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Superconductivity and specific heat of $Ru_{1-x}Ta_xSr_2GdCu_2O_8$ and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$

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Abstract. Samples of the compounds $Ru_{1-x}Ta_xSr_2GdCu_2O_8$ (Ru-1212) ($x = 0.0$, 0.05, 0.15) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ (Ru-1222) have been characterized by means of the resistivity, thermoelectric power, magnetic susceptibility and specific heat. Ta doping apparently suppresses superconductivity for the $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ system. The transport measurements show that the samples of Ru-1212 are typical underdoped cuprates; the sample of Ru-1222 is nearly at the optimal doping level. Specific heat measurements show an anomalous peak around T_c, indicating bulk superconductivity, whilst magnetic measurements seem to show the absence of a bulk Meissner state, which results because the susceptibility from the impurity Sr_2GdRuO_6 counters the diamagnetization from the bulk superconductivity.

1. Introduction

Recently, much attention has been paid to the ruthenate–cuprate layered compounds Ru-1212 and Ru-1222, in which long-range ferromagnetic (FM) order and superconductivity coexist [1–8]. In contrast to the case for ferromagnetic superconductors, in which the superconducting transition temperature (T_c) is higher than the magnetic transition temperature T_m, for Ru-1212 and Ru-1222, T_m is higher than T_c; these compounds are called superconducting ferromagnets [3]. The superconductivity in superconducting ferromagnets arises in a state with a well developed magnetic order, unlike in previous studies in which ferromagnetism arises in the superconducting state. Tetragonal Ru-1212 and Ru-1222 are both derived from the $LnBa_2Cu_3O_7$ (LnBCO) structure (Ln: lanthanide): the Ru ions replace Cu(1), and only one distinct Cu site (corresponding to Cu(2)) exists, with fivefold pyramidal coordination. For Ru-1212, the Cu–O layers are connected by perovskite $SrRuO_3$ layers through the apical oxygen atoms. For Ru-1222, the Ln layer in LnBCO is replaced by inserting a fluorite-type (Ln, Ce)O_2 layer, thus shifting alternate perovskite blocks by $(a + b)/2$. It is now established that all of these Ru-1212 and Ru-1222 samples are derived from substitution with a lanthanide [1], such as Gd, Sm, Ce, Eu.

The remaining unresolved question concerns the homogeneity of the superconducting (SC) phase. Evidence in favour of a bulk SC state has been obtained for Ru-1212 from differential heat capacity measurements [9]. However, Chu *et al* recently raised doubts as to whether Ru-1212 reveals bulk superconductivity [10]. They find that a bulk Meissner effect does not exist in Ru-1212. They argue that the SC signal might be due to an impurity phase which is

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not even detectable in x-ray or neutron diffraction experiments. Alternatively, they suggest that the absence of a Meissner effect could be attributed to the creation of a spontaneous vortex phase (SVP). Such a SVP can be expected to form in a FM superconductor if the spontaneous magnetization, $4\pi M$, exceeds the lower critical field H_{c1} (e.g. $4\pi M > H_{c1}(T = 0)$) [7,10,11]. Otherwise, if $H_{c1}(T = 0) > 4\pi M$, the Meissner state will be stable at low temperature. More recently, Bernhard *et al* [12] presented low-field dc magnetization measurements on polycrystalline Ru-1212 samples, which show evidence that a bulk Meissner state develops in the pure compounds at low temperature, with $T^{ms} \leqslant 30$ K varying from sample to sample. They showed that the SVP, which forms at intermediate temperature $T^{ms} < T < T_c$, is characterized by unique thermal hysteresis effects. They believed that the absence of a Meissner phase in Ru-1212 as reported by Chu *et al* [10] can be explained in terms of a moderate reduction of H_{c1} due to impurity scattering or grain size effects.

Since Ta-1212 is isostructural with Ru-1212 [13], we synthesized $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$, 0.05, 0.15), aiming to investigate the superconductivity of Ru-1212 when Ru is partially substituted for with Ta. At the same time, $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ was also synthesized. Suppression of superconductivity by Ta doping has also been observed in the $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ system. The samples of $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$, 0.05, 0.15) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ were characterized by magnetic susceptibility and specific heat measurements. Specific heat measurements show an anomalous peak around T_c, indicating bulk superconductivity, whilst magnetic measurements seem to show the absence of a bulk Meissner state. The difference could arise from the existence of the impurity phase Sr_2GdRuO_6.

2. Experiment

Like the synthesis of $RuSr_2GdCu_2O_8$ previously reported [1, 4, 5, 10], the synthesis of the $Ru_{1-x}Ta_xSr_2GdCu_2O_8$ ($x = 0.0, 0.05, 0.15$) compounds was carried out by solid-state reaction of stoichiometric powders, of RuO_2, Ta_2O_3, $SrCO_3$, Gd_2O_3 and CuO. The required amounts of these materials were ground, preheated at 960 °C in air for ten hours, then reground and reacted as pellets at 1010 °C in flowing nitrogen for 24 hours to obtain precursor material (Sr_2GdRuO_6 and Cu_2O) and minimize the formation of $SrRuO_3$ [1]. These resulting samples were pulverized, pressed into pellets and calcined at 1050 °C in air for 24 hours with an intermediate grinding. In each reaction, the samples were cooled to room temperature in a furnace. Subsequently, as-prepared samples of $Ru_{1-x}Ta_xSr_2GdCu_2O_8$ ($x = 0.00$, 0.05) were sintered in flowing oxygen at 1050 °C for 72 hours. $Ru_{0.85}Ta_{0.15}Sr_2GdCu_2O_8$ was sintered in flowing oxygen at 1050 °C for 24 hours, then annealed for 24 hours at 1050 °C under a high oxygen pressure of 50 bars. To synthesize $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$, stoichiometric powders of Ta_2O_3, $SrCO_3$, Gd_2O_3, CeO_2 and CuO were preheated in air and calcined in flowing nitrogen (this is similar to the synthesis of $Ru_{1-x}Ta_xSr_2GdCu_2O_8$), then the samples were reground, pressed into pellets and calcined in flowing oxygen. Finally, the samples were annealed under an oxygen pressure of 50 bars. Except that the preheating in air was at 960 °C for ten hours, all reactions were performed at 1050 °C for 24 hours. Powder x-ray diffraction (XRD) measurements were carried out in Rigaku D/max-rA x-ray diffractometer with graphite-monochromatized Cu Kα radiation ($\lambda = 1.5406$ Å). Resistivity measurements were performed by the standard four-probe method down to 4.2 K. The thermoelectric power (TEP) coefficient was measured by a dc method. The temperature gradient (ΔT) in the sample was measured using two pairs of rhodium–iron thermocouples. The sample was mounted on the top of two well separated copper blocks with silver paint. During the measurement, the temperature gradient ΔT of the two separated copper blocks was kept at 1 K. To eliminate the effects of the reference leads, the absolute thermoelectric power of copper was subtracted from

the measured thermoelectric voltage. The TEP result displayed in this paper is an average of five data measured at the same temperature. The specific heats were measured between 4.2 K and 300 K in an adiabatic, continuous-heating-type calorimeter using platinum thermometry. Its absolute accuracy is 0.8% from 15 K to 300 K and the precision is about 0.05%. The heating rate is 15 mK s^{-1} over the whole temperature range.

3. Results and discussion

Powder XRD measurements indicate that all of the samples of $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ (x = 0.0, 0.05, 0.15) are nearly single-phase (>95%) materials and have the tetragonal structure. Both the a-axis and the c-axis of the $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ system expand with increasing Ta content. For Ru-1212, the average valence of Ru ions is greater than 4; both Ru^{4+} and Ru^{5+} exist. The ionic radius of Ta^{5+} (0.68 Å) is larger than that of Ru^{4+} (0.64 Å) and that of Ru^{5+} (0.565 Å), so the expansion of the lattice can be due to Ru ions with small radius being replaced by larger Ta ions. The XRD pattern for the sample $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ can be indexed assuming a tetragonal unit cell with lattice parameters a = 3.844(1) Å and c = 28.615(7) Å. This suggests that the crystalline phase obtained is a layered cuprate with 1222-type structure. In XRD patterns for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ (x = 0.0, 0.05, 0.15) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$, an impurity phase is observed and identified as being the double perovskite Sr_2GdRuO_6 orthorhombic structure. Our attempts to completely get rid of the impurity phase were unsuccessful. It should be pointed out that the amount of impurity phase apparently increases with increasing Ta content.

Figure 1(a) shows the temperature dependence of the resistivity for the four samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ (x = 0.0, 0.05, 0.15) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$. The sample with formula $RuSr_2GdCu_2O_y$ shows a superconducting transition at 45 K with a very slight upturn in the vicinity of T_c and exhibits zero resistivity at 26 K; metallic behaviour with a dependence that is T-linear in character above 100 K is observed. This is consistent with that reported by McCrone *et al* [8]. According to reference [8], the very slight upturn in the vicinity of T_c is due to grain boundary effects. Since the oxygen stoichiometry remains fixed at about 8 for Ru-1212, the annealing mainly influences the granularity [3]. The temperature dependence of the normal-state resistivity is characteristic of a strongly underdoped superconducting cuprate compound. It should be pointed out that the ferromagnetic transition at T_c = 132 K causes a small yet noticeable drop in the resistivity for the as-grown sample with a semiconductor-like behaviour in the normal state, while no anomaly in the resistivity is observed for the post-annealed sample. The sample $Ru_{0.95}Ta_{0.05}Sr_2GdCu_2O_y$ displays a superconducting transition at 35 K, and exhibits zero resistivity at 10 K. The resistivity of the normal state shows metallic behaviour down to 80 K, below which a slight upturn in resistivity is also observed. The sample $Ru_{0.85}Ta_{0.15}Sr_2GdCu_2O_y$ shows metallic behaviour above 80 K, and a semiconductor-like behaviour at low temperature. It does not display a superconducting transition until 4.2 K is reached. These results indicate that Ta doping apparently destroys the superconductivity of the Ru-1212 system. Neither of the samples $Ru_{0.95}Ta_{0.05}Sr_2GdCu_2O_y$ and $Ru_{0.85}Ta_{0.15}Sr_2GdCu_2O_y$ follows a T-linear dependence for the resistivity at high temperature, although they show metallic behaviour. This is different from the case for the pure Ru-1212 sample. Figure 1(b) shows the same data plotted against T^2 for the samples $Ru_{0.95}Ta_{0.05}Sr_2GdCu_2O_y$ and $Ru_{0.85}Ta_{0.15}Sr_2GdCu_2O_y$. It is very clear that the temperature dependence of the resistivity shows a T^2-behaviour above 80 K for the two samples. This suggests that Ta doping changes the normal-state behaviour from T-linear to T^2 for the $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ system except for a suppression of the superconductivity. There are three possible ways to explain how the substitution of Ta for Ru suppresses the

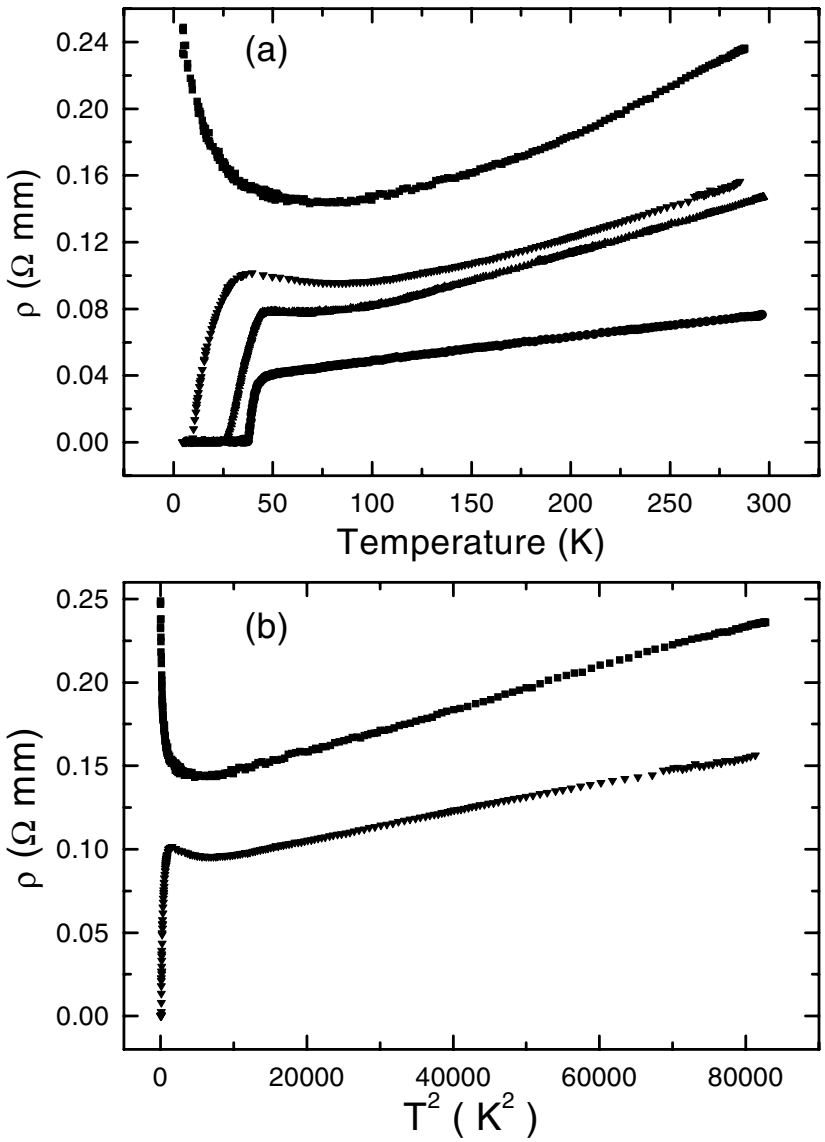

Figure 1. (a) The temperature dependence of the resistivity for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ with $x = 0.0$ (upwards-pointing triangles), 0.05 (downwards-pointing triangles), 0.15 (squares) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ (circles). (b) The same data but plotted against T^2 for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ with $x = 0.05$ (downwards-pointing triangles) and 0.15 (squares).

superconductivity: (1) the concentration of the carrier decreases, which is due to the doped Ta ions having valence higher than that of Ru ions; (2) the presence of Ta ions disorders the arrangement of Ru ions; (3) the expansion of the lattice, which is due to the radius of Ta ions being greater than that of Ru ions, weakens the coupling between the CuO_2 layer and RuO_2 plane. However, the temperature dependence of the resistivity for Ta-doped samples is very similar to that in the $La_{2-x}Sr_xCuO_4$ system within the small-x region [14]. This suggests that Ta doping seems to result in a decrease in the concentration of carriers. On the other hand,

upon Ta doping, the temperature dependence of the resistivity gradually crosses over from $\propto T$ to $\propto T^2$ at high temperature. This behaviour is usually considered as going to the 'overdoped' region. The temperature dependence of the resistivity for $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ is also shown in figure 1(a). The sample shows the characteristic of an optimally doped region, because a T-linear dependence persists until the superconducting transition occurs. The sample shows a superconducting onset at about 45 K and exhibits zero resistance at 38 K, typical for 1222 cuprates. The zero-resistance temperature for Ru-1222 is higher than that for Ru-1212.

Figure 2 shows the temperature dependence of the thermoelectric power (TEP), $S(T)$, for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$ and 0.05) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$. The magnitude and shape for the sample $RuSr_2GdCu_2O_y$ are typical for underdoped cuprates, being consistent with the TEP value of about 70 μV K^{-1} reported previously [5, 10]. It is found that long annealing leads to a rather small shift in the room temperature TEP, suggesting that there is little change in the concentration of carriers with annealing. This is consistent with the fixed oxygen stoichiometry at about 8 for Ru-1212 [3]. In our experiments, the TEP behaviour is consistent with the resistivity. $S(T)$ falls to zero at a temperature of about 26 K, corresponding to the zero-resistance temperature; while as will be seen in figure 2, the thermodynamic SC transition temperature is about 45 K, which is the same as the resistive onset temperature. This is quite different from that reported by Tallon *et al* [9]. They reported that $S(T)$ falls to zero at 43 to 45 K—significantly higher than the $T_c(R = 0)$ value determined by resistivity measurement, but close to the resistive onset at 46 K; the difference between the TEP and resistivity measurements is ascribed to the substantial granularity of the Ru-1212 samples. So the thermopower, in comparison to the resistivity, is much less sensitive to granularity

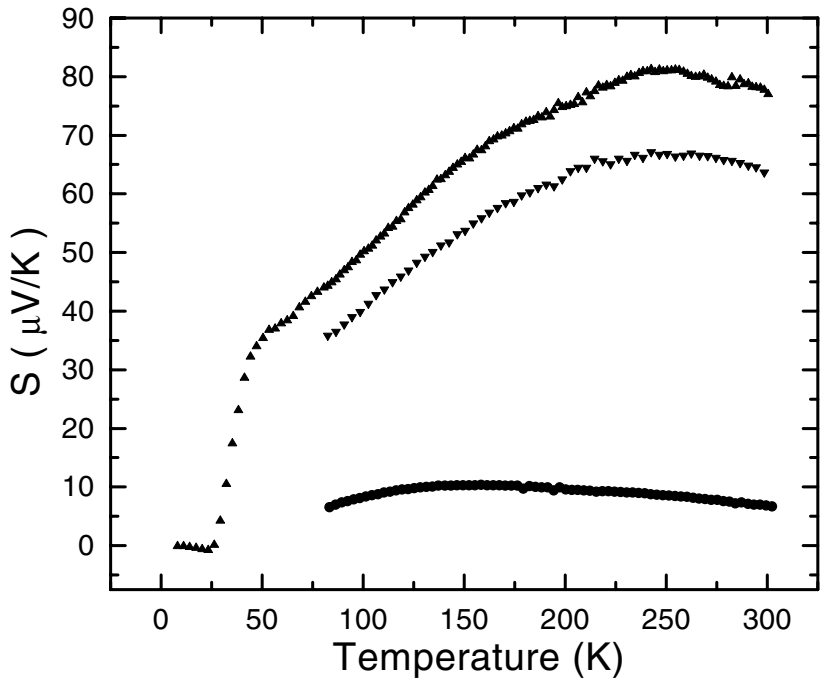

Figure 2. The temperature dependence of the thermoelectric power for the three samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$: upwards-pointing triangles; $x = 0.05$: downwards-pointing triangles) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ (circles).

and thus falls to zero close to the thermodynamic T_c. The sample $Ru_{0.95}Ta_{0.05}Sr_2GdCu_2O_y$ shows an $S(T)$ behaviour similar to that of the sample Ru-1212. This suggests that the sample is still in the underdoped region. For $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$, the TEP value increases monotonically with temperature decrease above 150 K. In addition, the TEP value at 300 K is about 6 μV K^{-1}. These results are typical for an optimally doped sample, which is consistent with the resistivity result.

Magnetic susceptibility measurements on the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$, 0.05, 0.15) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ were made with the field of 10 Oe between 2 and 300 K using a Quantum Design SQUID magnetometer. The temperature dependence of the susceptibility in the field-cooled (FC: open symbols) and zero-field-cooled (ZFC: solid symbols) modes for $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$, 0.05, 0.15) is shown in figure 3(a). All measurements displayed are on warming curves. For the three samples spontaneous magnetization develops at temperatures of about 135 K and below about 110 K it rises almost linearly with decreasing temperature for the FC susceptibility. A clear difference among the three samples is observed. For the sample without Ta, a sudden increase in moment and separation of the ZFC and FC branches are observed at about 135 K; these are considered to arise from the Ru spin ordering in the RuO_2 plane. Upon doping with Ta, the moment decreases monotonically with Ta content from 135 to 35 K, especially at the spin-ordering temperature. For the sample with $x = 0.15$, no apparent increase in moment at about 135 K is shown in the ZFC curve although the separation of the ZFC and FC branches is still observed. These results suggest that Ta substitution for Ru could damage the Ru spin ordering in the RuO_2 plane. In addition, another clear difference among the three samples appears below 35 K. A diamagnetic shift starting at 26 and 10 K for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$, 0.05) is clearly evident in the ZFC susceptibility but not in the FC susceptibility; this is similar to the previous report [10]. For each of the FC curves there exists a magnetic anomaly at about 30 K for the three samples—except for the spontaneous magnetization, where it occurs at about 135 K. The magnetic anomaly could arise from Sr_2GdRuO_6 which appears often in Ru-1212 and Ru-1222 samples as an impurity phase. It has been found that Sr_2GdRuO_6 is an antiferromagnet with a Néel temperature of about 30 K [10]. For non-superconducting $Ru_{0.85}Ta_{0.15}Sr_2GdCu_2O_y$ the moment increases sharply with decreasing temperature below 30 K. For the superconducting samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$, 0.05), there exists a crossover for the slope at the temperatures corresponding to the diamagnetic onset observed in the ZFC curves. This is different from the case for the non-superconducting sample in which no crossover in slope is observed. The crossover for the slope could arise from the cooperative effect of a rapid increase in moment caused by the impurity Sr_2GdRuO_6 and diamagnetization caused by the superconductivity, with the result that no bulk Meissner effect is observed in the FC curves. A similar behaviour is also observed for the sample $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$. Both ZFC and FC branches exhibit two magnetic anomalies at 85 K and at about 150 K—which is defined as the merging temperature of the ZFC and FC branches, and is similar to that of the previous report [2]. For the ZFC curve two diamagnetic transitions, at 40 K and at 20 K, are observed. This behaviour is similar to that reported by Bernhard *et al* for Ru-1212 [12]. The high onset temperature is that of the thermodynamic superconducting transition (T_c) and is marked by a weak diamagnetic shift. However, a sizable diamagnetic shift occurs at $T^{ms} = 20$ K. Bernhard *et al* [12] argued that the observed behaviour is indicative of a transition from a bulk Meissner phase at $T < T^{ms}$ to a spontaneous vortex phase at $T^{ms} < T < T_c$.

Figure 4 shows the specific heat C/T and $\Delta C/T$ as functions of temperature for the three samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0$, 0.05) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$. $\Delta C/T$ is obtained by subtracting $C_{\rm fit}/T$ from the C/T. It is very clear that there exists a specific heat jump at the temperatures of about 34 K, 29 K and 38 K for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$

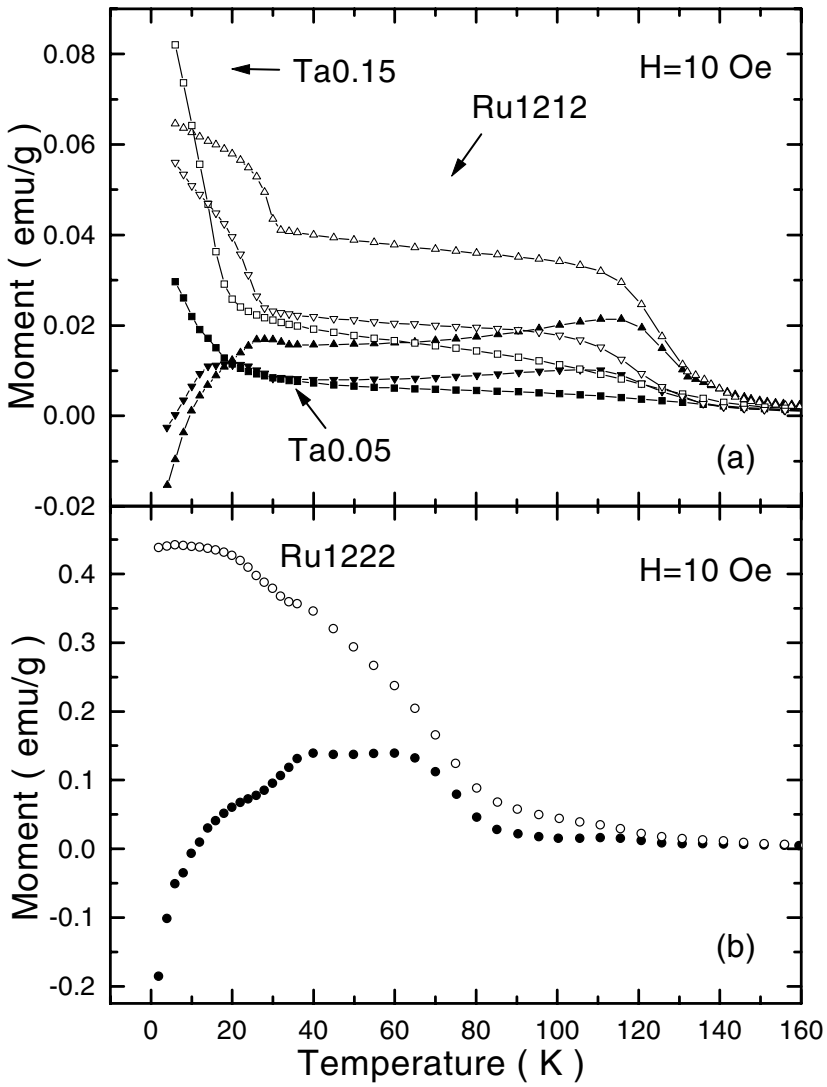

Figure 3. (a) The temperature dependence of the susceptibility (open symbols: field-cooled susceptibility; solid symbols: zero-field-cooled susceptibility) with the field of 10 G for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ with $x = 0.0$ (upwards-pointing triangles), 0.05 (downwards-pointing triangles), 0.15 (squares). (b) The temperature dependence of the susceptibility (open symbols: field-cooled susceptibility; solid symbols: zero-field-cooled susceptibility) with the field of 10 G for the sample $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$.

($x = 0.0$, 0.05) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$, respectively. The temperature corresponding to the specific heat anomaly is nearly the same as that of the superconducting transition onset. The magnitude of $\Delta C/T$ is about 0.04–0.07 mJ g^{-1} K^{-2} for the three samples, which is comparable to those for other underdoped cuprates. The existence of a sizable specific heat confirms the presence of bulk superconductivity. Chu *et al* [10] argued that the possible inclusion of a very small amount of the antiferromagnetic phase Sr_2GdRuO_6 in Ru-1212 and Ru-1222 samples can give rise to a C_p of magnitude similar to that of

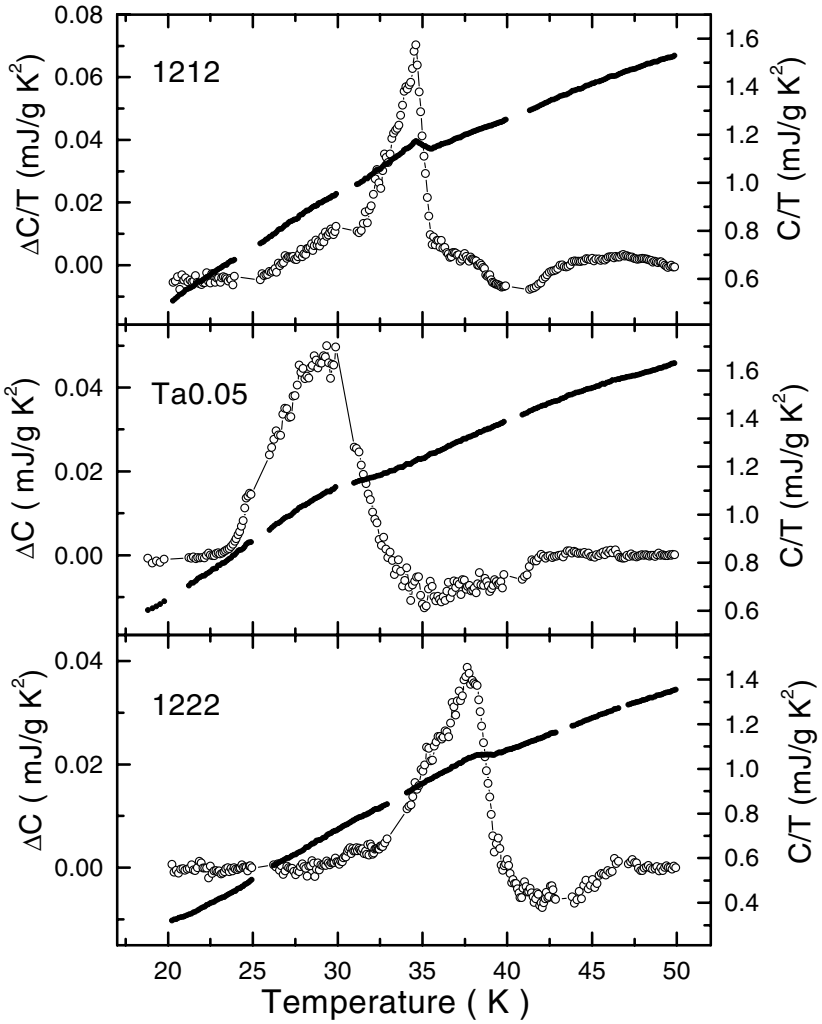

Figure 4. The specific heat C/T (solid symbols) and $\Delta C/T$ (open symbols) as functions of temperature for the samples $Ru_{1-x}Ta_xSr_2GdCu_2O_y$ ($x = 0.0, 0.05$) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$.

the underdoped cuprate superconductor, because of the large entropy associated with the transition. So we pay special attention to this. We also measured the specific heat for the non-superconducting sample $Ru_{0.85}Ta_{0.15}Sr_2GdCu_2O_y$. However, no specific heat anomaly is observed in the temperature range between 20 K and 50 K. As pointed out above, among all of the samples that we studied, the non-superconducting $Ru_{0.85}Ta_{0.15}Sr_2GdCu_2O_y$ had the highest content of the impurity phase Sr_2GdCuO_6. This is consistent with the susceptibility results: the susceptibility of the non-superconducting sample increases with decreasing temperature around 30 K much more rapidly than those of the superconducting samples. In addition, the temperature corresponding to the specific heat jump is different but not fixed for the three samples, and higher than that determined from the susceptibility anomaly arising from the impurity phase Sr_2GdRuO_6. These results suggest that the specific heat jump arises from the superconducting transition.

4. Conclusions

$Ru_{1-x}Ta_xSr_2GdCu_2O_8$ (Ru-1212) ($x = 0.0, 0.05, 0.15$) and $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_y$ (Ru-1222) samples have been studied by means of x-ray diffraction, resistivity, TEP, magnetic susceptibility and specific heat. It is found that Ta doping apparently suppresses the superconductivity for the Ru-1212 system. The superconducting temperature determined from TEP measurements is nearly the same as that determined from resistivity measurements. This is different from the behaviour arising from the granularity in Ru-1212 samples reported by Tallon *et al* [9]. For the superconducting samples, diamagnetization is observed in the ZFC susceptibility but not in the FC susceptibility. However, a slope crossover in the FC χ–T curve is observed at the temperature corresponding to the onset of the diamagnetic shift. The behaviour of the slope crossover could arise from the fact that the susceptibility arising from the impurity Sr_2GdRuO_6 counters the diamagnetization arising from the bulk superconductivity. The specific heat jump around T_c observed for our samples, comparable to those for the other underdoped cuprates and not deriving from the impurity phase Sr_2GdRuO_6, confirms the bulk superconductivity.

Acknowledgments

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