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Magnetization, vortex state and specific heat in the superconducting state of $RuSr_2GdCu_2O_8$

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Abstract. Based on field-cooled dc-magnetization measurements in a SQUID magnetometer with carefully controlled magnetic-field profiles, we present evidence that diamagnetism is missing in the superconducting state ($T < 50$ K) of the (weakly) ferromagnetic ($T_M \approx 130$ K) superconductor $RuSr_2GdCu_2O_8$ (Ru1212). Nevertheless, taking into account the granular nature of the samples investigated so far, this cannot be taken as evidence for the lack of a Meissner state or bulk superconductivity. It is shown that for low applied magnetic fields a vortex state most probably involves the intergrain area (area between the grains) rather than the individual grains (bulk Ru1212). Furthermore, the wide superconducting transition of Ru1212 ($T_{c,onset} = T_c \approx 48$ K, $T_s(R = 0) = T_s = 32$ K) realized in resistance measurements in zero applied magnetic field can be readily understood as the effect of resistive grain contacts and is not necessarily related to the movement of spontaneously induced vortices in bulk Ru1212, as it has been suggested previously. A comparison of the low-temperature specific heat of Sr_2GdRuO_6 (Sr2116), the precursor for the preparation of Ru1212 and thus a possible impurity phase, with previously reported data for Ru1212 shows that it is unlikely that Sr2116 is responsible for the specific-heat features attributed to the superconductivity of Ru1212 and supports the existence of a bulk superconducting state in the latter compound.

PACS. 74.72.-h Cuprate superconductors (high-T_c and insulating parent compounds) – 74.25.Ha Magnetic properties – 75.50.-y Studies of specific magnetic materials

1 Introduction

The high-temperature superconducting cuprate $RuSr_2GdCu_2O_8$ (Ru1212) has been the subject of intense investigations as it offers the rare opportunity to study how superconductivity develops in an already magnetically ordered state [1]. A clear picture of the magnetic and superconducting properties of this compound though is not available yet. On the one hand, for example, neutron diffraction studies [2] suggest a G-type antiferromagnetic structure for both the Ru and Gd moments with ordering temperatures of 136 and 2.5 K, respectively. NMR [3] and magnetization [4] investigations on the other hand favor a ferrimagnetic [3] or type-I antiferromagnetic [4] arrangement of the Ru moments with antiferromagnetically coupled ferromagnetic RuO_2 planes.

The superconducting properties of Ru1212 are also far from being understood. It is known that they are significantly affected by the preparation conditions and long annealing in oxygen atmosphere is necessary to obtain good quality samples [5]. In this case, resistance measurements in zero applied magnetic field show a rather wide superconducting transition with $T_{c,onset} = T_c \approx 50$ K and $T_s(R = 0) = T_s \approx 30$ K. The superconducting state of the sample though is not clear. Bernhard et al. [6] suggest a Meissner state at $T < T_s$. Contrary, the absence of such a state is suggested by Xue et al. [7]. Another interesting possibility arises from the fact that magnetic order is already developed at $T > T_c$. This could lead to the creation of vortices, when the sample enters the superconducting state, even in the absence of external magnetic field, if the internal magnetic field is greater than H_{c1}. The wide superconducting transition that characterizes Ru1212 has been recently attributed to the movement of spontaneously induced vortices in the sample [8].

In the present paper, we concentrate our attention to the superconducting properties of Ru1212. We provide experimental evidence that a bulk diamagnetic signal is generally missing in field-cooled dc-magnetization measurements of Ru1212 below T_c. Nevertheless, this cannot exclude the existence of a Meissner state or bulk superconductivity since the dc-magnetization investigations so far involved granular samples. This is true also for the dc-magnetization data reported by Lin at al. [9], where a lump of many micrometer-sized single crystals was

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measured. We show that an induced vortex state for low applied magnetic field or even a spontaneous vortex state (SVS) for zero applied magnetic field would more probably be formed in the intergrain area (area between the grains). Furthermore, the wide superconducting transition of Ru1212 can as well be attributed to resistive grain contacts in the temperature range $T_s < T < T_c$.

Specific-heat measurements represent an alternative way to investigate the bulk character of superconductivity in Ru1212, which is not expected to be affected by granularity. It has been suggested though [10], that the specific-heat features reported previously [11] and supporting the existence of bulk superconductivity in Ru1212, might as well be due to Sr_2GdRuO_6 (Sr2116), the precursor for the preparation of Ru1212 [12] and thus a possible impurity phase, which is known to order magnetically in the temperature range where Ru1212 becomes superconducting [13]. We present specific-heat measurements of a Sr2116 sample and show that this is most probably not the case.

2 Experimental

Details about the preparation and characterization in terms of X-ray powder diffraction of the granular (polycrystalline) Ru1212 and Sr2116 samples can be found in a previous work [13]. As we will show below, the investigated Ru1212 sample shows the typical properties of good-quality samples. The magnetic transition takes place at $T_M \approx 130$ K whereas $T_c \approx 48$ K and $T_s \approx 32$ K.

The presented dc-magnetization and ac-susceptibility measurements were performed in a Quantum Design magnetic properties measurement system (MPMS). The ultra-low-field option of this instrument allows the measurement of the magnetic-field profile along the axis of the superconducting magnet and can be used to achieve highly homogeneous low magnetic fields. For the dc-resistance and specific-heat measurements the physical property measurement system (PPMS) of the same manufacturer was used. The resistance measurements were performed using a standard four-wire configuration whereas the specific-heat measurements involve a thermal-relaxation calorimeter [14]. Both instruments are part of the equipment of the new Dresden High Magnetic Field Laboratory (a user facility for pulsed high magnetic fields up to 100 T/10 ms) [15,16].

3 Results and discussion

3.1 dc-magnetization and lack of diamagnetism in the superconducting state of Ru1212

One of the factors that complicates a deeper insight into the superconducting state of Ru1212 is the variety of behaviors observed in dc-magnetization measurements below T_s (e.g. Refs. [6,7,10,17–19]), that led to corresponding diverse interpretations (Meissner state, SVS, crypto-superconducting structure etc.). In a series of previous

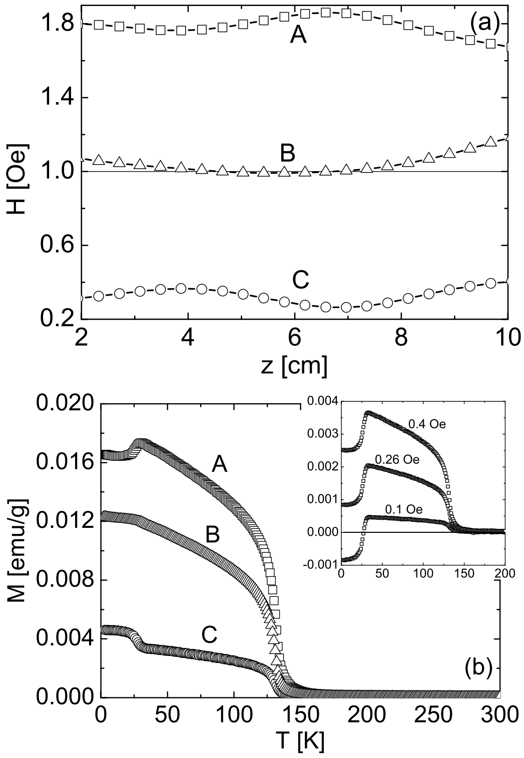

Fig. 1. (a) Measured magnetic-field profiles along the axis of the superconducting magnet in the MPMS. The center of the magnet is located at $z = 6$ cm. (b) Mass magnetization of Ru1212 measured in the magnetic-field profiles A, B, and C, respectively. Curves A and C were measured moving the sample over the whole corresponding field profile. Curve B was measured with a scan length of only 0.5 cm around the center of the magnet. Inset: measurements in magnetic-field profiles similar to profile A. The magnetic-field values given are those measured in the center of the magnet. All data were collected with increasing temperature after the sample was field cooled.

investigations [20–23] though, we were able to show that when the measurements are performed in a SQUID setup moving the sample in an inhomogeneous magnetic field then artifacts in the dc-magnetization may arise below T_s. Our approach implied that the variety of reported behaviors could be the result of measurements performed in different magnetic-field profiles, although the physics that governs the response of the investigated samples is essentially the same. Nevertheless, direct experimental evidence to support this point of view was missing and will be presented in the following.

In Figure 1a, we show three different magnetic-field profiles measured along the axis of the superconducting magnet in the MPMS. The magnetic-field profiles A and C were measured after a field value of 1 Oe was set to the magnet. Depending on the "history" of the superconducting magnet the remanent field is affected. In one case (profile A), the real value of the magnetic field was closer to 1.8 Oe and in the second case (profile C) closer to 0.3 Oe. The maximum field change measured over the distance of

8 cm around the center of the magnet shown in Figure 1a was about 0.2 Oe for profiles A and C.

In Figure 1b, we show the dc magnetization of the same Ru1212 sample measured in the magnetic-field profiles A and C. The sample was moved over the whole distance shown in Figure 1a (scan length 8 cm) during the measurement. At $T > T_s = 32$ K both measurements show the same features with the magnetic transition setting in at about 130 K. The differences in the measured values of the dc-magnetization in this temperature range is because of the different values of the real applied magnetic field. At $T < T_s$, though, different features are observed. The data collected in the magnetic-field profile A show a drop of the magnetization close to T_s followed by a kind of plateau at lower temperatures whereas those collected in profile C look rather like the "mirrored" feature with an increase of the magnetization close to T_s. We have also observed that small changes in the set value of the magnetic field simply shifted the field profile A, but did not change its shape. As shown in the inset of Figure 1b, for sufficiently low magnetic fields the measured magnetization even turned negative. Our findings are direct experimental evidence that the magnetic-field profile in which the dc-magnetization measurements are performed determines the features that will be observed below T_s although the underlying physics characterizing the superconducting state of Ru1212 is the same. Furthermore, it has been demonstrated that apparently systematic features can be simply the result of a reproducible field profile. We should note that by modifying the magnetic-field profile in the magnet we obtained measurements that resemble many of the previously reported data. The measurements in the field profiles A and C of Figure 1b resemble data reported in references [6] and [17], respectively.

Thus, an interesting question arises concerning the real behavior of Ru1212 at $T < T_s$. From the above discussions it becomes obvious that for reliable dc-magnetization measurements inhomogeneous magnetic-field profiles should be avoided. The real behavior of the magnetization of Ru1212 below T_s can be revealed either in a magnetometer that does not necessitate sample movement during the measurement [22] or, as in the case of the available MPMS, when the magnetic field is highly homogeneous. Using the ultra-low-field option of the MPMS we were able to achieve a high field homogeneity close to the center of the magnet. This is obvious in the field profile B shown in Figure 1a. The magnetic field is very close to the desired value of 1 Oe showing practically no inhomogeneity close to the center of the magnet ($z = 5$–7 cm). The magnetization of the sample measured in this field profile with a short scan length (0.5 cm) near the center of the magnet is very similar to the data obtained in a home-made magnetometer avoiding sample movement [22]. At $T < T_s$ a change of slope of the measured curve is observed, but the diamagnetic signature of a bulk Meissner state is missing. Nevertheless, as we will show below, the granular nature of the samples has to be carefully considered in the interpretation of the data.

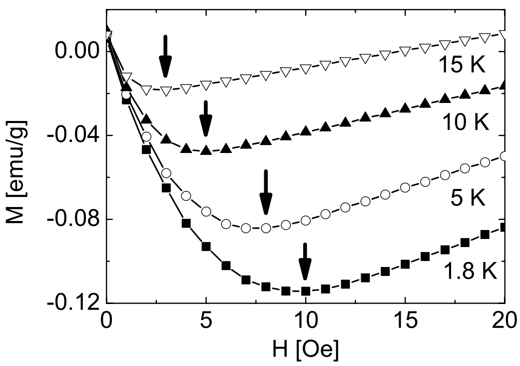

Fig. 2. Magnetization of our Ru1212 sample at low applied magnetic fields for $T = 1.8$ K, 5 K, 10 K, and 15 K.

We note that the lack of diamagnetism in the superconducting state of Ru1212 reported here refers to the field-cooled (FC) dc-magnetization measurements where one would look for evidence of magnetic-field expulsion from the sample (Meissner state). Zero-field-cooled (ZFC) dc-magnetization measurements can show diamagnetism [22] but this is related to the shielding of the magnetic field which might as well by caused by surface superconductivity and is not necessarily related to a bulk superconducting phase.

3.2 Granularity and vortex state

Apart from temperature-dependent dc-magnetization measurements, $M(T)$, magnetic-field-dependent measurements, $M(H)$, have also been used recently to define a Meissner region in the phase diagram of Ru1212 [8]. The average lower critical field, H_{c1}, was determined from the peaks of the virgin $M(H)$ curves measured at $T < T_c$. These peaks are marked with arrows in the $M(H)$ curves presented in Figure 2. This definition of H_{c1} is highly uncommon and in the following we will show that it is most probably not related to the bulk H_{c1}.

In Figure 3, we show the low-temperature part of the ZFC volume susceptibility, χ_V, of Ru1212 extracted from dc-magnetization measurements presented in a previous work [22]. There are two distinct points where χ_V rapidly changes its slope, marked with T_{intra} and T_{inter}. Similar features in reference [24] were attributed to the intragrain (T_{intra}) and intergrain (T_{inter}) transitions of the granular material. Indeed, it is well known [25] that granular high-temperature superconductors can be viewed as an array of Josephson-coupled superconducting grains. T_{intra} is the temperature where the grains become superconducting and start to shield the magnetic field. This shielding is expressed with the shallow decrease of χ_V below T_{intra}. The magnetic field remains in the intergrain area (area between the grains). At some lower temperature, T_{inter}, the coupling between the grains is established and the whole sample shields the magnetic field. This explains the rapid decrease of χ_V below T_{inter}. Actually, the definition of T_{inter} shown in Figure 3 is somewhat ambiguous since

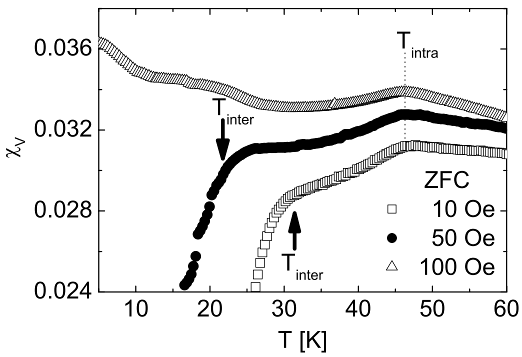

Fig. 3. Temperature dependence of the ZFC volume susceptibility of Ru1212 measured in 10, 50, and 100 Oe.

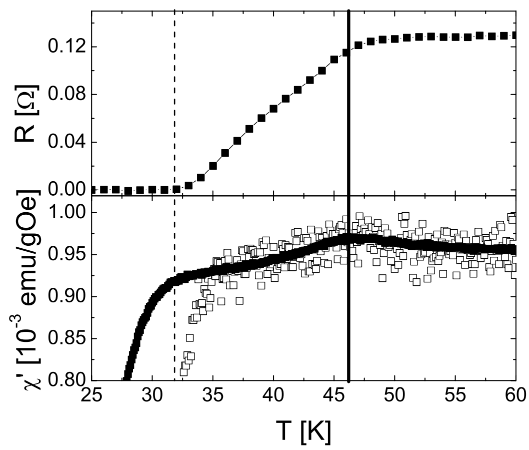

Fig. 4. (Top) Low-temperature resistance measurement of Ru1212 in zero applied magnetic field. (Bottom) Low-temperature details of the real part of the ac susceptibility of the same sample. The excitation-field amplitude was 0.5 Oe (open squares) and 3.9 Oe (filled squares). No dc field was applied. The excitation frequency was 22.2 Hz.

very often it is the point of maximum slope at lower temperatures which is defined as T_{inter}, but this does not alter the following discussion.

The application of low magnetic fields up to 100 Oe does not affect significantly the intragranular transition. On the other hand, the intergranular transition is clearly shifted to lower temperatures and no shielding of the magnetic field is observed at 100 Oe. These findings illustrate the well-known fact that it is much easier for the magnetic field to penetrate between the grains than into the grains and is consistent with the suggestion of Clem [25] that a granular material is characterised by two lower critical fields H_{c1}, denoted as H_{c1J} and H_{c1g} with $H_{c1J} < H_{c1g}$. At H_{c1J} the field forms vortices between the grains and at H_{c1g} within the grains. It is, therefore, most plausible that the main features in the magnetization curves of Figure 2 are caused by intergrain effects and not by the bulk Ru1212, as it is suggested in reference [8].

Thus, the lack of diamagnetism observed in the superconducting state of Ru1212 is not necessarily related with the lack of bulk superconductivity. It could be caused by a vortex state, spontaneous or induced, with vortices pinned in the intergrain area. This would be a particular vortex state related to the granularity of the samples investigated so far and the "softness" that the intergrain area shows to the penetration of the magnetic field. The lack of dc-magnetization measurements of single-crystalline samples complicates the elucidation of the state of the individual grains (bulk Ru1212). These might as well be in a Meissner state for the low magnetic fields used in the present study. The change of slope observed in the dc magnetization at $T < T_s$ (curve B in Fig. 1b) indicates that there is indeed field expulsion from the sample (no increase due to the Gd paramagnetic contribution is observed as for non-superconducting samples). We note that transport measurements on micrometer-sized single crystals are available [26] suggesting also the bulk character of superconductivity in Ru1212.

It should also be noted that the concept of SVS formation has been also used to explain the wide superconduction transition of Ru1212 as this is realized in resistance measurements in zero applied magnetic field. The wide transition is attributed to the movement of spontaneously induced vortices [8,27]. Concerning this issue an interesting situation is realized in Figure 4. The top part of this figure shows the resistive transition with a width of $\Delta T = T_c - T_s \approx 15$ K. The bottom part shows an ac-susceptibility measurement obtained with a very low excitation field of 0.5 Oe. This measurement was rather noisy above 35 K and in order to make the position of the intragranular transition more clear we have included in the same figure a measurement with much better resolution obtained with an excitation field of 3.9 Oe. As we have shown above, small changes of the magnetic field do not affect the position of the intragranular transition. It is obvious that T_c fits rather well with T_{intra} (solid line) whereas T_s with T_{inter} (dashed line). Thus the wide superconducting transition of Ru1212 also seems to be related to intergrain effects. It arises from resistive grain contacts and is not necessarily related to the movement of spontaneously induced vortices in bulk Ru1212 at $T_s < T < T_c$.

3.3 Specific heat

Granularity is not expected to influence specific-heat measurements which represent an alternative way to study the bulk character of superconductivity in Ru1212. In Figure 5, we show the specific heat, C/T, of our Ru1212 sample measured in zero applied magnetic field. The magnetic transition at $T_M \approx 130$ K is realized as a cusp in the specific heat which is more clearly seen in the inset. Nevertheless, no features related to the superconducting transition at T_c were observed. Given the large magnetic contribution to C/T near T_c, this result should not be taken to put in question the bulk character of superconductivity in Ru1212. The curve is actually very similar to those reported by Tallon et al. (see Fig. 5 in Ref. [11]). In this report, the authors subtracted from the specific heat of a superconducting sample that obtained for a

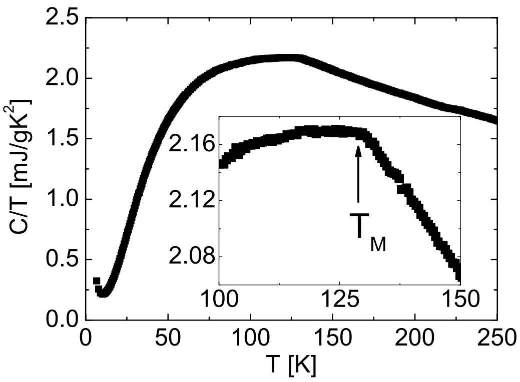

Fig. 5. Specific heat of Ru1212 measured in zero applied magnetic field.

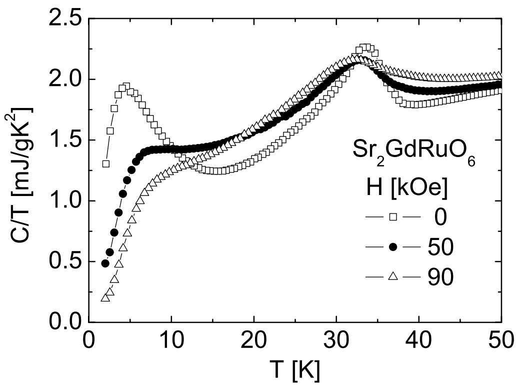

Fig. 6. Specific heat of Sr_2GdRuO_6 measured in different magnetic fields.

non-superconducting one (3% Zn-substituted Ru1212). They assumed that this second sample accounted for all contributions to the specific heat other than superconductivity. In that way they were able to observe peaks in the specific heat close to T_c. Nevertheless, in a previous work [13], we have presented evidence of magnetic ordering of both the Ru and Gd moments in the compound Sr_2GdRuO_6 (Sr2116) at about 30 and 3 K, respectively. Sr2116 was used as a precursor for the preparation of the Ru1212 samples on which the specific-heat measurements were performed [11]. Chu et al. [10] argued that this compound (Sr2116) could be responsible, as an impurity phase, for the specific-heat features observed for Ru1212 below T_c. In the following, we will discuss this possibility.

Figure 6 shows the specific heat of a Sr2116 sample measured in different magnetic fields up to 90 kOe. The curve measured in 0 Oe shows two peaks at 34 K and 4.5 K, respectively related to the magnetic-ordering effects reported previously [13]. We will concentrate our attention to the peak observed at 34 K since it is close to T_c of Ru1212. The size of this peak, $C/T(34\ \mathrm{K})$–$C/T(40\ \mathrm{K})$, is about 0.47 mJ/gK^2 at $H = 0$ and is obviously decreasing with the application of a magnetic field. The position of the peak is slightly shifting from 34 K at 0 Oe to 32 K at 90 kOe.

This behavior cannot account for the findings of Tallon et al. which report an increase of T_c by 4.5 K, when the magnetic field is increased to 50 kOe [11]. They consider this as evidence for triplet pairing in Ru1212. It should be noted though that in a report published later by Chen et al. [28] a more conventional behavior is reported for Ru1212 with a slight decrease of T_c when a magnetic field of 60 kOe was applied. This behavior resembles that reported above for Sr2116 and Chen et al. reported that their samples indeed contain Sr2116 impurities. Even so, it is difficult to attribute the features observed for Ru1212 to Sr2116 impurities. The size of the peak measured in zero field for the Ru1212 sample was about 0.08 mJ/gK^2 [28]. Taking into account the size of the Sr2116 peak in zero field given above, about 16% of the sample's mass should be attributed to Sr2116 impurities in order to explain this specific-heat anomaly. Such an amount of impurity would create more than one weak peak on the X-ray pattern of Ru1212, as it is stated in [28].

4 Conclusions

Field-cooled dc-magnetization measurements in well characterized magnetic-field profiles show that the observed features in the superconducting state of Ru1212 depend on the specific field profile. This explains the variety of behaviors reported previously and illustrates that the underlying physics is most probably the same.

Our findings suggest that the lack of diamagnetism in the field-cooled dc-magnetization below T_c is characteristic for the polycrystalline samples. This is not necessarily related with the lack of bulk superconductivity. A certain amount of field expulsion is evident by the slope change of the field-cooled dc-magnetization curves at low temperatures. Furthermore, the granular nature of the samples has also to be carefully taken into account since the magnetic field can penetrate more easily in the intergrain area than into the grains (bulk Ru1212).

A critical review of previously reported specific-heat data of Ru1212 with respect to the specific heat of Sr2116, the precursor for the preparation of Ru1212 and thus a possible impurity phase, also supports the existence of bulk superconductivity in Ru1212. We show that Sr2116 cannot account for the specific-heat anomalies reported for Ru1212 at T_c. The elucidation of the pairing mechanism in Ru1212 though requires further investigations.

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