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Paramagnetic Meissner effect, two-step peak-effect anomaly, and vortex phase diagram in the non-centrosymmetric superconductor Ru_7B_3

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Abstract

The non-centrosymmetric superconductors (NCSs) continue to draw significant attention owing to their exotic superconducting properties. Here, we present the results of our detailed magnetization and electrical resistivity measurements on a single crystal of the NCSs Ru_7B_3 . We observe a paramagnetic Meissner effect (PME) in the temperature-dependent field-cooled magnetization measurements ($M_{\text{FCC}}(T)$) in small magnetic fields (< 200 Oe) just below the superconducting transition temperature $T_c \sim 2.68(1)$ K. On further cooling, the positive $M_{\text{FCC}}(T)$ values cross over to the diamagnetic region indicating the usual Abrikosov lattice at lower temperatures. Across the temperature range of the PME, $2.6 \text{ K} < T < T_c$, the dc magnetization hysteresis loops ($M - H$) exhibit an unusual asymmetry indicating a deviation from the conventional type-II superconducting behavior and the occurrence of an unconventional vortex state. We associate the occurrence of PME in the present case to trapping of magnetic flux within the bulk of the sample and its subsequent compression on further cooling. The ac magnetic susceptibility data ($\chi'(H, T)$) exhibits another well-documented anomaly, namely the peak-effect (PE) transition close to the superconducting-normal phase boundary. Above a certain field (> 3.5 kOe), the PE transition splits into two well-resolved dips in $\chi'(H, T)$ which is suggestive of a step-wise amorphization of the vortex matter. We ascribe the split PE anomaly in Ru_7B_3 to the strong pinning mechanism associated with the unconventional vortex dynamics in the NCS superconductors. Both PE and the PME have been found to be robust features in Ru_7B_3 as they are observed for different crystal orientations with respect to the external field ($H \parallel [001]$ and $H \parallel [100]$). We present the vortex phase diagram of this superconductor displaying the phase boundaries of the split PE feature.

1. Introduction

One of the hallmarks of a superconductor is the ability to expel the magnetic field from its interior when it is cooled in small fields below its superconducting transition temperature T_c , an effect well known as the (diamagnetic) Meissner effect. In contrast to this usual behavior, it has also been seen that some superconductors show an unusual paramagnetic magnetization signal below T_c . This counter-intuitive phenomenon is called the paramagnetic Meissner effect (PME) or Wohleben effect [1]. Initially, the PME was observed in some high- T_c cuprate superconductors, such as $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ [2, 3] and $\text{YBa}_2\text{Cu}_3\text{O}_7$ [4–6] which had led to the proposition that the d -wave superconductivity may be the cause of the PME [7–9]. However, later the PME was also observed in a conventional s -wave low- T_c superconductor Nb which was suggestive of a more generic origin [10]. To understand the cause of the

PME, Koshelev and Larkin proposed a model which explains the occurrence of the PME due to the trapping of magnetic flux within the bulk of the superconductor due to inhomogeneous cooling [11]. Within the framework of the Ginzburg–Landau (GL) theory, the occurrence of the PME has been anticipated in mesoscopic-sized superconducting specimens due to the occurrence of a giant vortex state with multiple flux-quanta ($L\phi_0$, $\phi_0 = hc/2e$, $L > 1$) in the domain of surface superconductivity [12, 13]. The PME can also arise from the presence of odd-frequency pairing (OFP) states [14, 15] that emerge in a superconducting phase with broken time-reversal symmetry (TRS), as reported in certain superconducting-magnet and normal-metal–superconductor hetero-structures [14, 16, 17] as well as in multi-band superconductors [15, 18]. To date, the PME has been observed in a variety of superconductors, such as Nb [19–22], $2H-NbSe_2$ [23], $Ca_3Rh_4Sn_{13}$ [24], $Ca_3Ir_4Sn_{13}$ [25], $4H_a-NbSe_2$ [26], and other superconductors [14–18, 27–32].

Apart from the PME, another well-documented anomaly which has been commonly seen in a variety of superconductors is the peak effect (PE), which is related to an anomalous enhancement of the critical current density ($j_c(H, T)$) with field/temperature variation occurring at the edge of the superconducting-normal state transition [33]. The PE marks a faster collapse of the elasticity of the flux line lattice than the pinning strength [33] and is related to a transition from an ordered vortex lattice to an amorphous vortex solid [34]. The PE primarily depends on the amount of quenched random disorder [35, 36], a cleaner crystal usually depicts a pronounced and sharp PE feature. A plethora of research activities related to the novel features pertaining to the PE transition have been carried out over the past few decades which makes it an interesting subject matter of research [37–47] and the same continues to draw attention more recently [48–51].

Despite being so extensively studied in a variety of superconductors, both PE as well as PME have been rarely studied in the non-centrosymmetric superconductor (NCS) superconductors. To the best of our knowledge, amongst the NCS superconductors, the PE has been reported in a polycrystalline sample of Re_6Zr [52] while the PME has been known to occur in polycrystalline of $4H-NbSe_2$ [26]. However, whether there is any possible connection between NCS and the occurrence of the PE and PME is still lacking. Notably, in NCSs and in superconductors with broken TRS, the vortex dynamics exhibits behavior distinct from that of conventional superconductors. In such superconductors additional intrinsic sources of pinning arises from the domain walls which may play an important role in vortex motion [53–58]. Owing to lack of detailed theoretical and experimental studies of vortex dynamics in the NCS superconductors, further studies are required to fully understand the pinning mechanism in these important class of materials, particularly the possible role of intrinsic pinning in the occurrence of the PE in NCS systems.

Ru_7B_3 is a low- T_c (2.6–3.3 K) NCS superconductor which crystallizes in a hexagonal structure with space group $P6_3mc$ [59–64]. This compound has been in focus recently [65–68] for various studies including- (i) investigation of its superconducting properties [61, 62], (ii) observation of magnetic field-driven rotating vortex lattice accompanied by a pronounced hysteresis as evident in the results of small angle neutron scattering (SANS) [65, 68], (iii) presence of spontaneous magnetization arises from broken TRS [64], and (iv) singlet-triplet mixing in the superconducting order parameter [66, 67]. Amongst these, the vortex study by Cameron *et al* [65, 68] has shown interesting rotation of the vortex lattice where the observation was interpreted using a model based on a G-L framework. The study did not reveal any vortex lattice structural transition across the field-temperature phase-space ($T \sim 55$ mK, 1 K and $H > 20$ kOe) wherein they performed the SANS measurements. Whether this material exhibits an order-disorder transition (ODT) in the vortex matter is a challenging problem considering that the single crystals of this compound are disordered (residual resistivity ratio (RRR) defined as ρ_{300K}/ρ_{3K} , $RRR \sim 3.66$, as shown below) in comparison to the conventional superconductors, say $2H-NbSe_2$ [45] or $Ca_3Rh_4Sn_{13}$ [43] whose single-crystals are typically clean ($RRR \sim 120$ for $2H-NbSe_2$ [69]) and therefore, they display sharp well resolved PE transitions in both dc magnetization hysteresis loops ($M-H$) and ac magnetic susceptibility measurements $\chi'(H, T)$. In a disordered system containing significantly higher concentration of impurities or quenched random disorder, the PE transition may not be easily discernible in the magnetization measurements leading to difficulty in identifying the ODTs in the vortex matter [70]. Motivated by this, we have investigated a single crystal of the NCS superconductor Ru_7B_3 using detailed magnetization and electrical resistivity measurements. Our results of magnetization measurements have revealed the above-mentioned anomalies- (i) the occurrence of PME in low magnetic fields ($H < 200$ Oe) in the field-cooled dc magnetization measurements, $M(T)$. The isothermal $M-H$ scans in the domain of the PME deviates from the typical type-II superconducting behavior. (ii) The results of ac magnetic susceptibility measurements reveal a two-step PE feature at the edge of the superconducting-normal transition which is indicative of a step-wise amorphization of the vortex matter. The characteristic step PE in the NCSs Ru_7B_3 echoes very well with similar PE feature reported earlier

in a conventional low- T_c superconductor $2H\text{-NbSe}_2$ [45]. We ascribe the occurrence of PE in Ru_7B_3 to the pinning mechanism associated with non-centrosymmetric nature of the compound. In the end, we provide a vortex phase diagram of Ru_7B_3 marking the locus of the PE phase boundaries in the phase diagram of this compound.

2. Experimental details

The single crystals of Ru_7B_3 were grown by the optical floating zone technique at the University of Warwick, as described in detail in the [63]. X-ray Laue diffraction technique was utilized to orient the single crystals. After orientation, the crystals were cut along the principle crystallographic directions using a spark erosion cutter. A cuboidal-shaped crystal with dimensions $1.58 \times 1.45 \times 1.08 \text{ mm}^3$ was used for the magnetization measurements in a magnetic property measurements system Superconducting Quantum Interference Device Vibrating Sample Magnetometer (SQUID-VSM, Quantum Design, USA). The crystal geometry is such that the crystallographic [100] direction corresponds to the longest dimension while the [001] direction points along the width of the sample. For dc measurements, the amplitude of vibration of the sample was maintained at 2 mm. The magnetic field-inhomogeneity over the scan length of 8 mm is estimated to be small (10^{-2} Oe) [23] and therefore, the possibility of any artefact in the present measurements that may occur due to the field-inhomogeneity are negligible. We also repeated the measurements while varying the amplitude of vibration from 0.5 mm to 3 mm and did not observe any significant differences in the response showing that the field-inhomogeneity is negligibly small. These results confirm that the observations made for Ru_7B_3 are not due to any magnetic field inhomogeneity. The ac magnetic susceptibility measurements were performed in the presence of an ac driving field of magnitude $h_{ac} = 0.5$ Oe and a frequency $f = 211$ Hz. The ac field drive was superimposed on various dc magnetic fields applied in the different $\chi'(T)$ and $\chi'(H)$ scans. A platelet-shaped crystal of dimensions $3.14 \times 2.16 \times 0.5 \text{ mm}^3$ was utilized for the ac electrical resistivity measurements using the standard four-probe method using a Physical Property Measurement System (PPMS, Quantum Design, USA). The magnitude of the ac current drive (I_{ac}) was chosen to be 5 mA and the ac driving frequency was maintained at 17 Hz.

3. Experimental results

3.1. Electrical resistivity data of Ru_7B_3

The inset panel of figure 1 presents the temperature dependence of the resistivity recorded from 300 K down to 1.8 K in (nominal) zero field along $H||[1\bar{2}0]$. A sharp drop in resistivity below 2.8 K is clearly observed which is identified as the superconducting transition. A magnified view of this transition has been depicted in the main panel of figure 1. Using the 50% criterion, the superconducting transition temperature was estimated to be $T_c = 2.61(4)$ K marked in figure 1. The transition width $\Delta T_c \sim 0.08$ K was evaluated from the relation $\Delta T_c = T_{90\%} - T_{10\%}$, where $T_{90\%}$ and $T_{10\%}$ are the temperatures at which the resistivity drops by 90% and 10%, respectively. The RRR = $\rho(300 \text{ K})/\rho(3 \text{ K})$ is found to be approximately 3.66 which is closely matching with the RRR value reported earlier for this compound [62].

3.2. PME in Ru_7B_3

Figure 2(a) shows the ac magnetic susceptibility response $\chi'(T)$ as a function of temperature for Ru_7B_3 from 1.8 to 2.9 K. These data were recorded while warming the sample in (nominal) zero dc magnetic field and in an ac magnetic field of amplitude $h_{ac} = 0.5$ Oe with a frequency $f = 211$ Hz. A superconducting transition is well apparent with transition temperature $T_c(0) = 2.68(1)$ K for $H||[001]$, which agrees well with the values reported for the single crystals of Ru_7B_3 [62, 63]. The zero field $\chi''(T)$ data is also shown in the inset of figure 2(a) marking the T_c at 2.68 (1) K. For $H||[100]$, $T_c(0) = 2.71(1)$ K was obtained (the corresponding plot is not shown here). A slight variation in T_c is observed for different crystallographic orientations. Note that the T_c in $\chi'(T)$ data (figure 2(a)) is identified as the onset of diamagnetic drop whereas the same is identified using the 50% criteria in the resistivity data (figure 1). Given the broadening of the superconducting transition in both $\rho(T)$ and $\chi'(T)$ data, the small difference in the T_c measured in the two techniques is due to the different criteria used and of the different crystal orientations of the samples. It is worth mentioning here that the two pieces of crystals utilized in resistivity and magnetization measurements are originated from the same

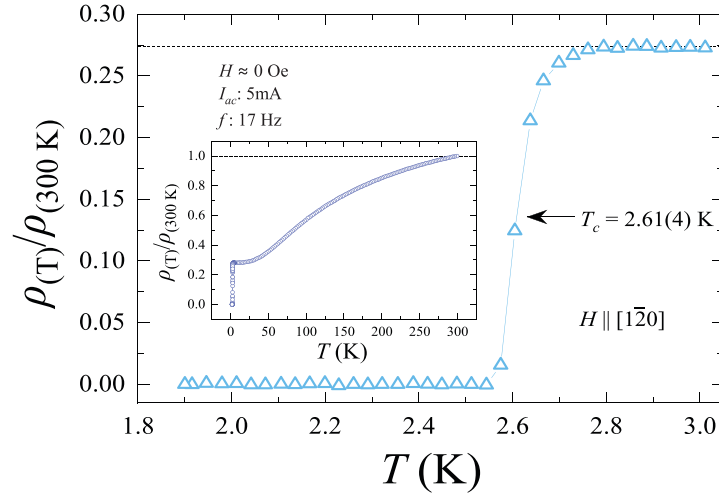


Figure 1. Temperature dependence of electrical resistivity measured in the temperature range $1.9 < T < 3$ K at $H = 0$ Oe. The inset shows the resistivity data for a temperature range $1.9 < T < 300$ K.

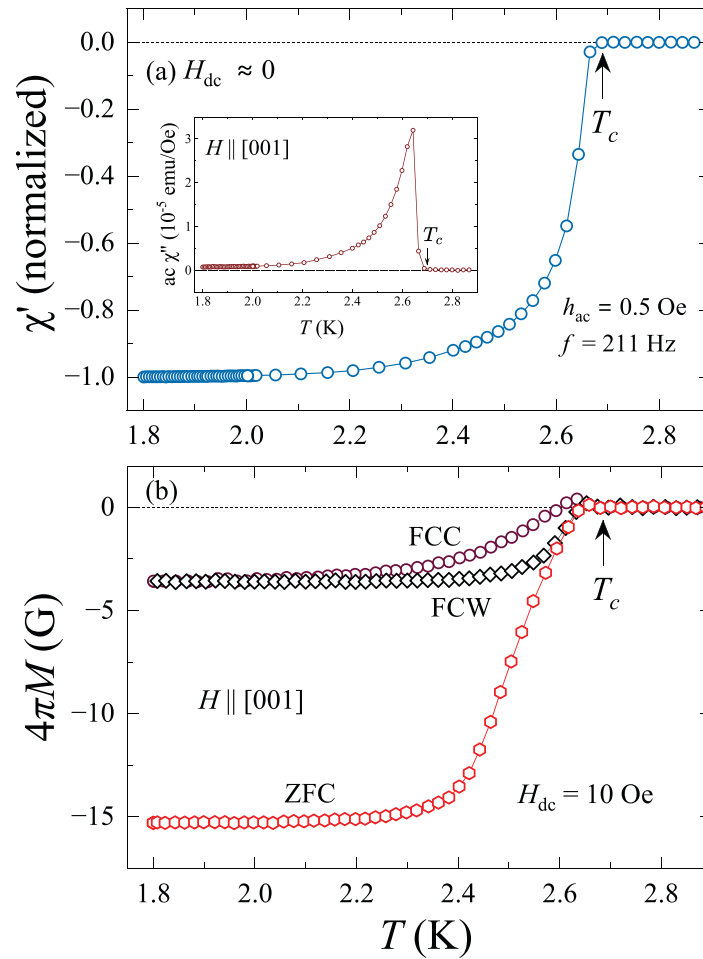


Figure 2. (a) Temperature dependency of real part of ac magnetic susceptibility $\chi'(T)$ and the inset shows the imaginary part of ac magnetic susceptibility $\chi''(T)$. (b) Variation of magnetization with temperature along [001] axis measured in ZFC, FCC and FCW mode.

batch of crystals. Note that a slightly higher value of T_c ($= 3.3$ K) has been reported for the polycrystalline samples of Ru_7B_3 [61]. Such variations in T_c may be attributed to differences in the boron content [71, 72].

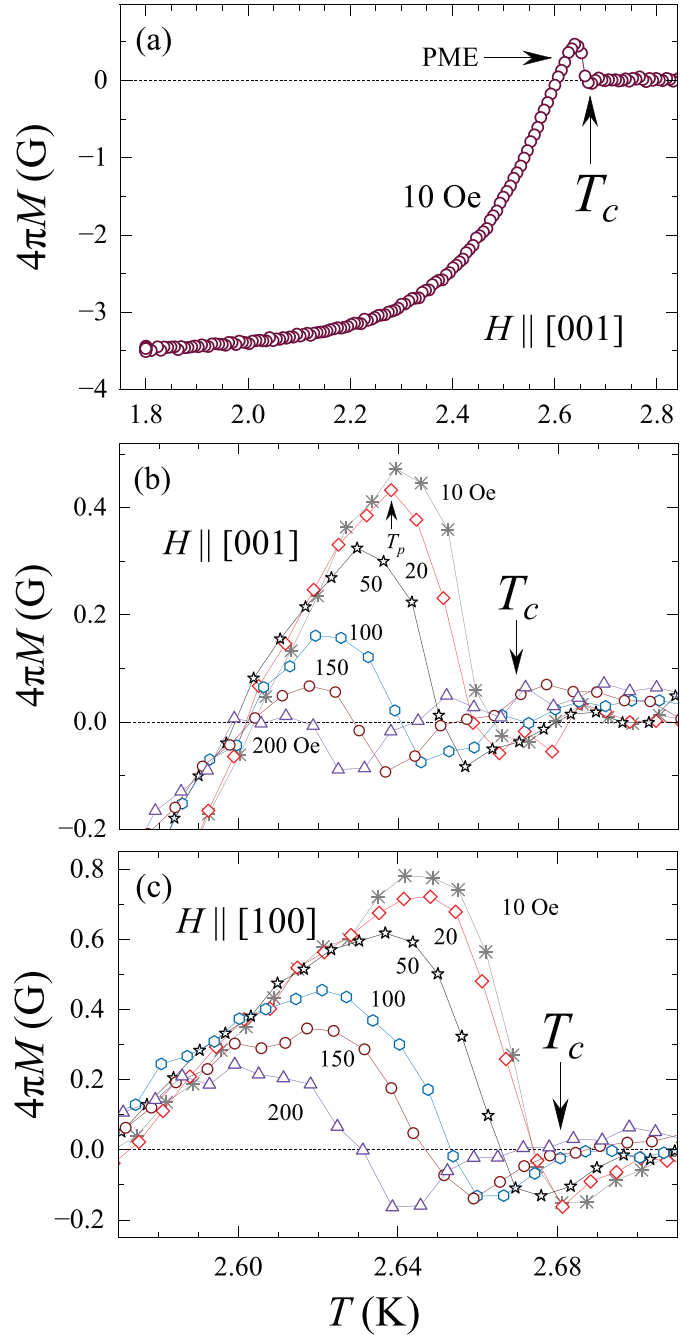


Figure 3. Manifestation of PME observed in $M_{\text{FCC}}(T)$ response (a) at $H = 10$ Oe along $H \parallel [001]$. Magnified view of the PME observed near T_c for the field range of $10 < H < 200$ Oe along (b) $H \parallel [001]$ and (c) $H \parallel [100]$ directions, respectively.

The superconducting transition is also evident in the temperature-dependent dc magnetization data $M(T)$ recorded in three modes: zero-field cooled (ZFC), field-cooled cool down (FCC) and field-cooled warm up (FCW), as shown in figure 2(b). The data were recorded in presence of a magnetic field $H = 10$ Oe applied along the $[001]$ direction. The ZFC magnetization $M_{\text{ZFC}}(T)$ exceeds full Meissner fraction (100%) which is due to the demagnetization factor. The field-cooled curves (FCC and FCW) saturate at lower temperatures below about 2.2 K. While the overall $M(T)$ response depicts a typical superconducting behavior, an anomalous positive magnetization in $M(T)$ has been observed close to T_c . Here, T_c is identified as the onset of the positive magnetization as discernible from figure 3(a). To elaborate this feature, we have shown the $M_{\text{FCC}}(T)$ curve in $H = 10$ Oe separately in figure 3(a), which shows an unusual paramagnetic peak in $M_{\text{FCC}}(T)$ in the superconducting state followed by a crossing over to diamagnetic values on further lowering of temperature. This is a clear manifestation of the PME known to occur in some high- T_c cuprate superconductors [2, 4] and in some conventional superconductors

[10, 23–25]. The PME was not reported in previous studies related to Ru_7B_3 [62, 63]. It is worth mentioning that the PME is highly sensitive to sample geometry [28], surface morphology [27, 30] and measurement conditions (specially on the cooling rate) [29]. Some studies also suggest that not all samples exhibit PME even with the same nominal composition [73]. This may explain why PME was not observed in the dc magnetization data reported in previous studies on Ru_7B_3 . The crystals utilized in our present studies belong to a different batch of crystal than that studied in [63].

Figure 3(b) shows the $M_{\text{FCC}}(T)$ in the vicinity of T_c for various magnetic fields, as indicated. As the applied field is sequentially increased from 10 Oe, the height of the paramagnetic peak in $M_{\text{FCC}}(T)$ decreases and eventually, the PME is suppressed in $H = 200$ Oe. The paramagnetic peak shifts to lower temperatures as the applied field increases. We also observe the PME for field applied along different directions, as shown for $H \parallel [100]$ in figure 3(c). Here we also observe a paramagnetic signal below T_c implying that the PME is not dependent on the orientation of field with respect to the crystallographic directions. There is very little difference between the $M_{\text{FCC}}(T)$ data in the two orientations except that the crossover from para to diamagnetic region occurs at slightly lower temperatures for $H \parallel [100]$ and thus the temperature range encompassing the PME is slightly broader for this orientation. The magnetization values across the PME remains almost the same in the two orientations for similar field values.

Another noteworthy feature that is seen in the $M_{\text{FCC}}(T)$ runs is the occurrence of an oscillatory behavior. For example, the $M_{\text{FCC}}(T)$ curves for $H = 100, 150$ and 200 Oe exhibit some characteristic undulations with the magnetization switching between paramagnetic and diamagnetic values as the sample is cooled below T_c . This oscillatory feature has also been observed across the regime of PME in some conventional low- T_c superconductors, such as, Nb [10], NbSe_2 [23], $\text{Ca}_3\text{Rh}_4\text{Sn}_{13}$ [24] and $\text{Ca}_3\text{Ir}_4\text{Sn}_{13}$ [25].

Based on the observation of the PME, it is now tempting to investigate the isothermal dc magnetization $M-H$ curves in the vicinity of T_c . In figure 4 we show the $M-H$ loops obtained at various temperatures. For each $M-H$ curve, the sample was cooled from the normal state (above T_c) to the target temperature in zero field and the magnetization was recorded while ramping the field to higher positive and negative values in a cyclic manner (four quadrants). The main panel of figure 4(a) shows a portion of the $M-H$ curve at $T = 1.8$ K for the field range, $-300 < H < 300$ Oe, demonstrating features typically expected for a type-II superconducting behavior. The inset in figure 4(a) shows a full view of this curve which is found to be mostly reversible with the forward and reverse legs of the $M-H$ curve almost coinciding with each other, suggesting a weak pinning strength for the crystal (very low j_c). On approaching the superconducting-normal transition, the $M-H$ loop, for example, at 2.62 K in figure 4(b) shows an unusual asymmetry as the magnetization curve for positive and negative fields are not the mirror image of each other. At a slightly higher temperature $T = 2.65$ K, where the peak of the paramagnetic signal was observed in $M_{\text{FCC}}(T)$, we observe that the $M-H$ curve (figure 4(c)) exhibits a positive magnetization near $H = 0$. The magnetization values decrease as the field is increased in either positive or negative directions. The peak height decreases on approaching closer to T_c , see for example, the $M-H$ curve at 2.67 K (figure 4(d)). For comparison, we also show the normal state $M-H$ data in the panels (figures 4(b)–(d)) which is found to be linear, as expected. Clearly, the features seen in the $M-H$ data close to the superconducting-normal transition (figures 4(b)–(d)) do not reflect a type-II superconducting behavior.

The evolution of $M-H$ curves with temperature from the normal state to PME region and thereafter to the usual diamagnetic region can be correlated with the $M_{\text{FCC}}(T)$ curve at a lower field say $H = 10$ Oe as shown as inset of figure 4(b). The $M-H$ at a temperature of 2.67 K close to $T_c(0)$ exhibits a positive peak at $H = 0$ which diminishes as the field is increased to positive or negative values (see figure 4(d)). On further cooling to 2.65 K (encircled in figure 4(b) 2.65 K), a temperature just above the peak temperature of PME the positive peak in $M-H$ increases. Such an increase is consistent with the increase in positive $M_{\text{FCC}}(T)$ values in this region of PME. Below the peak temperature (T_p) of PME, for example, at $T = 2.62$ K (main panel of figure 4(b)), the $M-H$ curves retain the asymmetry. Eventually, the $M-H$ loop at temperature far below the crossover temperature (T_{cross}) (marked in the inset of figure 4(b)) exhibits a type-II superconducting response showing a clear symmetry. Such a usual MH response is also anticipated based on the observation of a usual diamagnetic $M_{\text{FCC}}(T)$ behavior in this region (see figure 3(a)). The observed asymmetry in the $M-H$ curves and the occurrence of a positive magnetization at zero field bear similarities with the features reported for the conventional low- T_c superconductors, $\text{Ca}_3\text{Rh}_4\text{Sn}_{13}$ [24], and $\text{Ca}_3\text{Ir}_4\text{Sn}_{13}$ [25] where it has been associated with a giant vortex state. These observations suggest the occurrence of some unconventional vortex state across the regime of PME which is not anticipated for a conventional type-II superconductor.

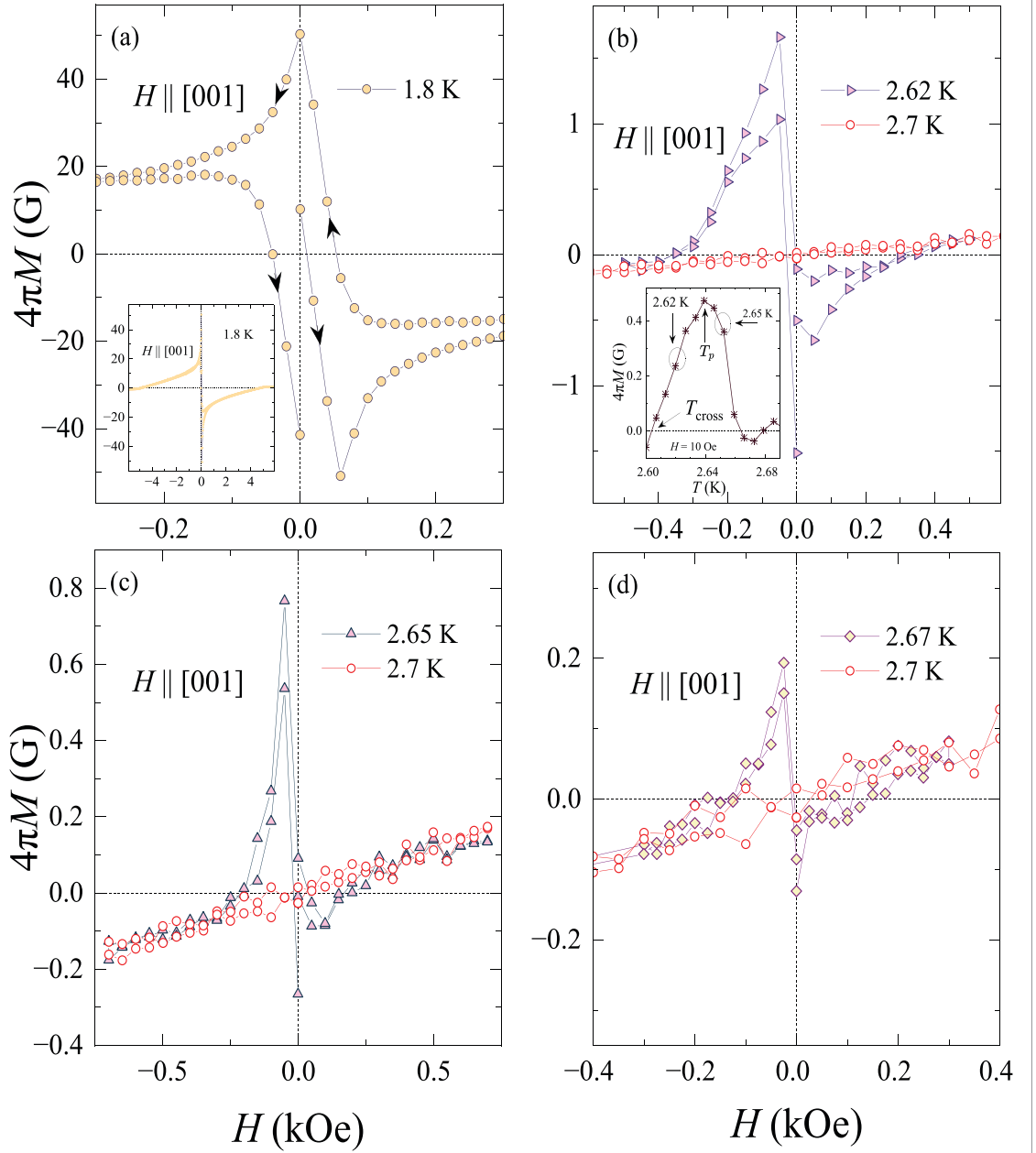


Figure 4. dc $M-H$ curve along $H \parallel [001]$ shown in panels (a)–(d). (a) $M-H$ curve at 1.8 K for typical type-II superconductor. $M-H$ curve at (b) 2.62 K (inset showing PME at $H = 10$ Oe where T_p and T_{cross} are marked), (c) 2.65 K and (d) 2.67 K showing unusual asymmetric behavior.

3.3. Two-step peak effect anomaly in Ru_7B_3

To explore the bulk pinning properties of Ru_7B_3 , we have recorded the ac magnetic susceptibility data at different magnetic fields. Figure 5 shows the real part of ac susceptibility $\chi'(T)$ as a function of temperature. The data were normalized with respect to the $\chi'(T)$ value at the lowest recordable temperature 1.8 K as shown for $H_{\text{dc}} = 2.5$ kOe in the inset panel of figure 5(a). As the temperature was swept from 1.8 K to higher values the magnitude of $\chi'(T)$ decreases. We recall here that the real part of ac susceptibility relates to the critical current density j_c by the following relations-

$$\chi' \sim -1 + \alpha \frac{h_{\text{ac}}}{j_c} \quad \text{for } h_{\text{ac}} < h^* \quad (1)$$

$$\chi' \sim -\beta \frac{j_c}{h_{\text{ac}}} \quad \text{for } h_{\text{ac}} > h^* \quad (2)$$

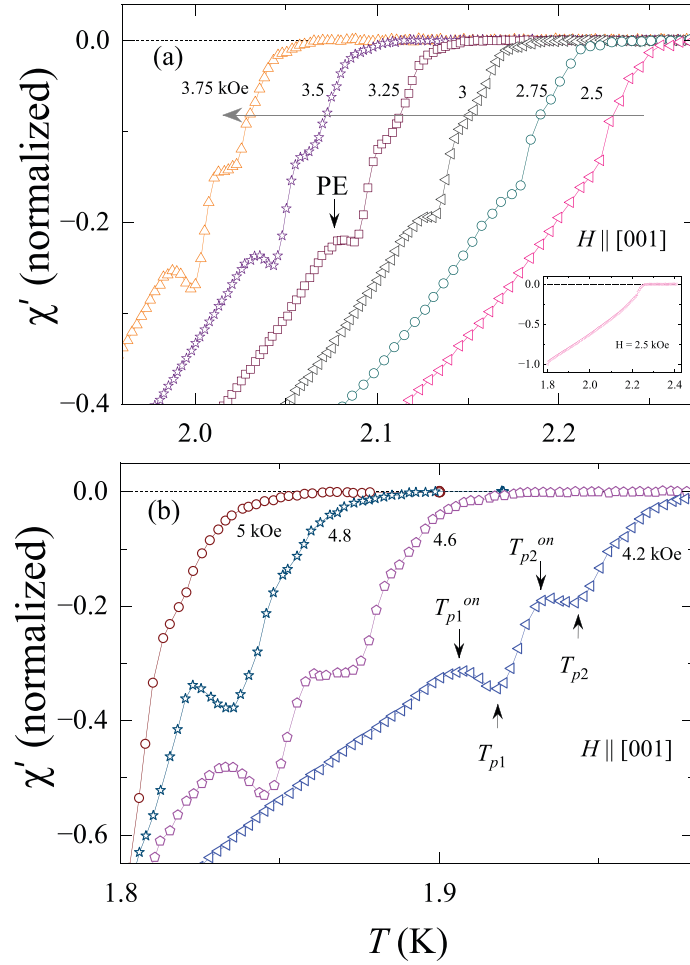


Figure 5. Variation of iso-field ac magnetic susceptibility $\chi'(T)$ for the field applied along $H \parallel [001]$ (a) $\chi'(T)$ data for fields in the range $2.5 < H < 3.75$ kOe. The inset shows the full view of $\chi'(T)$ curve in the temperature range $1.8 < T < 2.4$ K at $H = 2.5$ kOe. (b) $\chi'(T)$ data for fields in the range $4.2 < H < 5$ kOe, with the onset and peak positions of the split-peak effect marked for $H = 4.2$ kOe.

where, h^* is the ac field for full penetration in the sample and α, β are sample geometry dependent factor [74]. The observed fall in $|\chi'|$ with temperature (inset panel of figure 5(a)) implying a monotonic fall in j_c in accordance with eqs (1) and (2), is the usual scenario expected for a superconductor. However, a kink is observed in this curve, a magnified view of which can be seen in the main panel of figure 5(a). At higher fields, we note that the kink feature evolves into a dip in $\chi'(T)$, see for example, the $\chi'(T)$ data at $H = 3.5$ kOe. Such a dip feature reflects an unusual increase in the monotonically decreasing critical current density, j_c prior to the superconducting-normal transition which is the manifestation of the well documented PE anomaly [33, 34, 43–47]. Interestingly, at a higher field $H = 3.75$ kOe, we observe an additional kink above the PE (marked by an arrow) which evolves into another dip at higher fields as can be seen for $H = 4.2$ kOe and above (figure 5(b)). The two dips can clearly be seen for $H = 4.2$ kOe centered around $T_{p1} = 1.92$ K and $T_{p2} = 1.94$ K, respectively. The onset positions of the two dips (T_{p1}^{on} and T_{p2}^{on}) have also been marked for $H = 4.2$ kOe in figure 5(b). At $H = 4.8$ kOe and above, we could observe only one of the peaks and the other peak may have shifted below the lowest recordable temperature, 1.8 K. Considering that the PE marks the collapse of elasticity of the vortex matter [33], the observed two-peak PE anomaly in Ru_7B_3 is suggestive of a step-wise amorphization of the vortex matter [45].

Further evidence of the two-peak PE feature can be seen in the ac χ' vs H plots at various constant temperatures, as shown in figure 6(a). At each temperature, the sample was cooled in zero field to the target temperature, and the $\chi'(H)$ data were recorded while ramping the field to higher values. At lower temperature sides, a well-resolved two-peak PE anomaly is clearly observed. As the temperature increases from 1.8 to 2.2 K, the two-peak feature evolves into a composite kink feature, a behavior consistent with evolution of dip feature seen in the $\chi'(T)$ plots. The two-peak feature gets pronounced

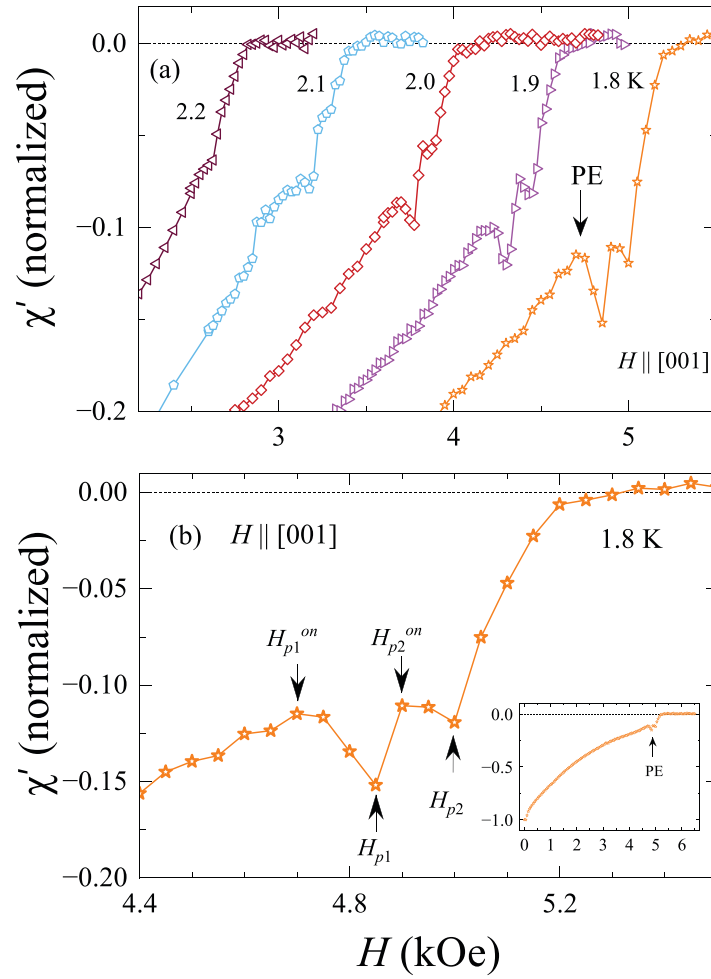
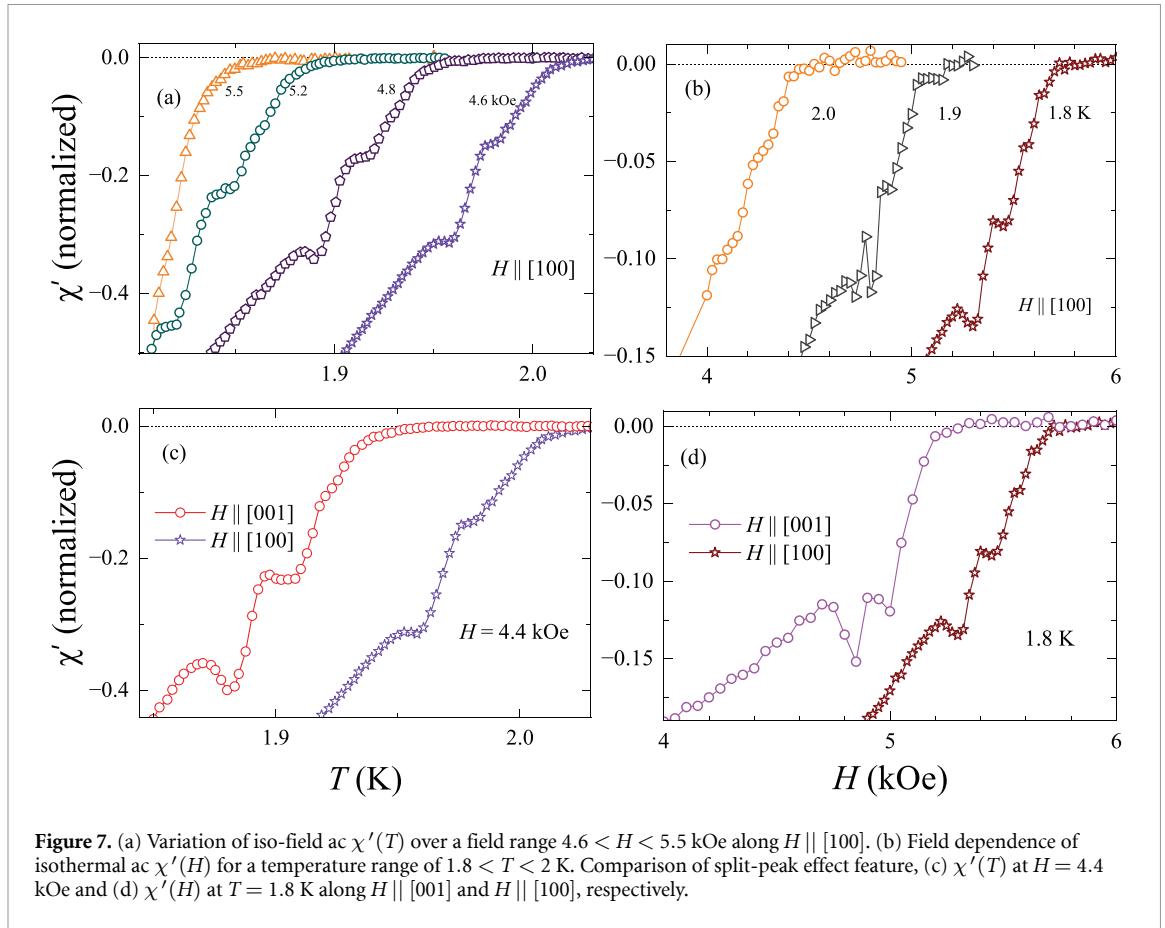


Figure 6. (a) Magnetic field variation of the (isothermal) ac magnetic susceptibility $\chi'(H)$ for a field applied along $H \parallel [001]$ in the temperature range $1.8 < T < 2.2$ K. (b) Magnified view of $\chi'(H)$ at 1.8 K showing the onset and peak positions of the split-peak effect. The inset shows the $\chi'(H)$ curve at 1.8 K over the field range $0 < H < 6.5$ kOe.

on approaching low-temperature high-field values. The onset (H_{p1}^{on} and H_{p2}^{on}) and peak field (H_{p1}^{on}) values of the two-peaks have been marked in figure 6(b) which shows a magnified view of the two-step PE region at 1.8 K. We also observed the PE anomaly for $H \parallel [100]$ as shown in figure 7. In panel 7(a), the $\chi'(T)$ plots in different fields depict the PE anomaly. Here also, we observe clear evidence of the two-peak feature across the PE region, a behavior consistent with the observed $\chi'(T)$ for $H \parallel [001]$. The isothermal scans $\chi'(H)$ shown in figure 7(b) also displays the two-peak PE feature as shown for $T = 1.8, 1.9$ and 2 K. A comparison of the $\chi'(T)$ and $\chi'(H)$ scans recorded for the two orientations $H \parallel [100]$ and $H \parallel [001]$ is shown in figures 7(c) and (d), respectively. Apparently, the superconducting-normal state transition [$H_{c2}(T)/T_c(H)$] occurs at lower field/temperature values for $H \parallel [001]$ and therefore, we observe the onset of PE occurs at lower field/temperature values in this orientation. Note that both the PME and PE appear in this compound irrespective of crystallographic orientations, showing only minor differences in peak height and position. This suggests that Ru_7B_3 exhibits weak anisotropic behavior.

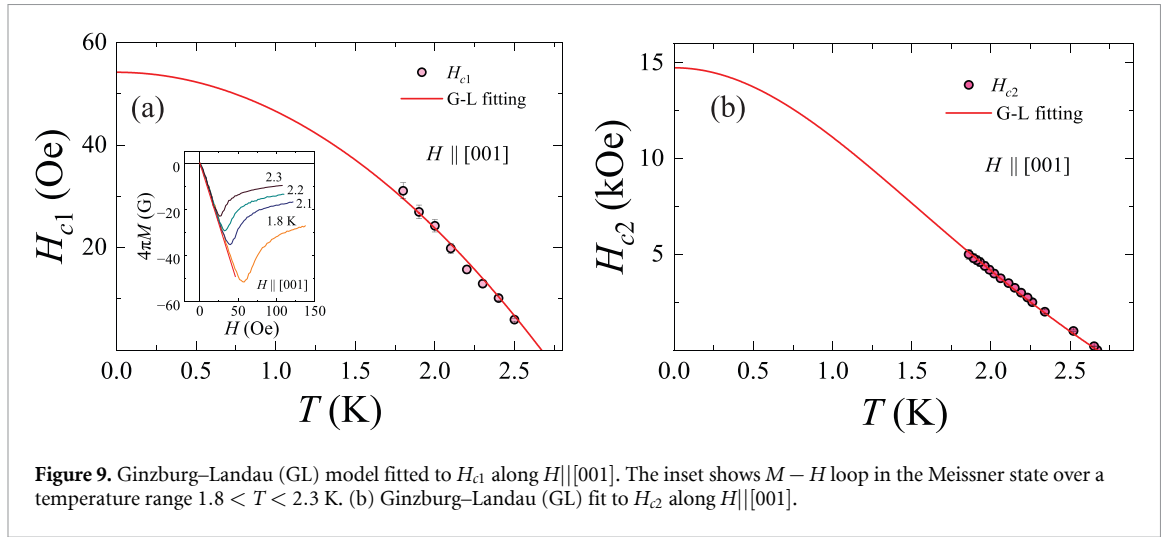
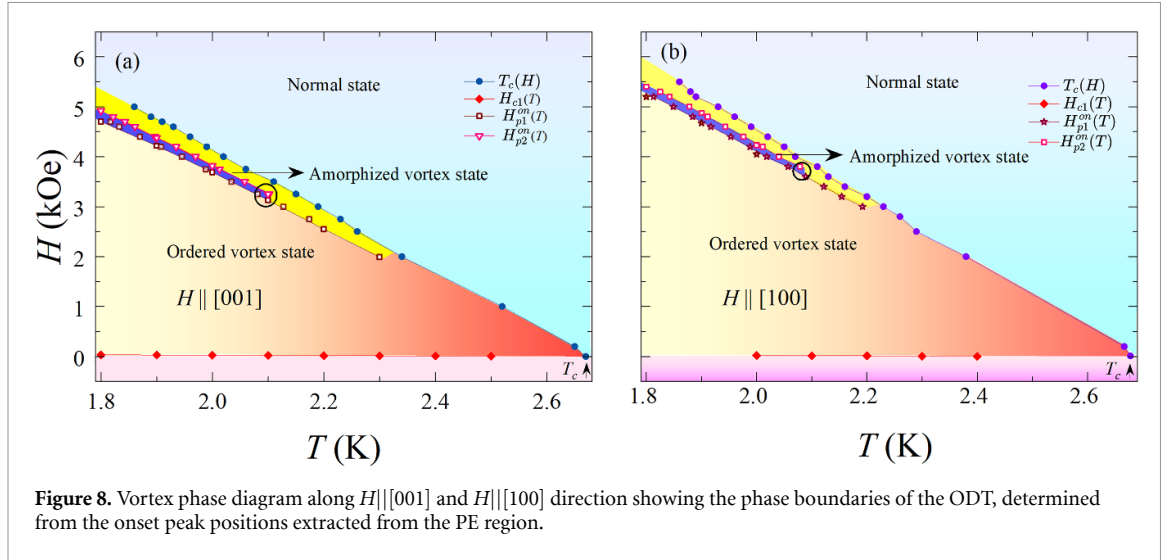
4. Discussion

We have presented our results of magnetization and electrical resistivity measurements on a single crystal of the NCS Ru_7B_3 which have led to the revelation of two unrelated anomalies- (i) the occurrence of PME in the field cooled-cool down magnetization runs $M(T)$ and (ii) a two-step PE feature encompassing the $H-T$ phase diagram close to the superconducting-normal state transition. The following possible scenarios may be considered to understand the origin of PME in Ru_7B_3 . The d -wave mechanism [7, 75] leading to the occurrence of PME in high- T_c superconductors is clearly ruled out in the present context as Ru_7B_3 exhibits $s + p$ wave symmetry [66]. Within the framework of Ginzburg-Landue theory,



the occurrence of PME has often been associated with the occurrence of giant vortex states with multiple flux quanta ($L > 1$, $\phi = hc/2e$) in various superconductors [76–79]. The magnetization due to such a giant vortex state has been anticipated to be metastable as per the theoretical work [12, 13]. However, such a scenario applies to the mesoscopic-sized samples and therefore, in the context of present study involving a macroscopic Ru_7B_3 crystal, such a possibility remains a rare proposition. Another possible explanation for the occurrence of PME involves unconventional OFP, which can emerge in superconductors with broken TRS [64]. However, the occurrence of PME due to OFP has been realized in multi-band superconductors [15, 18] or hetero-structures such as superconductor-magnet hybrid systems [14, 16, 17]. Hence the most appropriate theoretical model which comes closer to explaining our present findings relates to the trapping of magnetic flux within the bulk of the superconductor as proposed by Larkin and Koshelev [11]. As per this model, a superconducting specimen on field-cooling may exhibit an inhomogeneous cooling resulting in the nucleation of superconductivity near the edges of the sample leading to flux-free region near the edges. The magnetic flux remain trapped within the bulk of the sample as they remain in the normal state. Consequently, two oppositely flowing currents- (i) a paramagnetic current due to the trapped flux and (ii) a diamagnetic shielding current due to the onset of superconductivity at the surface compete with each other resulting in a paramagnetic signal in the $M_{\text{FCC}}(T)$ measurements [11]. The observed PME in Ru_7B_3 may therefore be ascribed to the trapping of magnetic flux [11] within the bulk of the superconducting Ru_7B_3 .

Apart from the PME, the occurrence of PE anomaly in the Ru_7B_3 is also noteworthy. In figure 8, we show the field-temperature ($H - T$) vortex phase diagram of Ru_7B_3 for the two orientations, $H \parallel [001]$ (figure 8(a)) and $H \parallel [100]$ (figure 8(b)) which includes the phase boundaries related to the onset positions of the ($H_{p1}^{\text{on}}(T)$ and $H_{p2}^{\text{on}}(T)$) of the two-peak PE feature extracted from the $\chi'(T)$ data. Both $H_{p1}^{\text{on}}(T)$ and $H_{p2}^{\text{on}}(T)$ decrease with increase in temperature displaying the typical temperature variation of the onset of the PE anomaly. Note that both these field lines ($H_{p1}^{\text{on}}(T)$ and $H_{p2}^{\text{on}}(T)$) are very close to each other and they encompass a narrow $H - T$ region between them. For clarity, we have not plotted the peak fields of the PE ($H_{p1}(T)$ and $H_{p2}(T)$) in figure 8 as they would be lying very close to each other. It is worth mentioning here that we did not observe any significant difference in the PE seen in different orientations except that their onset values being different which is obvious due to the anisotropy in the $H_{c2}(T)$ values and the fact that PE occurs at the edge of $H_{c2}(T)$ line in the $H - T$ phase space. Recalling



that the PE marks the ODT in the vortex matter [34, 80], we have identified the region below $H_{p1}^{\text{on}}(T)$ as the ordered vortex matter and that above this field line as the amorphous vortex matter. The two-peak PE feature converges into a composite peak below a certain field value (encircled in both the panels of figure 8). The observed split PE feature in Ru_7B_3 with similar two-step PE feature reported [45] in a conventional low- T_c superconductor 2H-NbSe_2 , a member whose single crystals are generally clean with a RRR ratio ranging from 30 to 120 [69, 81]. Our results bear a striking resemblance with the findings of [45], both these compounds exhibit the two-step features across the PE anomaly implying a step-wise amorphization of the vortex matter [45]. Evidence for the two-step PE in 2H-NbSe_2 is not limited to magnetic measurements. It was later investigated by Ganguli *et al* [82] using real-space imaging through scanning tunneling microscopy (STM) techniques, where they found that the introduction of cobalt into 2H-NbSe_2 creates random pinning centers for the vortices. Their investigation further revealed that the melting line of the vortex lattice also occurs in steps in the $\text{Co}_{0.0075}\text{NbSe}_2$ [82]. Interestingly, the RRR value (3.66) of Ru_7B_3 in the present single crystal lies far below that for the 2H-NbSe_2 crystal which is suggestive of Ru_7B_3 being more disordered system and therefore, it imposes challenges to identify the PE transition [70] in this compound. Clean crystals, such as those of 2H-NbSe_2 [69], on the other hand, are more likely to display the PE anomaly in the field-temperature variation of magnetization data including both dc $M - H$ and ac $\chi'(H, T)$ data [45]. Nevertheless, we could observe a clear indication of the PE feature for Ru_7B_3 in the ac $\chi'(H, T)$ although no signature of the same could be discernible in the dc $M - H$ loops recorded for the present crystal. Owing to the peculiarity of the ac susceptibility measurements to capture the information over a shorter time interval [83], the signature of the PE are evident in the present study.

To complete the vortex phase diagram (figure 8), we have extracted the lower critical field values $H_{c1}(T)$ from the dc $M - H$ loops recorded at different temperatures (see inset panel of figure 9(a)). The

Table 1. Superconducting parameters of Ru₇B₃ with applied field along [001] crystallographic axis.

Parameters	Unit	Value
T_c	K	2.68(1)
$H_{c1}(0)$	Oe	54.21(1)
$H_{c2}(0)$	kOe	14.73(5)
$\lambda_{GL}(0)$	Å	3020.74(8)
$\xi_{GL}(0)$	Å	149.47(3)
$\kappa_{GL}(0)$	—	20.21(4)
$H_c(0)$	Oe	515.39(2)
RRR value $\rho(300\text{ K})/\rho(3\text{ K})$	—	~ 3.66

field value at which the $M-H$ curve deviates from linearity yielded $H_{c1}(T)$ (shown for $T = 1.8 - 2.3$ K). Figure 9 shows the variation of H_{c1} with temperature, a fit according to the GL equation

$$H_{c1}(T) = H_{c1}(0) \left[1 - \left(\frac{T}{T_c} \right)^2 \right], \quad (3)$$

gave us the value of $H_{c1}(0)$, which compares reasonably well with the reported values [62]. We also extracted the upper critical field H_{c2} values from the $\chi'(T)$ data and plotted the same against temperature as shown in figure 9(b). A fit according to the GL equation

$$H_{c2}(T) = H_{c2}(0) \left(\frac{1-t^2}{1+t^2} \right), \quad \text{where } t = \frac{T}{T_c} \quad (4)$$

yields $H_{c2}(0)$. From our magnetization and resistivity measurements, we estimated the values of the superconducting penetration depth

$$\lambda_{GL}(0) = \sqrt{\frac{\phi_0 \ln \left(\frac{\lambda_{GL}(0)}{\xi_{GL}(0)} \right)}{4\pi H_{c1}(0)}}, \quad (5)$$

GL coherence length

$$\xi_{GL}(0) = \sqrt{\frac{\phi_0}{2\pi H_{c2}(0)}}, \quad (6)$$

GL ratio

$$\kappa(0) = \frac{\lambda_{GL}(0)}{\xi_{GL}(0)}, \quad (7)$$

thermodynamic critical magnetic field

$$H_c(0) = \frac{H_{c2}(0)}{\sqrt{2}\kappa(0)} \quad (8)$$

and tabulate these in table 1.

As stated earlier, the recent vortex study using SANS technique on Ru₇B₃ have revealed an interesting rotation of the vortex lattice without involving any structural transition deeper in the mixed state, $T = 55$ mK and $H > 2$ kOe [65]. It has been argued that the vortex matter is more disordered below this field value [65]. This is indeed the case in our present study as we did not observe any PE feature below 2 kOe for Ru₇B₃. The state of spatial order of the vortex matter may be disordered to an extent that the signature of PE may not be discernible in the $\chi'(H)$ or $\chi'(T)$ measurements for $H < 2$ kOe in this compound.

5. Summary and conclusion

We have presented our results of magnetization and electrical resistivity measurements in a single crystal of a low- T_c NCS Ru_7B_3 . This compound has recently been an interesting choice for vortex studies using SANS [65, 67, 68] and for studies related to TRS breaking [65]. Our present investigation led to the revelation of two well-documented anomalies- (i) observation of PME in the isofield $M(T)$ measurements and (ii) a split PE feature occurring close to the edge of the superconducting-normal transition. The PME has been observed in different orientations up to the field value as high as 200 Oe. Across the temperature range of the PME ($2.6 \text{ K} < T < T_c$), we also observe anomalous responses in isothermal $M-H$ runs revealing an unusual asymmetry in the shape of the $M-H$ curves. The occurrence of PME has been ascribed to the trapping of magnetic flux inside the bulk of superconductor due to inhomogeneous cooling [11].

Despite exhibiting a low RRR value of only 3.66, the signature of PE could be clearly observed in the ac $\chi'(H)$ and $\chi'(T)$ measurements, although no such fingerprints of the PE could be seen in the dc $M-H$ scans across the $H_{p1}^{\text{on}}(T)$ and $H_{p2}^{\text{on}}(T)$ phase boundaries. Interestingly, above a certain field value (3.7 kOe) the PE feature evolves into a two-peak feature suggesting stepwise amorphization of vortex matter. It is worth mentioning that both the PME and PE features which have been observed in the present study are not merely restricted to a particular orientation but they are also observed in different orientations showing that these features are robust in this compound. These results have allowed us to identify the phase boundaries ($H_{p1}^{\text{on}}(T)$ and $H_{p2}^{\text{on}}(T)$) of the ODT occurring step-wise in the vortex phase diagram of Ru_7B_3 .

In conclusion, to the best of our knowledge, no established theoretical framework directly correlates the role of broken inversion symmetry with the emergence of the PME or PE. However, it is well known that NCSs exhibit strong intrinsic pinning and the pinning mechanism in this class of superconductors is usually different from conventional superconductors [54, 55, 57, 58]. Therefore, we ascribe that the occurrence of the PE in the disordered system Ru_7B_3 may not be solely attributed to quenched random disorder, the intrinsic pinning associated with the non-centrosymmetric crystal structure may also play a role here. Nevertheless, a strong confirmation of this requires further theoretical analysis and experimental studies using advanced imaging techniques, such as, SANS and STM to comprehend the cause of the ODT of the vortex lattice in the NCS superconductors.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors. Data cannot be shared publicly as it forms part of ongoing research and will be made available upon reasonable request.

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