quasistatic fields vanish at T_C.

The SK model for the temperature dependence of $a_s(T)$ in a coupled spin system requires that both Mn and Fe spins develop a spontaneous magnetization below T_C. The temperature dependence is given by $a_s(T)=a_{s0}r(T)$, where $r(T)$ is the linewidth order parameter.[18] To calculate $r(T)$ we must specify the ratio of the mean to standard deviation of the exchange energy distribution $\bar{J}_0/\bar{J}$. This ratio was estimated by Verbeek *et al.*[4] from the ratio T_C/T_g to be $\bar{J}_0/\bar{J}=1.05$ for the concentration used here. Numerical calculations of $a_{s0}r(T)$ give the solid line shown in Fig. 1, where we have used $T_C=8.25$ K (see Sec. III). This estimate of $a_s(T)$ appears to be in rough agreement with the rapidly increasing rates observed.

Alternatively, the Mn and Fe spin systems may be only loosely coupled. This description is suggested by the field and temperature dependence of the resistivity[6] near T_g and T_C. Further support is provided by a comparison of nuclear orientation[7] and Mössbauer data,[21] which indicates that the Fe spins orient in an external field much more readily than the Mn spins. If the two spin systems are not well coupled they may make separate transitions to the ferromagnetic and spin-glass states. The separate-

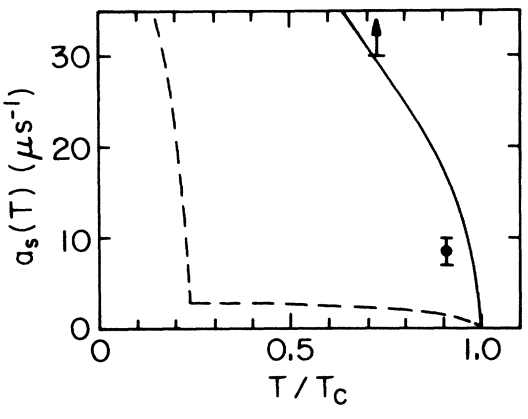

FIG. 1. Temperature dependence of the zero-field static linewidth $a_s(T)$. The data point and the lower limit on $a_s(T)$ were measured in $Pd\mathrm{Fe}_x\mathrm{Mn}_y$. Above T_C the estimated upper limits on $a_s(T)$ are coincident with the baseline and are not shown. The solid line is $a_{s0}r(T)$ calculated from the SK mean-field model for coupled Mn and Fe spin systems, while the dashed line assumes separate transitions in the two spin systems.

transition model is made more attractive by the observation that a $Pd\mathrm{Fe}_{0.0035}$ alloy[4] has $T_C=9.0$ K, while a $Pd\mathrm{Mn}_{0.05}$ alloy[3] has $T_g=3.0$ K. Both of these temperatures are only slightly above the corresponding transition temperatures observed in our $Pd\mathrm{Fe}_x\mathrm{Mn}_y$ specimen.

In the separate transition model the μSR linewidth would be expected to saturate at the Fe-only value, $a_{s0}=3.3\ \mu\mathrm{s}^{-1}$, at some temperature between T_C and T_g because the Fe spins will freeze at T_C, while the Mn spins would not be quasistatic in this temperature range. Below T_g the Mn spins would also freeze, and $a_s(T)$ should rise dramatically. This temperature dependence is shown by the dashed line in Fig. 1, calculated from the SK model for the two uncoupled spin systems using parameters determined from T_C and T_g, respectively. The point at 7.5 K and the observed lower limit of the linewidth at 6.0 K are clearly inconsistent with this picture, indicating that the Mn spins do participate in the freezing at T_C, despite the evidence for weak coupling between the Fe and Mn spins.

III. SPIN-LATTICE RELAXATION

A. Fitting functions

Time-dependent fluctuations of the impurity moments contribute to the muon depolarization through the magnetic dipole coupling to the muon moment. As noted above, the dynamic contribution to the depolarization dominates the observed relaxation at long times for $T<T_C$. Above T_C there is no quasistatic field and the observed relaxation is entirely dynamic in origin. The resulting relaxation function $P(t)$ is usually assumed to be either exponential or root exponential, $P(t)\propto\exp[-(\lambda t)^{1/2}]$. We will argue below that neither form adequately describes our data, so we briefly review the calculation of the depolarization function in the absence of muon diffusion. More complete discussions have been given by McHenry *et al.*,[22] and by Seymour and Sholl.[23]

In the motional narrowing limit, which is appropriate here, a muon at site j has an exponential depolarization function

$$P_j(t)=\exp\left[-t\sum_i{}'\frac{1}{T_1(r_{ij})}\right], \tag{3}$$

where the sum is restricted to the occupied impurity sites surrounding the muon site j. The overall depolarization function $P(t)$ observed in a μSR experiment is an average of the $P_j(t)$ over all muon sites. Since the $P_j(t)$ may depend strongly on j, the average $P(t)$ will in general be nonexponential. For dipolar or Ruderman-Kittel-Kasaya-Yosida (RKKY) coupling, $1/T_1(r_{ij})$ can be approximated[23] by an angularly averaged relaxation rate $1/T_1(r)=K/r^6$, where K is a temperature-dependent quantity containing the impurity fluctuation time and the coupling strength. Note that any concentration dependence observed in K is due to the concentration dependence of the impurity dynamics. All direct concentration dependence in the muon relaxation function has been removed through the sum in Eq. (3) and the average over muon sites.

When $1/T_1(r)=K/r^6$ the average relaxation function is given by[22,23]

$$P(t)=\prod_i[(1-c)+c\exp(-tK/r_i^6)], \tag{4}$$

where c is the impurity concentration and the r_i are the possible impurity site locations relative to the muon site. The general behavior of the relaxation function is determined by c and by $t_0=r_0^6/K$. The parameter r_0 is the minimum muon-impurity distance, so t_0^{-1} is the maximum muon relaxation rate due to a single impurity. When $c\to 1$, $P(t)$ is exponential at all times, but at the concentrations of interest here, $c\leqq 0.1$, $P(t)$ is exponential for $t\ll t_0$ and root exponential for $t\gg t_0$. Practically, it may be difficult to observe the exponential region at low concentration because very little depolarization will occur for $t\leqq t_0$. The root exponential will then be a good approximation to the observable depolarization function.

An explicit calculation of K is straightforward in the dipolar coupling case, yielding[22]

$$K=1.33(\hbar\gamma_\mu\gamma_s)^2S(S+1)\tau_{\mathrm{eff}}, \tag{5}$$

where γ_μ and γ_s are the muon and impurity gyromagnetic ratios, S is the impurity spin, and τ_{eff} is an effective impurity correlation time. The numerical factor arises from the angular averaging of the dipolar coupling.

Equation 4 is clumsy to use for fitting data, since evaluation of the lattice sum is lengthy. In the limit of small concentrations it is convenient to use a continuum approximation, replacing the lattice sum by an integral to obtain[22]

$$P(t)=\exp\left\{-\frac{4\pi}{3}r_0^3\rho c\left[e^{(-t/t_0)}-1+\left(\frac{\pi t}{t_0}\right)^{1/2}\mathrm{erf}\left(\frac{t}{t_0}\right)^{1/2}\right]\right\}, \tag{6}$$

where ρ is the number density of host sites. The quantity r_0 is an inner cutoff radius for evaluation of the integral. In terms of these parameters, the limiting root-exponential rate is $\lambda=(16\pi^3/9)\rho^2c^2K$, obtained when $r_0=0$. By adjusting r_0, one can also approximate the exact $P(t)$ for the case where the near-neighbor sites are excluded. These two situations are shown in Fig. 2, which compares $P(t)$ calculated for $c=0.05$ from the lattice sum Eq. (4), assuming that the muon occupies octahedral interstitial sites, and from Eq. (6). The correspondence is clearly adequate if r_0 is taken as a freely adjustable parameter.

B. Results and discussion

The muon relaxation data above T_C were initially fit with both exponential and root-exponential functions. Table I shows the resulting χ^2 per degree of freedom for zero-field measurements at three temperatures. The table demonstrates an apparent systematic change from an ex-

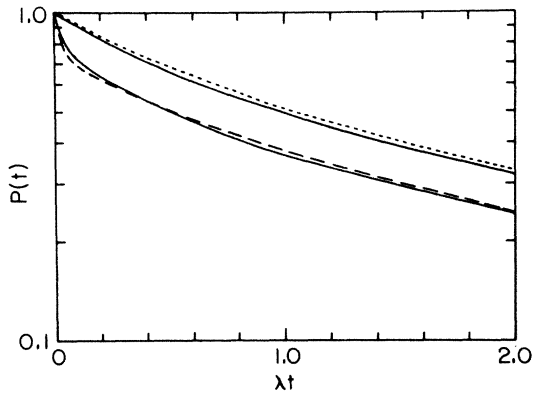

FIG. 2. Comparison of $P(t)$ from the lattice sum and continuum expressions for $c=0.05$, with time scaled by the root-exponential relaxation rate λ. The dashed line is calculated from Eq. (4) including all the octahedral interstitial sites, while the dotted line excludes sites adjacent to impurities. The corresponding solid lines are calculated from Eq. (6) with $r_0/a_0=0.0$ and 0.7, chosen to give the best agreement with the lattice sum calculation.

ponential form at the highest temperature (low relaxation rate) to a root-exponential form at lower temperature. Neither function is adequate to fit the entire range. The more general form of Eq. (6) requires a two-parameter fit to the data in a region where the relaxation function does not have either limiting form. The $P(t)$ data at $T=10$ K fulfill this requirement, as suggested by Table I. Accordingly, $P(t)$ from Eq. (6) was fit to the data by adjusting r_0 and t_0. We find $r_0/a_0=0.72\pm0.08$, where a_0 is the Pd fcc lattice parameter. This value of r_0 was then held fixed for the fits at other temperatures. The χ^2 values in the last column of Table I demonstrate that this procedure accurately describes our depolarization data.

Comparing the value of r_0 deduced from fitting the data to Eq. (6) with the lattice-sum results shown in Fig. 2, we infer that there is no contribution to $P(t)$ from the interstitial sites nearest to the impurities. In a normal resonance experiment, with a large applied field, this might be due to the shift of the muon resonance frequency produced by the polarized impurity moment. The shift would also entail a loss of signal amplitude relative to the pure host signal by a factor of $(1-c)^6=0.74$ since the six near-neighbor sites would not contribute to the intensity of the observed signal. Here, the muon $P(t)$ is measured in zero field and the full asymmetry is observed above T_C, so this mechanism cannot be significant. We suggest instead that the muon is physically excluded from the octahedral interstitial sites nearest the Mn impurities. This could be caused by a contraction of the host lattice around the Mn, or by a repulsive muon-Mn interaction of electronic origin. X-ray lattice parameter measurements on a series of *Pd*Mn alloys containing 2–10 at. % Mn do not show any significant deviation from the pure Pd lattice parameter, leading us to infer that the effect is electronic.

TABLE I. Comparison of χ^2 per degree of freedom for three fitting functions at each of three temperatures above T_C. In Eq. (6) a fixed r_0 is used, as discussed in the text.

T (K)	χ^2 (exp.)	χ^2 (root-exp.)	χ^2 [Eq.(6)]
12	1.09	1.64	1.05
10	1.15	1.29	1.00
9	1.50	0.74	0.75

Physical exclusion from certain muon sites will also account for earlier transverse-field results in *Pd*Mn alloys. The μSR linewidth well above the transition temperature is proportional to the impurity spin polarization, with a calculable[16] constant of proportionality. Assuming dipolar coupling and the free-ion Mn moment, the measured proportionality constants are 17% and 59% smaller than the calculated values for $Pd\text{Mn}_{0.02}$ and $Pd\text{Mn}_{0.07}$, respectively.[18,20] The full signal amplitude is seen in both alloys. A calculation of the linewidth allowing for exclusion from near-neighbor sites predicts reductions of 12 and 30% for the two hosts, assuming a completely random distribution of Mn sites. The remaining discrepancy might be partially explained by the known tendency of Mn to anticluster, or by exclusion beyond near-neighbor sites in the case of Mn pairs.

Assuming that $r_0/a_0=0.72$, we obtained values of t_0 by fitting Eq. (6) to the muon relaxation data for $Pd\text{Fe}_x\text{Mn}_y$. The results are plotted in Fig. 3, expressed

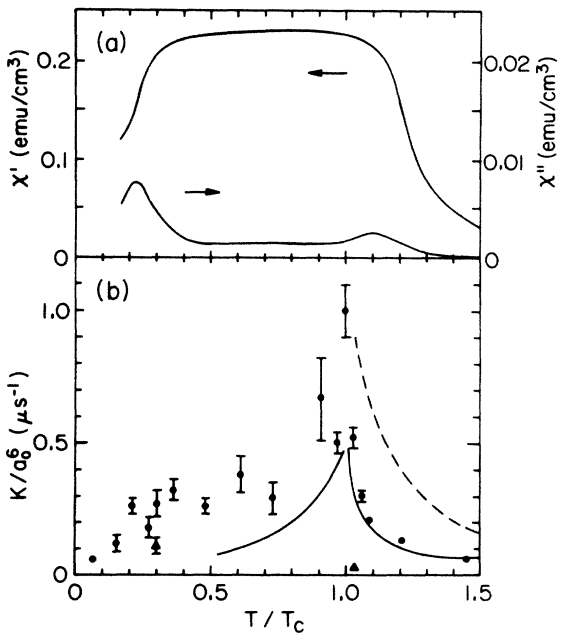

FIG. 3. (a) Temperature dependence of the real (χ') and imaginary (χ'') parts of the low-field ac susceptibility of the μSR sample, showing $T_g\approx1.9$ K from the peak of χ''. (b) Temperature dependence of the zero-field muon spin-lattice relaxation parameter, defined in Eq. (4), scaled by the Pd lattice parameter a_0. $T_C=8.25$ K, from the peak of the μSR relaxation rate. Circles: zero applied field. Triangles: 1 kOe longitudinal field. The solid line represents muon relaxation rates in ferromagnetic $Pd\text{Mn}_{0.02}$ with temperatures scaled by $T_C=5.8$ K (Ref. 18). The dashed line represents muon relaxation rates in spin-glass $Pd\text{Mn}_{0.07}$ with temperatures scaled by $T_g=5.0$ K (Ref. 20).

as a scaled spin-lattice relaxation rate constant $K/a_0^6=(r_0/a_0)^6 t_0$. Above T_C the fits have the full muon asymmetry and the whole time range is included in the fit. Below T_C the initial damping due to the quasistatic fields reduces the initial asymmetry for the dynamic part to $\frac{1}{3}$, and only the time range beyond the initial falloff is included in the fit. Two additional data points (triangles) were taken in an applied longitudinal field. The significance of these data will be discussed below. For comparison, we have also reanalyzed earlier data[18,20] on ferromagnetic $Pd\mathrm{Mn}_{0.02}$ and on spin-glass $Pd\mathrm{Mn}_{0.07}$, using Eq. (6) with $r_0=0.72a_0$. These results, scaled by their respective ordering temperatures, are plotted as the solid and dashed lines. Figure 3 also presents the temperature dependence of the low frequency (21.7 Hz) ac susceptibility on the same $Pd\mathrm{Fe}_x\mathrm{Mn}_y$ sample.

The onset of dynamic broadening above T_C is similar to that seen in ferromagnetic *Pd*Mn, as shown by the solid curve in Fig. 3. Furthermore, application of a longitudinal magnetic field strongly suppresses the muon relaxation for $T \geqq T_C$ in agreement with previous results in ferromagnetic *Pd*Mn.[18] By contrast, a field has only a modest effect on muon relaxation in a spin-glass.[20] It seems reasonable to conclude that the transition at T_C is to a ferromagnetic state, as previously reported.[4] It is not known why the Curie temperature determined from the peak of the μSR relaxation rate, $T_C=8.25\pm0.1$ K is somewhat lower than the temperature of the peak in χ'' or the inflection in χ'.

The transition to the spin-glass state is marked by the lower-temperature peak in χ'' and the falloff in χ'. There is no comparable signal in the muon relaxation, although the relaxation rate gradually decreases below T_g and is much less sensitive to applied fields near T_g than it is near T_C. A nonreentrant spin-glass, for example $Pd\mathrm{Mn}_{0.07}$, would display an increase in relaxation rate over a wide temperature range as $T \rightarrow T_g$ from above (indicated by the dashed line in Fig. 3). The rate near T_g would be similarly insensitive to applied fields.[20]

In the temperature region between T_C and T_g the relaxation rate constant remains substantially elevated, in contrast to the ferromagnet where the rate constant falls to unmeasurably low values at comparable reduced temperatures. This indicates the continued presence of slow fluctuations in this temperature range. From Eq. (5) we can deduce effective correlation times $\tau_{\mathrm{eff}} \approx 10^{-10}-10^{-11}$ s for these fluctuations. At the same time, the lack of an increase of K/a_0^6 near T_g in $Pd\mathrm{Fe}_x\mathrm{Mn}_y$ implies that the ferromagnet to spin-glass transition is not accompanied by critical fluctuations in the frequency range $\omega \approx 10^{10} s^{-1}$ probed by μSR. The low-frequency spin fluctuations of this reentrant system are therefore unlike those of either a disordered ferromagnet or a spin glass.

IV. CONCLUSIONS

Our main conclusions can be summarized as follows:

(1) The static muon linewidth in the ferromagnetic phase shows that the Mn spins participate in the spin freezing at T_C. This rules out the possibility that the ferromagnet to spin-glass transition at T_g might represent a simple spin-glass freezing of the Mn spins.

(2) A detailed analysis of the time dependence of the zero-field muon depolarization function shows that neither the simple exponential nor the commonly used root-exponential forms can fully describe the temperature dependence of the muon spin-lattice relaxation. We have used a more general function[22,23] which allows for the possibility that some of the host interstitial sites are not accessible to the muons. The fits to our data suggest that muon sites nearest to the impurity are in fact forbidden. The effect is also present in *Pd*Mn alloys previously studied by μSR.[18,20]

(3) The field and temperature dependence of the muon spin-lattice relaxation rate near T_C is consistent with a ferromagnetic transition at T_C involving the Mn spins. Below T_C, slow fluctuations persist to the lowest temperatures measured, but there is no strong dynamic signature of the transition at T_g. This indicates that the spin dynamics in this reentrant system are significantly different than the dynamics in either disordered ferromagnets or canonical spin glasses.

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