Metastable magnetic response across the antiferromagnetic to ferromagnetic transition in Gd_5Ge_4

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Results of isothermal magnetization and magnetic relaxation measurements are presented probing the nature of the magnetic-field-induced magnetostructural transition in the intermetallic compound Gd_5Ge_4. This transition shows the characteristics of a disorder-influenced first order transition including distinct metastable behavior. Below approximately 21 K, the transition from the magnetic-field-induced ferromagnetic state back to the antiferromagnetic state shows additional interesting features. Similarities with other classes of magnetic systems exhibiting magnetostructural transitions are pointed out.

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I. INTRODUCTION

The intermetallic compound Gd_5Ge_4 is drawing much attention in connection with the giant magnetocaloric effect, giant magnetoresistance, and colossal magnetostriction discovered in this and other members of the $Gd_5(Si_{4-x}Ge_x)$ family of materials.[1,2] It orders antiferromagnetically at $T_N \approx 130$ K,[3] and in magnetic fields lower than 10 kOe, the antiferromagnetic order is sustained at least down to 2 K.[4] Under applied magnetic fields exceeding 10 kOe (the precise field value is temperature dependent), Gd_5Ge_4 shows an interesting antiferromagnetic (AFM) to ferromagnetic (FM) transition that could be driven by both temperature (T) and magnetic field (H), and a detailed H-T phase diagram for Gd_5Ge_4 has been obtained through a series of magnetization and heat capacity measurements.[5] It has been suggested that the initial AFM state at the lowest temperature (below $\approx$10 K), labeled as AFM-2, is different from the higher-temperature AFM state between $\approx$21 and $\approx$130 K (Ref. 5). This latter AFM state is marked as AFM-1. The field-induced AFM-1 to FM transition is reversible in nature; the system reverts back to the AFM-1 state on reduction of H from the FM state. The AFM-1 to FM transition field (H_M) increases with the increase in T beginning from $\approx$10 kOe at $\approx$21 K. The field-induced AFM-2 to FM transition, on the other hand, is irreversible in nature. The AFM-2 state is lost after the virgin field cycle, and is recovered only after heating the sample above 21 K and subsequent cooling. H_M associated with the AFM-2 to FM transition decreases with the increase in T and reaches $\approx$10 kOe at $\approx$8.6 K. The reported temperature independence of H_M between 8.6 and 21 K is attributed to the possible phase coexistence of the AFM-1 and AFM-2 phases in this temperature regime.[5] High-resolution x-ray powder diffraction experiments performed *in situ* under applied H up to 35 kOe have shown that the AFM-FM transition in Gd_5Ge_4 is coupled to a martensiticlike structural transition in which the low H–low T Sm_5Ge_4-type structure transforms to a Gd_5Si_4-type orthorhombic structure.[6] Hints of this structural transition were earlier observed in magnetostriction[7] and electrical resistivity[3] experiments as well.

The magnetostructural AFM-FM transition is a subject of much scrutiny at least in two other classes of magnetic systems, namely, manganites showing colossal magnetoresistance[8,9] (CMR) and pseudobinary alloys of the C15–Laves phase ferromagnet $CeFe_2$;[10,11] the similarity of these systems to Gd_5Ge_4 has been pointed out recently.[7,12] Just as in Gd_5Ge_4 the AFM-FM transition in the two former systems is first order in nature. Metastability is a characteristic feature associated with the AFM-FM transition both in CMR manganites[13] and in the doped $CeFe_2$ alloys.[14] Magnetization relaxation experiments[14] along with micro-Hall probe imaging studies[15] revealed important aspects of phase nucleation and growth across the disorder-influenced first order AFM-FM transition in doped $CeFe_2$ alloys. The influence of intrinsic quenched-in statistical disorder on a first order phase transition in general[16] is a subject of current theoretical interest.[9,17] With much evidence pointing to the first order nature of the magnetostructural AFM-FM transition[3–7] in Gd_5Ge_4 it will now be interesting to investigate the character of this transition in more detail. Here we present results of isothermal magnetization and magnetic relaxation measurements in polycrystalline Gd_5Ge_4 highlighting the metastability of phases across the AFM-FM phase transition.

II. EXPERIMENT

Various samples of Gd_5Ge_4 obtained from different sources have been used in the present study. However, for the sake of clarity we shall present results obtained with a particularly well characterized sample. The details of preparation and characterization of this sample can be found in Ref. 2 and samples of the same batch were used earlier for various other measurements.[4–6] dc magnetization (M) measurements were performed at $T=5, 15$, and 25 K using a superconducting quantum interference device (SQUID) magne-

tometer (Quantum Design MPMS-5) with a scan length of 4 cm. The AFM-FM transition in Gd_5Ge_4 could be driven by both temperature and magnetic field. Isothermal field variation experiments were preferred in the present work so that thermal fluctuations would have the same magnitude across the entire phase transition regime. The isothermal *M*-*H* scans were started from the zero-field-cooled (ZFC) state. The magnetic field was then raised from 0 to 50 kOe, and cycled between ±50 kOe. The ZFC states were achieved by cooling the sample in a zero magnetic field from a temperature well above $T_N \approx 130$ K. The initial *M*-*H* curve obtained after ZFC and then increasing *H* from 0 to 50 kOe is termed the virgin curve, and those in the subsequent field cycling between ±50 kOe are labeled the envelope curves. The sample used here had irregular shape, and we have checked the effect of demagnetization assuming the sample to be a sphere and an ellipsoid. The differences between the applied fields and the internal fields in both cases were less than 2 kOe, and this does not lead to any qualitative change in the obtained results. Isothermal *M* versus time (*t*) measurements were carried out keeping the field constant at various *H* values on the virgin and envelope curves. At each *T*, the target *H* values for the magnetization relaxation measurements were reached starting from a ZFC state. To keep the field sweep rate constant for all target *H* values, we changed *H* in steps of 500 Oe with a pause time of 1 s at each step. *M* was measured immediately after reaching the target *H* (after pausing for 1 s to stabilize the electronics); subsequent measurements of *M* to obtain the *M* vs *t* data were carried out at approximately 2 min interval for 50 min while keeping *T* and *H* constant.

III. RESULTS AND DISCUSSION

We show in Fig. 1 the isothermal *M*-*H* curves obtained at 5 and 25 K, with the *M*-*H* curve for $T=15$ K shown in the inset panel of Fig. 1(a). For the sake of conciseness, the *M*-*H* data will be shown only in the first quadrant, noting that the forward envelope curve is obtained after reaching −50 kOe. The near linear rise of *M* with increasing *H*, observed in the low-field regime of the virgin *M*-*H* curves [Figs. 1(a) and 1(b)], is as expected in an antiferromagnet. The deviation from this linear behavior at an applied field H_M (≈13 kOe at $T=5$ K, ≈9 kOe at 15 K, and ≈14 kOe at 25 K) marks the onset of the AFM-FM transition. Beyond H_M, the magnetization rises rapidly until it saturates upon reaching the FM state. At $T=5$ K, the virgin *M*-*H* curve is found to lie totally outside the envelope *M*-*H* curves, and the ascending- and the descending-field legs of the envelope curve are found to overlap. At 15 and 25 K, the envelope curve exhibits a hysteretic opening up. The virgin curve at 15 K still lies outside the envelope, while at 25 K the virgin curve nearly coincides with the ascending-*H* leg of the envelope curve.

Figure 2 presents normalized *M* versus time (*t*) plots at $T=5$ K for different *H* values on the virgin and the descending-*H* envelope curve. Strong relaxation effects in *M* are found between $H=10$ and 27.5 kOe on the virgin curve at $T=5$ K [see Fig. 2(a)]. But *M* on both the ascending- and

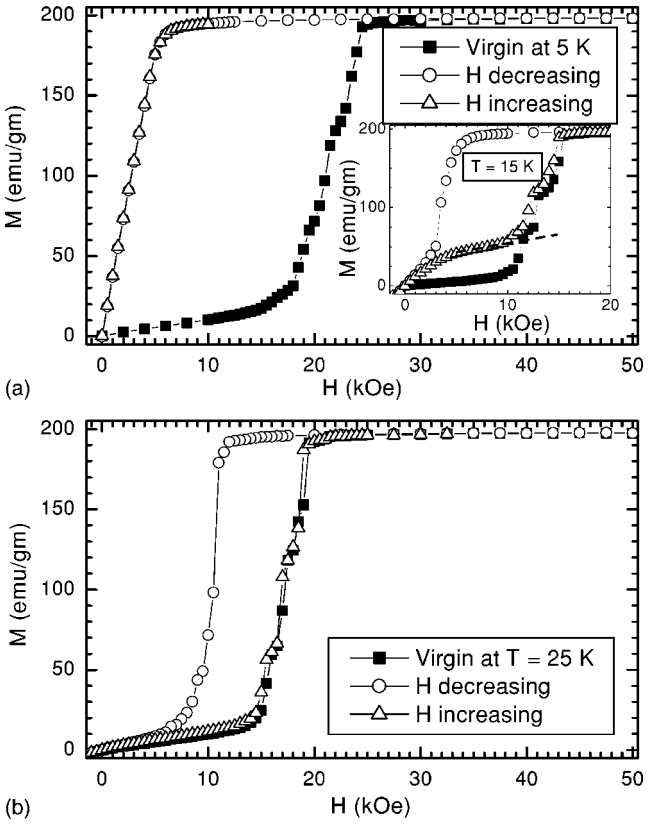

FIG. 1. Isothermal variation (starting from the ZFC state) of the magnetization of Gd_5Ge_4 with applied magnetic field.

descending-*H* envelope curves at the same temperature does not exhibit any relaxation within our experimental resolution for any value of *H* [see Fig. 2(b)]. The situation at $T=25$ K, however, is quite different, where *M* shows marked relaxation both on the virgin *M*-*H* curve between $H=12$ and 20 kOe [see Fig. 3(a)], and on the descending-*H* envelope curve between $H=11$ and 7 kOe [see Fig. 3(b)]. We note that at any of these temperatures (viz., 5, 25 K), no relaxation in *M* is observed on the virgin curve in the regime where $H < H_M$, i.e., in the AFM state, and also in the field regime well inside the FM state. Similar marked metastability in the AFM-FM transition region of CMR manganites[13] and doped $CeFe_2$ alloys[14] was considered to be a characteristic feature of a first order phase transition. Within this framework, H_M denotes the onset of a disorder-influenced first order phase transition process. The transformation (because of energy fluctuations) of the superheated AFM phase to the stable FM phase gives rise to relaxation in *M*. The transition from FM to AFM in the descending-*H* cycle is also expected to show evidence of metastability, i.e., supercooling. Presence of large relaxation provides such evidence across the FM to AFM transition in Gd_5Ge_4 at $T=25$ K; see Fig. 3(b). It is worth noting here that *H* is an intensive thermodynamic variable and within the phenomenology of first order phase transition, the generalized notations of supercooling and superheating are used without losing generality while exploring the *H*-*T* phase space.[18]

At $T=5$ K, on reducing *H* from the field-induced FM state, the magnetization of Gd_5Ge_4 starts decreasing rapidly

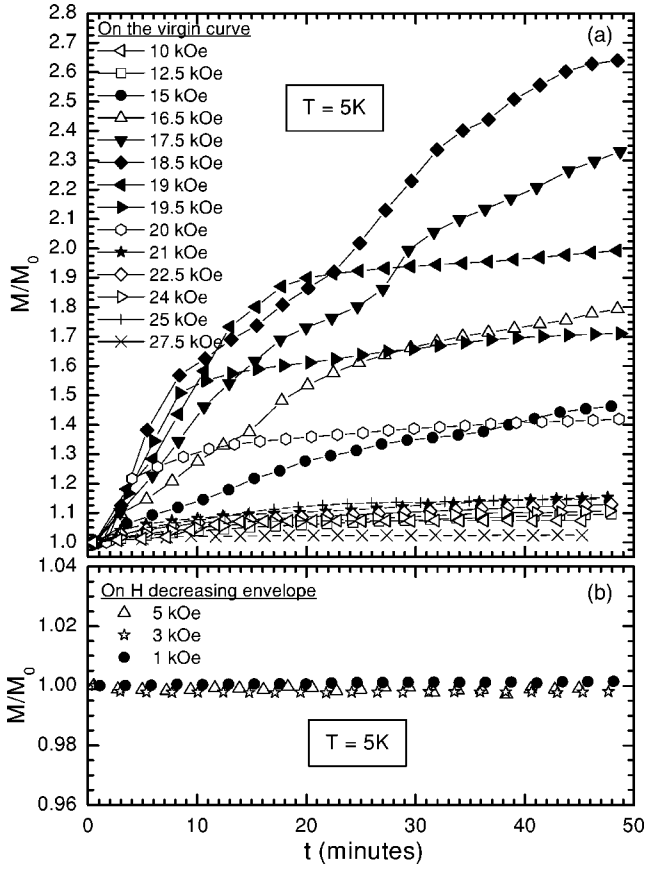

FIG. 2. Normalized magnetization vs time (t) for Gd_5Ge_4 measured in different constant magnetic fields, (a) in the virgin H cycle and (b) in the descending-H cycle at $T=5$ K. For each H, M_0 is the value of the magnetization recorded when the relaxation measurements were started, i.e., 1 s after the target H value was reached.

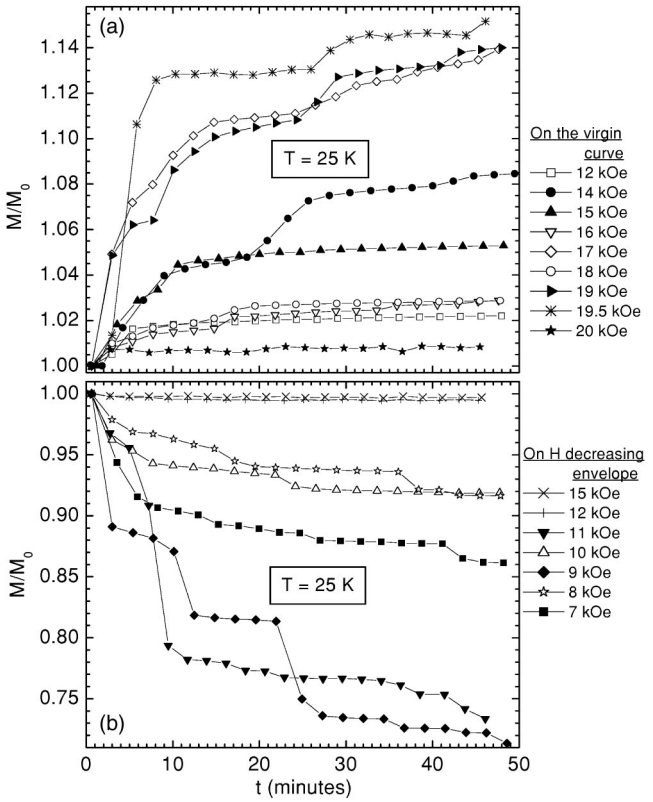

FIG. 3. Normalized magnetization vs time (t) for Gd_5Ge_4 measured in different constant magnetic fields, (a) in the virgin H cycle and (b) in the descending-H cycle at $T=25$ K. For each H, M_0 is the value of the magnetization recorded when the relaxation measurements were started, i.e., 1 s after the target H value was reached.

from its saturation value at around 10 kOe. This reduction of M on the descending-H envelope curve can be interpreted in terms of the normal domain reorientation in a ferromagnet. There is hardly any remanence at $H=0$ indicating the ferromagnetic state to be fairly soft. Our measurements detect no relaxation in M in this entire field regime [see Fig. 2(b)], and we accordingly infer that there is no supercooling (metastability) of the FM phase at $T=5$ K. These results, along with those reported earlier,[3–7] suggest that the FM to AFM transition does not take place at all in the descending-H cycle at this temperature. Thus, starting from a ZFC virgin state at 5 K, once the metamagnetic transition takes place, the system remains trapped in the FM phase. The initial AFM state cannot be recovered in any subsequent isothermal field cycling. Since the system remains in the FM state, its magnetization is higher than that in the AFM state. Hence, the virgin M-H curve lies well below the envelope curves. This behavior perhaps can be linked to the different AFM ground state AFM-2 below 10 K.[5] This would imply that the field-induced transition from the AFM-2 to the FM state is qualitatively different from the AFM-1 to FM transition taking place in the temperature region above 21 K. This assumption gains some support from the different nature of the temperature dependence of $H_M(T)$ in these two temperature regimes.[5] However, a complete understanding of the irreversible nature of this AFM-2 to FM transition will have to wait for the knowledge of the differences between the AFM-1 and AFM-2 phases, which is lacking at this moment. Such behavior of the virgin magnetization curve lying outside the envelopes in a first order AFM-FM transition was also reported in an Al-doped $CeFe_2$ alloy.[11] It was postulated that at sufficiently low T, the displacive motion of atoms involved in the structural distortion that was associated with the FM-AFM transition in the Al-doped $CeFe_2$ sample can slow down or even become arrested, as in the case of the transition from a supercooled liquid to a glass, where the characteristic time for structural relaxation becomes larger than experimental time scale.[19] The high temperature–high magnetic field FM phase is then frozen in. Accordingly it can be conjectured here that in the presence of a displacive structural transition (see Refs. 1 and 6 for more details on the mechanism of the magnetic-field-induced structural transition in Gd_5Ge_4), the kinetics of the first order magnetic transition in Gd_5Ge_4 is arrested in the low-T regime, thus providing a second plausible explanation for the persistence of the magnetic-field-induced FM state below ≈ 10 K.

The magnetic response in the descending-H cycle at $T=15$ K is intermediate between a fully arrested first order phase transition at $T=5$ K and a completed first order phase transition at $T=25$ K. This is in accord with the earlier studies.[5,7] In the descending-H cycle at $T=15$ K, there is a rapid decrease in M in the field regime $6>H>2$ kOe [Fig.

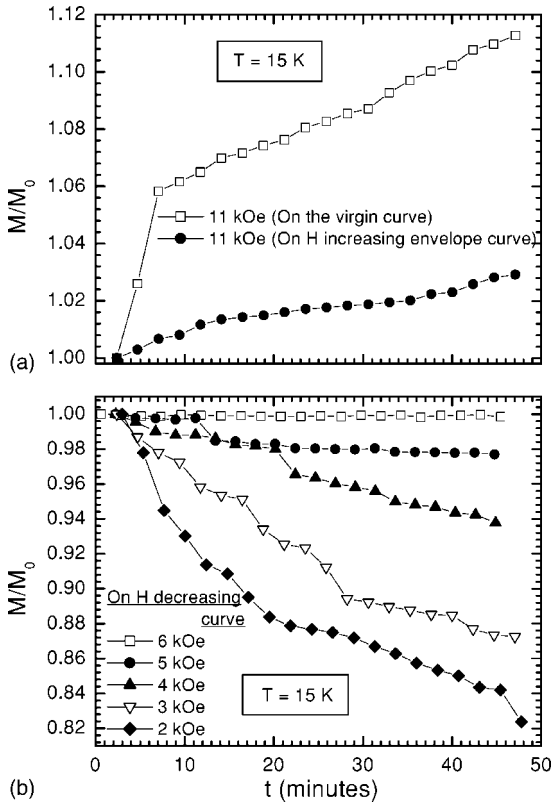

FIG. 4. Normalized magnetization vs time (t) for Gd_5Ge_4 measured in different constant magnetic fields, (a) in the virgin and ascending-H cycle and (b) in the descending-H cycle at $T=15$ K. For each H, M_0 is the value of the magnetization recorded when the relaxation measurements were started, i.e., 1 s after the target H value was reached.

1(a) inset]. This decrease of M, however, becomes less rapid below 2 kOe and it gradually merges with the ascending-H envelope curve at $H \approx 1$ kOe. A large time dependence of M is observed in the entire field regime mentioned above, until the merger with the ascending-H envelope curve takes place [see Fig. 4(b)]. This relaxation in M reinforces the idea that there exists a supercooled (metastable) FM state in this field regime, consistent with the first order nature of the FM to AFM transition taking place in the field regime between 10 and 1 kOe. We suggest that this FM to AFM transition is not completed and a kinetic arrest of this transition process starts around 1.5 kOe; a fraction of the FM phase remains unconverted in the sample even when the field is reduced to zero. Due to the presence of this arrested FM fraction, in the subsequent ascending-H cycle the envelope curve registers higher magnetization values than the virgin curve (the virgin state has only an AFM phase) until the AFM to FM transition is complete. This arrested FM fraction shows a magnetic-saturation-like character on the ascending-H envelope curve, in the field regime 4–8 kOe. Note that the arrested FM phase is different from the supercooled or metastable FM phase, as demonstrated by the fact that there is no relaxation in M in fields lower than $H=1$ kOe in the descending-H cycle, where the two envelope curves overlap. Even on the ascending-H envelope no relaxation in M is seen up to 8 kOe, i.e., before the AFM to FM transition sets in. Therefore the arrested FM fraction behaves as a stable phase within our present experimental time of 3000 s. Across the AFM to FM transition in the virgin sample, as well as the ascending-H cycle, however, the characteristic large relaxation associated with the first order phase transition process is observed at $T=15$ K [see Fig. 4(a)]. At 11 kOe on the ascending-H envelope curve the relaxation is well below that at the same field along the virgin curve, since there was no frozen fraction of the FM state in the virgin cycle.

It is important to note that the magnetic relaxation across the AFM to FM transition along the virgin M-H curve is much larger in magnitude at 5 K than that at 25 K. This clearly indicates that energy fluctuations of athermal origin play an important role in the phase transition process. The magnetic relaxation in Gd_5Ge_4 does not exhibit a logarithmic dependence on time (in contrast with the relaxation results reported for doped $CeFe_2$ alloys[14]). The time dependence of magnetization for a polycrystalline Gd_5Ge_4 in a 17 kOe magnetic field at $T=4.3$ K (the magnetic field was just under the critical $H=18$ kOe required to trigger a rapid AFM to FM transformation in the bulk) was also reported in Ref. 4. However, unlike the majority of the data depicted in Figs. 2 and 3, the results reported in Ref. 4 show no steplike behavior, and the magnetization was smoothly but nonmonotonically increasing over a 50 h period. The steplike behavior observed here for some $M(t)$ curves at 5 K and for the majority at 25 K may be related to minuscule temperature fluctuations experienced by the sample during the present measurements. While the sample in Ref. 4 was at the lowest attainable temperature without engaging the heater, the heater was operational in the present study for active temperature control. Thus, considering how the temperature of the exchange gas is controlled by a temperature controller (i.e., a heater and a feedback loop), and taking into account that the critical magnetic field in Gd_5Ge_4 is strongly dependent on temperature both at 5 and at 25 K,[3,5,7] we cannot rule out the possibility that temperature fluctuations on the order of 0.1 K trigger a transformation of a relatively large fraction of a material even when the magnetic field is held constant. It is also interesting to note here that distinct steplike features are observed on closer inspection during an isothermal field variation of M across the AFM-FM transition at $T=5, 15$, and 25 K (see Fig. 1). We have also studied some relatively impure samples. In such samples there is a marked nonlinearity observed in the M-H curve well below H_M, which is an indication of the presence of a ferromagnetic impurity phase. In the present context, importantly, no steplike features are observed in either $M(H)$ or $M(t)$ in a relatively disordered sample of Gd_5Ge_4 under the same experimental conditions. The rise in M at the AFM-FM transition under both temperature and field variation is less sharp in this sample. This suggests that the disorder profile of the samples may also be playing a role in the observed features across the AFM-FM transition.

Similar steps in the M-H curve across the metamagnetic transition and the associated metastability have been reported recently in CMR manganites and were claimed to be an intrinsic feature associated with the phase coexistence in such systems independent of their polycrystalline, single crystal, or epitaxial form.[20] It is also claimed that the appearance of

the steps depends on the nature of the magnetization measurements.[20] The stochastic nature of the M vs H steps observed in Fig. 1 may reflect an avalanchelike behavior similar to recently reported avalanchelike nucleation and growth of martensitic domains during the temperature-induced martensitic transition in a single crystalline Cu-Mn -Al.[21] Earlier, unusual and apparently random steps have also been reported during a temperature-induced first order phase transition in Er.[22] Taken together, these many features from quite distinct types of material systems are indications of common underlying physics associated with classic disorder-broadened first order phase transitions. This broadening of the first order phase transition can be attributed to the distribution of the local transition temperature or field across the physical dimension of the sample (Ref. 16), giving rise to a landscape. The disorder can be frozen in to the material by a number of mechanisms. They might arise due to the influence of intrinsic quenched-in disorder such as dislocations, vacancies, local composition, atomic configuration, etc., grain boundaries (in the case of polycrystalline samples), and strains. A landscape of transition fields created by the presence of disorder has been reported for the case of melting of a vortex solid in superconducting crystals.[23] In the present case the small temperature fluctuation described above can be sufficient to induce an excursion in this transition temperature–field landscape and trigger the phase conversion process, and hence the observed steps in the isothermal field and time variation measurements. The same landscape picture can probably explain the nonmonotonic behavior in dM/dt at various constant fields across the AFM-FM transition. The amount of the sample undergoing the AFM-FM transition at a particular field will depend on the nature of the local landscape. The same quenched-in disorder or strain also controls the actual distribution of energy barriers across the first order phase transition and as the system evolves it passes through a sequence of metastable states. Hence the observed metastability at a particular magnetic field across the AFM-FM transition is expected to depend on the nature of the landscape. Enhanced nonmonotonic behavior of the relaxation rate as a function of H at higher T can possibly be correlated with this landscape picture and the associated metastability. Apart from the higher intrinsic thermal fluctuation energy k_BT there is an added experimental complication. The sample heater will remain more active in temperature control in this higher-T regime, inducing also larger extrinsic temperature fluctuations. These will make the effects of landscape more visible in the higher-T regime and possibly explain the difference in the magnetization relaxation behavior at 5 and 25 K.

The presence of strain-disorder coupling can introduce additional interesting features in such phase coexistence.[24] The kinetics of phase transition in such systems where elastic forces play an important role, is expected to be different from that predicted by classical nucleation theory and is a subject of current research.[25] In this regard, the present results revealing metastability across the magnetostructural transition in Gd_5Ge_4 provide useful experimental input to these enquiries.

IV. CONCLUSION

In summary, upon field variation from the initial ZFC state the magnetic-field-induced AFM-FM transition observed in Gd_5Ge_4 is accompanied by distinct metastability. Evidence for this is presented in terms of the large magnetic relaxation at all temperatures where the field-induced AFM to FM transition occurs. The reverse transition from the FM state to AFM state on reducing H is also marked with the same kind of metastability in the temperature region above 21 K. Below 21 K, this FM to AFM transition process is hindered. At 5 K, the sample remains in the FM state at all fields including zero field after it has been magnetized at $H > 25$ kOe. This FM state is stable at least on the time scale of our magnetization relaxation measurements and is sustained on subsequent field cycling between ±50 kOe. At 15 K, the FM to AFM transition is initiated in the descending-H cycle but remains incomplete even when the magnetic field is reduced to zero. This leads to the interesting situation of phase coexistence between the converted stable AFM state and the unconverted FM state, which is also stable. This phase coexistence is to be contrasted with the phase coexistence observed across the AFM to FM transition in the virgin sample both below and above 21 K, and across the FM to AFM transition in the isothermal descending-H cycle above 21 K. Here, one of the phases—the AFM phase during the virgin cycle and the FM phase in the descending-H cycle—is metastable and relaxes toward the stable phase by energy fluctuations. The manifestation of such metastability is observed in the form of sizable magnetic relaxation. Similarity with the results in doped-$CeFe_2$ alloys[14] and CMR manganites[13,20] highlights the generality of the observed behavior, and suggests the possibility of disorder-influenced first order phase transition as the common underlying physics. A microscopic study of phase coexistence will be quite instructive here to verify assumptions of phase coexistence and its qualitative difference in various H-T regimes of Gd_5Ge_4.

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