

# Critical fields and the spontaneous vortex state in the weakly ferromagnetic superconductor $\text{RuSr}_2\text{GdCu}_2\text{O}_8$

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A spontaneous vortex state (SVS) between 30 and 56 K was observed for the weak-ferromagnetic superconductor  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  with the ferromagnetic Curie temperature  $T_C=131$  K and the superconducting transition temperature  $T_c=56$  K. The low-field ( $\pm 20$  G) superconducting hysteresis loop indicates a narrow Meissner state region within the average lower critical field  $B_{c1}(T)=B_{c1}(0)[1-(T/T_0)^2]$ , with average  $B_{c1}^{\text{ave}}(0)=12$  G and  $T_0=30$  K. Full Meissner shielding signal in very low applied field indicates an *ab* plane  $B_{c1}^{\text{ab}}(0)\sim 4$  G with an estimated anisotropic parameter  $\gamma\sim 7$  for this layered system. The existence of a spontaneous vortex state between 30 and 56 K is the result of weak-ferromagnetic order with a net spontaneous magnetic moment of  $\sim 0.1\mu_B/\text{Ru}$ , which generates a weak magnetic dipole field around 10 G in the  $\text{CuO}_2$  bilayers. The upper critical field  $B_{c2}$  varies linearly as  $(1-T/T_c)$  up to 7-T field. The vortex melting line  $B_m$  varies as  $(1-T/T_m)^{3.5}$  with melting transition temperature  $T_m=39$  K and a very broad vortex liquid region due to the coexistence and the interplay between superconductivity and weak-ferromagnetic order.

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## I. INTRODUCTION

Recently, high- $T_c$  superconductivity with anomalous magnetic properties was reported in the weak-ferromagnetic Ru-1212 system  $\text{RuSr}_2\text{RCu}_2\text{O}_8$  ( $R=\text{Sm}, \text{Eu}, \text{Gd}, \text{Y}$ ) with the tetragonal  $\text{TlBa}_2\text{CaCu}_2\text{O}_7$ -type structure.<sup>1-43</sup> For the Ca-substituted system, a possible superconductivity was also reported in the weak-ferromagnetic compounds  $\text{RuCa}_2\text{RCu}_2\text{O}_8$  ( $R=\text{Pr}-\text{Gd}$ ).<sup>44-46</sup> The metallic weak-ferromagnetic (WFM) order is originated from the long-range order of Ru moments in the  $\text{RuO}_6$  octahedra due to strong  $\text{Ru-}4d_{xy,yz,zx}\text{-O-}2p_{x,y,z}$  hybridization in this strongly correlated electron system. The Curie temperature  $T_C\sim 130$  K observed from magnetization measurement in the prototype compound  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  is probably a canted *G*-type antiferromagnetic order with  $\text{Ru}^{5+}$  moment  $\mu$  canted along the tetragonal basal plane resulting a small net spontaneous magnetic moment  $\mu_s\ll\mu(\text{Ru}^{5+})$  too small to be detected in neutron diffraction.<sup>4,5,9,10,21</sup> The occurrence of high- $T_c$  superconductivity with maximum resistivity onset  $T_c(\text{onset})\sim 60$  K in  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  is related with the quasi-two-dimensional  $\text{CuO}_2$  bilayers separated by a rare-earth layer in the Ru-1212 structure.<sup>1,2,4,5,29</sup> Broad resistivity transition width  $\Delta T_c=T_c(\text{onset})-T_c(\text{zero})\sim 15\text{--}20$  K observed is most likely originated from the coexistence and the interplay between superconductivity and weak-ferromagnetic order.<sup>1-43</sup> The diamagnetic  $T_c$  is observed anomalously at lower temperature near  $T_c(\text{zero})$  instead of at  $T_c(\text{onset})$ , and a reasonably large Meissner signal was reported using stationary sample magnetometer with diamagnetic  $T_c\sim 30$  K in  $\leq 1$  G applied field at zero-field-cooled (ZFC) mode.<sup>38</sup> Lower  $T_c(\text{onset})\sim 40$  and 12 K were observed for  $\text{RuSr}_2\text{EuCu}_2\text{O}_8$  and  $\text{RuSr}_2\text{SmCu}_2\text{O}_8$ , respectively.<sup>12,17</sup> No superconductivity can be detected in  $\text{RuSr}_2\text{RCu}_2\text{O}_8$

( $R=\text{Pr}, \text{Nd}$ ).<sup>3,16</sup> Superconducting  $\text{RuSr}_2\text{YCu}_2\text{O}_8$  phase is stable only under the high pressure.<sup>20,25</sup> The physics is still unclear in this system, and it will be interesting to investigate the effect of the weak-ferromagnetic order on the superconducting critical fields  $B_{c2}$  and  $B_{c1}$ , as well as on the possible existence of a spontaneous vortex state (SVS) at a higher temperature above the Meissner state.

## II. EXPERIMENTAL

The stoichiometric  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  samples were synthesized by the standard solid-state reaction method. High-purity  $\text{RuO}_2$  (99.99%),  $\text{SrCO}_3$  (99.99%),  $\text{Gd}_2\text{O}_3$  (99.99%), and  $\text{CuO}$  (99.99%) preheated powders with the nominal composition ratio of  $\text{Ru}:\text{Sr}:\text{Gd}:\text{Cu}=1:2:1:2$  were well mixed and calcined at 960 °C in air for 16 h. The calcined powders were then pressed into pellets and sintered in flowing  $\text{N}_2$  gas at 1015 °C for 10 h to form  $\text{RuSr}_2\text{GdO}_6$  and  $\text{Cu}_2\text{O}$  precursors. This step is crucial in order to avoid the formation of unwanted impurity phases. The  $\text{N}_2$ -sintered pellets were heated at 1060 °C in flowing  $\text{O}_2$  gas for 10 h to form the Ru-1212 phase. The pellets were oxygen annealed at slightly higher 1065 °C for 5 days and slowly furnace cooled to room temperature with a rate of 15 °C per h.<sup>15</sup>

The powder x-ray diffraction data were collected with a Rigaku Rotaflex 18-kW rotating anode diffractometer using graphite monochromatized  $\text{Cu-K}\alpha$  radiation with a scanning step of 0.02° (10 s counting time per step) in the  $2\theta$  ranges of 5°–100°. The electrical resistivity and magnetoresistivity measurements were performed using the standard four-probe method with a Linear Research LR-700 ac (16Hz) resistance bridge from 2 to 300 K with applied magnetic field up to 7 T. The magnetization, magnetic susceptibility, and magnetic hysteresis measurements from 2 to 300 K with applied fields

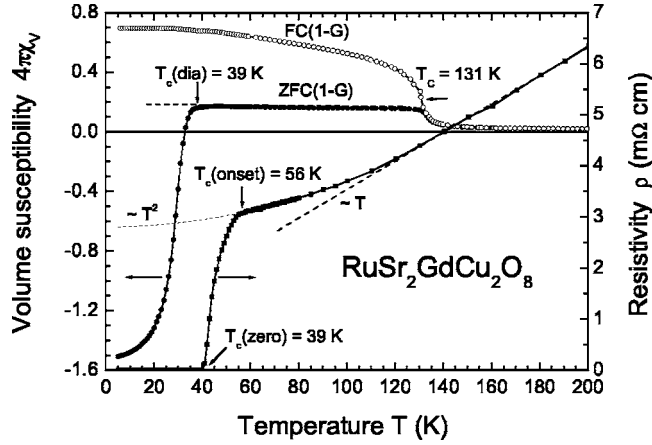


FIG. 1. Electrical resistivity  $\rho(T)$  and volume magnetic susceptibility  $\chi_V(T)$  at 1-G field-cooled (FC) and zero-field-cooled (ZFC) modes for oxygen-annealed  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ .

from 1 G to 7 T were carried out with a Quantum Design 1-T  $\mu$ -metal shielded MPMS2 or a 7-T MPMS superconducting quantum interference device (SQUID) magnetometer.

### III. RESULTS AND DISCUSSION

The powder x-ray diffraction pattern for the oxygen-annealed  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  polycrystalline sample indicates close to a single phase with the tetragonal lattice parameters of  $a=0.5428(5)\text{nm}$  and  $c=1.1589(9)\text{nm}$ . The space group  $P4/mbm$  is used for Rietveld refinement analysis, where neutron-diffraction data indicate that a  $\text{RuO}_6$  octahedra  $14^\circ$  rotation around the  $c$  axis is needed to accommodate physically reasonable Ru-O bond lengths.<sup>10</sup> The refinement with the fixed  $14^\circ$  rotation angle gives a good residual error  $R$  of 3.64%, weighted pattern error  $R_{\text{WP}}=6.07\%$ , and Bragg error  $R_B=5.05\%$ .

The temperature dependence of the electrical resistivity  $\rho(T)$  and the volume magnetic susceptibility  $\chi_V(T)$  at 1-G field-cooled (FC) and zero-field-cooled (ZFC) modes for  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  are shown collectively in Fig. 1. The high-temperature resistivity decreases monotonically from room temperature value of 9.2 m $\Omega$  cm (not shown) to 6.4 m $\Omega$  cm at 200 K, and extrapolated to 2.8 m $\Omega$  cm at 0 K with a good resistivity ratio  $\rho(300\text{ K})/\rho(0\text{ K})$  of 3.3 for the polycrystalline sample. The high-temperature resistivity shows a non-Fermi-liquid-like linear  $T$  dependence down to a Curie temperature  $T_C$  of 131 K, then changes to a  $T^2$  behavior below  $T_C$  due to magnetic order.

The superconducting onset temperature of 56 K is determined from the deviation from  $T^2$  behavior, with a zero resistivity  $T_{c(\text{zero})}$  at 39 K. The broad transition width  $\Delta T_C=17\text{ K}$  observed is the common feature for all reported Ru-1212 resistivity data, which indicates that the superconducting Josephson coupling along the tetragonal  $c$  axis between Cu-O bilayers may be partially blocked by the dipole field  $B_{\text{dipole}}$  of ordered Ru moments in the Ru-O layer.<sup>1,2,4,5,29,40</sup> The diamagnetic  $T_c$  at 39 K was observed in the 1-G ZFC susceptibility measurement. The full Meissner

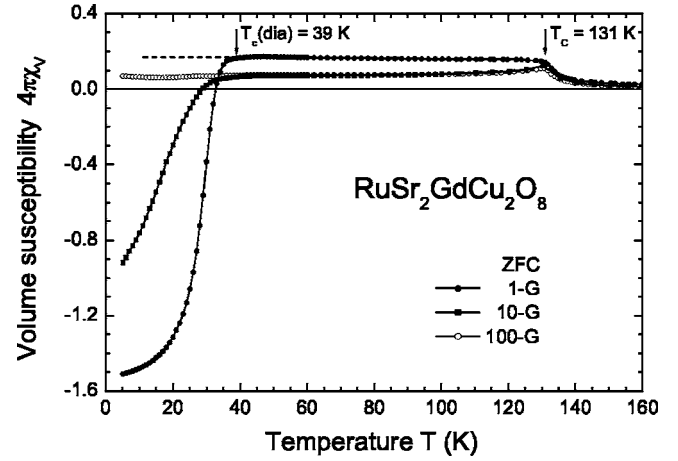


FIG. 2. ZFC volume susceptibility  $\chi_V(T)$  for  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  at 1, 10, and 100 G. Note that the full Meissner shielding signal was observed only at low applied field and low temperature.

shielding signal  $4\pi\chi_V=4\pi M/B_a \sim -1.5$  (Gaussian units) was recorded at 5 K. This value is identical to the Meissner shielding signal expected for a superconducting sphere with a demagnetization factor  $N$  of  $-4\pi/3$  and in an applied field  $B_a$  well below lower critical field  $B_{c1}$ . The large diamagnetic signal in 1-G ZFC mode is the best data observed so far from various reported susceptibility measurement techniques.<sup>4,5,28,29,38</sup> Since our measurements were performed with the standard moving-sample SQUID magnetometer, it is clear that sample quality is more crucial than measuring techniques. Both ZFC and FC data reveal a Curie temperature  $T_C$  of 131 K. However, in 1-G FC mode, no diamagnetic field-expulsion signal can be detected below 39 K due to strong flux pinning where superconductivity coexists with weak-ferromagnetic order.

The zero-field-cooled (ZFC) volume susceptibility  $\chi_V(T)$  at 1, 10, and 100 G applied fields are shown collectively in Fig. 2. All data show the same magnetic order  $T_C(\text{Ru})$  of 131 K. Although the diamagnetic  $T_c$  of 39 K was still observed at 10-G ZFC measurement, the diamagnetic signal at 5 K is reduced to 60% of the full Meissner signal. Consider the polycrystalline nature of sample with varying microcrystallite size and orientation, the average superconducting lower critical field  $B_{c1}$  at 5 K is estimated to be close to 10 G. No net diamagnetic signal can be detected at 100-G ZFC mode where the sample is already in the vortex glass or lattice state and the small diamagnetic signal is overshadowed by a large weak-ferromagnetic background.<sup>38</sup>

Based on this information, the low-field ( $\pm 20\text{ G}$ ) isothermal superconducting hysteresis loops  $M-B_a$  are measured and collectively shown in Figs. 3(a) (5, 10, 15, and 20 K) and 3(b) (25, 30, and 35 K). The initial magnetization curve deviates from straight line in 4 G at 5 K, 3.5 G at 10 K, 3 G at 15 K, 2 G at 20 K, and 1 G at 25 K. This is the narrow region that full Meissner signals are detected and is roughly corresponding to the anisotropic lower critical field in the  $ab$  plane  $B_{c1}^{ab}(T)$  with  $B_{c1}^{ab}(0) \sim 4\text{ G}$ . The average lower critical field  $B_{c1}^{\text{ave}}$  for the polycrystalline sample is determined from

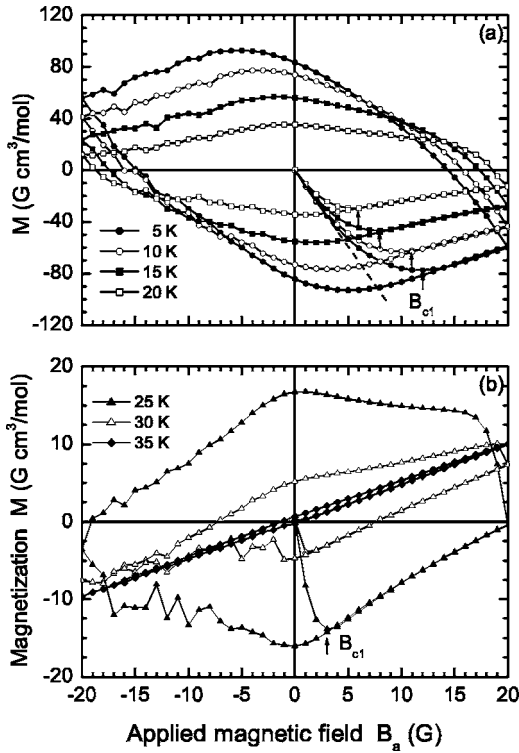


FIG. 3. Low-field superconducting hysteresis loops  $M$ - $B_a$  for  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ : (a) at 5, 10, 15, 20 K and (b) at 25, 30, and 35 K.

the peaks of initial diamagnetic magnetization curves. The effect on the exact peak value due to the surface barrier pinning is neglected.  $B_{c1}$  decreases steadily from 12 G at 5 K, 11 G at 10 K, 9 G at 15 K, 6 G at 20 K, 3 G at 25 K, and below 1 G at 30 K. A simple empirical parabolic fitting gives  $B_{c1}(T) = B_{c1}(0)[1 - (T/T_0)^2]$ , with average  $B_{c1}^{\text{ave}}(0) = 12$  G and  $T_0 = 30$  K (see Fig. 4). Using the anisotropic Ginzburg-Landau formula  $B_{c1}^{\text{ave}} = [2B_{c1}^{\text{ab}} + B_{c1}^c]/3$ ,  $c$ -axis  $B_{c1}^c \sim 28$  G and the anisotropy parameter  $\gamma \sim 7$  is estimated. This value is close to a reported anisotropy  $\gamma$  value for

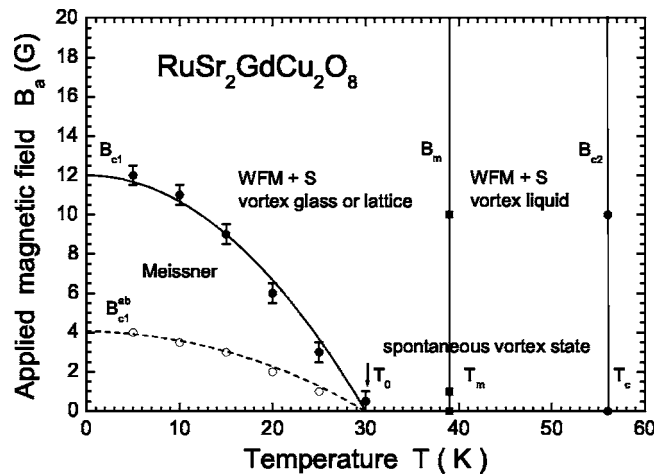


FIG. 4. The lower field, low-temperature superconducting phase diagram  $B_a(T)$  of  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ .

$\text{YBa}_2\text{Cu}_3\text{O}_7$  where the 123-type structure can be written as  $\text{Cu-1212 CuBa}_2\text{YCu}_2\text{O}_7$ . An average penetration depth  $\lambda_{\text{ave}}(0) = [\Phi_0/2\pi B_{c1}^{\text{ave}}(0)]^{1/2}$  of 520 nm was derived with estimated  $\lambda_{\text{ab}}(0) = 340$  nm and  $\lambda_c(0) = 2400$  nm from  $B_{c1}^c = \Phi_0/2\pi\lambda_{\text{ab}}^2$  and  $B_{c1}^{\text{ab}} = \Phi_0/2\pi\lambda_{\text{ab}}\lambda_c$ , where  $\Phi_0$  is flux quantum.

Since  $T_0 = 30$  K is well below  $T_c(\text{onset}) = 56$  K and  $T_c(\text{zero}) = 39$  K in zero applied field, a spontaneous vortex state (SVS) indeed exists between 30 and 56 K. The low-field phase diagram  $B_a(T)$  for the polycrystalline sample is shown in Fig. 4, with the average  $B_{c1}(T)$  separates the Meissner state from the vortex state and a smaller  $B_{c1}^{\text{ab}}(T)$  inside the Meissner region for reference.  $T_c(\text{zero}) = 39$  K in the broad resistive transition is the onset of vortex depinning by a driving current. This temperature is very close to the melting transition temperature  $T_m$  from the spontaneous vortex glass or lattice state to the spontaneous liquid state due to nonzero dipole field  $B_{\text{dipole}}$  of weak-ferromagnetic order. The upper critical field  $B_{c2}$  defined from  $T_c(\text{onset})$  and the vortex melting field  $B_m(T)$  defined from  $T_c(\text{zero})$  are temperature independent for small applied fields below 20 G. The internal dipole field generated by a weak-ferromagnetic order can be estimated using a simple extrapolation  $[B_{c1}(0) + B_{\text{dipole}}]/B_{c1}(0) = T_c/T_0 = 56 \text{ K}/30 \text{ K}$ , which results with a dipole field  $B_{\text{dipole}} \sim 10.4$  G on the  $\text{CuO}_2$  bilayers. A small net spontaneous magnetic moment  $\mu_s$  of  $\sim 0.11\mu_B$  per Ru is estimated using  $B_{\text{dipole}} \sim 2\mu_s/d^3$  with  $d = c/2 = 0.58$  nm which is the distance between midpoint of  $\text{CuO}_2$  bilayers and two nearest-neighbor Ru moments. If the weak-ferromagnetic structure is a canted  $G$ -type antiferromagnetic order with Ru moments  $\mu (= 1.5\mu_B$  for  $\text{Ru}^{5+}$  in  $t_{2g}$  states) canted along the tetragonal basal plane, the small net spontaneous magnetic moment gives a canting angle of  $4^\circ$  from the tetragonal  $c$  axis and is difficult to be detected in neutron diffraction with a resolution around  $0.1\mu_B$ .<sup>9,10,21</sup>

At 5 K, the shape of superconducting hysteresis loop with a large remanent molar magnetization  $M_r$  of  $83 \text{ G cm}^3/\text{mol}$  indicates a strong pinning as well as a good indication of bulk nature of superconductivity for the oxygen-annealed sample. The remanent  $M_r$  decreases to  $4 \text{ G cm}^3/\text{mol}$  at 30 K and  $1 \text{ G cm}^3/\text{mol}$  at 35 K, where a weak-ferromagnetic background can be clearly seen. Fluctuation in the hysteresis loop is probably also related to the weak-ferromagnetic order.

To study the high-field effect on superconductivity, the magnetoresistivity  $\rho(T, B_a)$  for  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  up to 7 T are collectively shown in Fig. 5. The broadening of the resistive transition in magnetic fields is the common features for all high- $T_c$  cuprate superconductors.<sup>47</sup> The normal-state resistivity is field independent and follows a  $T^2$  dependence below  $T_c$ , with the superconducting  $T_c(\text{onset})$  of 56 K in the zero field decreases slightly to 53 K in 7-T field. The temperature dependence of upper critical field  $B_{c2}(T)$  can be fitted with a linear function  $B_{c2}(0)[1 - T/T_c]$  with average  $B_{c2}(0) = 133 \text{ T}$ .<sup>47</sup> An average coherence length  $\xi_0^{\text{ave}} = [\Phi_0/2\pi B_{c2}^{\text{ave}}(0)]^{1/2}$  of 0.5 nm with the Ginzburg-Landau

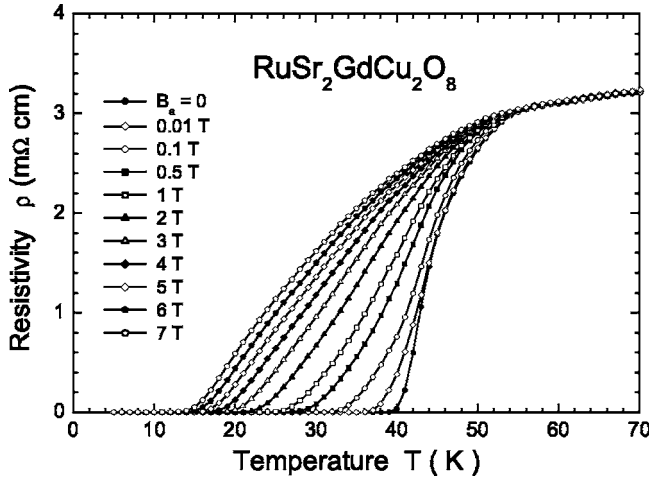


FIG. 5. Temperature dependence of magnetoresistivity  $\rho(T, B_a)$  for  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  in applied field up to 7 T.

parameter  $\kappa$  of 1040 and the thermodynamic critical field  $B_c(0) = (B_{c1}B_{c2})^{1/2} = 0.32$  T. No anisotropic  $\xi_{ab}$  and  $\xi_c$  values can be estimated from present data. The  $T_c(\text{zero})$  decreases from 39 K in zero applied field to 32 K in 1-kG, 28 K in 5-kG, 25 K in 1-T, 22 K in 2-T, 19 K in 3-T, 17 K in 4-T, 16 K in 5-T, 15 K in 6-T, and 14 K in 7-T field. If the zero resistivity is taken as the lower bound of the vortex melting temperature  $T_m$ , then the temperature dependence of the vortex melting transition line  $B_m(T)$  can be fitted roughly by the formula  $B_m(T) = B_m(0)[1 - T/T_m]^{3.5}$  with  $B_m(0) = 35$  T and large exponent 3.5. In the lower field region,  $B_m(T)$  rises as  $[1 - T/T_m]^2$  as predicted by the mean-field approximation for temperature near  $T_m = 39$  K.<sup>47</sup> The full phase diagram  $B_a(T)$  of  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  is shown in Fig. 6 to exhibit both the high- and low-field features. The very broad vortex liquid region with  $\Delta T = 17$  K in zero field and  $\Delta T = 42$  K in 7-T field is extraordinary and is most likely originated from the coexistence and the interplay between superconductivity and weak-ferromagnetic order. This magnetic order is so weak that superconductivity can coexist with the magnetic order, but the effect of a weak spontaneous magnetic moment  $\mu_s \sim 0.1 \mu_B$  is detected through the appearance of a spontane-

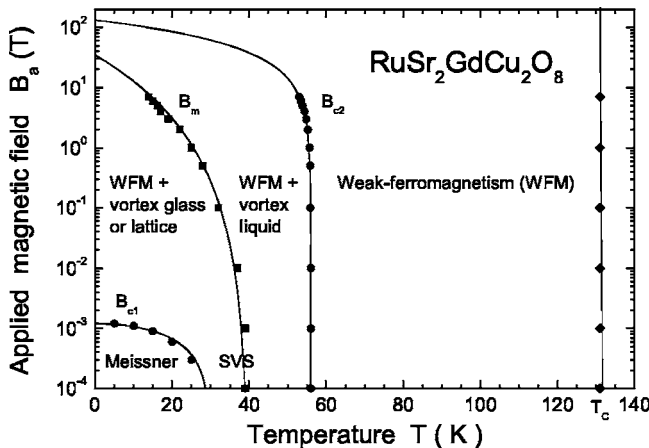


FIG. 6. Full phase diagram  $B_a(T)$  of  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ .

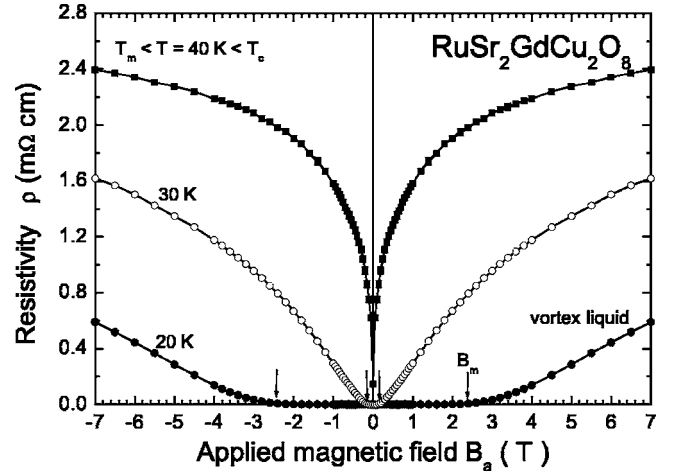


FIG. 7. Field dependence of magnetoresistivity  $\rho(B_a)$  for  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$  in the vortex state at 20, 30, and 40 K. The zero resistivity gives a lower bound of vortex melting field  $B_m$  at 20 K.

ous vortex state above 30 K with a broad spontaneous vortex liquid region above  $T_m$  of 39 K.

To study the broad vortex liquid region, the isothermal field-dependent magnetoresistivity  $\rho(B_a)$  for  $T < T_c$  are shown in Fig. 7, where the zero resistivity gives a lower bound of the vortex melting field  $B_m$ . In the resistive vortex liquid region, the magnetoresistivity increases with increasing applied magnetic field and temperature. At 40 K, the magnetoresistivity is rapidly approaching a saturation value in an extrapolated saturation field  $B_a \sim B_{c2}(40 \text{ K}) \sim 40$  T.

The last issue to be addressed is the depression of  $T_c$  by small spontaneous Ru magnetic moments. The weak-ferromagnetic order is actually a canted antiferromagnetic order that can coexist with superconductivity. However, the observed  $T_c$  of 56 K is too low as compared with 93 K for  $\text{YBa}_2\text{Cu}_3\text{O}_7$  or 103 K for  $\text{TlBa}_2\text{CaCu}_2\text{O}_7$ . The depression of  $T_c$  by small spontaneous magnetic moment can be partially recovered by substitution of nonmagnetic Cu ions at Ru site. For example, in the  $\text{Ru}_{1-x}\text{Cu}_x\text{Sr}_2\text{GdCu}_2\text{O}_8$  system,  $T_c$  onset up to 65 K for  $x = 0.1$  and 72 K for  $x = 0.4$  was reported.<sup>26,29</sup>

#### IV. CONCLUSION

The lower critical field with  $B_{c1}(0) = 12$  G and  $T_0 = 30$  K indicates the existence of a spontaneous vortex state (SVS) between 30 K and  $T_c$  of 56 K. This SVS state is closely related with the weak-ferromagnetic order with a net spontaneous magnetic moment of  $\sim 0.1 \mu_B$  per Ru. The broad vortex liquid region observed above vortex melting line  $B_m(T)$  is also due to the coexistence and the interplay between superconductivity and weak-ferromagnetic order. Indeed, a possible spontaneous vortex state was also reported in the weak ferromagnetic superconductor Ru-1222 compound  $\text{RuSr}_2(\text{Eu}_{1.5}\text{Ce}_{0.5})\text{Cu}_2\text{O}_{10}$ .<sup>48</sup>

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