Mössbauer study of the superspin glass transition in nanogranular $Al_{49}Fe_{30}Cu_{21}$

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The spin-glass-like behavior observed in mechanically alloyed $Al_{49}Fe_{30}Cu_{21}$ is attributed to the collective freezing of ferromagnetic nanoclusters, i.e., the appearance of a superspin glass phase. The presence of the clusters and their estimated size (~ 1 nm) were inferred from magnetization measurements. The transition critical exponent $z\nu = 13.8 \pm 0.4$, extracted from ac-susceptibility data, is similar to those obtained in other superspin glasses. Mössbauer spectroscopy offered more direct evidence: the component related to the clusters undergoes a transition from a high-temperature quadrupolar doublet to a distribution of sextets, whose mean hyperfine splitting grows with decreasing temperature in a similar fashion to spin glasses. The relation between these magnetic dynamics and the sample structure is addressed.

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I. INTRODUCTION

The magnetic properties of granular alloys and heterogeneous nanostructures built by ferromagnetic and nonmagnetic components have attracted much attention due both to the fundamental interest in their rich phenomenology and to their potential applications—for instance, in magnetoresistive devices and magnetic recording[1]. In particular, the role of interactions between single-domain particles has received a growing interest from the two perspectives. From the application viewpoint, the effects of intergranular interaction on the magnetoresistance, which before was treated assuming simple superparamagnetism, have been taken into account.[2] But more relevant to this paper are the attempts to describe the dynamics of random magnetic particle systems with a strong (compared to the anisotropy barrier) interparticle interaction. Most of such studies have been concerned with dipolar interactions, using the model system of frozen ferrofluids, where collective freezing, as opposed to superparamagnetic blocking, has been proved for sufficiently high particle concentrations.[3,4] The spin-glass-like (SGL) properties of such particle phases can be understood from the random position of the particles and the competition between parallel and antiparallel alignment introduced by the dipolar interaction, thereby receiving the name *superspin glasses*. In addition, in metallic granular systems, an indirect RKKY interaction between the clusters has been theoretically shown to be able to provide the necessary frustration in a similar fashion as it does in canonical spin glasses,[5] being dominant over dipolar interactions for sufficiently small cluster size.[6,7] Some of these granular systems, including discontinuous multilayers,[8] melt-spun Co-Cu,[9] or sputtered $Fe_{20}Ag_{80}$,[10] have been lately found to exhibit superspin glass phases at low temperatures, although the RKKY interaction has never been held responsible for their SGL behavior. Although designed and mostly used for other purposes,[11] mechanical alloying (MA) has proved to be an efficient technique for producing bulk spin-glass-like materials.[12–15] However, their analysis is particularly difficult due to the different possible sources for nonequilibrium magnetic behavior, including true amorphous spin glasses,[12] spin-boundary disorder,[16] and the commented superspin freezing.[13] For instance, the SGL behavior of an essentially amorphous alloy—namely, mechanically alloyed $Fe_{30}Al_{49}Cu_{21}$—was recently attributed by De Toro *et al.* to the presence of interacting fine ferromagnetic particles, or clusters, embedded in the majority amorphous paramagnetic matrix,[17] but in principle both the matrix and the suggested ''particle phase'' can be argued to be able to produce the observed behavior.

In this context, the presence in granular systems of two phases containing different Fe concentrations calls for the use of Mössbauer spectroscopy. This technique, besides detecting the freezing process, allows direct determination of the phase actually being frozen and its proportion in the sample, thus clarifying the interpretation of the SGL behavior in such materials. The sample choice in this work—the rather complex mechanically alloyed $Fe_{30}Al_{49}Cu_{21}$—is intended to exemplify these capabilities, while presenting, to our best knowledge, the first Mössbauer characterization of the superspin glass freezing in a granular alloy. Additionally, although mechanical alloying has been extensively investigated at room temperature (RT) by Mössbauer spectroscopy,[18] this work contributes to relieve the lack of complementary measurements at low temperatures.

II. EXPERIMENT

The sample was obtained by mechanically alloying Fe, Cu, and Al powders for 180 h using a high-energy planetary ball mill. Details of the milling conditions can be found in Ref. 17, where it was also shown by x-ray diffraction and transmission electron microscopy that the final product is mostly an amorphous solid solution. The precise structure of the sample will be discussed later considering the information presented here. Mössbauer spectra were recorded at temperatures ranging from 4.2 K to RT. The sources were ^{57}Co in a rhodium matrix with an activity of ~ 10 mCi. Transmission mode Mössbauer spectrometers were used in three con-

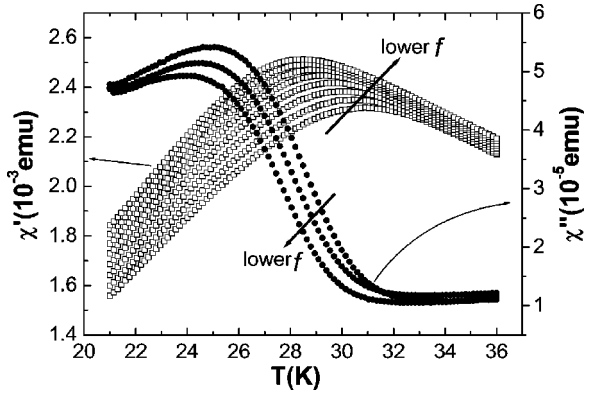

FIG. 1. Temperature dependence of the real (χ') and imaginary (χ'') components of the ac susceptibility measured at different frequencies $f=0.1$, 0.37, 1.39, 5.16, 19.24, 71.62, 267.09, 997.34 Hz. For clarity, only the first three frequencies are shown for χ''.

figurations: at room temperature, with a 4.2 K helium bath cryostat, and a liquid-helium flow cryostat for variable temperature measurements. The spectra were fitted using two separate packages: FFITA employing the FCFCORE and NAG libraries for spectra with discrete hyperfine fields and the NORMOS/DIST routines for spectra with distributions of hyperfine field. Calibration of all spectra were done relative to metallic Fe at room temperature. Prior to the Mössbauer analysis, we used a Quantum Design superconducting quantum interference device (SQUID) to accomplish ac-susceptibility measurements, with 1 Oe field amplitude as a function of temperature and frequency and dc magnetization measurements with fields up to 7 T.

III. RESULTS AND DISCUSSION

Figure 1 plots the real (χ') and imaginary (χ'') components of the ac susceptibility measured in the relevant temperature range using eight different frequencies logarithmically distributed in our available frequency spectrum of 0.1–1000 Hz. It exhibits the typical spin-glass characteristics:[19] the maximum in $\chi'(T)$, relatively sharp if the enlarged temperature scale is taken into account, shifts slightly to higher temperatures with increasing frequency (about 2% per decade), and $\chi''(T)$ undergoes a more sudden onset, with the maximum slope temperature coinciding with the maximum in $\chi'(T)$, T_{max}. Other spin-glass-like features, including the smearing of the ac-susceptibility peak upon application of moderate bias fields or the characteristic thermal irreversibility between the field-cooled (FC) and zero-field-cooled (ZFC) magnetizations, can be seen in Ref. 17. There, dealing with poorer ac data we showed that activated dynamics laws, i.e., Arrhenius like, could not fit the $T_{max}(f)$ data with physically meaningful parameters, whereas attempts to fit with the critical slowing down law

$$\tau = \tau_0 \left(\frac{T}{T_f} - 1 \right)^{-z\nu} \tag{1}$$

were inconclusive. Such a law has been used now to fit the more comprehensive data shown in Fig. 1, substituting (T,τ) by $(T_{max},\tau_m=1/f)$, and the results obtained are $\tau_0=(8\pm2)$ ps, $z\nu=13.8\pm0.4$, and $T_f=(25.9\pm0.1)$ K. Notice the relatively small errors due to the low data dispersion, as shown in Fig 2. The value of $z\nu$ is higher than those typically extracted from three-dimensional (3D) spin glasses, e.g., 10.5 in $Fe_{0.5}Mn_{0.5}TiO_3$. A similar large value of $z\nu$ (12.5 ± 1.5) was reported by Jurberg *et al.* in a ferrofluid consisting of strongly interacting FeC particles of about 5 nm diameter, where physically meaningful static critical coefficients were also obtained.[20] However, they found a decreased attempt time ($\sim10^{-6}$ s), whereas τ_0 in our sample has the usual value for conventional spin glasses.

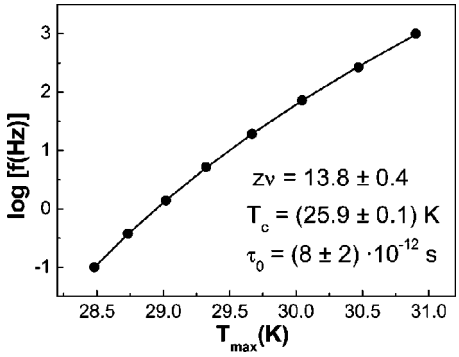

FIG. 2. Frequency dependence of the peak temperature in the real component of the ac susceptibility (T_{max}) and fit to the critical slowing down law (solid line), with the best fitting parameters.

Although the above ac-susceptibility analysis is valuable to investigate the magnetic moment correlated dynamics, the sample SGL behavior might be attributed to the presence of interacting ferromagnetic nanoparticles after analysis of the field dependence of the magnetization. A conventional spin-glass phase would require the homogeneous dilution of the Fe atoms, yielding a linear paramagnetic response at temperatures well above T_f. However (see Fig. 3), the magnetization measured at 200 K begins to bend over at relatively small fields, below 3 T, pointing to the existence of fine superparamagnetic moments. A small ferromagnetic component, which quickly saturates to 0.34 emu/g ($\sim$2% of the

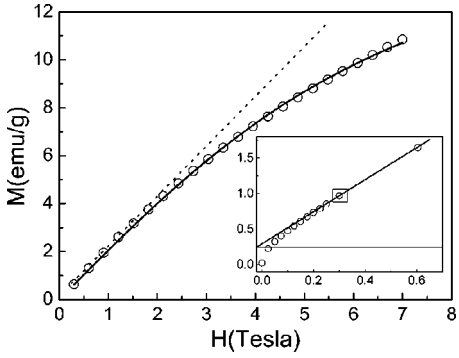

FIG. 3. Magnetization measured at $T=200$ K and fit (solid line) to the Langevin law. The dotted line indicates the initial susceptibility. The inset shows the small ferromagnetic component subtracted before fitting; the squared point corresponds to the first point in the fitted data (main figure).

total saturation magnetization), was subtracted from the data and the points below its saturation (3 kOe) removed (see inset, where the squared point corresponds to the first point in the main area). This contribution corresponds to about 0.5% of the Fe atoms, probably in larger, blocked particles, and thus can be neglected henceforth. The data were then successfully fitted (see the solid line in Fig. 3) to the Langevin expression

$$M = N\mu\left[\coth\left(\frac{\mu H}{k_B T}\right) - \frac{k_B T}{\mu H}\right], \tag{2}$$

where μ is some mean magnetic moment for the nanoparticles. The relatively high temperature justifies the use of the isolated particle approximation. The fit yielded the values $\mu = (105 \pm 2)\mu_B$ and $N\mu = (17.6 \pm 0.3)$ emu/g. Assuming spherical particles of pure bcc Fe with its bulk magnetic moment, the diameter would be as small as $d = 1.0$ nm. Although from the synthesis procedure it is natural to expect, however, not pure but Fe-rich and highly disordered clusters, this does not invalidate any of the following discussion. Such magnetic entities, embedded in a mostly amorphous paramagnetic matrix,[17] are difficult to resolve by conventional high-resolution microscopy due to their very fine size and the rather weak structural and compositional contrast with the bulk matrix.[21] The saturation moment obtained from the fit leads, assuming again the bulk moment for the Fe atoms in the particles, to a 21% fraction of the sample Fe taking part in the nanoparticle phase.

In this two-phase picture, the spin-glass behavior could be still ascribed, in principle, to either of them: the homogeneous solid solution matrix could account for it in a similar fashion to amorphous spin glasses,[22] while the introduction of competing interactions between the nanoparticles could lead to the formation of a superspin glass.[3,20] The magnitude of the magnetization involved in the FC-ZFC thermal irreversibility (not shown), in agreement with the data in Fig. 3, points towards the nanoparticle phase being responsible for the observed SGL behavior. Yet this issue is elucidated more conclusively in the following Mössbauer study, which also yielded relevant information about the nature of both the particles and the magnetic transition.

Figure 4 shows selected Mössbauer spectra taken between 10 and 40 K. The latter was best fitted using two doublets with similar isomer shifts $IS_1 = 0.36$ mm/s and $IS_2 = 0.39$ mm/s, both roughly corresponding to Fe^{3+}, and quadrupolar splittings $QS_1 = 0.72$ mm/s and $QS_2 = 0.31$ mm/s, where the subindex 1 refers to the less intense doublet. Spectra taken at 77 and 300 K (not shown) were well fitted with the same two components, i.e., essentially equal quadrupolar splittings and doublet areas ($A_2 \approx 62\%$), but linewidths narrowing with temperature (due to paramagnetic relaxation effects) and isomer shifts exhibiting the usual thermal shift. Since the blocking temperatures customarily estimated from Mössbauer are considerably larger than T_{max} from the ac-susceptibility peaks, because of the different time scales of the two techniques, the possibility of superparamagnetism (or weak interparticle interaction) in $Fe_{30}Al_{49}Cu_{21}$ can be already ruled out from the absence of any magnetic component

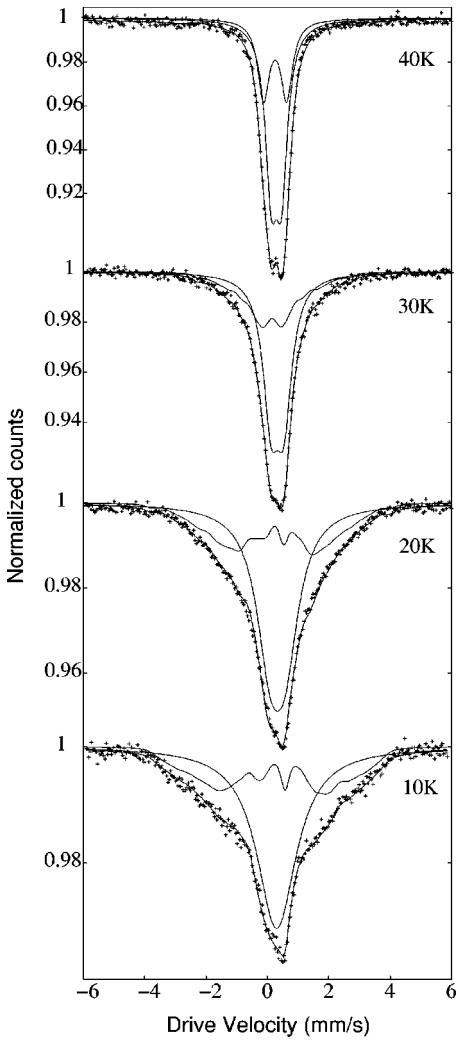

FIG. 4. Mössbauer spectra at selected temperatures. At high temperatures, here represented by the spectra at 40 K, the data are well fitted with two doublets. For the spectra at $T \leqslant 30$ K, one of the doublets is substituted by a distribution of magnetic sextets, whose splitting grows with decreasing temperature.

in the 40 K spectrum. However, such a component emerges at 30 K and becomes progressively more noticeable with decreasing temperature.

Figure 5 presents separately, for the sake of clarity, the spectrum taken at 4.2 K, where the magnetically split component is best resolved. This spectrum was fitted to a doublet plus a distribution of magnetic hyperfine fields and isomer shifts (MHFD). The fit involved six free parameters, including the relative area and quadrupolar splittings. The QS of the doublet component is the same as that of the large doublet in the high-temperature regime spectra, which together with the resemblance in area percentage, 66%, justifies their identification. Therefore, the small doublet in the high-temperature spectra splits into a broad MHFD at low temperatures. It is natural to associate the large component with the paramagnetic matrix, which remains paramagnetic at least down to 4.2 K, and ascribe the appearance of the hyperfine magnetic field distribution component to the freezing of the interacting nanoparticles. The discrepancy between the

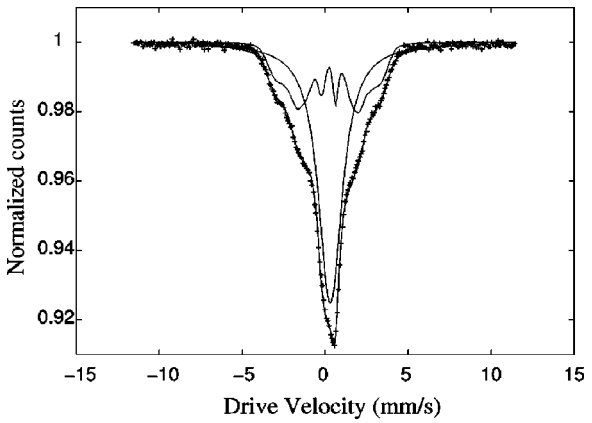

FIG. 5. Mössbauer spectrum at 4.2 K.

Fe fraction in the particle phase estimated from magnetization (Fig. 3), 21%, and Mössbauer measurements, 34%, has the expected sign: it can be accounted for by simply dismissing the assumption of the bulk value for the magnetic moments of the Fe atoms in the nanoparticles, which would thereby take the value 1.4 μ_B. This indicates the partial alloying of the Fe in the particles with a certain amount of Al and Cu, consistently with the diminished value of the mean magnetic hyperfine field, 16.1 T, in comparison with the bulk pure Fe value (33 T).

The spectra at 10 K, 20 K, and 30 K were fitted in the same way. Significantly, the area percentages for the doublet and MHFD components were very similar ($\pm 3\%$) to those obtained in the 4.2 K spectrum, decidedly pointing to collective dynamics. Difficulties related to the decreased hyperfine splitting in the 25 K and 30 K spectra obliged us to fix the area there to 34%. Self-consistency was checked using this alternative fitting procedure too in the other low-temperature spectra, yielding approximately the same parameter values. A progressive increase of the mean hyperfine field (MHF) of the MHFD component upon lowering the temperature below 30 K is manifest in Fig. 4. Figure 6 shows explicitly such a variation, clearly revealing a phase transition at approximately 35 K, since it is well known that the magnetic hyperfine field is proportional to the order parameter. In superparamagnets, the so-called collective magnetic excitations around the blocked direction can lead to a maximum variation of about 15% in the hyperfine field.[23] The open circle in Fig. 6 is the value of the Mössbauer transition temperature, $T_M = 37.7$ K, obtained from the extrapolation of the critical slowing down curve fit (Fig. 2) up to the Larmor frequency $f_L \approx 2\times 10^8$ Hz. Here T_M is about 25% higher than the freezing temperatures showed by the susceptibility peaks, close to the 20% typical of conventional spin glasses,[18] and seems consistent with the MHF data. The fit of the data closer to T_c using the power law

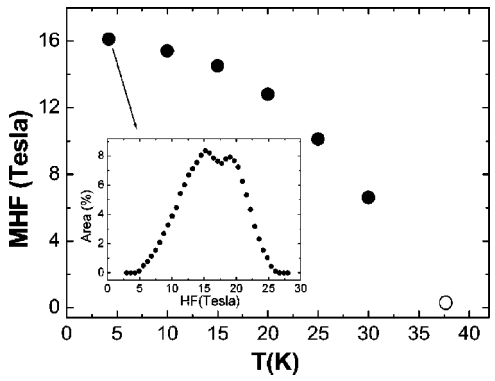

FIG. 6. Mean magnetic hyperfine field (MHF) as a function of temperature below the freezing temperature. The empty point has been obtained from the extrapolation of the fit in Fig. 2 as explained in the text. The inset plots the hyperfine field distribution for the magnetic component of the 4.2 K spectrum (shown in Fig. 5).

$$\mathrm{MHF} \propto \left(1 - \frac{T}{T_c}\right)^{\beta}, \tag{3}$$

with $T_c = T_M$, yielded $\beta = 0.72$, which lies between the value obtained in 3D short range spin glasses, $\beta = 0.5$,[19] and those found in dipolar superspin glasses, $\beta \approx 1.0$.[8,20] This intermediate value may arise from a presumable mixture of dipolar and RKKY interactions. It is sometimes considered that techniques sensitive to the local magnetic interactions can distinguish unequivocally between intrinsic (atomic) spin glasses and granular ferromagnets,[24] however, this Mössbauer study illustrates how certain strongly interacting granular systems can exhibit essentially the same temperature dependence of the hyperfine splitting as intrinsic spin glasses.

Regarding the shape of the hyperfine magnetic field distribution, it is apparent from the poorly resolved aspect of the magnetic component that we are dealing with a broad distribution. For instance, the inset of Fig. 6 shows the hyperfine magnetic field distribution of the spectrum at 4.2 K. The MHFD's at higher temperatures are shifted to lower values of hyperfine fields, but show a similar relative width. The reason for the large width of the distributions can be found in (a) the probable internal structural disorder of the magnetic particles and (b) local variations in the strength of the interparticle interactions.[23] In fact, it has been shown in frozen ferrofluids that the Mössbauer spectra of interacting particles typically exhibit much broader lines than the spectra of noninteracting particles.[25]

Despite all the experimental data presented here being consistent with the onset of a superspin glass phase at $T_c = 25.9$ K, it must be noticed that the nonlinear dc susceptibility failed to show any divergence resembling that of spin glasses,[17] which we now attribute to the high magnetic fields employed in order to avoid the nonsaturated region of the small ferromagnetic component.

The term *superspin glass* is preferred here to the older *cluster glass* or *mictomagnetism* to remark on the existence of ferromagnetic particles at temperatures well above the freezing process, yielding a superparamagnetic response. Further, the displaced hysteresis loops typical of cluster glasses[19] were not observed in $Al_{49}Fe_{30}Cu_{21}$.[17] The name ''superspin glass'' seems to be sometimes linked in the literature to the dominance (or exclusiveness) of dipolar over RKKY interactions[3,8] in providing magnetic frustration. The classical dipolar interaction between neighboring particles

can be estimated directly from the particles concentration and their mean magnetic moment,

$$T_d \approx \frac{\mu_0 \mu^2}{k_b D^3} \approx 16 \text{ K},$$

where

$$D = \frac{d}{\left(\frac{6c}{\pi}\right)^{1/3}} = 1.8 \text{ nm}, \quad (4)$$

with D being the mean distance between particles and $c = 0.10$ the particle volume concentration (calculated from the magnetic component area of the 4.2 K spectra, 34%). This value of T_d is of the same order of magnitude as T_c, but still rather small. The only other magnetic interaction that can provide the necessary parallel-antiparallel competition is the RKKY indirect exchange, which has been theoretically shown to conserve its oscillatory character independently of the ferromagnetic particle size.[5] The sample structure, a metallic alloy, does indeed allow such an interaction, but a direct proof of its predominance can only be obtained in experiments dealing with changes in the electrical conductivity of the matrix. As far as we know, no superspin glass has been reported yet to arise from interparticle RKKY interaction. However, its contribution in our sample seems plausible after the latest theoretical predictions: Skomski concluded that magnetostatic interactions tend to suppress the RKKY effect in particles larger than about 1 nm,[7] and before, Altbir *et al.* had established in a discrete calculation that the upper cluster size limit for the dominance of the RKKY interaction is about $100\mu_B$.[6] The clusters present in $Fe_{30}Al_{49}Cu_{21}$ were estimated above to have $105\mu_B$ and about 1 nm diameter, both values falling in the mentioned theoretical crossovers between the RKKY and dipolar interactions dominance.

Our results are in agreement with the conclusions of Le Caër *et al.*,[18] who used room-temperature Mössbauer spectroscopy to deduce a universal behavior in their ball-milled Fe-based samples: in many systems, they detected the presence of a component consisting of nanometer-sized Fe-rich heterogeneities which gave rise to a distribution of hyperfine magnetic fields. This fraction remains stationary and still magnetically active even after very long milling times. However, compositional clustering is an often ignored question in the field of mechanically alloyed materials.[26] The discovery of Fe-rich nanoclusters in most of the alloys prepared by our group,[13,17,27] leading to spin-glass-like behavior at low temperatures, should serve to increase caution in this regard. The same can be applicable to other nonequilibrium synthesis techniques: for instance, the presence of segregated structures near the subnanometric scale has been recently reported in amorphous alloys obtained using sputtering deposition.[28] In bulk samples, this very fine compositional clustering is hard to detect by conventional x-ray diffraction (XRD) or high-resolution transmission electron microscopy (TEM); thereby, detailed knowledge of the magnetic behavior derived from the possible fine compositional granularity can serve to diagnose the presence and characteristics of the responsible nanoscale magnetic entities.

In summary, we have proved by Mössbauer spectroscopy and high-field magnetization that the spin-glass-like properties of mechanically alloyed $Fe_{30}Al_{49}Cu_{21}$ are due to the presence of interacting magnetic nanoparticles, and not to the homogeneous amorphous matrix where they are embedded. The temperature dependence of the mean magnetic hyperfine field confirms the collective character of the transition from superparamagnetism to a superspin glass state, as suggested by the frequency dependence of the ac-susceptibility peak.

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