Spin-glass-like state in GdCu: role of phase separation and magnetic frustration

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We report investigations on the ground state magnetic properties of intermetallic compound GdCu through dc magnetization measurements. GdCu undergoes first order martensitic type structural transition over a wide temperature window of coexisting phases. The high temperature cubic and the low temperature orthorhombic phases have different magnetic character and they show antiferromagnetic and helimagnetic orderings below 145 K and 45 K respectively. We observe clear signature of a glassy magnetic phase below the helimagnetic ordering temperature, which is marked by thermomagnetic irreversibility, aging and memory effects. The glassy magnetic phase in GdCu is found to be rather intriguing with its origin lies in the interfacial frustration due to distinct magnetic character of the coexisting phases.

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I. INTRODUCTION

Spin glass is an intriguing example of non-ergodic state marked by several physical properties such as thermomagnetic irreversibility, slow dynamics, non-exponential decay, magnetic memory effect, etc. The non-equilibrium state arises from frustration due to competing magnetic interactions among atomic spins as well as disorder capable of pining the spins. In recent time, spin glass like non-equilibrium dynamics and time dependent phenomena have been observed in several magnetic systems, where the basic building blocks responsible for the ‘glassy’ behavior are not really the atomic spins, rather spin cluster or bigger spin entity. In particular, such behavior have been widely observed in phase separated manganites and cobaltites. The observation of slow dynamics resembling classical spin glass is also extended to intermetallic alloys showing first order magnetostructural transition and also to magnetic nanoparticles. Since, the basic building blocks are bigger spin entity, they are often termed as cluster glass, superspin glass or magnetic glass.

The coexisting magnetic phases related to glassyness in manganites and other bulk solids are often related to first order phase transition (FOPT). In presence of static disorder, FOPT can lead to coexistence of high-temperature (T) parent and low-T product phases within the region of transition. In certain circumstances, there can also be structural freezing of the parent phase below the transition point. The glassyness in such phase separated system can have two likely origin. Firstly, the slow dynamics of the coexisting phases due to their metastability, and secondly due to frustration arising from the magnetic interaction between two clusters having distinct magnetic nature. The spin glass like state (or often called cluster glass) in phase separated manganites has often been attributed to the coexisting ferromagnetic (FM) and charged order antiferromagnetic (AFM) phases of micrometer size due to FOPT. It is worth noting that in case of polydispersive non-interacting nanoparticle system (where the magnetic interaction between two particles is negligible), a simple model based on the distribution of superparamagnetic relaxation time can explain the observed glassy magnetic behavior including the memory effect.

Considering the fact that a large number of bulk phase separated materials show glassy magnetic behavior, it is pertinent to investigate the role of inter-cluster magnetic interaction toward the observed state. However, unlike magnetic nanoparticles one can not tune the strength of the magnetic interaction in a bulk material. More importantly, the FOPT in such systems is of magnetostructural type with strong interplay between magnetic and structural degrees of freedom. As a result, the structural and the magnetic transitions occur below the same T, and their individual role toward the glassyness becomes difficult to differentiate.

Here we report the magnetic investigation on GdCu intermetallic compound, which was reported to show phase coexistence due to first order martensitic transition (MT). The fact that prompted us to investigate GdCu is that the magnetic and structural transitions occur at distinctly different T. Therefore, it provides an opportunity to investigate the role of magnetic interaction and structural phase separation in determining the ground state magnetic character.

The RCu (R= rare-earth) series of compounds with heavy rare-earth (R ≥ Gd) crystallize in the cubic CsCl-type structure (hereafter called C-phase) at high-T. Some of the members (R = Gd, Tb and Y) show lattice instability and undergo MT at low T to an orthorhombic FeB-type structure (hereafter called O-phase). GdCu shows long range AFM ordering below $T_N$ in the C-phase, while it undergoes a second transition below $T_N^O$ to a helimagnetic (HM) spin structure in the low-T O-phase. Our investigation shows the existence of an unconventional glassy magnetic phase in GdCu below $T_N^O$, which presumably arises out of the magnetic frustration from coexisting phases.
II. EXPERIMENTAL DETAILS

Polycrystalline samples of GdCu and DyCu were prepared by argon arc melting with the constituent elements of purity better than 99.9 wt%. The ingots were homogenized at 800°C for 120 h. Room temperature powder x-ray diffraction (XRD) patterns were recorded using a Bruker AXS diffractometer (Cu Kα radiation, 2θ range from 20° to 80° with a step size of 0.02° and 5 s/step counting time). The collected powder patterns were used for phase identification of the given compound using the GSAS software package. Rietveld refinement data along with XRD pattern of GdCu are shown in the inset of fig. 1. Lattice parameter (a = 3.518 Å) and unit cell volume (V = 43.54 Å³) are in good agreement with the previously reported data. The analysis indicates that the annealed samples are single phase with cubic CsCl structure at room temperature.

The T variation of resistivity (ρ) in zero as well as in presence of external magnetic field (H) were measured down to 5 K in a commercial cryogen free high magnetic field system from Cryogenic Ltd., U. K. Magnetization (M) was measured using a commercial Quantum Design SQUID magnetometer (MPMS XL Ever Cool model).

III. RESULTS

Fig. 1 represents the T dependence of ρ of GdCu measured during cooling and heating. A sharp anomaly is observed around T_{MT} = 250 K in the cooling data which signifies the MT from the high-T C-phase to low-T O-phase as reported in previous studies. Only a part of the thermal hysteresis loop associated with the MT is visible in the measured T range, because the transition extends up to 650 K in the high-T side. Similar hysteresis is also present between the field-cooling (FCC) and subsequent field-cooled heating (FCH) protocols with the MT from the high-T C-phase to low-T O-phase.

FIG. 1: (Color online) The main panel shows the zero field resistivity as a function of temperature for cooling and subsequent heating legs for GdCu. The sample was cooled from 300 K down to 5 K and heated back to 300 K again. The inset shows the Rietveld refinement of x-ray powder diffraction pattern of GdCu compound. Open circle represent observed data and the lines drawn through the data points correspond to the calculated patterns.

FIG. 2: (a) Inverse magnetization as a function of temperature measured in the field-cooling (FCC) and subsequent field-cooled heating (FCH) protocols for GdCu. The FCC and FCH data show thermal hysteresis down to about 140 K. The inset shows the magnetization as a function of field recorded at 4 K. (b) Zero field-cooled-heating (ZFCH) and field-cooled-heating (FCH) magnetizations as a function of temperature in 1 kOe of applied field.
are found to show $\beta$ evolving, and it approaches 1 when the number of such intermediate states through which the system should pass is large. Such broad feature associated with thermomagnetic irreversibility can be an indication of disordered and/or glassy magnetic phase below $T_N^O$.

The inset of fig. 2 (a) shows the $M - H$ curve of GdCu measured at 4 K, which is found to be linear without any signature of field induced transition. The $\rho$ versus $H$ curves (not shown here) also do not show any signature of metamagnetism. The magnetoresistance (MR = $(\rho(H) - \rho(0))/\rho(0)$) at 4 K is found to be very small and positive (about 4% for $H = 50$ kOe), which is common among bulk AFM materials. Therefore, one can rule out the possibility of any major role of $H$ in stabilizing different magnetic and structural phases in GdCu. This is possibly indicative of a weak magneto-structural coupling in GdCu unlike manganites or magnetic shape memory alloys.

A. Magnetic relaxation

Considering magnetic irreversibility in GdCu, the time ($t$) evolution of $M$ was investigated at low $T$. The measurement was performed in two protocols, namely zero-field-cooled (ZFC) and field-cooled (FC) as described in figs. 3 (a) and (b). The $t$ dependence of $M$ for both the protocols were measured under 100 Oe of applied field. The ZFC measurement was performed at different temperatures above and below $T_N^O$. However, only strong relaxation effect was observed below $T_N^O$. The $M(t)$ data collected in the ZFC mode at 30 K (below $T_N^O$) shows 1% change in 3600 s, while at 60 K (just above $T_N^O$) the change is only 0.1%.

The time evolution of $M$ has been analyzed on the basis of various available models of slow dynamics applicable to magnetic system. It can often show a power law ($\sim t^{\pm \alpha}$) or a Kohlrausch-Williams-Watt (KWW) stretched exponential behavior ($\sim \exp \left[-(t/\tau)^{\beta}\right]$) where $\tau$ is the characteristic relaxation time and $\beta$ is the shape parameter. We have used both the relations to fit our data, and found that the best fit is obtained with the KWW model. Such model was widely used to analyze the data for spin glass and other disordered magnets. In this model, $\beta$ lies between 0 and 1 and class of disordered materials. The value of $\beta$ was found to be 0.51 and 0.59 for the ZFC and FC data respectively at 4 K. The exponent $\beta$ in the KWW model signifies the number of intermediate states through which the system should evolve, and it approaches 1 when the number of such intermediate states diminishes. Glassy magnetic systems are found to show $\beta$ values over a wide range between 0.2 to 0.6 below the freezing temperature ($T_f$). For example, the spin glass alloy La-Fe-Mn-Si has $\beta$ value close to 0.5 well below $T_f$, while CuMn (4.0 at %) shows $\beta$ to be between 0.2 to 0.4 depending upon the temperature of measurement.

B. Aging

We studied the aging effect in GdCu below $T_N^O$ (see fig. 4). The sample was first zero-field cooled down to 15 K, and was allowed to age there for a certain waiting time of $t_w$. Subsequently $M$ was measured as a function of time in the presence of $H = 10$ kOe. The measurement was performed for three different values of $t_w$, namely 600, 2000 and 5000 s. From the $t$ variation of $M$ in the resulting states, we have calculated the magnetic viscosity ($S(t) = \frac{1}{M} \frac{\partial M(t)}{\partial t}$) as shown in fig. 4. It is clear that the $S(t)$ behavior is strongly influenced by the waiting
time. The $S(t)$ plots show clear peak at the respective $t_w$ values, which is typical experimental evidence of the non-equilibrium states in classical spin glass system.\textsuperscript{2} It is worth noting that such perfect match between $t_w$ and the peak position of $S(t)$ in case of GdCu is absent in several glassy magnetic materials indicating a deviation from the classical spin glass behavior (see for example\textsuperscript{38,39}).

C. Memory in the temperature variation of $M$

We probed the memory in dc magnetization in GdCu through different modes of measurements.\textsuperscript{4,17,22,38,39} First we depict the memory in the $T$ dependence of $M$ measured in the FC state following the protocol by Sun et al.\textsuperscript{17} (see fig. 5 (a)). Chronologically, the measurement was performed in the following steps: (i) the sample was cooled in $H = 100$ Oe from 100 K to 2 K with intermediate stops of duration $t_w = 1$ h each at $T_{stop} = 30$, 20 and 10 K. During each stop, $T$ was kept constant, while $H$ was reduced to zero. After each stop, we reapplied $H$ and resumed cooling, which resulted a step-like $M - T$ curve ($M^{\text{stop}}_{\text{FC}}$). (ii) After reaching 2 K, the sample was heated back in $H = 100$ Oe, which produces the so called ‘memory’ curves ($M^{\text{mem}}_{\text{FC}}$). (iii) A reference curve was also recorded by simply allowing the sample to heat in $H = 100$ Oe after being field-cooled in the same field without any stop ($M^{\text{ref}}_{\text{FC}}$). The $T$ ramp rate in all the measurement (as well as during field cooling) was kept fixed at 1 K/min. While heating (curve $M^{\text{mem}}_{\text{FC}}$ in fig. 5 (a)), we observe striking memory effect signifying nonergodic behavior of the low-$T$ phase. At each $T_{stop}$, the sample produces clear upturn revealing the previous history of stops in zero field at that $T$. This is a manifestation of memory in the low-$T$ phase. However, it is interesting to note that no memory was observed above $T^G_0$ (i.e. $T_{stop} > T^G_0$), which once again proves the role of HM ordering for the observed arrested dynamics in GdCu.

We also looked for memory in DyCu, which belongs to the same RCu series of compounds. However, unlike GdCu, DyCu does not undergo MT and it remains in the C-phase down to low $T$. DyCu undergoes AFM ordering below 63 K\textsuperscript{40} and shows a second magnetic transition below 20 K. We performed the same field experiment on DyCu, and no signature of magnetic memory was observed (see fig. 5 (b)). This is in contrast with the behavior of GdCu, and presumably related to the absence of MT related phase coexistence.

Memory in the FC magnetization can also occur due to a distribution of relaxation time for a poly-dispersive nanoparticle system. GdCu being a phase separated system, similar effect may also arise from the independent relaxation of metastable phase clusters. A probable way to rule out such possibility is to investigate the memory in the ZFC magnetization, which is an unequivocal signature of glassy magnetic state originated from cooperative spin-spin interaction.\textsuperscript{23} Here the protocol is the same as that of FC memory measurement, only the sample was cooled in zero field and allowed to relax at selected stops (here 20 and 12 K) in zero field only. While heating in 100 Oe, the sample shows characteristic features at the point of stops. This is clearer in fig. 6 (b), where we have subtracted the reference ZFC curve ($M^{\text{ref}}_{\text{ZFC}}$, measured without stops) from the memory curve ($M^{\text{mem}}_{\text{ZFC}}$ measured with stops). The present observation clearly strengthens the view of spin glass like state in GdCu. Notably, the reference and the ‘memory’ curves coincide exactly at the low and high $T$ end points, which is a signature of the complete eradication of the memory upon heating and cooling.\textsuperscript{20}

D. Memory in the time variation of $M$

The arrested dynamics of the glassy magnetic phase can be described by a ‘free energy landscape picture’, which consists of many local minima of the free energy separated by a distribution of finite energy barriers.\textsuperscript{21} If the system gets trapped in one such local minimum while cooling through the glass transition, the subsequent dynamics is governed by the thermal activation across the energy barriers. The memory effect and aging are the manifestation of the evolution from one free energy minimum to another with time.

In order to strengthen the observed memory ‘dips’ in the $M(T)$ data, we investigated the relaxation behavior with negative $T$ cycling as shown in fig. 7 (a). The relaxation data was recorded in the ZFC mode. The sample was first cooled down to $T_0 = 15$ K in zero field. Subsequently, $M$ was recorded as a function of $t$ in presence...
of $H = 100$ Oe [segment $ab$ in fig. 7 (a)]. After 1 h, the sample was quenched in constant field to a lower $T$, $T_0 - \Delta T = 10$ K and $M(t)$ was recorded further for a time $\sim 1$ h [segment $cd$]. Finally, $T$ is turned back to $T_0$ and $M$ was recorded for further 1 h [segment $ef$]. Relaxation process during $ef$ is simply a continuation of the process during $ab$. This memory effect reflects that the state of the system before cooling is recovered when the sample is cycled back to the initial $T$. We also performed simultaneous $H$ and $T$ cycling to check the robustness of magnetic memory in the system [fig. 7 (b)]. Here the protocol is very similar to that of the relaxation experiment depicted in fig. 7 (a). The only difference is that during the temperature quench to $T_0 - \Delta T = 10$ K, $H$ was also reduced to zero. The continuity of the $M(t)$ curves before and after $T$ and $H$ cycling indicates that the sample is capable of retaining the history even for large change in $M$ (about 10 times).

**E. Rejuvenation**

It is known that for a nonergodic system, a small positive $T$ cycling can destroy the previous memory and rejuvenates the system.\textsuperscript{43} We performed such positive $T$ cycling in the relaxation data as shown in fig. 8. The difference between the present protocol and that of fig. 7 (a) is that intermittently the sample was heated to a higher $T$, $T_0 + \Delta T = 20$ K instead of cooling. A sharp contrast is observed between the results of fig. 7 (a) and the present data: the relaxation curves $a''b'\prime$ (before heating) and $e''f'\prime$ (after heating) are found to have different nature and do not continue to posses similar $M$ values. This indicates that heating to a higher $T$ erases the memory and it is similar to the rejuvenation observed in various spin glasses and other nonergodic systems.\textsuperscript{43}
IV. DISCUSSION

GdCu is a phase separated system and in analogy with the well known example of manganites, the glassy phase is likely to be connected to the coexisting phases. The phase separation in GdCu originates from the FOPT which drives the system from high-\(T\) C-phase to low-\(T\) O-phase. The role of FOPT related phase coexistence is evident from our experiment with analogous compound DyCu. The Dy-compound does not undergo FOPT, although like GdCu, it has two magnetic transitions. The failure to observe memory in DyCu indicates that low-\(T\) C-phase to low-\(T\) O-phase. The role of FOPT related phase coexistence is evident from our experiment with analogous compound DyCu. The Dy-compound does not undergo FOPT, although like GdCu, it has two magnetic transitions. The failure to observe memory in DyCu indicates that low-\(T\) C-phase to low-\(T\) O-phase.

The C-phase becomes antiferromagnetically ordered below about 145 K, and the slow dynamics can originate from the dependent magnetization of the frozen phase. Arrested dynamics in structurally frozen phase has been observed in Ti-Ni shape memory alloys. However, our relaxation and memory experiments identify the glassy phase only below \(T_{\text{N}}^{\text{O}} = 45\) K, which is the onset point of HM order of the O-phase. Therefore, it is not the metastability of the structurally frozen phases, rather the cooperative magnetic interaction which decides the glassyness. Otherwise, one would expect the glassy behavior to originate right below \(T_{\text{MT}}\) out of the frozen dynamics of the coexisting structural phases.

Our data also rules out the possibility of a glassy state originating from a distribution of relaxation time of non-interacting spin clusters, as observed in weakly interacting nanoparticles. Had this been the scenario, we would not expect memory in the ZFC measurement (see fig. 6). Notably, unlike nanoparticles, the magnetic anisotropy is rather weak in a Gd-based system with total orbital quantum number \(L = 0\). The likely mechanism behind the observed glassyness in GdCu lies in the magnetic correlation. Since it occurs only below \(T_{\text{N}}^{\text{O}}\), the low-\(T\) HM state plays a deciding role. The glassy phase below \(T_{\text{N}}^{\text{O}}\) resembles the classical spin glass state, particularly from the evidence of ZFC memory and aging (figs. 6 and 4 respectively). In GdCu (both in the O and C phases), there is only one crystallographic site\(^{22}\) for Gd which may not be sufficient enough for competing magnetic interaction among atomic spins leading to frustration. In addition, geometrical frustration can be ruled out in a cubic or orthorhombic crystal structure (both orthogonal). The only possibility that seems viable in the present case is the coexisting structural phases with contrasting magnetic character, which

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FIG. 7: (Color online) (a) Magnetic relaxation data at \(H = 100\) Oe in the zero-field-cooled state measured at 15 K (\(ab\) and \(ef\)) along with an intermediate measurement at 10 K (\(cd\)) for GdCu. (b) shows a similar measurement, with only exception is that the intermediate 10 K data was recorded in zero field. Here duration of all the relaxation measurements is \(\sim 1\) h.

FIG. 8: (Color online) Magnetic relaxation data measured in the zero field cooled state at 15 K with an intermediate increases in temperature from 15 to 20 K for GdCu. The duration of all the relaxation studies is \(\sim 1\) h.
nucleate. As the system goes below $T_{\text{trans}}$, the O-phase increases. Due to arrested kinetics, the GdCu introduces frustration and disorder leading to spin glass state.

We propose following scenario for the observed glassiness, which has been depicted in fig. 9. As evident, GdCu has several magnetic and structural phases. Above $T_{\text{MT}}$ (see fig. 1), the system is purely cubic and paramagnetic (PM) [see fig. 9(a)]. According to the terminology of MT in metallic alloys, $T_{\text{MT}}$ is called the martensitic start temperature, below which the low-$T$ martensite starts to nucleate. As the system goes below $T_{\text{MT}}$, the O-phase develops within the matrix of C-phase, however both are PM in nature. When $T$ is further lowered, the fraction of O-phase increases. Due to arrested kinetics, the complete transformation of the C-phase $\rightarrow$ O-phase does not take place and a fraction of C-phase exists even at the lowest $T$. Now below $T_N^c$, the C-phase orders antiferromagnetically, but the O-phase fraction remains PM, and eventually it orders below $T_N^O$. Within the temperature region $T_{\text{MT}} < T_N^O$, mixed phase prevails in the sample. However, unless one goes below $T_N^c$, the O-phase remains PM and can not introduce magnetic frustration. Eventually at $T < T_N^O$, both the coexisting phases become magnetically ordered and interfaces between two phases give rise to frustration owing to their conflicting magnetic character. The effect is analogous to the surface spin glass state in interacting $\gamma$-Fe$_2$O$_3$ nanoparticles with phase boundaries in GdCu playing the part of surface layers of the nanoparticles. The interesting point seen in case of GdCu is that the spin glass like state does not originates right below the first order transition, rather it develops below the low-$T$ HM transition temperature point. The presence of C-phase and O-phase clusters may give rise to a frozen local strain order as observed in Ti-Ni alloys, but that does not gives rise to a magnetic glassy phase as long as $T > T_N^O$. It is to be noted that previous heat capacity study showed broad peak around HM transition point, which is also a signature of the onset of glassy phase in a material.

In conclusion, we observe glassy magnetic phase in GdCu marked by slow dynamics and clear memory effect. Our results suggest that cooperative magnetic correlation between the coexisting phases is the primary cause of the observed spin glass like state. Such understanding can be quite useful in interpreting the spin glass like state in other phase separated systems including manganites.

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