

Minima of the muon depolarization rate in $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$

A. Golnik

Institute of Experimental Physics, Warsaw University, Hoża 69, PL-00-681 Warsaw, Poland

A. Weidinger

Hahn-Meitner-Institut Berlin, Glienickerstrasse 100, D-14109 Berlin, Germany

Ch. Niedermayer, C. Bernhard, and E. Recknagel

Universität Konstanz, Fakultät für Physik, Universitätsstrasse 1, D-78434 Konstanz, Germany

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Muon-spin rotation experiments were carried out on $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ samples with different Mn concentrations x and different annealing treatments (in Cd vapor or in vacuum). The purpose of the experiment was to elucidate the origin of the pronounced minima in the temperature dependence of the muon depolarization rate. We found that the data can be analyzed in a two-state model with the assumption that the muon has a high ($10\text{--}30\ \mu\text{s}^{-1}$) depolarization rate in state one and a low ($0.1\text{--}0.3\ \mu\text{s}^{-1}$) depolarization rate in state two. The minima are attributed to a change from state one (presumably a paramagnetic center) to state two (presumably a diamagnetic center). The state change is either purely electronic or caused by a site change of the muon. [S0163-1829(97)05319-8]

INTRODUCTION

The $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ mixed crystals are the classical example of the wide gap diluted magnetic semiconductors (DMS's) and belong to the most intensively studied representatives of this class of magnetic materials. The magneto-optical and magnetotransport properties of DMS materials are determined by the strong exchange interaction between charge carriers (delocalized p - and s -type electrons) and localized spins of magnetic ions (d -shell electrons).¹⁻³ The magnetic properties are governed by the antiferromagnetic d - d exchange^{4,5} between magnetic Mn ions at the cation sublattice of CdTe. The exchange interaction leads to *short range antiferromagnetic ordering* for higher Mn concentrations⁶ and *spin-glass-like behavior* for lower Mn contents.⁷

The muon-spin rotation (μSR) technique^{8,9} has been already successfully applied to study the spin freezing in $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ crystals.¹⁰⁻¹⁴ The divergent increase of the μSR depolarization rate, when the crystal was cooled down to the spin-freezing temperature was interpreted as a sign of the critical slowing down of spin fluctuations at the freezing temperature. The analysis of the temperature dependence of the μSR depolarization rate in this temperature region enabled the determination of critical exponents^{11,12} and estimates of spin fluctuation rates.¹³

At intermediate temperatures, the muon depolarization rate exhibits unusual minima as first observed by Ansaldo *et al.*^{11,12} There were already several investigations on these minima^{11,12,14} but no clear conclusions could be reached. The aim of present work is to obtain more information on these minima and to contribute to a better understanding of their origin.

It has been suggested^{11,12} that the minima are caused by muon trapping at defects. In this case, a change of the concentration of these defects should influence the location of

the minima. A good candidate for a trapping center is the cation vacancy which in CdTe and $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ forms acceptor centers when pairing with impurities.¹⁵⁻¹⁹ The anomalies observed in the region of the minima may then result from the muon trapping and detrapping from such acceptors.

In order to check this hypothesis, we performed a series of μSR experiments on the $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ crystals annealed under different conditions. Annealing in Cd vapor was used to decrease the concentration of the cadmium vacancy acceptors, whereas vacuum annealing was used to produce more Cd vacancies.

The results of the present work rule-out the Cd-vacancy model. We will show, however, that a two-states model involving changes of the electronic configuration of the muon center gives an adequate description of the data.

I. EXPERIMENT

All $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ samples used in the present μSR experiment were single crystals grown at the Institute of Physics of the Polish Academy of Sciences. The crystal dimensions were in the order of 14 mm in diameter and 1-5 mm in thickness. The $x=0.40$ crystal was especially grown for the μSR experiments and had a diameter of 20 mm. Since the muon beam was collimated to 10 mm diameter, all muons stopped in the sample and no background signal was expected.

After measurements on as grown crystals, selected samples were annealed under various conditions. The Mn concentration was checked before and after annealing by x-ray fluorescence and was found to agree with the nominal concentration within the accuracy of the method (0.02).

The samples with $x=0.30$ and 0.40 were annealed in vacuum for 24 h at 740°C and then quenched rapidly in water. The $x=0.20$ sample and a part of a previously vacuum annealed $x=0.40$ sample were annealed in Cd vapor

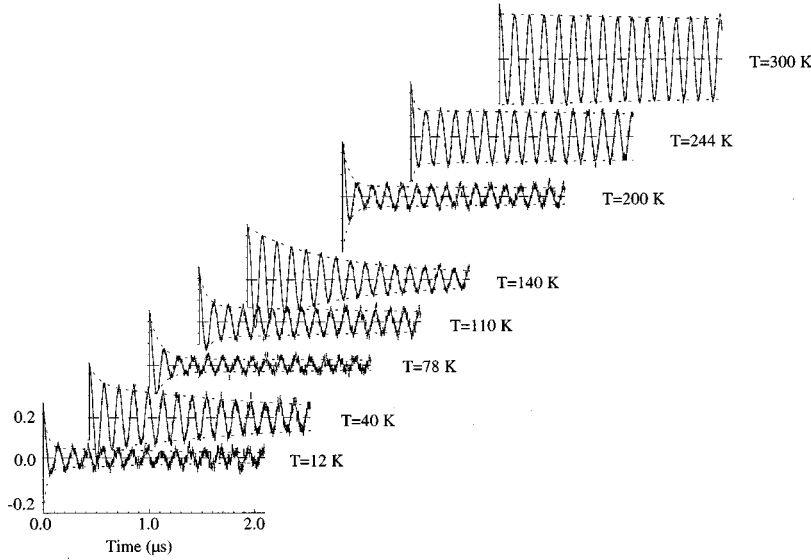


FIG. 1. μ SR spectra (asymmetry part) of a $\text{Cd}_{0.8}\text{Mn}_{0.2}\text{Te}$ crystal at different temperatures.

at 540 °C and slowly cooled in the furnace. It is expected¹⁷ that the Cd-annealed samples have a significantly lower free-carrier concentration than the untreated samples and that the conductivity is dominated by acceptor levels which are somewhat deeper than in the virgin sample.

The carrier concentration of the annealed samples was checked by infrared transmission measurements. This method was applied by Wojtal *et al.*¹⁶ in the studies of $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ crystals with $x=0.05$ and 0.1. In our crystals, with significantly higher Mn contents, the IR spectra were not well fitable with the formulas used in Ref. 16, thus we were able to determine quantitatively only the free-carrier concentration. The information about acceptor concentration and ionization energy was more qualitative. We found that the hole concentrations at room temperature was between $1 \times 10^{16} \text{ cm}^{-3}$ and $6 \times 10^{16} \text{ cm}^{-3}$ for as-grown $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ crystals used in this work. After vacuum annealing the concentration increased by a factor of 2–5. In the Cd-annealed samples the free-carrier concentration at room temperature was below the detection limit of the IR method. Namely, for $\text{Cd}_{0.6}\text{Mn}_{0.4}\text{Te}$ sample we found the room-temperature hole concentration to be $(0.8 \pm 0.3) \times 10^{16} \text{ cm}^{-3}$, $(1.6 \pm 0.5) \times 10^{16} \text{ cm}^{-3}$ and less than $0.3 \times 10^{16} \text{ cm}^{-3}$ in the as grown, vacuum annealed, and Cd-annealed sample, respectively.

Most of the μ SR experiments were done with the surface muon beam at the Paul-Scherrer Institute in Villigen, Switzerland. Some additional measurements were performed at the TRIUMF facility in Vancouver, Canada. The transverse field μ SR configuration (TF- μ SR) (Refs. 8 and 9) was chosen for the present experiments since the analysis of TF- μ SR spectra is simpler and gives more reliable information on amplitudes. We have checked previously^{13,14} that for the $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ crystals the same double exponential depolarization is observed in both TF- μ SR and in the zero-field μ SR (ZF- μ SR) method, which is the standard method to study the spin-glass freezing.²⁰

Spin polarized positive muons are implanted into the $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ samples with an energy of 4 MeV and come to rest without losing their spin polarization. Only one muon is present in the sample at a given time. In the TF- μ SR

experiments the external field is perpendicular to the spin polarization of the muon beam, thus the spin of the implanted μ^+ precesses around the field direction. The μ^+ -spin precession frequency is given by the muon gyromagnetic ratio $\gamma_\mu/2\pi = 135.534 \text{ MHz/T}$ and the value of the internal magnetic field at the muon site.

The positive muon decays with a time constant of $\tau_\mu = 2.1971 \mu\text{s}$ into the positron and two neutrinos. Since the positron is emitted preferentially in the direction of muon spin, the spin precession can be registered by detecting the positrons as a function of time between the muon implantation and its decay. If the positron counter is placed at the angle θ to the initial direction of the muon spin, the obtained histogram obeys the relation

$$N(t) = N_0 \exp(-t/\tau_\mu) [1 + A G_x(t) \cos(\omega t + \theta)] + B_0, \quad (1)$$

where B_0 and N_0 are the background and normalization constants, respectively.

The theory of weak interaction predicts the value of $\frac{1}{3}$ for the muon decay asymmetry A , but the observed value of A is usually lower, mainly because of the finite solid angle covered by the positron counter. The physical information on the internal field distribution and dynamics is contained in the precession term $\cos(\omega t + \theta)$ and depolarization function $G_x(t)$.

The standard TF- μ SR spectra (like those shown in Fig. 1) display only the asymmetry part of the above formula, i.e., the oscillatory term $A G_x(t) \cos(\omega t + \theta)$. If the muons form different species or stop at different sites, the asymmetry term takes the form of the sum of different components $\sum_i A_i G_{x_i}(t) \cos(\omega_i t + \theta)$. The amplitudes A_i determine the muon fractions which contribute to the different species or sites. Such a simple picture becomes more complicated when the positive muon in semiconductor captures an electron forming a muonium (Mu) center.^{21,22} However, in some special cases, when the electron in Mu center exhibits strong depolarization (e.g., due to the spin exchange), the observed TF- μ SR signal might be almost identical to that of bare muon.^{21,23}

II. RESULTS

Typical μ SR spectra for a $\text{Cd}_{0.8}\text{Mn}_{0.2}\text{Te}$ crystal are shown in Fig. 1. It can be seen that the form of the spectra and the depolarization rates change strongly and nonmonotonously as a function of temperature. At 12 K, the initial signal is strongly damped and goes over at later times to an almost undamped signal with a small amplitude. Similar spectra are observed at 78 K and 200 K. However, at temperatures between these points, the spectra are very different, showing much weaker and more continuous damping.

The precise analysis of μ SR spectra always involves some arbitrary choice of the fitting function. The best description of the μ SR spectra observed for $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ in our experiments can be achieved using a sum of two exponentially damped oscillations $\sum_{i=1}^2 A_i \exp(-\lambda_i t) \cos(\omega_i t + \theta)$. Fits with a single component but with a stretched exponential damping $G_x(t) = \exp(-(\delta t)^\beta)$ (like recently proposed for spin glasses³⁴) were not satisfactory and generally inferior to the fits with two exponentials.

In the following discussions we will refer also to the three-component fits. Such fits with three exponential components give in principle more continuous behavior of the relative amplitudes of the components. However, when the relaxation rates of two weakly damped components become comparable (like in the temperature region of the minima) the separation of the components becomes ambiguous, and requires human intervention into the fitting routine (fixing of some parameters). Therefore, in the following we will present and discuss mainly the two component fits, which are more reliable. The only constraint put into the two exponential fit was that the sum of the amplitudes was not allowed to exceed the amplitude derived from the calibration run.

The two exponentials are characterized by the mean precession frequencies ω_1, ω_2 , the depolarization rates λ_1, λ_2 , and the relative amplitudes A_1, A_2 . The mean frequencies are, except near the spin-freezing temperature,¹⁰ given by the external field, so in the following only the depolarization rates and relative amplitudes of the components will be discussed in details.

Figure 2 shows the depolarization rates of the strongly damped component for a set of as-grown $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ samples with different Mn concentrations (measured in our previous experiments¹⁴). The pronounced minima mentioned in the introduction and reported previously by Ansaldo *et al.*^{11,12} are clearly seen. One kind of minima is observed for all samples. The location of these minima shifts from about 100 K for $x=0.15$ to about 200 K for $x=0.4$. A second minimum at lower temperatures appears for $x=0.2$ and 0.15 samples, but seems to be washed out for the higher Mn concentrations. For crystals with $x \leq 0.05$ the strongly damped component was not observed at any temperature.

The relative amplitude of the weakly damped component (the other is one minus that) and the two relaxation rates are displayed by open and solid symbols in Figs. 3–5 for the as grown, vacuum-, and Cd-annealed $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ samples with $x=0.4, 0.3$, and 0.2, respectively.

One could note that the data for as grown $\text{Cd}_{0.60}\text{Mn}_{0.40}\text{Te}$ sample shown in Fig. 2 are different from that shown in Fig. 3 for same sample. Data from Fig. 2, are more complete and cover the whole temperature range, but

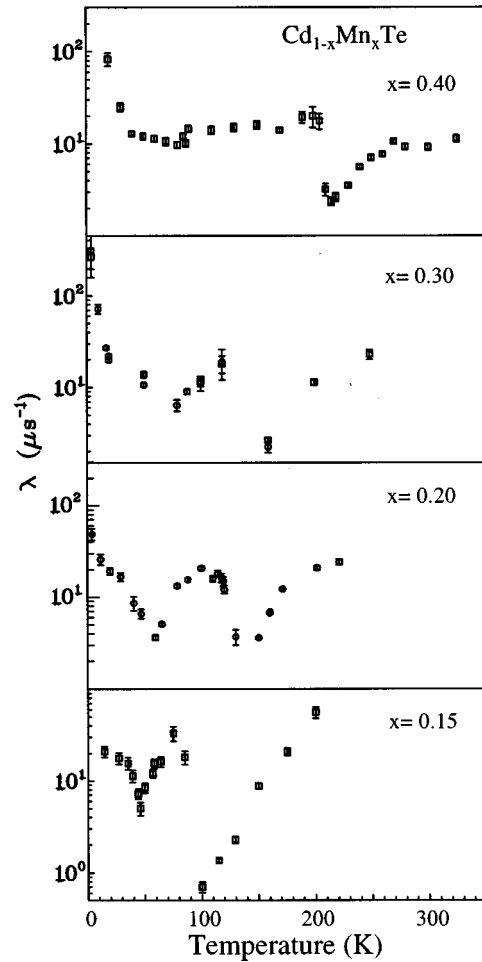


FIG. 2. Temperature dependence of the depolarization rate λ for the strongly damped component of the μ SR signal in $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$.

were taken on the high momentum muon beam with high background signal (about 40%). The analysis of the amplitudes for this data was then unreliable. The data shown in Fig. 3 for as grown sample, although less complete, are background free, thus might be compared with later data obtained for the same sample after annealing.

The main features, which could be deduced from Figs. 3–5, are as follows:

(i) The depolarization rate of the strongly damped component outside of the minima and not too close to the spin-freezing temperature is on the order of $10\text{--}30 \mu\text{s}^{-1}$, whereas the depolarization rate of the weakly damped component is on the order of $0.1\text{--}0.3 \mu\text{s}^{-1}$. Both are slightly higher for the smaller Mn concentrations than for the larger concentrations. They exhibit a moderate-temperature dependence, except at the anomalies.

(ii) The relative fractions of the two components exhibit strong temperature variations. It can be seen in Figs. 3–5 (left-hand side), where the relative amplitude of the weakly damped component is shown. The rapid changes of the amplitude of that component are the most significant signatures of the minima.

(iii) The minima are then characterized by a gradual increase of the slowly damped fraction (open symbols) fol-

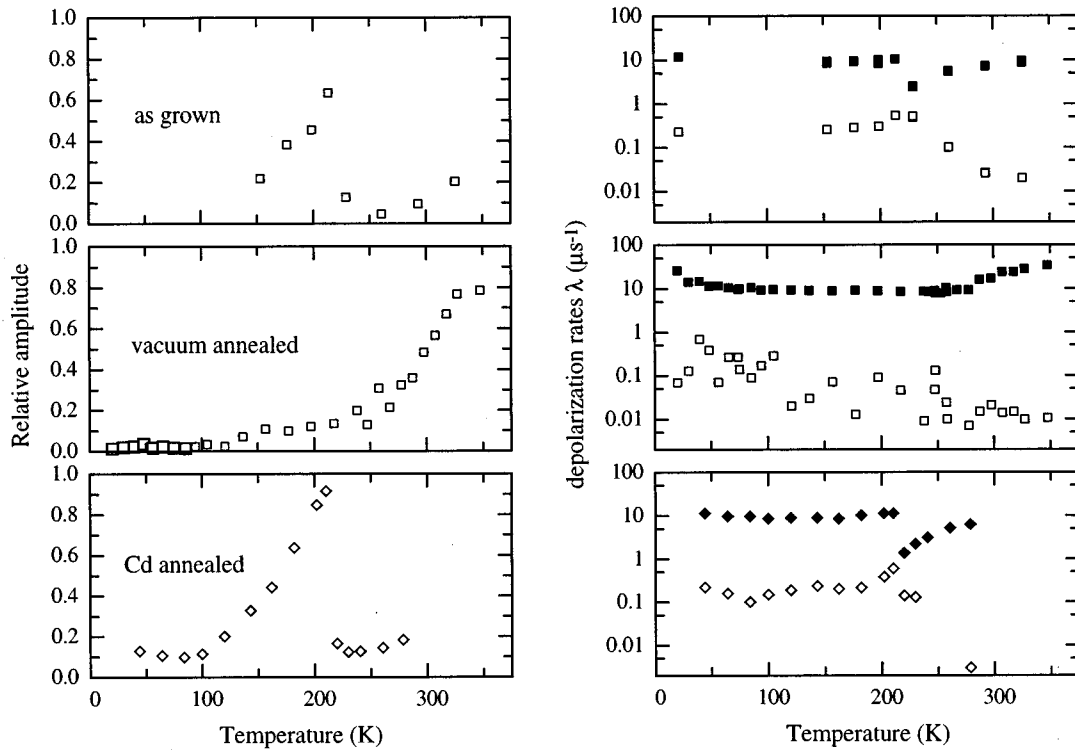


FIG. 3. Temperature dependence of the relative amplitude of the weakly relaxing component (left) and the depolarization rates of the two components (right) of the μ SR signal for $\text{Cd}_{0.6}\text{Mn}_{0.4}\text{Te}$ (as grown and after vacuum and Cd annealing).

lowed by a fairly abrupt falloff, which approximately coincides with the minimum in the depolarization rate of the strongly damped component (closed symbols).

(iv) Annealing in Cd vapor has a minor influence on the behavior of the anomalies, whereas vacuum annealing induces significant changes, in particular in the $x=0.4$ sample (Fig. 3).

The above description of the data was based on the “classical” interpretation, where the relaxation rate is the main characteristics of the two components (solid and open symbols in Figs. 3–5). There exists, however, another possible interpretation of the same experimental data, which in our

opinion is less reasonable. This interpretation is based on the three-component fit, mentioned before, which is depicted by the lines in Fig. 5. Please note that the fit is truly three-component only in certain temperature ranges where the minima of relaxation rate occurred within previous interpretation. When approaching these temperature ranges, the strongly damped component (solid lines) disappears — its amplitude decreases to zero, its relaxation rate increases slightly. The amplitude of the remaining fraction (weakly damped-dotted lines) increases gradually (as before), but then remains almost constant. The relaxation rate of this fraction starts to increase, so that at a sufficiently high damping

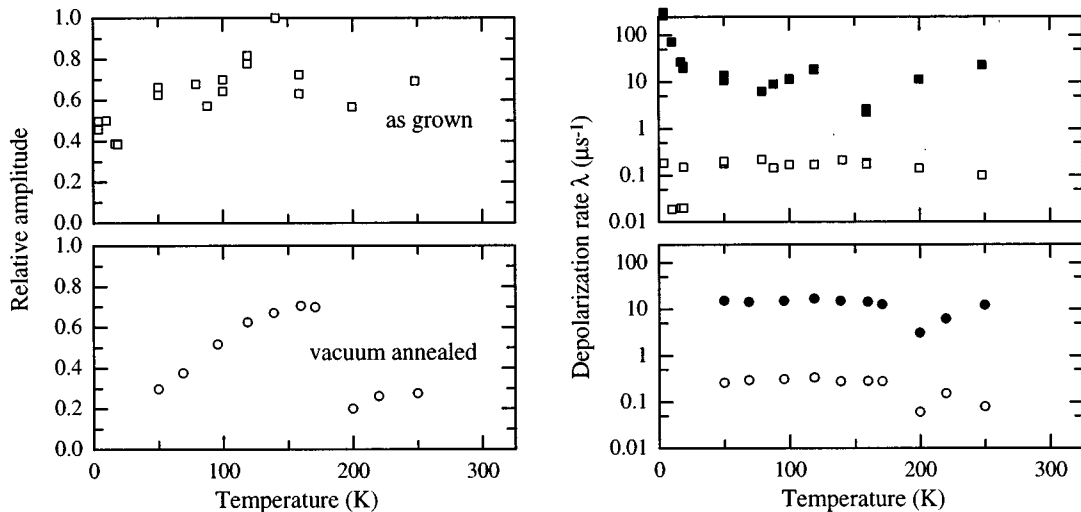


FIG. 4. Temperature dependence of the relative amplitude of the weakly relaxing component (left) and depolarization rates of the two components (right) of the μ SR signal for $\text{Cd}_{0.8}\text{Mn}_{0.3}\text{Te}$ (as grown and after vacuum annealing).

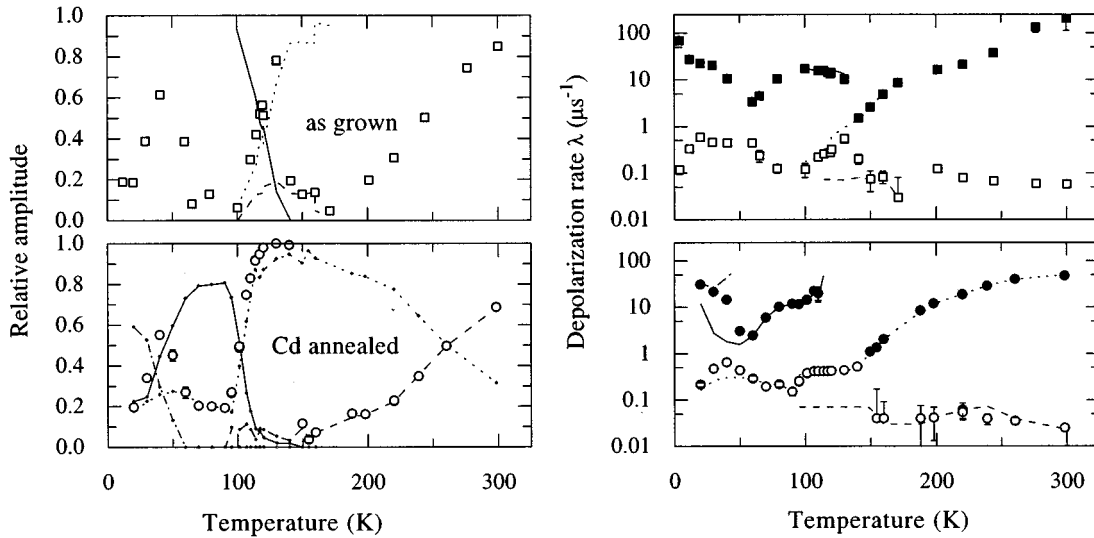


FIG. 5. Temperature dependence of the relative amplitude of the weakly relaxing component (left) and the depolarization rates of the two components (right) of the μ SR signal for $\text{Cd}_{0.8}\text{Mn}_{0.2}\text{Te}$ (as grown and after Cd annealing). The lines illustrate a three-component fit (see text).

rate the third almost undamped component (dashed lines) becomes clearly visible. Although, this interpretation gives a more continuous temperature dependence of both amplitudes and relaxation rates, it remains questionable since the three-component fits are ambiguous.

III. ANALYSIS AND DISCUSSION

The experimental data can be described consistently in the framework of a two-state model, in which one state has a high and the second one has a low depolarization rate. In this model, the minima are due to a change in the occupation of these two states. In the following, we give a characterization of the two states followed by an analysis of the two-state model.

We assume that the muon depolarization in both states is caused by the fluctuating Mn spins and that, except at very low temperatures, the motional narrowing formula can be applied. Then the muon depolarization $\lambda_{1,2}$ in the two states is given by

$$\lambda_{1,2} = \omega_{1,2}^2 \tau_c, \quad (2)$$

where $\tau_c = 1/\omega_{\text{ex}}$ is the correlation time of the spin fluctuations. Its high-temperature limit is given by the exchange interaction of the Mn spins ($\omega_{\text{ex}} \approx 10^{12} \text{ s}^{-1}$ for nearest neighbors). The experimentally observed trend that $\lambda_{1,2}$ decreases as the Mn concentration increases can be traced back to a corresponding behavior of ω_{ex} .

The interaction strengths ω_1 and ω_2 between the muon and Mn-spin systems in formula (2) contain information about the nature of these centers. Our earlier estimates of the width of dipolar field distributions for the two most probable muon sites, as well as the more precise Monte Carlo simulations done by Noakes^{12,25} for same sites, showed that the dipolar fields from the Mn magnetic moments are too small and cannot account for the experimental values of λ_1 (close to the spin-freezing temperature). This means that in the case of the strongly damped component, a hyperfine field from an unpaired electron must be involved. We therefore assign the

strongly damped component to a paramagnetic center. On the other hand, the weak relaxation of the μ SR signal in the second state is consistent with the dipolar field calculations and we therefore assign this state to a diamagnetic center.

In our opinion the most plausible explanation of the minima is that they are caused by a change of the muon from the paramagnetic to the diamagnetic state. The origin of such a change could be merely electronic or could be induced by a site change of the muon. In the following, this later possibility will be examined assuming that the site change is due to trapping.

In order to be in accordance with the data, we must assume that the free state corresponds to the fast relaxing and the trapped to the slowly relaxing component. The amplitude of the two components is determined by the trapping rate which depends on the temperature and trap concentration: the higher the trap concentration the lower the temperature at which trapping occurs and vice versa. In order to check whether trapping at Cd vacancies could cause the minima, we performed measurements with differently annealed samples.

According to the above considerations we would expect that the vacuum annealing, which increases the Cd-vacancy concentration, shifts the anomalies to lower temperatures. As can be seen clearly in Fig. 3 and also, but less pronounced, in Fig. 4, this is apparently not the case. The vacuum annealed sample (Fig. 3) shows no anomaly up to 250 K. In particular, the anomaly at 200 K, which has been seen in the as grown and in the Cd-annealed sample, does not show up. Only the high-temperature increase of weakly damped fraction is observed for this vacuum-annealed sample.

We conclude from this finding that the trapping at Cd vacancies is not the cause of the minima below room temperature. We speculate that at room temperature or somewhat above, the muon trapping at Cd vacancies may begin and that the increase of the slowly relaxing fraction in this temperature range, which is observed in most of the samples, may be an indication of such trapping.

If not vacancies, then perhaps other defects act as muon trapping centers and cause the minima. However, such an explanation is also not plausible since the shift of the minima with the Mn concentration (see Fig. 2) cannot be explained satisfactorily. If we assume that for some reason, e.g., because of a higher diffusivity or higher defect concentration, the trapping shifts to lower temperatures for a decreasing Mn concentration x , then the fact that also the detrapping shifts in the same way would still be a mere accident and not very plausible.

Therefore, it seems that the state change of the muon at the anomalies is of electronic origin, although trapping cannot be excluded completely. At the low-temperature side of the anomalies, the fraction of the slowly relaxing (diamagnetic) component gradually increases at the expense of the fast relaxing (paramagnetic) component. The fact that the change is not abrupt might be explained by slight local inhomogeneities, which cause the transition to occur at different temperatures. At the high-temperature side of the anomalies a fluctuation between the two states can best describe the data, which show an intermediate depolarization rate.

The remaining question is the nature of the paramagnetic center. Two kinds of paramagnetic muonium centers were well identified in classic semiconductors. These are, the normal muonium Mu_T — located at a tetrahedral interstitial and the anomalous muonium Mu_{BC} — anisotropic, located at the bond center.²² In both cases, however, the observed TF- μSR signal contains multiple oscillation frequencies, which depend in rather complicated manner on the values of electron-muon hyperfine coupling, ω_0 and on the Zeeman frequencies, ω_e and ω_μ , for electron and muon spins, respectively.

The only case described in the literature, when the muonium signal resembles those of diamagnetic μ^+ , occurs, when the electron depolarization rate ν is much higher than ω_0 .^{21,23} Additionally, the electron has to be depolarized independently from the muon spin, e.g., by the rapid spin or charge exchange. The dominant μSR frequency of Mu_T , ω_{12} , would then have almost the same value as for the bare μ^+ . The signal would be exponentially damped with a relaxation rate, λ_{12} , roughly proportional to ω_0^2/ν and would show a small negative frequency shift of the order of $(\omega_e\omega_0^2)/\nu^2$. In fact, the negative frequency shift was observed for the strongly damped component¹⁰.

The independent depolarization of the electron and muon spins in the Mu center should indeed be expected in the semi-insulating $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ DMS crystals. The strength of the exchange interaction between the spins of the electron from conduction band and the localized Mn ions (s - d exchange) might be characterized by the value of exchange constant,² $N_0\alpha = 0.22$ eV. Thus, the influence of the fluctuating spins of neighboring Mn ions on the Mu center will be dominated by the exchange fields of the amplitude δ_{ex} . The upper limit of δ_{ex} is on the order of 10^{15} s⁻¹, but anyhow δ_{ex} might be easily much higher than ω_0 . Unlike the dipolar fields, these exchange fields act solely on the electron spin and scale differently with the distance. Both dipolar and exchange fields should fluctuate with the same characteristic time τ_c given by Mn-spin fluctuations, but there is no reason

to assume any phase coherence between them. So the fluctuating exchange fields should decouple electron and muon spins.

For sufficiently rapid fluctuations ($\tau_c \gg \delta_{\text{ex}}$) it is expected²¹ that $\nu = \delta_{\text{ex}}^2 \tau_c$, but then we should observe stronger damping, when τ_c decreases with increasing temperature, which is not the case experimentally. It is much more probable that in $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ $\delta_{\text{ex}} \gg \tau_c$, $\nu \propto 1/\tau_c$, and finally $\lambda_{12} \propto \tau_c$, however, such a case was not explicitly considered in theoretical works on muonium relaxation.

The other argument for the muonium hypothesis would come from the fact that the fast relaxing component was observed only in the $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ samples with relatively high Mn concentration $x \geq 0.10$, when the samples were semi-insulating. Also for the semi-metallic $\text{Cd}_{0.7}\text{Mn}_{0.3}\text{Se}$ (n -type) and $\text{Hg}_{0.65}\text{Mn}_{0.35}\text{Te}$ (p -type) samples no fast relaxing signal was observed even in the vicinity of the spin-freezing temperature. No muonium frequencies (other than the μ^+) were found in our DMS samples.

One might further speculate that the changes of the relative amplitudes of different components is the sign of the Mu_T to Mu_{BC} and Mu_{BC} to Mu_{BC}^+ (or μ^+) transitions. In order to account for the observed dependence of the conversion temperature on Mn concentration, we have to assume that the ionization energies of different Mu centers change when the energy gap of the $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ opens with increasing x . Although, this interpretation looks promising from the point of view of the three-component picture, we have no strong arguments supporting such a hypothesis.

CONCLUSION

The present data indicate that the muon in $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ can exist in two different states: the first state has a high depolarization rate and is assigned to a paramagnetic configuration, the second state has a low depolarization rate and is assigned to a diamagnetic configuration. The minima in the depolarization rate are attributed to a change from the paramagnetic to the diamagnetic state.

The experiments performed on the annealed $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ samples clearly show that the earlier hypothesis of the muon trapping at the Cd vacancies cannot be the cause for the minima below the room temperature. We argue that trapping at other defects is also unlikely so that a mere electronic configuration change is the most probable origin of the minima.

The exact microscopic picture of the paramagnetic and diamagnetic muon states in $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ mixed crystals and the reason for the state change remains unexplained. The experiments described in this paper provide, however, important data, which should facilitate the planning of the future experiments on these DMS crystals.

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