PHYSICAL REVIEW B **71**, 024523 (2005)

Supercooling and giant relaxation of the disordered vortex state in a doped $CeRu_2$ alloy

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(Received 23 January 2004; revised manuscript received 21 May 2004; published 28 January 2005)

Giant relaxation in magnetization of metastable (supercooled) states in $Ce(Ru_{0.95}Nd_{0.05})_2$ alloy in the vortex solid-solid transition regime is reported. The metastable states were prepared by cooling the alloy sample from a temperature well above the T_C, in the presence of different magnetic fields. This was followed by an isothermal field change to set up a critical state in the entire bulk of the sample. Relaxation in magnetization was measured isothermally in this critical state. Through such a choice of experimental protocol, the relaxation in magnetization contributed by supercooled states is clearly distinguished. Variation of the relaxation rates with respect to the fields applied for producing the field-cooled states is explained in terms of supercooling of the disordered vortex phase.

DOI: 10.1103/PhysRevB.71.024523 PACS number(s): 64.60.My, 64.70.−p

I. INTRODUCTION

To establish the first order nature of a phase transition, one needs to measure discontinuities in entropy and volume (or magnetization), and show that these discontinuities are related through the Clausius-Clapeyron relation. It often becomes difficult, experimentally, to estimate a small entropy discontinuity (small latent heat) and even to distinguish it from a peak in specific heat.[1] In such cases the characteristic feature of hysteresis, associated with supercooling and/or superheating,[2] is used to identify a first order transition.[3] In our earlier studies on the C 15 Laves phase superconductor $CeRu_2$, we have used the existence of supercooled states to argue that the onset of peak effect (PE) was associated with a first order transition (FOT) in vortex matter [from one kind of (quasiordered) vortex solid phase to another kind of (disordered) solid phase].[3,4] This became necessary because pinning of vortices in hard superconductors results in producing variation in local magnetic fields across the sample volume. This in turn broadens the transition and smudges out discontinuities in magnetization and entropy. Supercooling was observed across this transition in $CeRu_2$ both by decreasing field H and by decreasing temperature T.[5] Standard phenomenology of FOT was used to study metastability in different samples under different paths in the H-T space and under different extents of supercooling.[6,7] Measurements on $CeRu_2$ have supported the prediction that supercooling by reducing H reduces the effective free energy barrier seen by the metastable state, and supercooling is best achieved by reducing T in constant H, i.e., by field cooling. Signatures of supercooling across a FOT in vortex matter have also been reported in various other superconductors such as YBCO,[8,9] $NbSe_2$,[10] BSCCO,[11] Nb,[12] V_3Si,[13,14] etc. Measurements on V_3Si (Ref. 13) have also supported the expectation[15] that a more deeply supercooled state is unstable to a smaller fluctuation.

In a parallel effort, we have been studying first order antiferromagnetic to ferromagnetic transition in doped $CeFe_2$ alloys.[16–18] This transition is broadened by the inherent microscopic random quenched disorder associated with doping. Supercooling and superheating were used to identify this field and the temperature-induced transition as first order.[16] In consonance with the idea that a more deeply supercooled state is more susceptible to energy fluctuations, one expects to see enhanced relaxation as one approaches the limits of metastability (supercooling and superheating) in a FOT. Such a giant relaxation was recently observed in doped $CeFe_2$ (Ref. 18) near the limits of both supercooling and superheating. Having already established the phenomenon of supercooling of vortex matter in $CeRu_2$, we now investigate in this paper whether we can see enhanced relaxation in this system of vortex matter as we move further below the equilibrium transition line $[T_C(H)]$ in the H-T space.

It has been pointed out that supercooling of vortex matter is best achieved by the protocol of field cooling.[5,6] Again, it is known that hard superconductors show temporal relaxation whenever an isothermal field change is made. Such relaxation is due to the Lorentz force that acts on the vortices as a result of the field change. It has been shown that the relaxation rate in the absence of a Lorentz force, as in a field-cooled (FC) state, is more than one order of magnitude smaller than when a full critical state is established.[19] The measurement process in a superconducting quantum interference device (SQUID) magnetometer (used for the present measurements) can result in the creation of a partial critical state, and the resulting relaxation might give rise to unknown complication if the relaxation in the FC state is intrinsically small. To eliminate such uncertainty, we measure relaxation always in the full critical state, which is created by applying a suitable isothermal field change (greater than the field for full penetration) after field cooling. If the vortex state created by field cooling is an equilibrium (stable) state, then the observed relaxation would be due to the usual Anderson flux creep. If the FC state is a supercooled state, however, then the relaxation resulting from this metastability should add on to that due to flux creep. In our measurements, we observe a large enhancement of relaxation in M near the PE regime. From our analysis we detect the enhancement in relaxation contributed by the metastability of FC states.We also obtain a clear demarcation regarding the H limits within which metastability (supercooling) is observed.

II. EXPERIMENTS

In the present measurements we have used a $Ce(Ru_{0.95}Nd_{0.05})_2$ sample that has been used in our earlier

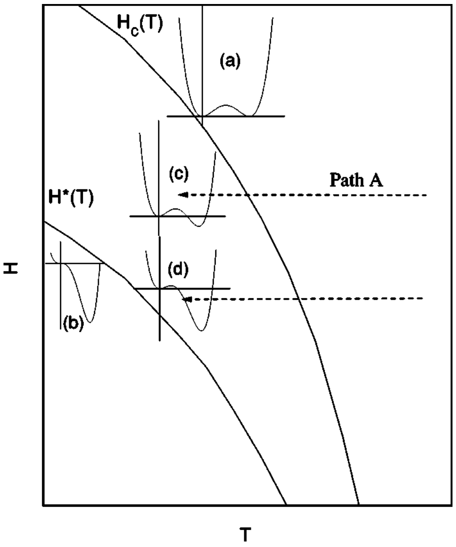

FIG. 1. Schematic depicting the extent of metastable regime. Supercooling (metastability) could be observed in the region bounded by $H_C(T)$ and $H^*(T)$ lines. The four inserted curves show the variation of free energy (along the *y* axis) with order parameter (along the *x* axis), following Landau's theory, (Ref. 24) at different positions in the *H*-*T* plane. The equilibrium situation at the first order transition line $H_C(T)$ is represented by the topmost curve (a). The inserted curve (b) represents the limit of supercooling $H^*(T)$. Curves (c) and (d) depict the extents of supercooling at the end points of the arrow heads, while the dashed lines show the path traversed to prepare the supercooled state.

measurements.[3,4,20] This sample has been used as a representative of the $CeRu_2$ family in many of our earlier measurements as well, since in the pure sample the PE region encompasses both paramagnetic and diamagnetic regimes over a small change in applied magnetic field, and SQUID measurements involving such a small variation in applied field can lead to uncertainties in the crossover regime. In the presently used sample, the PE region which possesses all the characteristics observed in pure $CeRu_2$ (Refs. 3 and 4) is confined to the paramagnetic regime alone. dc magnetization measurements were performed at $T=4.5$ K and $T=5$ K using a Quantum Design MPMS-5 SQUID magnetometer with a scan length of 2 cm. For relaxation studies (at $T=4.5$ K and $T=5$ K) in the disordered state under various extents of supercooling the field H was applied at $T=10$ K, well above T_C(=6.8 K), and the temperature was lowered monotonically to 4.5 K or 5 K, thus following path *A* indicated in Fig. 1. (See the figure caption for details.) For *H* values lying within the schematic $H_C(T)$ and $H^*(T)$ lines in Fig. 1, this prepares the initial supercooled state with lower values of *H*, giving states that are more deeply supercooled. We waited for 1800 s (to exclude any creep in the superconducting magnet) after the temperature was stable at 4.5 K or 5 K and then applied a field change *h* of either sign. The typical values of *h* (20–60 Oe) were selected in such a way that they are greater than the fields for full penetration at all values of *H* studied,[20] and thus a critical state was set up in the entire bulk of the sample.[21,22] Relaxation of magnetization was measured in this critical state for various values of H and for both signs of *h* at each *H*. *M* was measured at 100-s intervals for ~50 min for each value of *H* (for $T=4.5$ K and $T=5$ K) to get a complete idea of the field dependence of relaxation in *M* in the PE regime of $Ce(Ru_{0.95}Nd_{0.05})_2$.

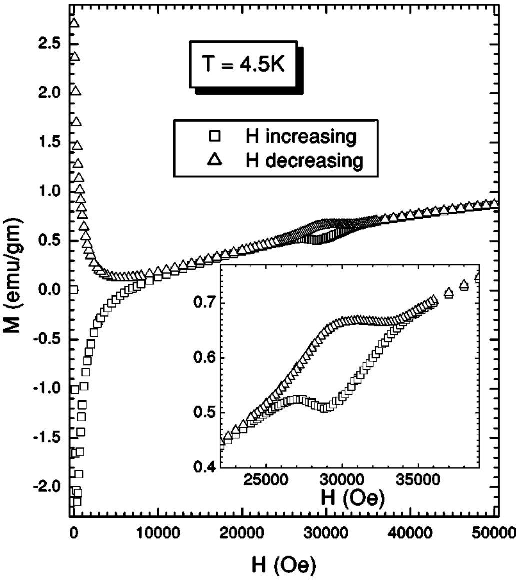

FIG. 2. Isothermal ($T=4.5$ K) variation (starting from the ZFC state) of magnetization with the applied magnetic field in the peak effect regime.

III. RESULTS

We show in Fig. 2 the isothermal *M*-*H* scan at 4.5 K, highlighting the PE regime in the inserted panel. This *M*-*H* scan was started from a zero field-cooled[5] (ZFC) state. One can estimate the J_C of the superconductor from the difference in the values of *M* between *H*-decreasing and *H*-increasing envelopes. We plot this difference as ΔM_{ZFC} against *H* in Fig. 3(b). ΔM_{ZFC} shows a peak at 29.3 kOe in the PE regime, at $T=4.5$ K. In Fig. 3(a) we have shown the values of *M* in the critical state created at 4.5 K after field cooling. The isothermal (starting from ZFC) *M*-*H* curve is also shown along with it, for comparison. The difference in the *M* values for the two signs of *h* provides a measure of J_C in the FC state. Following Steingart *et al.*[23] we define[20] the enhancement factor (ϵ) due to field cooling as $\epsilon(H)=[[\Delta M_{FC}(H)-\Delta M_{ZFC}(H)]/\Delta M_{ZFC}(H)]$, where ΔM_{FC} is the difference in *M* values between the critical states created by $+ve$ and $-ve$ field jerks. [See Fig. 3(a) and 4.] ϵ for 4.5 K is plotted against H in Fig. 3(c), and it shows a peak at 19 kOe. Qualitatively similar results were obtained for $T=5$ K as well. Figure 4 shows the values of *M* in the critical state created at 5 K after field cooling. The isothermal *M*-*H* curve at the same temperature in the PE regime is also shown in Fig. 4. At $T=5$ K, the field variations of ΔM_{ZFC} and ϵ show peaks, respectively, at 22.5 kOe, and 17.5 kOe.[20] The ΔM_{ZFC} vs H and ϵ vs H graphs for $T=5$ K are not shown here for conciseness.

We find from the isothermal *M*-*H* scan following ZFC [Fig. 3(a)] that the extent of the PE regime at $T=4.5$ K is

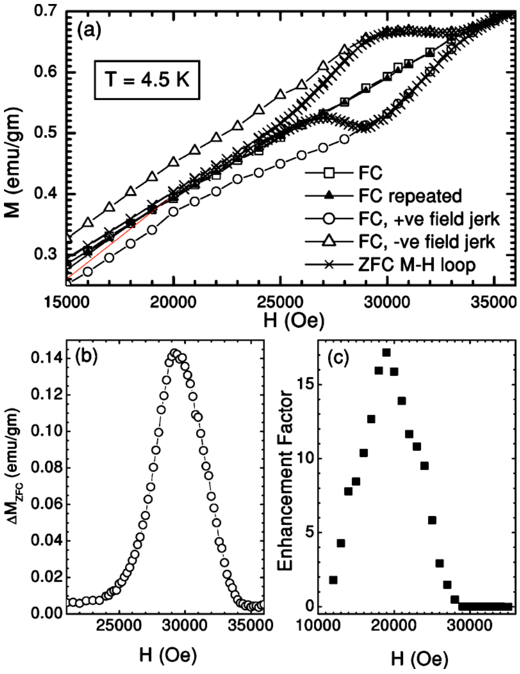

FIG. 3. (a) Variation of magnetization with the applied field in the peak effect regime at $T=4.5$ K. Field jerks of either sign were applied in the FC state to produce the critical state. Since the FC states were prepared separately for each case, two sets of FC data points were obtained. Both sets of FC data are plotted here in order to get a measure of the experimental uncertainty involved in the process. (b) Variation of ΔM_{ZFC} with magnetic field at $T=4.5$ K. (c) Variation of the enhancementfactor ϵ with magnetic field at $T=4.5$ K.

from 23 to 36 kOe approximately. Within the field limit 29 to 36 kOe in the PE regime, the J_C obtained in the isothermal (following ZFC) scan is nearly equal to the J_C obtained in the critical state prepared over the FC state. But below 29 kOe magnetic field, at $T=4.5$ K, the FC critical state J_C registers an enhancement compared to the isothermal ZFC J_C. Similarly, the extent of PE at $T=5$ K is from 18 to 26 kOe. (See Fig. 4 and Ref. 20.) And within the field limit 22.5 to 26 kOe in the PE regime, the J_C obtained in the isothermal (following ZFC) scan is nearly equal to the J_C obtained in the critical state prepared over the FC state; below 22.5 kOe magnetic field, at $T=5$ K, however, the FC critical state J_C is greater in magnitude compared to the isothermal ZFC J_C. We can thus state that J_C in the FC critical state is greater than the isothermal ZFC J_C below the field at which ΔM_{ZFC} exhibits a peak. [See Fig. 3(b) and Ref. 20.] In fact, we observe that J_C in the FC critical state exhibits a rapid enhancement compared to the isothermal J_C at H values that are well outside the PE regime (obtained in the isothermal H scan) both for $T=4.5$ K and $T=5$ K.

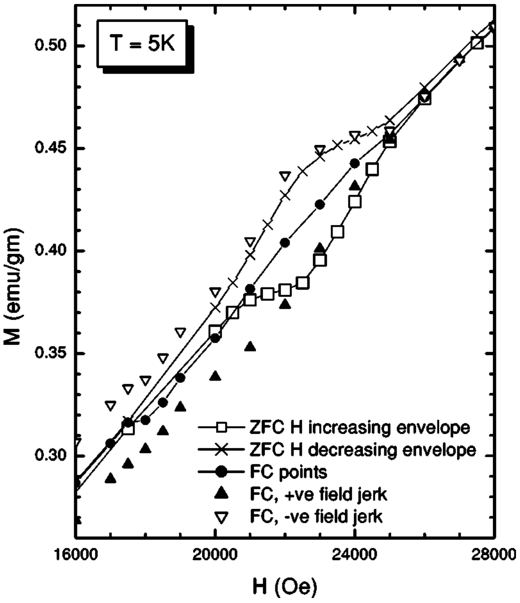

FIG. 4. Variation of magnetization with applied field in the peak effect regime at $T=5$ K.

We now present our relaxation results. Figure 5 shows a few representative curves obtained at 5 K. (See the figure caption for details.) In our experiments, relaxation in magnetization (for $T=4.5$ K and $T=5$ K) is found to be very small in the FC state as compared to that obtained in the critical state prepared by both $+ve$ and $-ve$ field jerks. (All the curves are not shown here for clarity and conciseness.) This is in consonance with Ref. 19. Magnetization is found to vary logarithmically with time (t) in the critical states, as well as in the FC states. In view of the uncertainties (pointed out in the Introduction) involved in the relaxation results in FC state, we would concentrate only in the M vs t data obtained in the full critical state created over the FC states. Relaxation in M shows considerable variation with varying H. To obtain the complete picture regarding the H dependence of relaxation in M, we need to calculate the rates at which M varies with respect to time for all values of H. For the FC states that are stable, relaxation in M in the mixed state of a superconductor is expected due to Anderson creep. Such a creep would be governed by k_BT/U_0, where U_0 is the pinning potential.[21] In the understanding of Anderson creep, magnetization varies logarithmically with time, and this decay rate is also proportional to the magnetization of the critical state. The relevant relaxation rate is thus

$$S = \frac{1}{\Delta M}\frac{dM}{d(\ln t)}, \tag{1}$$

where ΔM is the hysteresis in the isothermal M-H curve, and is proportional to the isothermal J_C (or U_0). Hence, we calculate the relaxation rates (for each value of H and for either signs of h at each H) as

$$S(H) = \frac{1}{\Delta M_{ZFC}(H)}\frac{dM}{d(\ln t)}, \tag{2}$$

where $dM/d(\ln t)$ is a function of both H and h, but is independent of h once h is large enough to establish the full critical state. We plot $S(H)$ with respect to H in Fig. 6 both for $T=4.5$ K and $T=5$ K. The $dM/d(\ln t)$ values are actually negative for $-ve$ h. But here, for clarity, we plot the magnitudes only. It is worthwhile to add here that the relaxation rates plotted in Fig. 6 are actually average values calculated

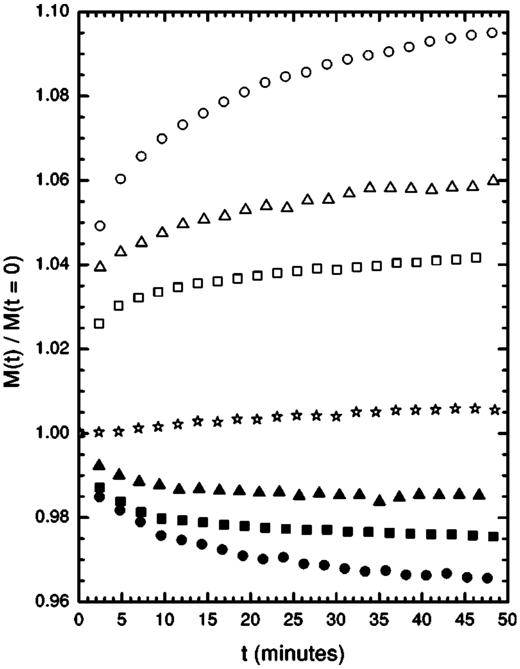

FIG. 5. Selected $M(t)/M(t=0)$ vs time (t) results on $\mathrm{Ce(Ru_{0.95}Nd_{0.05})_2}$ alloy for different constant H values. These relaxation measurements were performed at 5 K. For each H value, $M(t=0)$ is the value of magnetization (emu/gm) when the relaxation measurements are started. $M(t)$ is the magnetization (emu/gm) after an interval t. The stars represent relaxation of field-cooled (in $H=22$ kOe, from 10 K) magnetization. Open squares, open circles, and open triangles, respectively, represent relaxation of magnetization in the critical state obtained after field cooling the sample from 10 K in fields 9 kOe, 18 kOe, and 22 kOe, and then applying a field jerk of +20 Oe in each case. In the supercooled regime (see the Sec. IV), the positive field jerk takes the magnetization to values well below the H increasing envelope magnetization curve. M subsequently tends to return towards the (higher) FC values through relaxation. The filled squares, filled circles, and filled triangles, respectively, represent relaxation of magnetization in the critical state obtained after field cooling the sample from 10 K in fields 9 kOe, 18 kOe, and 22 kOe, and then applying a field jerk of −20 Oe in each case. In the supercooled regime, the negative field jerk takes the magnetization to values well above the H decreasing envelope magnetization curve. M subsequently tends to return towards the (lower) FC values through relaxation.

after repeating the whole set of experiments three times for each value of H and h. The results of the relaxation experiments are summarized in the following text.

(1) The curves for the two different signs of h in Fig. 6 show very small and similar magnitudes of relaxation rates in the high field regime both for $T=4.5$ K and $T=5$ K. S is nearly independent of a variation of H in this regime.

(2) As H is reduced, relaxation rates register rapid rises in magnitude both for $T=4.5$ K and $T=5$ K for either signs of h. The curves show a peak (at ~20 kOe for $T=4.5$ K and ~17.5 kOe for $T=5$ K), and then with further lowering of H the relaxation rates once again fall back to small values that are nearly independent of variation of H.

(3) From Fig. 6 one notices that the relaxation rates are higher at $T=4.5$ K as compared to $T=5$ K.

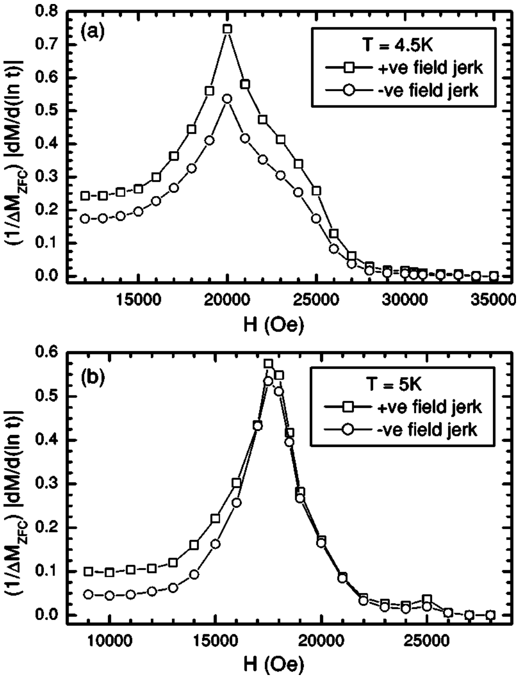

FIG. 6. Relaxation rates of magnetization of $\mathrm{Ce(Ru_{0.95}Nd_{0.05})_2}$ alloy measured at (a) $T=4.5$ K and (b) $T=5$ K in the critical state (see text for details) created in the PE regime.

(4) For both temperatures ($T=4.5$ K and $T=5$ K) the $S(H)$vs H curve for $+ve\ h$ registers higher magnitudes (as compared to $-ve\ h$) for nearly the entire range of H scanned (except the high field regime studied).

IV. DISCUSSION

In the following text, we analyze our results on the basis of standard phenomenology of FOT.[6,7,24]

(1) If the relaxation is contributed only by Anderson creep, then it follows from Eq. (2) of Ref. 21 that the relaxation rate (as defined here) could only show a smooth monotonic variation with the variation of H (depending on the H dependence of J_C). In Fig. 6(a) we see that the relaxation rates for $H \geqslant 29$ kOe and $H \leqslant 15$ kOe for $T=4.5$ K are almost independent of H. Similarly in Fig. 6(b) we see that for $H \geqslant 23$ kOe and $H \leqslant 12$ kOe, S is nearly independent of H. We argue that the relaxations in M in these field regimes are contributed by Anderson creep, but the peak in $S(H)$ cannot be explained by Anderson creep. In our understanding, it is contributed by metastable FC states associated with first order vortex phase transition. This statement is further justified by the arguments that follow.

(2) For both the temperatures $T=4.5$ K and $T=5$ K, the H values corresponding to the peaks in relaxation rates nearly coincide with the fields at which ϵ vs H curves show their respective peaks. Again (both for $T=4.5$ K and $T=5$ K) the values of H at which the relaxation rates start increasing (see the high field regime of the H axis of Fig. 6) with the lowering of H nearly tallies with the H values at which ΔM_{ZFC} vs H curves show their respective peaks. We conjecture that the fields $H \approx 29$ kOe for $T=4.5$ K, and H

≈ 22.5 kOe for $T=5$ K, where ΔM_{ZFC} is maximum, are close to the H_C for the FOT (Ref. 24) between the two vortex phases referred to earlier. The enhancement factor ϵ is negligible in this H regime. The energy barrier separating the high field disordered vortex phase and the low field quasiordered vortex phase is maximum for this field. As H is decreased below H_C, the energy barrier is effectively reduced. Because of thermal fluctuations, portions (heterogeneous nucleation) of the disordered vortex phase can then cross the energy barrier and transform to the quasiordered phase. The remaining fraction of the disordered phase exists in the sample as a supercooled (metastable) phase. Transformation of the metastable (supercooled disordered vortex solid) phase to the stable one contributes to relaxation in M. As H is reduced further below H_C the disordered phase is more deeply supercooled (see Fig. 1), i.e., more and more fractions of the disordered phase have thermal energies greater than the energy barrier's. Hence there are more and more metastable to stable transformations, and as a result there is an enhancement in the relaxation rates. This enhancement in relaxation can go on until the limit of supercooling H^* is reached.[24] We conjecture that H^* for the present $\mathrm{Ce(Ru_{0.95}Nd_{0.05})_2}$ sample is close to the H value where $S(H)$ or (ϵ) registers a peak. Since there is no metastable phase at fields lower than H^*, the relaxation rates register a drop in magnitude until they fall back to their values characteristic of Anderson creep in the mixed state of the superconductor.

(3) The peaks in relaxation rates and ϵ for both $T=4.5$ K and $T=5$ K are observed in the H regime where the signature of PE in the isothermal (starting from ZFC) M-H scan is not even initiated. We argue that this is a result of supercooling. In the FC measurements the high- J_C disordered vortex phase is supercooled to fields much lower than the fields at which ΔM_{ZFC} registers peaks for both $T=4.5$ K and $T=5$ K. ΔM_{ZFC} drops sharply with lowering of H in this regime,while the FC J_C does not. Hence, from the definition of ϵ,[23] it is quite obvious that the enhancement factor would register a rise in this field regime with lowering of H. As H is reduced below H^*, the FC J_C would drop as there is no supercooled high- J_C phase left in the sample in this field regime.

(4) In the present interpretation of the relaxation results in terms of H_C and H^*, the relaxation rates at H values below the peak should fall abruptly. The finite width in H [$\sim$5 kOe, both for $T=4.5$ K and $T=5$ K (see Fig. 6)] over which $S(H)$ falls with lowering of H could be because of a broadened transition, where we see a distribution of H_C and H^* over the sample. In samples of nonzero J_C, the local fields are not same as the applied H. This, along with the effects of quenched disorder[25] due to alloying, might as well result in broadened bands for H_C and H^*.

(5) Higher magnitudes of relaxation rates for *+ve* field jerks (Fig. 6) probably hint towards an asymmetry in the transition. The asymmetry is also indicated in Fig. 5. While we understand that this asymmetry is different from the phenomenological asymmetry observed in a FOT,[18] its source is not quite clear at the moment.

The giant enhancement in the relaxation rate in the present alloy originates from the metastability of states. Such a signature of metastability observed over the wide H regime, and the facts considered in point (4) in the discussion above clearly indicate that the quasiorder to disorder transition in vortex matter in the present alloy is a disorder-broadened FOT. This further justifies our conjecture that the FOT in the present material is initiated through heterogeneous nucleation. Kalisky *et al.*,[26] have shown that transient vortex states give rise to nonequilibrium order-disorder vortex transition in Bi-2212 single crystals. They assumed heterogeneous nucleation and dynamic coexistence of stable and unstable vortex phases,[27] and that the two coexisting phases are separated by a sharp interface that exhibits a nontrivial front motion depending on the rate of change of the external field, induction at the front, temperature, and annealing time.[28] We argue that this annealing time and its temperature and field dependence cannot be compared with those of the relaxation time periods of our magnetization data because of the following reasons.

(i) The target fields for our relaxation experiments are applied when the sample is in the normal state (above T_C). Sufficient delay is allowed (while field cooling and waiting in the FC state, see Sec. II) to exclude any effect due to creep in the magnet. A field change of $20-60$ Oe is then needed to create the full critical state. In an MPMS-5 magnetometer, in the no-overshoot mode, this takes a couple of seconds. The magnetometer then waits for 15 s before declaring the field stable. The actual magnetization measurements took $27-46$ s more in the configuration used in the present experiments. After the first magnetization measurement in each relaxation experiment, the subsequent magnetization measurements were taken with a pause time of 100 s—excluding the time consumed in each magnetization measurement. The annealing times mentioned by Kalisky *et al.*[26,28] are much smaller than all these time scales. Therefore, the formation of transient states, if any, was not observable in our experiments.

(ii) The typical time periods of our relaxation data are several orders of magnitude higher than the annealing time expected according to the work done by Kalisky and her co-workers. Annealing time in Kalisky's work is mainly relevant to the H-T regime where the system approaches the equilibrium phase boundary. The present paper deals with the system when the disordered phase is supercooled below the equilibrium phase boundary in the H-T phase space. (Refer to Fig. 1.)

Heterogeneous nucleation and phase coexistence in a macroscopic scale has been shown in a $2H$-$\mathrm{NbSe_2}$ single crystal,[29] in the PE regime, using scanning Hall probe microscopy. The ordered phase was found to nucleate and grow in isolated regions inside the sample. Recently we have mapped a first order magnetic transition in a doped $\mathrm{CeFe_2}$ alloy through the Hall imaging technique. We have actually observed heterogeneous nucleation and phase coexistence across the FOT, without any single sharp front separating ordered and disordered magnetic phases. Instead of the motion of a single sharp front, what we have observed is nucleation and growth of the new phase in pockets and their subsequent coalescence beyond certain values of the control variables (H,T).[30] We also have similar Hall imaging data in the first order magnetic transition in a $\mathrm{Gd_5Ge_4}$ compound. We believe that such a heterogeneous nucleation is also possible in the present Nd-doped $\mathrm{CeRu_2}$ alloy. The formation of a single sharp front and its subsequent motion across the sample is not an absolutely essential requirement for the FOT. Finally, we would like to state that the magnetic relaxation in the present sample does not have any appreciable dependence on surface contamination. This has been explicitly shown in our previous work on this same sample.[31]

V. SUMMARY AND CONCLUSIONS

In summary, we have measured relaxation in magnetization of metastable states in a Ce$(\mathrm{Ru}_{0.95}\mathrm{Nd}_{0.05})_2$ alloy. The metastable states were prepared by cooling the alloy sample from a temperature well above the T_C of the material, in the presence of different magnetic fields of magnitudes in and around the peak effect regime. This was followed by the application of field jerks of a few tens of Oe of either sign so as to prepare a critical state in the entire sample, over the FC states. Based on the understanding of Anderson creep of vortices in a mixed state of a superconductor, we devised a method to separate the relaxation in magnetization due to supercooling of the disordered vortex phase from the relaxation resulting from creep. We have observed a large enhancement of relaxation in magnetization near the field limit of supercooling. Interesting variations of the relaxation rates with respect to the fields applied for producing the FC states are explained on the basis of the standard phenomenology of first order phase transition, in terms of supercooling of the disordered vortex phase.

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