

## Experimental evidence for spatial inhomogeneous spin freezing in $\text{CuMn}$

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Although the time correlation function of the impurity spins in spin glasses around the ordering temperature  $T_f$  has been well determined by various experiments, microscopic insight into the development of spatial correlations during freezing is still lacking. In order to obtain direct information on this aspect we undertook a high-precision study of the muon-spin-relaxation function for the  $\text{CuMn}$  system just above  $T_f$  by means of the zero-field muon-spin-relaxation ( $\mu\text{SR}$ ) technique. The data were analyzed on the basis of either a spatially homogeneous or inhomogeneous spin-freezing model. While the former failed to fit the data the latter provided an excellent description. This is a strong indication for the coexistence of regions of fast spin fluctuations with regions of more strongly correlated spins at about  $1.5T_f$ . The zero-field  $\mu\text{SR}$  technique is shown to be advantageous as a method for investigating spatial inhomogeneity in detail.

### I. INTRODUCTION

Since the discovery of the susceptibility cusp in spin glasses at the freezing temperature  $T_f$  by Cannella and Mydosh,<sup>1</sup> there has been much theoretical and experimental work done trying to understand the main features of the freezing process. Despite great progress made in understanding details, a generally accepted view is still missing.

There is agreement that the frozen state of a spin glass (SG) with competing interactions is characterized by a majority of spins which are kept more or less fixed in random orientations while the rest, called "frustrated" spins, remain nearly free. The central question concerning the transition then is: How does this state develop during cooling down through  $T_f$ , or, more precisely, what are the correlations of the impurity spins in *time and space* in the temperature region around  $T_f$ ?

After sketching some of the answers currently given by theory and experiment we shall present new results on the SG system  $\text{CuMn}$  provided by the muon-spin-relaxation ( $\mu\text{SR}$ ) method. They show that the muon essentially probes two different environments which we assign to areas of correlated and uncorrelated spins.

### II. THEORY

The theory of spin glasses has recently been comprehensively reviewed by Fischer.<sup>2</sup> After the strong start by Edwards and Anderson<sup>3</sup> who introduced the order parameter  $q_{\text{EA}}$  describing local long-time correlations, theoretical progress became entangled in mathematical difficulties even in the case of static mean-field theories applied to infinite-range models.<sup>4</sup> Morgenstern and Binder<sup>5,6</sup> argue against a phase transition at  $T_f$ , showing that in the short-range case for two and three dimensions,  $q_{\text{EA}}$  as

well as any other order parameter<sup>7</sup> vanishes for  $T > 0$ . Consequently they explain the transition at  $T_f$  as a dynamic process.

A spin glass can qualitatively be interpreted in terms of a generalized Edwards-Anderson model of magnetic clusters with random interactions,<sup>8</sup> the cluster approach explaining the sharp cusp in  $\chi(T)$  as well as the broad maximum in the magnetic part of the specific heat.<sup>9</sup> Recently Hertz<sup>10</sup> justified cluster models by introducing the concept of condensing, localized modes which may be viewed as spin clusters.

Because of the simpler mathematical treatment, spin glasses are usually treated as a problem of random bonds<sup>3</sup> between the magnetic atoms, rather than one of random sites. There are, however, a few approaches of the latter kind<sup>11-13</sup> which have in common the basic idea introduced by Smith<sup>14</sup> that the spin-glass transition can be explained as percolation of clusters which increase with decreasing temperature. At present, none of the theories permits a satisfactory description of the correlations between the spins in time and space across the spin-glass transition.

### III. NEUTRON SPIN ECHO, SUSCEPTIBILITY AND MÖSSBAUER EFFECT EXPERIMENTS

From the experimental point of view,<sup>15</sup> spin glasses do not exhibit "simple" magnetic behavior even far above  $T_f$ . The static susceptibility  $\chi_{\text{dc}}$ , measured in higher external fields for systems such as  $\text{CuMn}$  and  $\text{AuMn}$ ,<sup>16</sup> begins to deviate from a Curie-Weiss law even at  $T = 5T_f$ . Taking into account the atomic short-range order found by neutron scattering,<sup>17,18</sup> the authors explain their results by short-range ferromagnetic correlations of Mn spins preceding the freezing at  $T_f$ .

Another interesting aspect emerging from magnetiza-

tion measurements concerns the first nonlinear terms in the field dependence of the magnetization.<sup>19–21</sup> The anomaly of the corresponding terms in the susceptibility at  $T_f$  and their scaling behavior were taken as an argument in favor of phase-transition behavior. In the following, however, we shall restrict ourselves to the results of *zero-field* methods which do not suffer from any disturbance of the system by an applied external field (as apparent for instance in the field dependence of the  $\chi$  cusp<sup>22</sup>). The most powerful methods have proved to be neutron spin echo (NSE), complex ac susceptibility ( $\chi_{ac}$ ), Mössbauer effect (ME) and muon-spin-relaxation.

The NSE method<sup>23</sup> directly measures the time-dependent spin-correlation function  $\xi(\mathbf{q}, t)$  for a time scale  $10^{-12}$ – $10^{-8}$  s. The results of Mezei and Murani<sup>24,25</sup> for CuMn show a distinct change of  $\xi(\mathbf{q}, t)$  from a simple exponential shape (with a correlation time  $\tau_c$  around  $10^{-11}$  s) to a logarithmic time dependence (covering  $\tau_c$  values between  $10^{-11}$  and  $10^{-8}$  s) within the temperature interval  $1.3T_f > T > 0.7T_f$ . This corresponds to the appearance of a broad distribution of correlation times.  $\xi(\mathbf{q}, t)$  turns out to be independent of the wave number  $\mathbf{q}$  in the range used ( $0.045 \text{ \AA}^{-1} < |\mathbf{q}| < 0.36 \text{ \AA}^{-1}$ ), i.e., spatial correlations in the corresponding range (about 9–70 Å) are not detected by the technique.<sup>26</sup>

Precise measurements of the complex ac susceptibility  $\chi_{ac}$  (Refs. 22, 27, and 28) yield a logarithmic frequency dependence of the real part below  $T_f$ . From the mutual relation of the real and imaginary parts the authors conclude the existence of a wide spectral distribution of relaxation times below  $T_f$ . The cusp is tentatively interpreted to arise from the evolution of viscous, possibly percolating clusters at  $T_f$ .<sup>27</sup>

Recent ME studies on AuFe spin glasses<sup>29</sup> show a rather smeared out transition over the temperature range of about  $1.2T_f > T > 0.9T_f$ . The authors found an unsplit paramagnetic component present still below  $T_f$ . They claim that their results cannot be interpreted in terms of critical fluctuations and use instead a distribution of transition temperatures to fit their data.

Interpreting the latter as a consequence of thermally activated relaxation processes governed by a distribution of energy barriers, the ME results are compatible with the existence of distributed correlation times for the local hyperfine field probed by the <sup>57</sup>Fe nucleus. Because of its inherently narrow time window, ME is less appropriate for studying the detailed shape of the correlation function.

The importance of dynamical effects for the spin-glass transition is apparent. The methods cited above either directly measure a very fast change of the impurity spin-correlation function near  $T_f$  or the assumption of this change in  $\xi(t)$  at least permits an interpretation of the results.

The next section will show how, using the  $\mu$ SR method, one can investigate dynamical aspects of the transition and provide additional information on the development of spatial correlations during freezing.

#### IV. $\mu$ SR EXPERIMENTS

The muon-spin-relaxation ( $\mu$ SR) technique measures the relaxation function  $G(t)$  of the muon spin which

probes the local magnetic field at interstitial lattice sites in crystalline samples.<sup>30–33</sup> In random systems such as spin glasses,  $G(t)$  depends on the probability distribution  $p(H)$  of the local fields and, very sensitively, on their dynamics characterized by the correlation function  $\xi(t)$ . Once  $p(H)$  and  $\xi(t)$  have been specified within a given model,  $G(t)$  can be calculated, at least numerically.<sup>34,35</sup>

Muon-spin relaxation in zero external field (ZF- $\mu$ SR) is particularly appropriate to distinguish between static and dynamic effects in spin glasses.<sup>36</sup> Assuming a single correlation time  $\tau_c$  [i.e.,  $\xi(t) = \exp(-t/\tau_c)$ ], the first ZF- $\mu$ SR measurements on AuFe, CuMn, and AgMn spin glasses<sup>37,38</sup> reveal a rapid slowing down of spin fluctuations from  $\tau_c = 10^{-10}$  s to  $\tau_c = 10^{-7}$  s within the temperature interval  $1.4T_f > T > 0.7T_f$ . More elaborate  $\mu$ SR studies on the systems CuMn, AuFe, and AgMn either support the existence of nonexponential correlation functions<sup>39,40</sup> or directly derive them under certain assumptions.<sup>41,42</sup>

Following Edwards and Anderson, Uemura<sup>43</sup> developed a model where below  $T_f$  the impurity moments fluctuate rapidly around a local preferred direction. These directions are randomly distributed but the expectation value of the spin component along this direction is uniform. In short, this model is denoted by “homogeneous static polarization.” The local field originating from the impurity spins is simulated by a static random part (mean amplitude  $a_s$ ) and a superimposed dynamic random part (mean amplitude  $a_d$ ). The correlation function for the local fields then exhibits a time-independent part  $q \propto a_s^2$ , i.e., an Edwards-Anderson-type order parameter.

Uemura *et al.*<sup>40</sup> measured  $a_s$  as a function of temperature for the systems AuFe and CuMn and found nonzero values only below  $T_f$ . If the temperature is increased the static amplitude  $a_s$  abruptly drops to zero near  $T_f$ . Above  $T_f$  all local fields completely reorient with a fairly fast reorientation frequency of the order of  $10^{10}$  Hz. In this limit, the relaxation function  $G_{SG}(t)$ , caused by the SG impurity spins, obeys an “exponential root”-law<sup>43,44</sup>

$$G_{SG}(t) = \exp(-\sqrt{\lambda t}) \quad (\text{homogeneous model}) \quad (1)$$

In contrast to these results, we were led to interpret our previous data for the system CuMn,<sup>39</sup> assuming that different regions of the sample show quite different dynamics of the local field. In other words the distribution of correlation times is assumed to be inhomogeneous in space.

This can cause distinct changes in the shape of  $G_{SG}(t)$ , because the dynamical effect depends on the characteristic correlation time  $\tau_c$  compared to the time window of the  $\mu$ SR method (about  $10^{-10}$ – $10^{-6}$  s). In regions where the local field fluctuates very rapidly with correlation times  $\tau_c < 10^{-10}$  s, the average local field experienced by the muon within its lifetime is zero and the spin is not relaxed, i.e.,  $G_{SG}(t) = 1$  within the observation time of about 7  $\mu$ s.

In regions with slower fluctuations  $G_{SG}(t)$  is not constant, but shows an observable decay [for example according to Eq. (1)]. Consequently the relaxation function consists of two parts:

$$G_{SG}^{\text{tot}} = A + (1 - A)G_{SG}(t) \quad (\text{inhomogeneous model}), \quad (2)$$

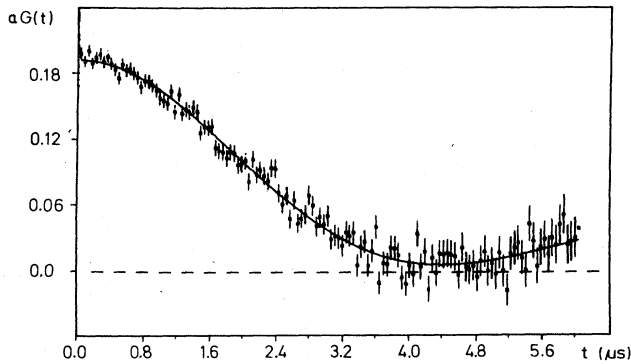


FIG. 1. Measured zero-field muon-spin relaxation in the spin glass CuMn (0.87 at.%) ( $T_f=9.5$  K) at  $T=39$  K  $=3.6T_f$ .  $G(t)$  follows the Kubo-Toyabe function [Eq. (3)] with a linewidth  $\Delta=0.38 \mu\text{s}^{-1}$  found in pure Cu (see text). The factor  $a$  denotes the experimentally observed asymmetry of the muon decay.

where  $A$  corresponds to the volume fraction of the “paramagnetic” regions in the sample.  $G_{\text{SG}}(t)$  accounts for the relaxation in all other parts, the “spin-glass-like” regions, where  $\tau_c > 10^{-9}$  s. Here we emphasize that the terms paramagnetic and spin-glass-like are defined by the time window of the method. Usually it is difficult to observe a time-independent part of the relaxation function, i.e., the first term in Eq. (2). Here the system CuMn has an advantage compared to others like AgMn and AuFe. At temperatures far above  $T_f$  where the rapidly fluctuating Mn moments have negligible effect, the muon spin is still relaxed by the Cu nuclear dipoles according to the Kubo-Toyabe formula:<sup>45</sup>

$$G_{\text{KT}}(t) = \frac{1}{3} + \frac{2}{3}(1 - \Delta^2 t^2) \exp(-\Delta^2 t^2/2). \quad (3)$$

Figure 1 shows an example for CuMn (0.87 at.%) at  $T=3.6T_f$ .  $G_{\text{KT}}(t)$  passes a minimum at  $\sqrt{3}/\Delta = 4.4 \pm 0.1 \mu\text{s}$  and recovers toward  $\frac{1}{3}$  for longer times. The Gaussian linewidth  $\Delta$  agrees well with the measured value of  $\Delta=0.38 \pm 0.01 \mu\text{s}^{-1}$  for pure Cu.<sup>46,47</sup> To account for this additional mechanism we multiply every SG relaxation function  $G_{\text{SG}}(t)$ , which describes the Mn moments in the limit of fast fluctuations ( $T > T_f$ ), by  $G_{\text{KT}}(t)$  for pure Cu to obtain the complete relaxation function  $G(t)$  for CuMn:<sup>48</sup>

$$G(t) = \exp(-\sqrt{\lambda t}) G_{\text{KT}}(t) \quad (\text{homogeneous model}), \quad (1a)$$

$$G(t) = [A + (1-A)\exp(-\sqrt{\lambda t})] G_{\text{KT}}(t) \quad (\text{inhomogeneous model}). \quad (2a)$$

In the present paper we report on new measurements which were designed to test unambiguously the validity of either the homogeneous model [Eq. (1a)] or the inhomogeneous model [Eq. (2a)].

In order to distinguish the different long-time behavior of Eqs. (1a) and (2a) the  $\mu\text{SR}$  spectrum must be extended to longer times which requires at the same time much higher counting statistics. Consequently, the time range

was extended to  $7.5 \mu\text{s}$  and the statistics were increased from our previous  $(4-6) \times 10^6$  counts per spectrum to  $5 \times 10^7$  counts per spectrum.

Spatial inhomogeneity, if existent, should be most pronounced in the vicinity of  $T_f$ . Here we expect both contributions to  $G(t)$  in Eq. (2a) to be comparable and the paramagnetic regions should leave their unambiguous fingerprints via the Cu signal.

The experiments were performed with a conventional  $\mu\text{SR}$  spectrometer at the Swiss Institute for Nuclear Research (SIN) in Villigen, Switzerland. The samples used were CuMn single crystals slowly cooled from the melting point with Mn concentrations 0.54 and 0.87 at. % (orientation: [100] axis parallel to the initial muon spin). A careful x-ray microanalysis showed that no concentration inhomogeneity was present in the samples down to a scale of about  $1 \mu\text{m}$ . In fact, no differences were found between slowly cooled and quenched samples in contrast to results in a former work by Uemura *et al.*<sup>49</sup> where fluctuations of the Mn concentration up to 30% and extending over  $40-50 \mu\text{m}$  were reported for slowly cooled samples.

Of course differences between the slowly cooled and quenched state still can exist in our samples on scales below  $1 \mu\text{m}$ . However, “simple” quenching of bulk samples from temperatures near  $T_m$  does not produce

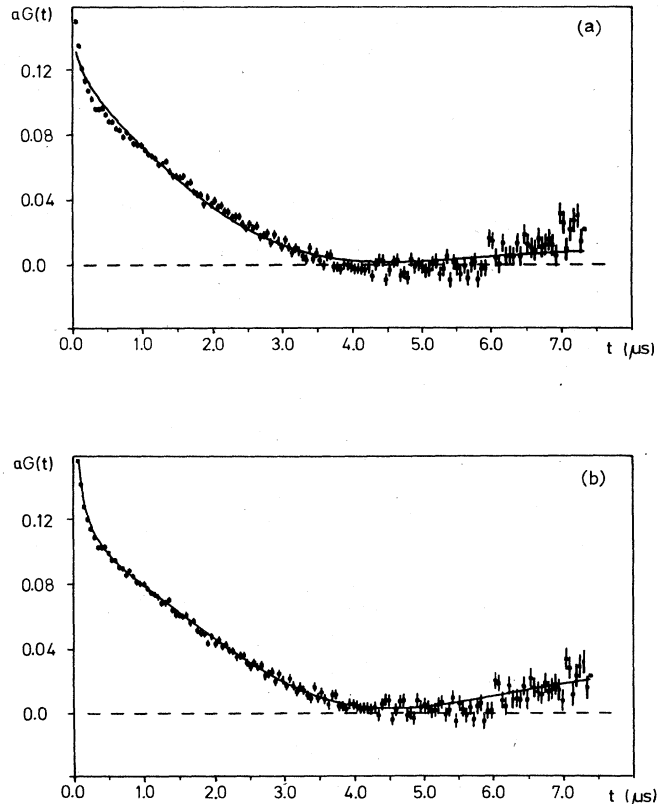


FIG. 2. Experimental muon-spin relaxation function in zero field for CuMn (0.54 at.%) ( $T_f=6.5$  K) at  $T=7.6$  K  $=1.17T_f$ . The curves represent fits of the homogeneous model function [Eq. (1a); Fig. 2(a)] and the inhomogeneous model function [Eq. (2a); Fig. 2(b)]. Normalized  $\chi^2$  are  $\chi^2(1a)=3.26$ ;  $\chi^2(2a)=1.04$ .

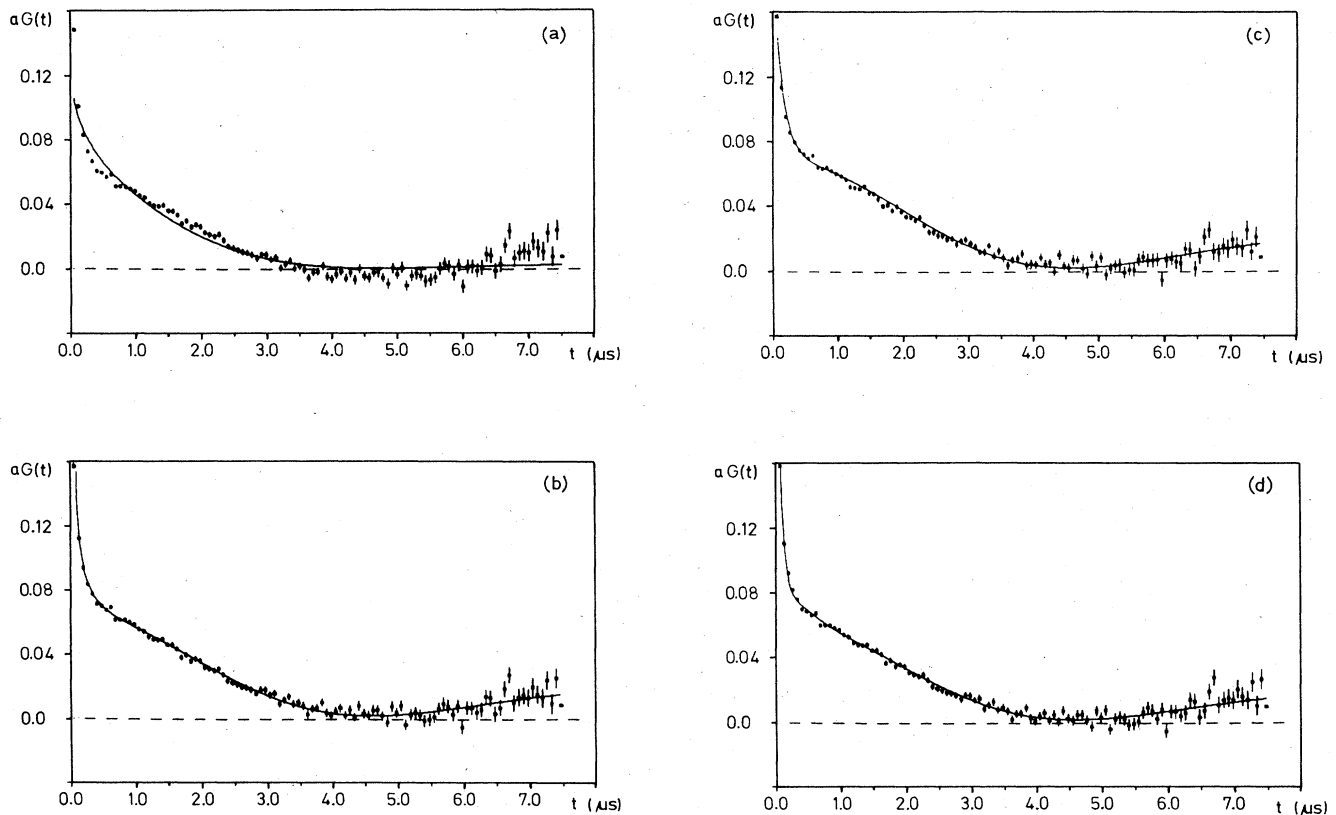


FIG. 3. Measured muon-spin relaxation in zero field for CuMn (0.54 at. %) at  $T = 6.8 \text{ K} = 1.05 T_f$ . Solid curves (a)–(d) represent best fits of the relaxation functions Eqs. (1a), (2a), (4), and (5), respectively. Three different inhomogeneous models (2a), (4), and (5) yield good fits, the homogeneous model Eq. (1a) does not. Normalized  $\chi^2$  are  $\chi^2$  (1a)=4.72;  $\chi^2$  (2a)=1.11;  $\chi^2$  (4)=1.49;  $\chi^2$  (5)=1.28.

“better” spin glasses in the sense of a true random distribution of the impurity atoms for the following reasoning.

(i) For systems known to show short-range ordering or clustering, quenching from *high* temperatures does not lead to the least ordered state,<sup>50</sup> not even if the samples are thin.

(ii) Quenching of thick samples, i.e., with thickness of the order of 1 mm, produces appreciable thermal stresses and gradients in the density of vacancies across the sample cross section. These inevitable spatial inhomogeneities might even be accompanied by inhomogeneities in the degree of short-range order or clustering due to the spread in cooling rates inside the sample during the quench. Thus, samples quenched in this manner are likely to have no well defined spatial structure.

The use of slowly cooled single crystals avoids the difficulties sketched under (ii) and in addition excludes any inhomogeneities possibly arising from grain boundaries. But in any case, existing differences between slowly cooled and quenched samples do not affect the general features of the measured zero-field muon-spin relaxation function discussed here.<sup>51</sup>

Figure 2 shows data points for a CuMn (0.54 at. %) sample (slowly cooled,  $T_f = 6.5 \text{ K}$ ) at  $T = 7.6 \text{ K} = 1.17 T_f$ , fitted by the functions (1a) [Fig. 2(a)] and (2a) [Fig. 2(b)]. All subsequent fits were evaluated under ex-

actly the same conditions: The instrumental dead time was approximately 30 ns, so the time range was set to 0.05–7.5  $\mu\text{s}$ . The Cu linewidth  $\Delta$  was held fixed to the value of  $\Delta = 0.38 \mu\text{s}^{-1}$  cited above. The typical feature of the relaxation is a quick initial decay for  $0.05 < t < 0.3 \mu\text{s}$  followed by a much slower one, a minimum around 4.5  $\mu\text{s}$  and a final recovery up to  $t = 7.5 \mu\text{s}$ .

The best least-squares fit of the homogeneous model function Eq. (1a) distinctly fails: It is unable to reproduce the observed fast decay in the beginning and the long time recovery simultaneously [Fig. 2(a)]. The two-component function Eq. (2a) fits the same data very well [Fig. 2(b)].

To test whether this agreement depends on the particular choice of  $G_{\text{SG}}(t) = \exp(-\sqrt{\lambda}t)$  we also tried to fit the following relaxation functions to the data (valid again for the temperature range  $T > T_f$ ):

$$G(t) = [A + (1-A)\exp(-\Lambda t)]G_{\text{KT}}(t), \quad (4)$$

$$G(t) = [A + (1-A)G_{\text{SG}}^d(t)]G_{\text{KT}}(t). \quad (5)$$

The simple exponential function in Eq. (4) avoids fit problems due to the infinite slope in Eq. (1) for  $t \rightarrow 0$ .  $G_{\text{SG}}^d(t)$  denotes a model function based on a distribution of correlation times inside the spin-glass-like phase.<sup>39,52</sup>

For comparison Fig. 3 shows the best fits of all four functions (1a), (2a), (4), and (5) to a high statistics run tak-

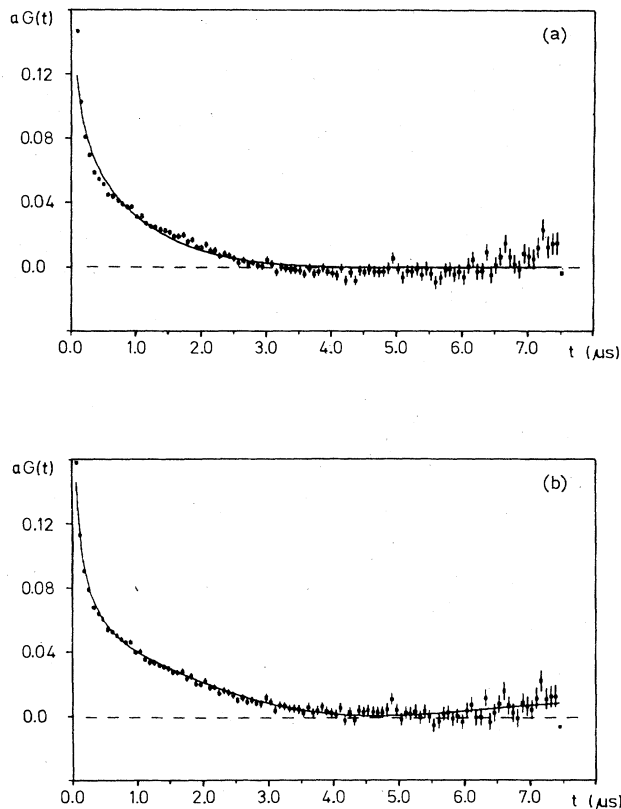


FIG. 4. Measured muon-spin relaxation in zero field for CuMn (0.87 at. %) at  $T=10$  K  $=1.06T_f$ . Solid lines represent best fits to Eq. (1a) [homogeneous model, Fig. 4(a)] and Eq. (2a) [inhomogeneous model, Fig. 4(b)].  $\chi^2$  (1a) = 2.65;  $\chi^2$  (2a) = 1.20.

en at  $T=6.8$  K  $=1.05T_f$  on the CuMn (0.54 at. %) sample. All three functions with the sum structure of Eq. (2a) yield good fits to the data whereas Eq. (1a) again fails. This indicates that the assumption of spatial inhomogeneity is independent of the particular model of the spin-glass-like phase within the present accuracy.

Finally, Fig. 4 shows spectra from a CuMn sample with a somewhat higher Mn concentration of 0.87 at. % taken at  $T=10.0$  K ( $T_f=9.5$  K). The results are very similar to those for CuMn (0.54 at. %) in Fig. 2. Again

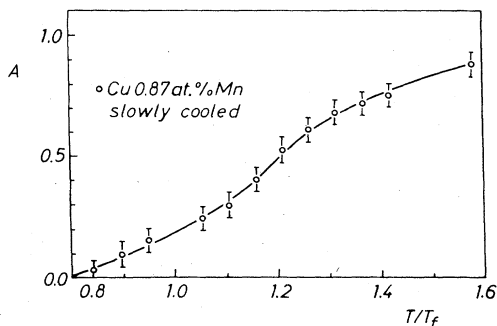


FIG. 5. Volume fraction  $A(T)$  of the paramagnetic phase vs reduced temperature  $T/T_f$  for the slowly cooled CuMn (0.84 at. %) sample.

Eq. (1a) does not give a satisfactory fit, whereas the other functions do.

As a result of the analysis Fig. 5 shows the volume fraction  $A(T)$  of the paramagnetic phase as a function of temperature for the slowly cooled CuMn (0.84 at. %) sample. The formation of regions with correlated spins already begins at  $T=1.6T_f$  as  $A(T)$  deviates from unity.  $A(T)$  shows a smooth decrease to zero within the temperature interval  $1.6T_f > T > 0.8T_f$  without any observable discontinuity at  $T_f$ . These results disagree with Monte Carlo calculations by Kinzel<sup>53</sup> for a two-dimensional Ising spin glass, where frozen spins appear in small clusters only just below  $T_f$ .

## V. DISCUSSION AND CONCLUSIONS

Regarding the results of all four zero-field methods, NSE,  $\chi_{ac}$ , ME, and  $\mu\text{SR}$ , the importance of dynamical effects for the spin-glass freezing process is apparent. None of these methods reveals critical fluctuations, i.e., there is no usual type of phase transition present in the SG systems investigated. Rather the common feature of these systems is a broad spectral distribution of correlation times for the impurity spins in the temperature region of the transition.

With regard to spatial correlations our analysis for the CuMn system clearly shows that the transition in the vicinity of  $T_f$  cannot be explained by the onset of a homogeneous static polarization of the impurity spins. Instead we find: The distribution of correlation times for the local fields in CuMn probed by the muon is inhomogeneous in space. This result does not depend on the choice of particular fitting functions, i.e., the analysis does not depend on a specific model for the spin-glass-like phase. The idea that this spatial inhomogeneity found in the distribution of correlation times may arise from separate spin clusters fluctuating with different relaxation times was already assumed in the interpretation of other experimental results (e.g., in Refs. 27 and 28) but there was no direct experimental evidence.

The development of the correlated regions extends over a finite temperature interval starting markedly above and ending slightly below  $T_f$ . Partly this is a consequence of the dynamic nature of the transition. The definition of the transition region depends on the time window of the particular experimental method and consequently different methods find different transition intervals (see for comparison, e.g., Ref. 29). From this point of view the "freezing temperature"  $T_f$  determined from the ac susceptibility is not of general physical importance. Regarding a spectral distribution of correlation times as a main feature of the metallic SG systems the ac susceptibility need not necessarily show a pronounced sharp cusp.<sup>27</sup>

From the  $\chi_{dc}$  and neutron scattering measurements it is known that far above  $T_f$  a few spins form small correlated areas with diameters in the range of a few lattice constants. Because the  $\mu\text{SR}$  method uses a localized probe, it is in general not possible to deduce from the data the spa-

tial extension of the correlated areas (i.e., the correlation length). We believe that the decrease of the paramagnetic fraction  $A(T)$  with temperature reflects a growing of clusters which fill nearly the whole volume of the sample below  $T_f$ . Thus our results, in comparison with those of the other zero-field methods, strongly support the model of a spatial inhomogeneous percolation-like transition in the system  $\text{CuMn}$ . Therefore the development of theories which more explicitly take account of spatial correlations between spins and elucidate the formation and interaction of magnetic clusters deserves high interest. Corresponding trends in present theories were mentioned in Sec. II. Up to now  $\mu\text{SR}$  seems to be the only method known to give direct experimental evidence for the reported inhomogeneity.

*Note added.* In a very recent paper, published in *Phys. Rev. B* 31, 546 (1985), Uemura *et al.* used exactly the relaxation function Eq. (1a) of the homogeneous freezing model [i.e., Eq. (25) of that paper] for analyzing data on  $\text{CuMn}$  and stated that "... the data above  $T_g$ ... actually followed this function,..." In the present publication, however, clear evidence is given for the failure of this model in fitting our data.

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