Towards universal magnetization curves in the superconducting state of $RuSr_2GdCu_2O_8$

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The reported dc magnetization measurements on the magnetic ($T_M \sim 130$ K) superconductor ($T_c \sim 30$ K) $RuSr_2GdCu_2O_8$ (Ru-1212Gd) reveal a variety of behaviors below T_c. The fact that, for magnetometers which require sample motion during the measurements, artifacts can arise in the measured magnetic moment, when the movement of the sample is done in an inhomogeneous field, complicates even more the analysis of the existing data. In order to avoid the generation of artifacts, we did measurements on a stationary Ru-1212Gd sample employing a homemade magnetometer. The measured curves showed none of the suspicious "symptoms" present in the curves measured with a magnetometer employing sample movement, and if verified by measurements on stationary samples by other groups, a universal behavior in the superconducting state of Ru-1212Gd could be revealed by the dc magnetization measurements. Our considerations support the existence of bulk superconductivity for Ru-1212Gd.

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I. INTRODUCTION

The ruthenium cuprates of the general chemical formulas $RuSr_2(R)Cu_2O_8$ (1212 type) and $RuSr_2(R_{1+x}Ce_{1-x})Cu_2O_8$ (1222 type), where $R=$ Sm, Eu, and Gd, synthesized in 1995 (Ref. 1–3) have attracted a lot of attention because, as was shown for the first time by Bauernfeind,[2,4] in these compounds superconductivity arises in a state in which magnetic order is already developed. The difference between the superconducting transition temperature T_c and the magnetic transition temperature T_M is of the order of 100 K. This is in contrast to what is known for other magnetic superconductors like the molybdenum sulfides[5,6] and selenides,[7,8] the rhodium borides,[9,10] and the borocarbides,[11,12] where T_c and T_M are close, with the magnetic transition appearing usually below the superconducting one.

A large number of investigations was undertaken in an attempt to determine the type of superconductivity and magnetic ordering and whether the two phenomena coexist on a microscopic scale. Magnetic studies include muon spin rotation experiments,[13] electron paramagnetic resonance (EPR) experiments,[14] neutron powder diffraction (NPD),[15–17] and NMR investigations[18] as well as dc magnetization and ac susceptibility measurements.[19–22] Some of the findings contradict each other [e.g., NPD (Refs. 16 and 17) and NMR (Ref. 18) investigations] and still there is no agreement on the type of magnetic ordering in the ruthenium cuprates. Nevertheless, it seems to be widely accepted that magnetism represents a bulk property of these compounds.

Whether superconductivity as well represents a bulk property of the ruthenium cuprates has been investigated by both specific heat and dc magnetization measurements. Nevertheless, concentrating on $RuSr_2GdCu_2O_8$ (Ru-1212Gd), the subject of the present paper, interpretation of the specific heat data is rather difficult, since the existing reports[23,24] are contradictive concerning the magnetic field dependence of the specific heat peaks below T_c.

Much more complicated is the interpretation of the dc magnetization data. In principle, field expulsion shown in a field-cooled dc magnetization measurement, corresponding to a bulk Meissner effect, is generally considered as the key indicator for bulk superconductivity. However, a variety of behaviors have been reported for Ru-1212Gd below its superconducting transition temperature T_c (see, for example, Refs. 25–28), leaving the question of bulk superconductivity for this compound open. Nevertheless, it has been shown[29,30] that Ru-1212Gd is sensitive to field inhomogeneities in the superconducting magnet of the superconducting quantum interference device (SQUID) magnetometer, which affect the SQUID response as the sample is moved in the magnet during the measurements and can create artifacts in the measured magnetic moment. In order to eliminate possible artifacts, we did measurements on a stationary Ru-1212Gd sample. We suggest that, if similar measurements are done by other groups also, universal dc magnetization curves indicating bulk superconductivity for Ru-1212Gd could be obtained.

II. EXPERIMENT

Details about the sample preparation and characterization in terms of x-ray powder diffraction can be found in our previous work.[25] Here we note only that all our samples belong to the same batch, meaning that they were prepared and heat treated together.

Two SQUID magnetometers were employed for the dc magnetization measurements. One of them was a commercial (Cryogenic Consultants Ltd. S600) rf-SQUID magnetometer, which allows measurements in the temperature range $1.6\ \mathrm{K} \leqslant T \leqslant 300\ \mathrm{K}$ in magnetic fields $-6\ \mathrm{T} \leqslant B \leqslant 6\ \mathrm{T}$. This magnetometer necessitates the movement of the sample through a pickup coil system (second-order gradiometer) for the measurements. The SQUID response to this movement is fitted using the ideal response for a point dipole of constant magnetic moment and the sample's magnetic moment at the temperature of the measurement is calculated. In the following, this magnetometer will be denoted as MSM (moving sample magnetometer).

The second magnetometer was a homemade system employing a niobium rf-SQUID of the type SHE 330 (SHE Co., San Diego). With this system we did measurements in the temperature range $4.5\ \mathrm{K} \leqslant T \leqslant 150\ \mathrm{K}$. The cryostat is equipped with a superconducting solenoid, made from NbTi-based wire, that was used for measurements in magnetic fields up to 100 G. In this second magnetometer the sample is kept stationary during the measurements and what is actually measured, using a flux counter from SHE Co., is the flux change through the pickup coil system, which can be transformed to the corresponding change of the magnetic moment of the sample during the measurement. Thus, measurements of absolute values of the magnetic moment require a reference point. In our case, since Ru-1212Gd is in a paramagnetic state above the magnetic transition temperature $T_M \sim 130\ \mathrm{K}$, we assumed that the magnetic moment of the sample M is zero at 150 K. In the following this second magnetometer will be denoted as SSM (stationary sample magnetometer). This magnetometer has been used for several studies in the past.[31–33] A similar system is described in detail by Vandervoort *et al.*[34]

Two types of measurements were done with the SSM. For the zero-field-cooled (ZFC) measurements the sample was cooled from above 150 K to the lowest temperature in zero (set value) magnetic field; then the magnetic field was applied and the measurements were taken during warm-up. For the field-cooled (FC) measurements the samples were agained cooled from above 150 K, but in the desired magnetic field. The FC measurements were taken also during warm-up, since exchange He gas was required to cool the sample to the lowest temperature, which made temperature controlling for measurements on cooling difficult.

III. RESULTS AND DISCUSSION

A. Magnetism and superconductivity of our $RuSr_2GdCu_2O_8$ samples

1. Magnetism

In Fig. 1, $M(T)$ and $M(B)$ measurements for our Ru-1212Gd samples are shown. A magnetic transition is obvious at $T_M \sim 133$ K with significant hysteresis between the ZFC and FC branches of the $M(T)$ measurement starting at this temperature. Hysteresis loops indicative of a ferromagnetic component in the magnetic behavior of the samples are revealed in the $M(B)$ measurements. The loops become wider as the measuring temperature decreases, with the remanent moment reaching $\sim 0.1\mu_B$ per formula unit at low tempeartures. In view of the contradicting reports cited in Sec. I it is difficult to propose an origin for the observed properties.

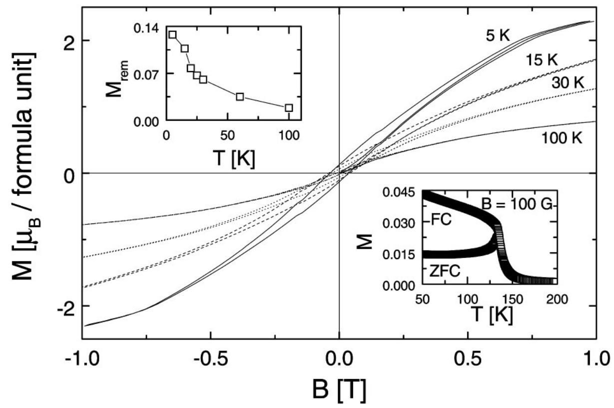

FIG. 1. High-field magnetic hysteresis loops for Ru-1212Gd taken with the MSM. The field was changed between -6 and 6 T but for clarity only the lower-field part is shown. Insets: In the lower right side, magnetic moment measurements as a function of temperature are shown. In the upper left side, the remanent magnetic moment as determined by hysteresis loops at different temperatures is given.

The behavior observed for our samples, though, is the typical one observed in similar measurements by many other groups. For comparison, $M(T)$ and $M(B)$ measurements on Ru-1212Gd samples can also be found, for example, in Refs. 13 and 17.

2. Superconductivity

Typical resistance and ac susceptibility measurements for our samples can be found in our previous works.[25,29] A comparison with other published ac susceptibility measurements,[15,27,35,36] which, like in our case, are usually done with the sample stationary in the pickup coil system, and resistivity measurements[13,23,24,27,35] shows that the superconducting properties of our samples are the usual ones for "good quality" Ru-1212Gd samples ($T_{c,onset} = 50$ K, $T_c(R=0) = T_c = 30$ K).

B. dc magnetization measurements using a MSM

As described above, our samples show the typical magnetic and superconducting, in terms of resistance and ac susceptibility measurements, behavior that all "good quality" Ru-1212Gd samples show and thus could be considered as universal. On the other hand, the superconducting behavior of the Ru-1212Gd samples in terms of dc magnetization measurements is far from universal. The measurements we did on our samples with the MSM have shown below T_c features similar to many of the different behaviors reported by other research groups.[26–28] Nevertheless, we have shown[29,30] that the measuring procedure in a MSM can create artifacts in the measured magnetic moment below T_c, when magnetic field inhomogeneities are present over the distance that the sample is moved during the measurements. As was shown by Libbrecht *et al.*,[37] the form that these artifacts will have for a superconducting sample will be determined by the shape of the field profile, which in turn determines the field change that the sample will experience during

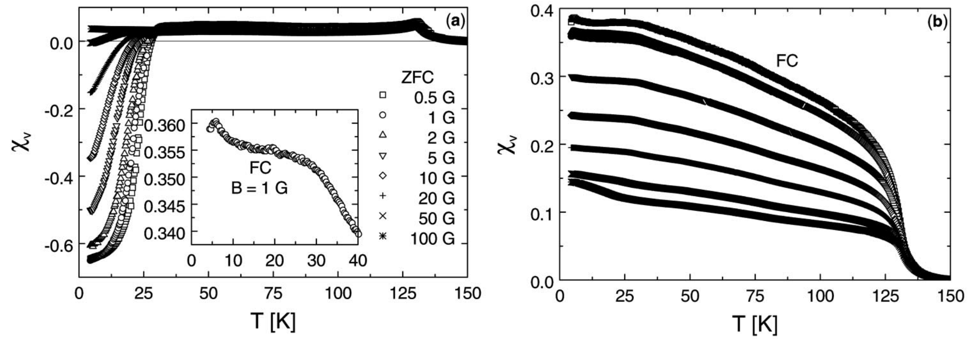

FIG. 2. (a) Volume susceptibility calculated from ZFC dc magnetization measurements on Ru-1212Gd taken with the SSM. Inset: the low-temperature part of the FC curve measured in a field of 1 G. (b) Volume susceptibility calculated from FC dc magnetization measurements on the same sample. Since in this set of measurements the high density of points makes it difficult to distinguish between the different symbols, we should note that the higher the field, the lower the measured susceptibility. Only the curve measured in a set field of 2 G shows slightly higher values of χ_V compared to that measured in 1 G.

its movement. Thus, the differences in the reported dc magnetization of Ru-1212Gd below T_c could be the result of different field profiles during the measurements and not of different superconducting properties.

The above discussion indicates that dc magnetization measurements on stationary Ru-1212Gd samples are required to clarify the superconducting properties of this compound. Such measurements could reveal a universal superconducting behavior for Ru-1212Gd in terms of dc magnetization also. We will present dc magnetization measurements on a stationary Ru-1212Gd sample in the next section. Nevertheless, the establishment of a universal behavior requires that our measurements be verified by measurements on stationary ''good quality'' Ru-1212Gd samples done by other research groups.

C. Measurements on a stationary $RuSr_2GdCu_2O_8$ sample

We have already presented preliminary measurements on a stationary Ru-1212Gd sample in Fig. 8(b) of Ref. 29. Nevertheless, those measurements were confined to only one field value and were also noncalibrated as far as both the magnetic moment and temperature are concerned. Here, calibrated measurements in several magnetic fields will be discussed. These measurements, done with the SSM, are shown in Fig. 2. In this figure we chose to show the volume susceptibility in SI units so that estimations of the superconducting volume of the sample can be made. For the calculation we used a value of the density $\rho = 6.7$ g/cm^3, estimated using the lattice parameters measured previously.[25] The susceptibility of the spherical sample was corrected for geometric demagnetization using the demagnetization factor $N = 1/3$.

1. No suspicious "symptoms"

We have observed that the measurements taken with the MSM show several suspicious ''symptoms,'' when artifacts are present. These will be discussed in detail in a separate article.[38] Here, as an example, we mention the nonreversal of the features observed in the superconducting state of Ru-1212Gd by a field reversal, as we have shown in Ref. 29. This point was independently verified by Cimberle *et al.*[39] In their case,[39] clear dips in both ZFC and FC curves, indicative of bulk superconductivity in Ru-1212Gd, were not reversed by the application of a negative field. The authors attributed the nonreversal of the ZFC dips to effects related with the remanent field in the superconducting magnet, but provided no explanation for the nonreversal of the dips in the FC curves. Nevertheless, they state clearly that at the superconducting transition their SQUID magnetometer, also a MSM, indicates a worsening of the quality of the measurement through the regression factor and the answer function that tends to lose its symmetry.

Contrary to the measurements with the MSM, no suspicious ''symptoms'' were observed for the measurement with the SSM. Going back to the previous example, in Fig. 3 it can be seen that the ZFC and FC measurements taken with opposite field directions are almost ''symmetric'' with respect to zero. The small differences can be attributed to not quite identical field values in the superconducting magnet of

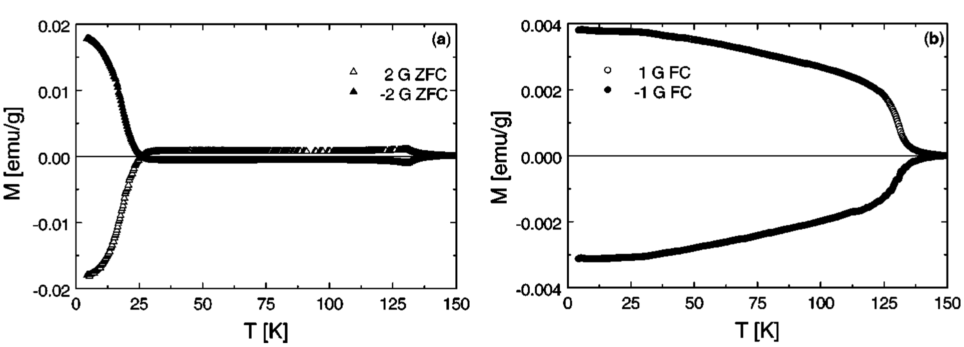

FIG. 3. (a) ZFC dc magnetic moment of Ru-1212Gd in 2 G and −2 G. (b) FC dc magnetic moment of the same sample in 1 G and −1 G. All measurements were taken with the SSM.

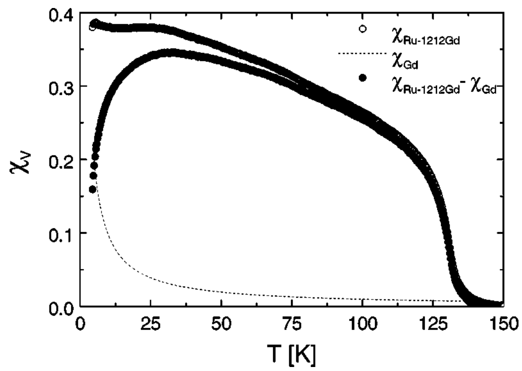

FIG. 4. Volume susceptibility of Ru-1212Gd (solid circles) after the Gd paramagnetic contribution (dashed line) was subtracted from the measured FC curve in a field of 0.5 G (open circles).

the SSM for the positive and negative directions during the measurements. The absence of any peculiar ‘‘symptoms’’ from the measurements taken with the SSM underlines the validity of these measurements.

2. Question of bulk superconductivity for $RuSr_2GdCu_2O_8$

From the above discussion, it is obvious that the measurements on stationary samples are probably the most trustworthy for a discussion whether Ru-1212Gd is a bulk superconductor or not. The ZFC measurements of Fig. 2(a) show that at low fields more than 60% of the sample is shielded from the magnetic field. This alone, however, cannot be considered as an indication for bulk superconductivity. Although it would be very difficult to create the observed shielding signal by a superconducting impurity in a concentration nondetectable with x-ray diffraction, surface superconductivity could not be excluded.

The signature of bulk superconductivity is the Meissner effect which, if present, appears in the FC curves as a magnetization decrease consistent with field expulsion from the sample. Such a magnetization decrease does not appear in the measurements of Fig. 2(b), but neither is the paramagnetic contribution from the Gd moments apparent in the low-field measurements; it is obvious only in the 100-G measurement. Instead, a shallower slope of the susceptibility is observed below T_c, indicating a competition between the field expulsion due to superconductivity and the contributions from the Gd and Ru moments. This is more clearly seen in the inset of Fig. 2(a). In order to estimate the contribution from the superconducting part of the sample we have subtracted from the data the Gd paramagnetic contribution. Assuming noninteracting Gd moments, we have calculated their contribution to the measured susceptibility using the Brillouin function.[40] For Gd we used the data in tables 31.2 and 31.3 of Ref. 40. The result of this procedure for the measurement in a field of 0.5 G is shown in Fig. 4. The Ru contribution at low temperatures corresponds to the ferromagnetic component and varies slowly as a function of temperature. The variation is small compared to that due to the Gd. Thus, assuming a constant Ru contribution at low temperatures, it can be easily seen in Fig. 4 that its subtraction (from the data where the Gd contribution is already subtracted) will lead to a diamagnetic signal indicative of field expulsion from about 20% of the sample’s volume.

Although field expulsion from 20% of the sample’s volume represents an indication of bulk superconductivity, it rises the question of coexistence of superconductivity and magnetism on a microscopic scale. Muon spin rotation experiments,[13] for example, indicate that the magnetic phase in Ru-1212Gd is homogeneous and accounts for at least 80% of the sample volume. Although this is a lower limit,[13] one could propose a phase separation model where bulk magnetism coexists with bulk superconductivity in Ru-1212Gd, not on a microscopic scale, but rather in different areas of the sample. There are several reasons, though, which could keep the FC superconducting contribution to the magnetization low despite superconductivity in the full sample volume. Bernhard *et al.*[26] report for polycrystalline Ru-1212Gd samples a grain size between 2 and 10 μm, while Chu *et al.*[41] estimate an unusually large penetration depth of about 50 μm. Grains, or clusters of grains, with size smaller than the penetration depth will not expel the magnetic field in a FC process and a reduced diamagnetic signal will be recorded. Thus, the reduced FC superconducting contribution can be the result of grain size effects while magnetism and superconductivity coexist on a microscopic scale. Furthermore, a Meissner state is not the only superconducting state which could be considered for Ru-1212Gd. In a magnetic superconductor, if the internal field exceeds the first critical field H_{c1}, then this will be accommodated in the sample in the form of vortices (spontaneous vortex phase).[42,43] A vortex phase will result in a reduced diamagnetic signal compared to a Meissner state, but it is a bulk superconducting state which could coexist with magnetism also on a microscopic scale.

Indications that Ru-1212Gd is a bulk superconductor can be found also in the measurements taken with the MSM. In Ref. 29 it can be seen that artifacts related to the movement of the sample in a nonhomogeneous field dominate the low-temperature part of the FC curves. We expect that, if surface superconductivity was present, then the Gd contribution, from the interior of the grains, would dominate the behavior of the sample at low temperatures. On the other hand, a bulk superconducting state, possibly in the form of weakly pinned vortices, as is indicated by the narrow hysteresis loops below T_c,[4,30] is much more sensitive to field inhomogeneities, which will affect the measured magnetic moment.

IV. SUMMARY

Whereas resistivity and ac susceptibility measurements reveal a universal superconducting behavior for good quality Ru-1212Gd samples, this is not the case with the dc magnetization measurements. The reported SQUID measurements in the superconducting state of Ru-1212Gd reveal a variety of behaviors, leaving the question of bulk superconductivity for this compound open. Based on the observed[29] sensitivity of Ru-1212Gd to magnetic field inhomogeneities, when it is moved in the superconducting magnet of the SQUID magnetometer during the measurements, we suggest that the reported different behaviors can be the result of different field profiles in the superconducting magnet and not of different superconducting properties. In order to avoid the artifacts arising from moving the sample in an inhomogeneous field, we did measurements on a stationary Ru-1212Gd sample employing a homemade SQUID magnetometer. Our data showed none of the suspicious ''symptoms'' that measurements with a magnetometer employing sample movement show when artifacts are present (e.g., no reversal of the features observed in the superconducting state of Ru-1212Gd by a field reversal). This underlines the validity of our measurements. To the best of our knowledge, this is the first time that measurements on stationary Ru-1212Gd samples were presented. Verification of our measurements by measurements on stationary good quality samples from other groups could reveal a universal behavior in the superconducting state of Ru-1212Gd in terms of dc magnetization measurements also. Our considerations support the existence of bulk superconductivity for Ru-1212Gd.

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