Magnetization study of $RuSr_2EuCu_2O_8$

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We report the results from a magnetization study on $RuSr_2EuCu_2O_8$. This compound has recently been found to exhibit the coexistence of superconductivity and magnetic order. We show that the low-field data can be interpreted in terms of antiferromagnetic order with a small ferromagnetic component ($\sim 0.05\mu_B$/Ru at 5 K) consistent with a recent neutron-diffraction study on $RuSr_2GdCu_2O_8$. However, the high-field data imply a significant ferromagnetic component.

There have been a number of recent reports of the coexistence of superconductivity and magnetic order in the ruthenate-cuprates.[1–4] The first compound reported was $R_{1.4}Ce_{0.6}RuSr_2Cu_2O_{10-\delta}$ where R is Eu or Gd.[1] Recent reports have focused on a similar compound, $RuSr_2GdCu_2O_8$, where a magnetic transition is observed at 133 K and a bulk Meissner state exists for temperatures less than 30 K.[3–11] A muon spin-rotation study showed that the magnetic order is spatially uniform and exists in both the normal and superconducting states.[4] This compound was first synthesized by Bauernfeind *et al.*[12] The unit cell is similar to $YBa_2Cu_3O_{7-\delta}$ except that the CuO chains are replaced by a RuO_2 layer. However, its structure is more complex than $YBa_2Cu_3O_{7-\delta}$ in that there is a coherent rotation of the RuO_6 octahedra about the c axis. The coherent rotations occur within small domains where the domain diameters are less than 20 nm.[5] All current studies have focused on powder samples because, unlike other high-temperature superconducting cuprates, it is not possible to align $RuSr_2GdCu_2O_8$ in a magnetic field, and there are as yet no single crystals available. The temperature dependence of the heat capacity, thermopower, resistivity, and magnetization data have also been analyzed in terms of a thermodynamic superconducting transition temperature at 45 K in $RuSr_2GdCu_2O_8$.[6] The appearance of a bulk Meissner state at a lower temperature (<30 K) is attributed to granularity[6] or a spontaneous vortex phase.[13]

There have been a number of conflicting reports concerning the magnetic order in $RuSr_2GdCu_2O_8$. It was initially speculated that the magnetic order is antiferromagnetic in the RuO_2 layers.[14] It was later reported that the magnetic order in $RuSr_2GdCu_2O_8$ is ferromagnetic with the Ru moments possibly aligned along the *ab* plane[3,4] or the *c*-axis direction.[11] A magnetization and zero-field muon spin-rotation study reported low-field ferromagnetic order in the *ab* plane with a dipolar field at the muon site of ~ 700 G to ~ 1000 G.[4] A Gd electron spin-rotation study on $RuSr_2GdCu_2O_8$ found a ~ 600 G shift in the Gd electron spin-resonance spectra, but this was interpreted in terms of Ru-Gd ferromagnetic exchange rather than a dipole field,[11] However, a powder neutron-diffraction study on $RuSr_2GdCu_2O_8$ has recently found evidence of low-field antiferromagnetic order in all directions in the RuO_2 layers with the Ru moments aligned along the c axis.[15] Furthermore, the antiferromagnetic order at 80 K was observed to weaken for magnetic fields above ~ 4 kG.[15] The appearance of antiferromagnetic order in $RuSr_2GdCu_2O_8$ is not intuitively expected because other ruthenate compounds, such as metallic $Sr_3Ru_2O_7$ and $SrRuO_3$, are known to display ferromagnetic order.[16] It is possible that the additional effect of the large Gd moment is the origin of the different interpretations of the magnetic order.

An understanding of the magnetic order in the ruthenate-cuprates is important for determining how superconductivity and magnetism can coexist in these compounds. For example, antiferromagnetic order can coexist with uniform superconducting order and result in complete diamagnetic shielding (i.e., the Meissner state). However, long-range ferromagnetic order is expected to destroy the Meissner state for samples much larger than the magnetic penetration depth.[17] It is possible for ferromagnetic and superconducting order to coexist in $RuSr_2GdCu_2O_8$ provided that there is some accommodation, for example, via spatial modulations of the respective order parameters[7] or via a spontaneous vortex phase to locally screen the internal magnetic field.[2,13]

In this paper, we report a magnetization study of $RuSr_2EuCu_2O_8$ where nonmagnetic Eu has replaced magnetic Gd. It has recently been shown that this compound also exhibits magnetic order and superconductivity.[18] The advantage of $RuSr_2EuCu_2O_8$ is that the magnetic behavior of the ruthenate lattice (reported low-field and low-temperature moment of $1.18\mu_B$/Ru (Ref. 15) in $RuSr_2GdCu_2O_8$) is not hidden by the magnetism from Gd [moment of $7.94\mu_B$ and ordering temperature of 2.5 K (Ref. 4)]. We show below that the magnetic order in $RuSr_2EuCu_2O_8$ can be interpreted in a manner consistent with a recent powder-neutron-diffraction study of $RuSr_2GdCu_2O_8$.[15]

The $RuSr_2EuCu_2O_8$ sample preparation is discussed elsewhere.[18] The dc magnetization data were obtained using a superconducting quantum interference device (SQUID) magnetometer in the temperature range of 5 K to 300 K and magnetic-field range of -60 to 60 kG. The measurements were made on a sintered ceramic sample. As mentioned above, $RuSr_2EuCu_2O_8$ exhibits bulk superconductivity[18] with the zero-field superconductivity onset being ~ 10 K in the current sample. It is known that the Meissner phase is rapidly suppressed in $RuSr_2GdCu_2O_8$ and $RuSr_2EuCu_2O_8$ where it has been shown from dc magnetization measurements that a bulk Meissner phase is not evident in the field-cooled dc

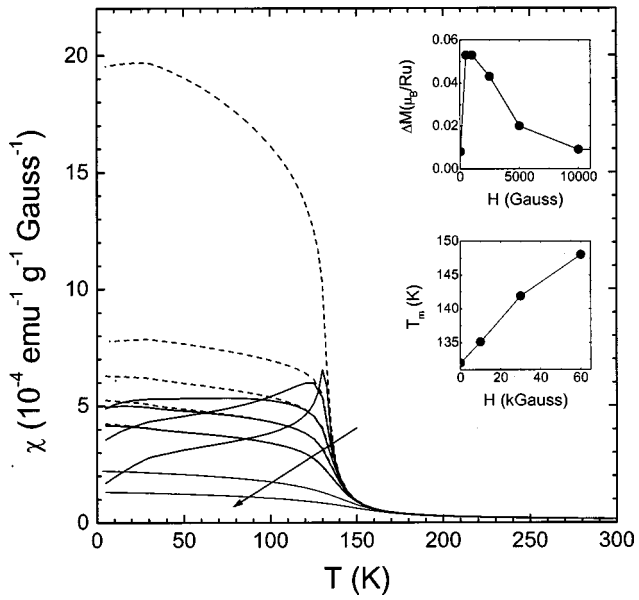

FIG. 1. Plot of the dc susceptibility from $RuSr_2EuCu_2O_8$ against temperature. The curves are for field-cooled (dashed curves) or zero-field-cooled (solid curves) in an applied magnetic field of 40 G, 1 kG, 2.5 kG, 5 kG, 10 kG, 30 kG, and 60 kG. The arrow indicates increasing magnetic field. Inset: Plot of the magnetization difference at 5 K for applied magnetic fields of 40 G and higher.

susceptibility for applied magnetic fields greater than 10 G. This suppression was attributed to a spontaneous vortex phase.[13,18]

We plot in Fig. 1 the zero-field-cooled (solid curves) and field-cooled (dashed curves) dc susceptibility from the $RuSr_2EuCu_2O_8$ sample where the applied field was 40 G, 1000 G, 2500 G, 5000 G, 10 000 G, 30 000 G, and 60 000 G. The main features observed in Fig. 2 are (i) the decrease in the susceptibility below 32 K for magnetic fields less than ~1000 G, (ii) the increase in the field-cooled susceptibility for temperatures less than ~150 K as well as the peak in the zero-field-cooled susceptibility data near 132 K for low magnetic fields and (iii) the magnetic irreversibility where the field-cooled susceptibility is greater than the zero-field-cooled susceptibility data.

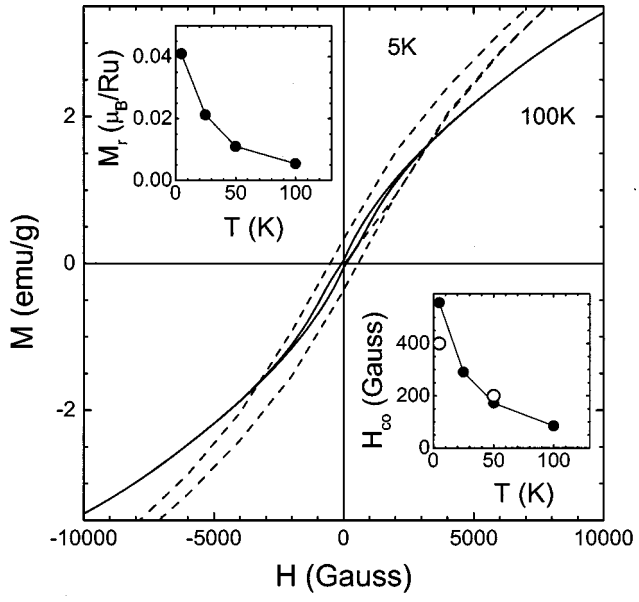

FIG. 2. Plot of the magnetization from $RuSr_2EuCu_2O_8$ against H at 5 K (dashed curve) and 100 K (solid curve). Upper left inset: Plot of remanent magnetization H_r against temperature from $RuSr_2EuCu_2O_8$ (filled circles). Lower right inset: Plot of coercive field H_{co} against temperature from $RuSr_2EuCu_2O_8$ (filled circles) and $RuSr_2GdCu_2O_8$ [open circles (Ref. 4)].

We first discuss the decrease in the susceptibility for temperatures below 32 K. A similar decrease was observed at 32 K in $Eu_{1.4}Ce_{0.6}RuSr_2Cu_2O_{10-\delta}$,[1,2] at 42 K in $Gd_{1.4}Ce_{0.6}RuSr_2Cu_2O_{10-\delta}$,[2] and at 45 K in $RuSr_2GdCu_2O_8$.[6,13] It has been argued that this feature is due to the appearance of superconductivity, where the bulk Meissner phase is suppressed due to granularity[6] or a spontaneous vortex phase.[2,13] This feature was not observed by Felner *et al.* in a previous study on a nonsuperconducting $RuSr_2EuCu_2O_8$ sample.[14] The apparent disappearance of this feature in our $RuSr_2EuCu_2O_8$ sample for high fields may be due to the large magnetic component from the RuO_2 layers when compared with the small magnetic contribution from the CuO_2 layers.

We now consider the increase in the field-cooled susceptibility below ~150 K and the zero-field-cooled peak in the susceptibility near 132 K observed for low magnetic fields. Similar to previous studies on magnetic compounds, we define the magnetic-ordering temperature, T_M, as the temperature where the maximum slope in the field-cooled susceptibility is observed. We show in the lower insert to Fig. 1 that T_M increases with increasing magnetic field. This magnetic-ordering temperature is in-between that of $SrRuO_3$ (165 K) and $Sr_3Ru_2O_7$ (104 K).[6] The peak in the zero-field-cooled susceptibility of $RuSr_2EuCu_2O_8$ also occurs at 132 K. A similar peak in the zero-field-cooled susceptibility data was observed in measurements on $RuSr_2GdCu_2O_8$ at 131 K (Refs. 3 and 4) and on $R_{1.4}Ce_{0.6}RuSr_2Cu_2O_{10-\delta}$ at ~90 K.[1] However, the zero-field-cooled peak was not observed by Felner *et al.* in their study of a nonsuperconducting $RuSr_2RuCu_2O_8$ sample.

A zero-field-cooled peak is not expected in a ferromagnetic compound. In fact, a peak in the zero-field-cooled susceptibility near the magnetic-ordering temperature is observed in antiferromagnetic compounds.[19,20] Thus, the susceptibility data suggest that the dominant low-field magnetic order is antiferromagnetic, consistent with the neutron-diffraction study on $RuSr_2GdCu_2O_8$.[15] The disappearance of this peak with increasing applied magnetic field may be related to the spin-reorientation interpretation of the neutron-diffraction data. We show later that the data can be interpreted in terms of the high-field magnetic order containing a significant ferromagnetic component. This is also suggested from the temperature dependence of the high-field susceptibility data plotted in Fig. 1, which is similar to that found in ferromagnetic compounds.

The third feature observed in Fig. 1 is the magnetic irreversibility, where there is a significant difference between the field-cooled and zero-field-cooled susceptibility. This implies a low-field ferromagnetic component. Ferromagnetic compounds are observed to display a large difference between the field-cooled and zero-field-cooled magnetization

curves at low temperatures and moderate magnetic fields, which can be a sizable fraction of the saturation magnetization.[16] For this reason, we plot the magnetization difference between the field-cooled and zero-field-cooled magnetization from $RuSr_2EuCu_2O_8$ at 5 K in the upper inset to Fig. 1. It can be seen that the magnetization difference increases to $\sim 0.05\mu_B$/Ru with increasing magnetic field and then decreases, eventually reaching zero for magnetic fields greater than 60 kG. The maximum magnetization difference is significantly less than that observed in ferromagnetic $Sr_3Ru_2O_7$ where the magnetization difference is $0.21\mu_B$/Ru[16] for an applied magnetic field of 100 G. However, the small intrinsic ferromagnetic component of $\sim 0.05\mu_B$/Ru is consistent with the analysis of the neutron-diffraction measurements on $RuSr_2GdCu_2O_8$, where an upper limit of $\sim 0.1\mu_B$/Ru was placed on any ferromagnetic component.[15] It is possible that the ferromagnetic component arises from a ferromagnetic spin canting in the *ab* plane. We note that a small canting of a $\sim 1\mu_B$ Ru moment by $\sim 3°$ could produce the observed ferromagnetic component of $\sim 0.05\mu_B$/Ru.

A small low-field ferromagnetic component could also explain the low remanent magnetization seen in the $M(H)$ curves plotted in Fig. 2. Here we plot the $M(H)$ data at 5 K and 100 K. The resultant coercive field H_{co}, and the remanent magnetization, M_r, obtained from the $M(H)$ curves at different temperatures are plotted as insets to Fig. 2. We find that M_r at 5 K is slightly less that the maximum magnetization difference at 5 K obtained from the difference between the field-cooled and zero-field-cooled susceptibility data. However, M_r is much less than that observed in ferromagnetic $SrRuO_3$ and $Sr_3Ru_2O_7$.[14,16] These compounds exhibit a large remanent magnetization ($\sim 1.3\mu_B$/Ru and $\sim 0.75\mu_B$/Ru respectively).

The magnitude of the remanent magnetization in $RuSr_2EuCu_2O_8$ is different from $M_r(T)$ reported in other ruthenate-cuprate compounds. In the case of $RuSr_2GdCu_2O_8$, $M_r(T)$ is ~4 times greater ($\sim 0.14\mu_B$/Ru at 5 K and $\sim 0.05\mu_B$/Ru at 50 K).[4] However, the ferromagnetic component is still within the limits determined from the powder neutron-diffraction study on $RuSr_2GdCu_2O_8$. Felner *et al.* did not find any evidence of remanent magnetization in their study of a nonsuperconducting $RuSr_2EuCu_2O_8$ sample.[14] This may be due to the large magnetic-field steps used in their study. As noted above, Felner *et al.* also did not observe the low-field peak in the zero-field-cooled susceptibility data. It is possible that the differences in the magnitudes of the remanent magnetization are due to small differences in the Ru moment spin-canting angle.

The largest low-temperature M_r was reported in $Eu_{1.4}Ce_{0.6}RuSr_2Cu_2O_{10-\delta}$ ($\sim 0.3\mu_B$/Ru at 5 K).[2] However, unlike $RuSr_2EuCu_2O_8$ and $RuSr_2GdCu_2O_8$, both H_{co} and M_r are only significant for $T < 40$ K[2] even though the magnetic-ordering temperature was reported as ~122 K. This was interpreted in terms of a spontaneous vortex phase existing below the superconducting onset temperature of ~33 K, and hence, H_r is assumed to be of superconducting origin. It is clear in the insets to Fig. 2 that there are no well-defined changes in H_{co} and M_r from $RuSr_2EuCu_2O_8$ that could be related to superconductivity. Rather, both H_{co} and M_r decrease monotonically with increasing temperature and disappear at the magnetic-ordering temperature. We show in the lower right inset to Fig. 2 that H_{co} from $RuSr_2GdCu_2O_8$ (open circles[4]) is comparable to that observed in $RuSr_2EuCu_2O_8$.

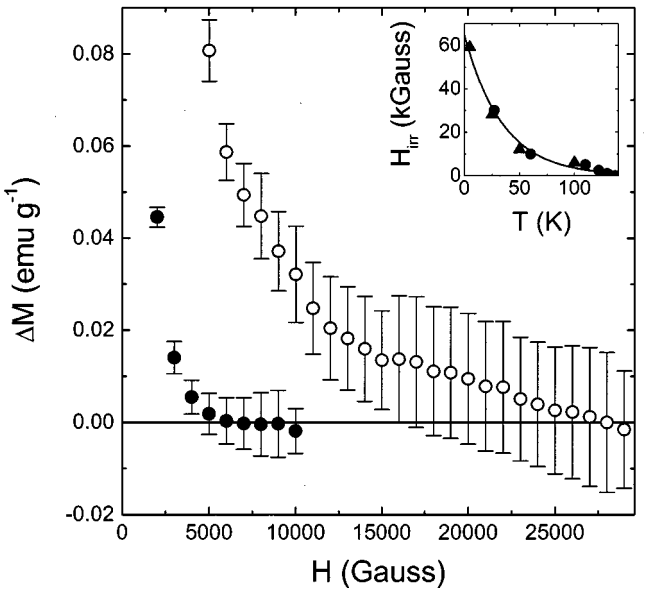

FIG. 3. Plot of the magnetization difference from $RuSr_2EuCu_2O_8$ for H increasing or H decreasing against H at 100 K (filled circles) and 25 K (open circles). Inset: Plot of the magnetic irreversibility field against temperature as determined from the $M(H)$ data (filled up triangles). Also plotted is the irreversibility temperature as determined from the $M(T)$ data. The solid curve is a fit to the function described in the text.

It is apparent in Figs. 1 and 2 that both $M(T)$ and $M(H)$ display irreversibility behavior. The $M(H)$ irreversibility is clearer in Fig. 3, where we plot the difference between $M(H)$ for H increasing or H decreasing at 100 K (filled circles) and 50 K (open circles). It can be seen that the irreversibility field $H_{\rm irr}$ decreases rapidly with increasing temperature. The resultant irreversibility field and irreversibility temperature are plotted in the insert to Fig. 3. It is apparent in the inset to Fig. 3 that both the magnetic irreversibility field (solid up triangles) and the irreversibility temperature (solid circles) are consistent with each other. The rapid decrease in $H_{\rm irr}$ with increasing temperature can be characterized by fitting the data in the insert to Fig. 3 to $H_{\rm irr}(T) = a\exp(-bT/T_0)$ for $T < 0.8T_0$ where T_0 is the irreversibility onset temperature (138 K). We show later that the irreversibility could arise from a field-induced spin-reorientation transition from antiferromagnetic order to ferromagnetic order.

To enable a comparison with previous reports, we estimate the *high-temperature* Ru moment by fitting the susceptibility data in Fig. 4 to $\chi = c/(T+\theta)+\chi_0$ for temperatures above 200 K where c is the Curie constant, θ is the Curie-Weiss temperature and χ_0 is the temperature-independent susceptibility. Here, we plot the dc susceptibility for magnetic fields increasing from 100 G. to 60 kG. It can be seen that the dc susceptibility is field independent for temperatures above 200 K, which indicates that spin-fluctuation ef-

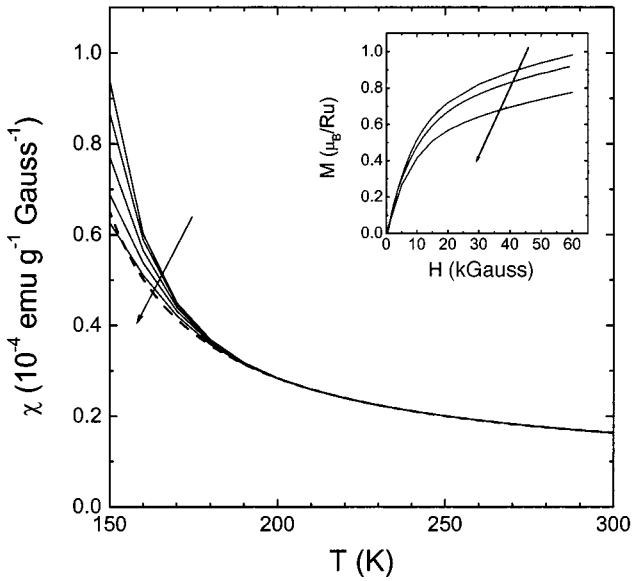

FIG. 4. Plot of the susceptibility against temperature above the magnetic-ordering temperature in $RuSr_2EuCu_2O_8$ for applied magnetic fields of 100 G, 15 kG, 30 kG, 45 kG, and 60 kG. The arrow indicates increasing magnetic field. The dashed curve is the best fit to the data for temperatures greater than 200 K using the function described in the text. Inset: Plot of the magnetization against applied magnetic field at 5 K, 50 K, and 100 K. The arrow indicates increasing temperature.

fects occur for temperatures of up to 70 K above the magnetic-ordering temperature. We show by the dashed curve in Fig. 4 that this Curie-Weiss function does fit the data, where we obtain $\theta = 122$ K, an effective moment of $3.0\mu_B$/Ru and $\chi_0 = 4.9\times10^{-3}$ emu g^{-1} mol^{-1}. The fitted value of θ is 10 K less than the temperature where the low-field peak in the susceptibility data is observed. The effective moment is slightly larger than that found in $SrRuO_3$ [$2.76\mu_B$/Ru (Ref. 16)] and $Sr_3Ru_2O_7$ [$2.63\mu_B$/Ru (Ref. 16)] crystals with the magnetic field applied along the c axis. However, it is less than that observed in $CaRuO_3$ over the same temperature range [$3.4\mu_B$/Ru (Ref. 19)]. We also find that χ_0 is comparable to χ_0 in $SrRuO_3$ [3.9×10^{-3} emu g^{-1} mol^{-1} (Ref. 21)] and $Sr_3Ru_2O_7$ [3.2×10^{-3} emu g^{-1} mol^{-1} (Ref. 16)] crystals. These results indicate that the magnetic behavior in the 200 to 300 K temperature range in $RuSr_2EuCu_2O_8$ is similar than that in $SrRuO_3$ and $Sr_3Ru_2O_7$. The high-temperature magnetic moment in $RuSr_2EuCu_2O_8$ is larger than that estimated in $RuSr_2GdCu_2O_8$ [$1.05\mu_B$/Ru (Ref. 4)]. It is possible that the lower moment obtained from magnetization measurements on $RuSr_2GdCu_2O_8$ is due to the fitting function and the dominant effect of the Gd moment. The susceptibility was fitted to a Curie-Weiss (from Ru) and a Curie (from Gd) function and no temperature-independent offset. It is possible to fit the $RuSr_2EuCu_2O_8$ susceptibility to a similar function, where the Curie function dominates and results in a Ru moment of $1.9\mu_B$/Ru. However, in the case of $RuSr_2EuCu_2O_8$, Eu is expected to be nonmagnetic for magnetic fields less than 60 kG.

We estimate the *low-temperature* Ru moment from the $M(H)$ data plotted in the inset to Fig. 4. It can be seen that the magnetization from the $RuSr_2EuCu_2O_8$ sample has not saturated even for applied magnetic fields of 60 kG. The Ru moment at 5 K and 60 kG is estimated to be $0.98\mu_B$/Ru. This is comparable to the Ru moment estimated from the magnetization data from superconducting $RuSr_2GdCu_2O_8$ at the same magnetic field and at 2 K and after the large Gd contribution has been subtracted.[4] It is also comparable to that observed in nonsuperconducting $RuSr_2EuCu_2O_8$ (Ref. 14) and $R_{1.4}Ce_{0.6}RuSr_2Cu_2O_{10-\delta}$.[2] It is only slightly less than the low-field moment estimated from a neutron-diffraction study on $RuSr_2GdCu_2O_8$ at 80 K [$1.18\mu_B$/Ru (Ref. 15)] and it is slightly less than that the saturation moment in $SrRuO_3$ [1.1 to $1.4\mu_B$/Ru (Ref. 16)] and $Sr_3Ru_2O_7$ [$1.3\mu_B$/Ru (Ref. 16)]. The comparable magnitudes of the high-field moment in $RuSr_2EuCu_2O_8$ and the saturation moment in ferromagnetic $SrRuO_3$ and ferromagnetic $Sr_3Ru_2O_7$ would seem to suggest that the dominant high-field magnetic order is ferromagnetic and that the high-field magnetization is dominated by the RuO_2 layers.

The data above suggest that the low-field magnetic order is predominately antiferromagnetic, while the high-field magnetic order is predominately ferromagnetic. It is possible that there is a spin reorientation to a mixed magnetic state and then a ferromagnetic state at high fields. A mixed magnetic state at intermediate fields would explain the irreversibility and the absence of a well-defined spin-reorientation field. It is also possible that the spin-reorientation field is averaged out owing to the random microcrystallite orientations to the applied field. As mentioned earlier, Lynn *et al.* also find evidence at 80 K from a powder neutron-diffraction study on $RuSr_2GdCu_2O_8$ of a spin reorientation for magnetic fields above ~4 kG. (Ref. 15). This spin reorientation is gradual and does not appear to be complete even for fields as high as 60 kG. This is interpreted in terms of a reorientation into another antiferromagnetic structure. However, as noted above, in the case of $RuSr_2EuCu_2O_8$ the data would seem to suggest a dominant ferromagnetic component at high magnetic fields. Magnetic-field-induced spin reorientations into a ferromagnetic state are not unusual[19,20] and have been observed in a number of compounds including $Sr_3Ru_2O_7$ (Ref. 16) and $La_{1.4}Sr_{1.6}Mn_2O_7$ (Ref. 22) single crystals. In the case of $La_{1.4}Sr_{1.6}Mn_2O_7$ single crystals the spin reorientation is from antiferromagnetic to ferromagnetic order occurring via a mixed state, and it is accompanied by a small hysteresis. A spin reorientation is also observed in antiferromagnetic Y_2CuO_4, where the weak ferromagnetic component is compensated for at low fields.[23] The zero-field-cooled and field-cooled susceptibility of Y_2CuO_4 displays a remarkable resemblance to that observed in $RuSr_2EuCu_2O_8$, where the zero-field-cooled peak is not evident at high fields, and the irreversibility temperature decreases with increasing magnetic field.

In conclusion, we have performed a magnetization study on $RuSr_2EuCu_2O_8$, which displays magnetic ordering and superconductivity. We find that the high-temperature Ru moment and the low-temperature-high-field Ru moment are comparable to the Ru moment observed in ferromagnetic $SrRuO_3$ and $Sr_3Ru_2O_7$. However, the remanent magnetization at low temperatures is more than 15 times less than that observed in $SrRuO_3$ and $Sr_3Ru_2O_7$, and we also find a

temperature-dependent magnetic irreversibility. One possible interpretation of the low-field data is antiferromagnetic ordering of the Ru moments in the *c*-axis direction and spin canting along the *ab*-plane direction. This interpretation is consistent with a recent neutron-diffraction study on $RuSr_2GdCu_2O_8$. The high-field data suggest that the dominant high magnetic-field order is ferromagnetic.

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