The discovery of oxypnictide superconductors has triggered the development of new superconducting materials based on FeAs and BiS\(_2\) as the superconducting layers.\(^{1-3}\) Since iron is a magnetic element, much attention has been paid to the magnetism and superconductivity of iron-based superconductors. Furthermore, the coexistence of the antiferromagnetic order of Eu\(^{3+}\) and superconductivity has been reported in the EuFe\(_2\)(As,P)\(_2\) system.\(^{4,5}\) In addition, simultaneous superconductivity and spontaneous magnetization were recently observed in Ce(O,F)BiS\(_2\) and EuA\(_2\)Fe\(_4\)As\(_4\) \((A = Rb\) and Cs).\(^{6,7}\) Therefore, the coexistence of magnetism and superconductivity originates from the Ru\(_{2}\)(RuRE-1212) and RE\(_2\) \((A = Rb\) and Cs)\(^{6,7}\) therefore, the coexistence of magnetism and superconductivity has attracted renewed attention.

Ruthenocuprate materials such as RESr\(_2\)RuCu\(_2\)O\(_{8−δ}\) (RuRE-1212) and RE\(_2\)Ce\(_x\)Sr\(_{2−x}\)RuCu\(_2\)O\(_{10−δ}\) (RuRE-1222), where RE denotes a rare-earth element, have also been shown to behave as superconducting ferromagnets. RuRE-1212 and RuRE-1222 were first synthesized and determined to be superconducting by Braun et al. in 1995.\(^{9}\) Subsequently, Felner et al. demonstrated the coexistence of magnetism and superconductivity in RuRE-1222.\(^{9}\) The crystal structure of RuRE-1212 is similar to that of YBa\(_2\)Cu\(_3\)O\(_{7−δ}\) (Y-123),\(^{8}\) in which the CuO\(_2\) chain, the Y ions sandwiched between the CuO\(_2\) planes, and the Ba ions of the blocking layer are replaced by RuO\(_2\) octahedra, rare-earth ions, and Sr ions, respectively. The magnetic ordering temperature, \(T_M\) (130–150 K), of RuRE-1212 is higher than its superconducting transition temperature, \(T_c\) (30–50 K). The \(T_M\) value of RuRE-1212 is much higher than those of the aforementioned EuFe\(_2\)(As,P)\(_2\) and Ce(O,F)BiS\(_2\). Thus, various investigations have been performed to understand the origin of the coexistence of magnetism and superconductivity in ruthenocuprates.\(^{10-16}\) These studies revealed that the observed ferromagnetism and superconductivity originate from the Ru and the CuO\(_2\) planes, respectively. Specifically, the magnetism and superconductivity of RuRE-1212 were understood from the perspective of the stacking structure of SrRuO\(_3\) (an itinerant ferromagnet with a \(T_M\) of approximately 160 K) and the CuO\(_2\) planes (responsible for the superconductivity of high-\(T_c\) cuprates). Furthermore, ruthenocuprates are considered to be potential candidates for \(π\) junctions, and it has been experimentally demonstrated that \(π\)-junction qubits exhibit coherence times comparable with traditional superconducting phase qubits.\(^{17}\) In particular, RuGd-1212 has the potential to facilitate the realization of robust qubits in the future. However, numerous questions concerning ruthenocuprates remain unanswered, such as their broad superconducting transition width and the absence of a bulk Meissner effect. Subtle differences in the sample preparation conditions can greatly affect the superconducting and magnetic properties of ruthenocuprates, which complicates the investigation of these materials.\(^{18-21}\) Furthermore, it was reported that the superconductivity of the RuEu-1222 system may be attributable to the intergrowth of the satellite EuSr\(_2\)Cu\(_3\)-(CO\(_3\))\(_2\)O\(_7+\) phase.\(^{22}\) It was also suggested that the superconductivity of RuGd-1212 may originate from its decomposition at high temperature.\(^{23}\) Therefore, it is still unclear whether the observed superconductivity is truly a bulk intrinsic property or not. The investigation of high-quality single crystals of RuRE-1212 compounds would be indispensable with regard to answering these questions; however, the difficulty of crystal growth and the variability of the growth conditions have hindered progress in the study of these systems.

Lin et al. reported on the growth of RuGd-1212 single crystals with dimensions up to approximately 200 × 200 × 50 \(\mu\)m\(^3\) using the self-flux method.\(^{24}\) Furthermore, bulk superconductivity and intrinsic Josephson junction properties were successfully demonstrated.\(^{15,25}\) It is surprising that the SrRuO\(_3\) layer serves as a blocking layer given that SrRuO\(_3\) is a metallic itinerant ferromagnet. It is plausible that RuGd-1212 exists in an underdoped state in analogy with the Y-123 system.\(^{26-28}\) This possibility appears to be consistent with the dispersion of \(T_c\) in the RuRE-1212 systems. However, the quality of the previously obtained RuGd-1212 single crystals\(^{15,24,25}\) was not high, as demonstrated by the fluctuation of the \(T_c\) value and the broad transition width. The observed \(T_c\) value exceeds 50 K, which suggests the partial substitution of Cu at the Ru sites.\(^{20,29}\) It should be noted that for GdSr\(_2\)-Ru\(_{1−x}\)Cu\(_{2+x}\)O\(_{8−δ}\) maximum \(T_c\) of 72 K was observed at \(x = 0.4\), whereas \(T_c\) of stoichiometric RuGd-1212 is 30–50 K. Such variations in \(T_c\) are ideally understood in relation to the crystal structure. However, a detailed structural analysis of a RuRE-1212 single crystal has not been reported to date.

**Reference**

1. Kazuhiro Yamaki, Takashi Mochiku, Shugo Funahashi, Yoshihiro Bamba, Michihide Kitamura, Yoshitaka Matsuhashi, and Akinobu Irie. Synthesis and structural characterization of a superconducting GdSr\(_2\)RuCu\(_2\)O\(_{8−δ}\) single crystal grown by partial melting. *Applied Physics Express* 11, 113101 (2018). https://doi.org/10.3585/apex.11.113101
Recently, we successfully synthesized cubic-shaped single crystals of $\text{Gd}_{1+\delta}\text{Sr}_{2-x}\text{Ru}_{1-y}\text{Cu}_{2+y}\text{O}_{8+\delta}$ with typical lengths of 100–150$\mu$m using the partial melting technique.\textsuperscript{30,31} However, these single crystals did not exhibit superconductivity.\textsuperscript{31} Single-crystal X-ray diffraction (XRD) measurements and structural refinement indicated that the absence of superconductivity may have originated from the increase in the lattice parameter $a$.\textsuperscript{31} Since the superconductivity of the RuRE-1212 materials is highly sensitive to the sample preparation conditions, we considered that it would be valuable to reexamine the synthetic conditions used to prepare RuGd-1212 by the partial melting technique, and to characterize the acquired single crystals in more detail.

Polycrystalline precursors of $\text{Gd}_2\text{Sr}_{2-y}\text{Cu}_{2+y}\text{O}_{8+\delta}$ with $n = 0, 1, 2$, and 3 were prepared by conventional solid-state reactions using $\text{RuO}_2$ (Ru 76.45 wt %), $\text{SrCO}_3$, $\text{Gd}_2\text{O}_3$, and CuO (3N) powders. Subsequently, RuGd-1212 single crystals were synthesized using the partial melting technique. In this synthesis process, $n$ represents the ratio of additional Sr: Gd: Cu flux. In other words, $n = 0$ corresponds to a Sr : Gd : Cu flux ratio of 2 : 1 : 2, whereas $n = 3$ corresponds to a Sr : Gd : Cu flux ratio of 2 : 1 : 5. The partially melted flux promotes the growth of single crystals. In this study, the crystal structures and conductivities of the RuGd-1212 single crystals were evaluated with a focus on superconductivity. The detailed synthesis procedure is described elsewhere.\textsuperscript{30,31}

Figure 1(a) shows the XRD patterns of the GdSr$_2$Ru$_{0.7}$Cu$_{2+0.3}$O$_{6+0.3n}$ samples with $n = 0, 1, 2$, and 3 prepared by the partial melting technique, along with that of polycrystalline RuGd-1212 as a reference. A two-step sintering procedure was used for all samples with $n = 0$ samples, decomposition of the RuGd-1212 phase was clearly observed, where “$+$” and “$-$” denote the peaks corresponding to the SrRuO$_3$ and SrGdRuO$_6$ impurity phases, respectively. (b) SEM images of the $n = 1$ (left) and $n = 3$ (right) samples, showing the RuGd-1212 single crystals.

**Table I.** Maximum size and composition of the single crystals grown from various nominal compositions of GdSr$_2$Ru$_{0.7}$Cu$_{2+0.3}$O$_{6+0.3n}$ with $n = 0, 1, 2, 3$.

| $n$ | Maximum size ($\mu$m$^2$) | Ru | Sr | Gd | Cu |
|-----|-------------------------|----|----|----|----|
| 0   | 100 × 100               | 0.56 | 1.65 | 1.33 | 2.46 |
| 1   | 150 × 150               | 1.06 | 2.14 | 0.99 | 1.80 |
| 2   | 120 × 100               | 1.01 | 2.16 | 0.96 | 1.87 |
| 3   | 90 × 90                 | 0.92 | 2.09 | 1.08 | 1.90 |

Note: The constraint used for the elemental ratio was Ru + Sr + Gd + Cu = 6.

Sintering of this sample at $T_2$ for 150 h led to $T_{c-\text{onset}}$ of 23 K. However, zero resistance was not observed down to 4.2 K. This sample contained approximately two to five single crystals, as observed using an optical microscope. XRD measurements revealed that the RuGd-1212 phase was retained even after prolonged sintering at $T_2$ for 150 h. Therefore, prolonged sintering is considered to be useful for achieving superconductivity in RuGd-1212. To investigate the origin of superconductivity in RuGd-1212, the crystal structure was refined based on single-crystal XRD measurement data for a single crystal selected from a region of the superconducting sample. The intensity data were collected using a charge-coupled device (CCD)-type single-crystal diffractometer at 295 K. In the analysis, the initial model was determined using the SHELXT software\textsuperscript{34} and the final values of $R$ and $R_w$ were 2.81 and 6.58%, respectively. The Sr and Ru sites were

**Fig. 1.** (a) XRD patterns for the RuGd-1212 (GdSr$_2$Ru$_{0.7}$Cu$_{2+0.3}$O$_{6+0.3n}$ with $n = 0, 1, 2, 3$) samples prepared by the partial melting technique, along with that for polycrystalline RuGd-1212 as a reference. For the $n = 0, 2$ samples, decomposition of the RuGd-1212 phase was clearly observed, where “$+$” and “$-$” denote the peaks corresponding to the SrRuO$_3$ and SrGdRuO$_6$ impurity phases, respectively. (b) SEM images of the $n = 1$ (left) and $n = 3$ (right) samples, showing the RuGd-1212 single crystals.
Note 3: No Ru substitution at Cu sites forming the CuO$_2$ plane was observed on re-
structural comparison of the superconducting single crystal of 
nonsuperconducting sample in our previous report,\textsuperscript{31} and also 
prolonged sintering at Sr sites is consistent with the results of our single-crystal 
Elongation of the lattice parameter 
partially substituted by Gd and Cu, respectively, and a slight 
substitution at Cu sites forming the CuO$_2$ plane was not 
reported previously for polycrystalline RuGd-1212 samples doped with La at the Sr sites.\textsuperscript{35}

Furthermore, the superconducting 
Ru/Co$_2$Cu$_2$O$_{6.4}$ sample was determined to 
have a lattice parameter $a$ that was larger than that of the 
nonsuperconducting sample in our previous report,\textsuperscript{31} and also 
similar to that reported previously for nonsuperconducting 
RuGd-1212 samples doped with La at the Sr sites.\textsuperscript{35}

Elongation of the lattice parameter $a$ by doping with La at 
Sr sites is consistent with the results of our single-crystal 
analysis in which Gd substitutes at Sr sites. Since some of 
the samples with an elongated lattice parameter $a$ exhibits 
superconductivity, it is necessary to reconsider the origin of 
superconductivity in RuGd-1212. Table III presents a struc-
tural comparison of the superconducting single crystal of 
RuGd-1212 synthesized in this work, two previously reported 
superconducting polycrystals,\textsuperscript{12,29} and our previously re-
ported non-superconducting single crystal.\textsuperscript{31} Polycrystal I 
has a stoichiometric composition,\textsuperscript{12} whereas polycrystal II 
was synthesized under high pressure and contains a partial 
substitution of Gd at the Ru sites.\textsuperscript{29} The superconducting and 
nonsuperconducting single crystals have a larger lattice para-
meter $a$ than polycrystals I and II owing to the substitution of 
Gd at the Sr sites. It is considered that the partial melting 
technique leads to a different site occupancy compared with 
the typical solid-state reaction because in the former method, 
the sample is sintered at the decomposition temperature of the 
RuRE-1212 phase for a short time. Furthermore, since no 
substitution of Ru on the CuO$_2$ plane was detected for the 
superconducting samples, the occupancy of Ru on the CuO$_2$ 
plane appears to be correlated with the emergence of super-
conductivity in RuGd-1212 single crystals. There is also the 
possibility that the substitution of Gd at the Sr sites inhibits 
superconductivity, because the $T_c$ value of the super-
conducting single crystal synthesized in this work is lower than 
that reported for polycrystal II. This is because the Sr in 
the blocking layer is adjacent to the CuO$_2$ plane and hence, 
the large magnetic moment of Gd at the Sr sites may prevent 
superconductivity. In general, high-$T_c$ cuprates are relatively 
robust against impurities such as Co, Ni, and Zn,\textsuperscript{36–39} but the 
results of this study indicate that the presence of Ru in the 
CuO$_2$ layer or Gd at the Sr sites may destroy the super-
conductivity of the CuO$_2$ plane. This loss of superconduct-

\begin{table}[h]
\centering
\caption{Refined structural parameters for the single crystal of RuGd-1212 (space group $P4/mmm$).}
\begin{tabular}{lccccc}
\hline
Site & Wyckoff position & Occupancy, g & $x/a$ & $y/b$ & $z/c$ & $R_{el}$/Å$^2$ \\
\hline
Sr/Gd$^+$ & 2h & 0.920(6)/0.080(6) & 1/2 & 1/2 & 0.19162(6) & 0.01297(15) \\
Gd & 1d & 1.00 (fixed) & 1/2 & 1/2 & 1/2 & 0.00710(9) \\
Ru/Cu$^-$ & 2a & 0.452(18)/0.548(18) & 0 & 0 & 0 & 0.0064(2) \\
Cu/Ru$^-$ & 2g & 1.0 (fixed) & 0 & 0 & 0.35416(7) & 0.0075(2) \\
O1 & 2f & 0.96(3) & 0 & 1/2 & 0 & 0.149(12) \\
O2 & 2g & 1.00 (fixed) & 0 & 0 & 0.1655(5) & 0.0222(12) \\
O3 & 4i & 1.00 (fixed) & 0 & 1/2 & 0.3708(3) & 0.0115(5) \\
\hline
\end{tabular}
\end{table}

Note 1: Final $R$ values: $R = 2.81\%$, $R_p = 6.58\%$, $\alpha = 3.8420(2)$ Å, and $c = 11.4918(6)$ Å (295 K).
Note 2: The constraints used for the cation occupancies were $g$(Sr) + $g$(Gd$^+$) = 1 and $g$(Ru) + $g$(Cu$^-$) = 1.
Note 3: No Ru substitution at Cu sites forming the CuO$_2$ plane was observed on refinement [Cu : Ru = 1.000(13) : 0.000(13)].

\begin{table}[h]
\centering
\caption{Structural comparison of polycrystals and single crystals of RuGd-1212.}
\begin{tabular}{lccc}
\hline
 & Single crystal & Polycrystal I & Polycrystal II \\
 & (superconducting) & (this work) & (non-superconducting) \\
\hline
 & & & \\
$T_c$ & 23 K (onset) & 37 K ($R = 0$) & 72 K (onset) & — \\
\hline
 & Lattice parameter (Å) & & & \\
$\alpha$ & 3.8420(2) & 3.83841(2) & 3.8262 & 3.84010(10) \\
$c$ & 11.4918(6) & 11.5731(1) & 11.518 & 11.5026(4) \\
$c/\alpha$ & 2.991 & 3.015 & 3.010 & 2.995 \\
\hline
 & Cation occupancy, g & & & \\
Sr/Gd$^+$ & 0.920/0.080(6) & 1.0 (fixed) & 1.0 (fixed) & 0.911/0.089(6) \\
Ru/Cu$^-$ & 0.453/0.547(18) & 1.0 (fixed) & 0.7/0.3 (fixed) & 0.534/0.466 (19) \\
Cu/Ru$^-$ & 1.000/0.000(13) & 1.0 (fixed) & 1.0 (fixed) & 0.989/0.011(15) \\
\hline
 & Distance (Å) & & & \\
Sr/Gd$^+$–O1 & 2.9222(5) & 2.661(4) & — & 2.9199(5) \\
Ru/Cu$^-$–O1 & 1.92100(10) & 1.969(2) & — & 1.92005(5) \\
Cu/Ru$^-$–Cu/Ru$^-$ & 3.3519(12) & 3.3624(23) & — & 3.3588(14) \\
Cu–O2 & 2.168(6) & 2.184(6) & — & 2.173(7) \\
\hline
\end{tabular}
\end{table}
ity due to Ru and Gd substitution is reasonable given the broad transition width of rutheno-cuprate magnetic superconductors; that is, the heterogeneity with respect to site substitution may exert a strong influence on $T_c$. Therefore, since the Ru and Gd substitution in the single crystal is heterogeneous, the transition width becomes broad. Furthermore, the Cu–Cu distance between the CuO$_2$ planes is shorter for the superconducting single crystal than that of the non-superconducting single crystal. This suggests that the valence of Cu in the CuO$_2$ plane decreased. A bond valence sum analysis indicates a high hole concentration of ~0.4 in the CuO$_2$ plane.$^{12,40}$ Since the Ru-NMR study of pure RuGd-1212 revealed that 40 and 60% of Ru have a valence of 4 and 5, respectively, the valence of Cu in the CuO$_2$ plane results in 2.20.$^{41}$ However, the electrical properties of pure RuGd-1212 are similar to those of heavily underdoped high-$T_c$ materials.$^{35}$ In our results, the decreased valence of Cu was observed for the superconducting RuGd-1212 single crystal.

Since the results presented above indicate that prolonged sintering leads to superconductivity, the $n = 1$ sample was sintered at $T_2$ for 300 h. Figure 2(a) shows the $R$–$T$ characteristic for this RuGd-1212 single-crystal sample, as measured using the four-probe method. The inset shows an optical microscopy image of the sample, with a silver paste attached to the edge of the crystal surface along the $ab$ plane. A superconducting transition with $T_{c\text{-onset}}$ of 23.0 K and $T_{c\text{-zero}}$ of 6.2 K was successfully observed. This constitutes the first observation of zero resistance for a single crystal of RuGd-1212 prepared using the partial melting technique. The superconductivity of this sample may originate from the prevention of Gd substitution at the Sr sites by the prolonged sintering, when compared with the structural analysis of the superconducting single crystal obtained after sintering for 150 h. However, the observed $T_c$ value was lower than those of polycrystalline samples in which the Ru site is partially substituted by Gd. This seems to be strongly related to the substitution of Gd to the Sr sites.$^{35}$ The $R$–$T$ characteristic of the RuGd-1212 single crystal revealed metallic behavior above 85 K and semiconducting behavior below 85 K. This temperature dependence is consistent with the results obtained for our previous non-superconducting single crystal.$^{31}$ However, there is also the possibility that the electrical component of the $c$-axis contributed to the resistance, since the absolute value of resistance was relatively high compared with our previous samples. Figure 2(b) shows the $I$–$V$ characteristic at 4.2 K. The $I_c$ value was estimated to be approximately 3 mA. The temperature dependence of the critical current was found to be linear (data not shown), which may reflect an underdoped state and/or electrical inhomogeneity in this single crystal. The inset in Fig. 2(b) shows the magnetic field dependence of the zero-field-cooled magnetization ($M$–$H$) for the same RuGd-1212 single crystal. Since no diamagnetism was observed, this single crystal did not exhibit bulk superconductivity. The results appear to be consistent with those reported for a superconducting RuGd-1212 single crystal prepared by the self-flux method in a platinum crucible.$^{15}$ Since the $T_c$ value of our sample is low, further investigations should be performed to elucidate the correlation between the magnetic order, superconductivity, and crystal structure in more detail.

In conclusion, we have successfully grown single crystals of RuGd-1212 using the partial melting technique. The obtained single crystals possessed a cubic shape with a typical length exceeding 100 µm. Prolonged sintering was found to be effective for realizing superconductivity. The structural analysis results for the single crystal revealed that the Ru and Sr sites were partially substituted by Cu and Gd ions, respectively. The loss of superconductivity in non-superconducting samples may be attributable to Ru and Gd substitution of the CuO$_2$ planes and Sr sites, respectively. Furthermore, the distance between the CuO$_2$ planes in the superconducting sample was shorter than that in a non-superconducting single crystal, which suggests that the valence of Cu in the CuO$_2$ plane decreases. Finally, a superconducting transition with $T_{c\text{-onset}}$ of 23.0 K and $T_{c\text{-zero}}$ of 6.2 K was observed for the first time for a RuGd-1212 single crystal prepared by partial melting.

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