High-temperature thermoelectric properties of the double-perovskite ruthenium oxide 
\((\text{Sr}_1-x\text{La}_x)_2\text{ErRuO}_6\)

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We have prepared polycrystalline samples of \((\text{Sr}_1-x\text{La}_x)_2\text{ErRuO}_6\) and \((\text{Sr}_1-x\text{La}_x)_2\text{YRuO}_6\), and have measured the resistivity, Seebeck coefficient, thermal conductivity, susceptibility and x-ray absorption in order to evaluate the electronic states and thermoelectric properties of the doped double-perovskite ruthenates. We have observed a large Seebeck coefficient of \(-160\,\mu\text{V/K}\) and a low thermal conductivity of \(7\,\text{mW/cmK}\) for \(x=0.1\) at 800 K in air. These two values are suitable for efficient oxide thermoelectrics, although the resistivity is still as high as \(1\,\Omega\text{cm}\). From the susceptibility and x-ray absorption measurements, we find that the doped electrons exist as \(\text{Ru}^{4+}\) in the low spin state. On the basis of the measured results, the electronic states and the conduction mechanism are discussed.

I. INTRODUCTION
Thermoelectricity is a technology that converts heat into electric power or vice versa through the thermoelectric phenomena in solids. Since this technology is a direct energy conversion in solids, it has attracted a renewed interest as a fundamental technology for environmentally-friendly energy conversion. In particular, thermoelectric power generation has been now considered as a possible renewable energy resource.

Oxide thermoelectrics has been extensively investigated as a promising thermoelectric power generator, for oxides are stable at high temperatures in air. Oxides were considered to be poor thermoelectric materials, but after the discovery of a large thermoelectric power factor in \(\text{Na}_x\text{CoO}_2\), some cobalt oxides are recognized as good thermoelectric oxides of p-type. In contrast, not yet discovered is an n-type counterpart to the cobalt oxides. Some of the transparent conductors such as \(\text{ZnO}\) and \(\text{In}_2\text{O}_3\) show indeed good thermoelectric performance above 1000 K but the lattice thermal conductivity is much higher than the conventional thermoelectric materials. The doped titanates and niobates are fairly good n-type thermoelectric materials at room temperature, but they are easily oxidized at high temperature to lose conductivity in air.

Recently a large Seebeck coefficient and a low thermal conductivity have been reported in polycrystalline samples of the double perovskite ruthenate \(\text{Sr}_2\text{LuRuO}_6\) \((L;\text{ rare-earth})\). This particular ruthenate was first synthesized by Donalne and McCann whose crystal structure and physical properties were investigated by Battle and Wacklyn. It crystallizes in the \(B\)-site ordered perovskite structure of \(A_2BB'O_6\), where the two different cations of \(L\) and \(B\) occupy the \(B\) and \(B'\) sites to form a \(\text{NaCl}\) type ordered structure. As a unique feature, the Ru ion is pentavalent \((\text{Ru}^{5+})\) with the electronic configuration of \((4d)^3\), which acts as a local moment of \(S=3/2\) to show an antiferromagnetic order below 26 K in \(\text{Sr}_2\text{YRuO}_6\). When magnetic rare-earth ions occupy the \(B\) site, the transition temperature and the magnetic structure change depending on the species of the rare-earth ions. In addition, a possible high-temperature superconductivity has been observed in \(\text{Sr}_2\text{LuRu}_{1-x}\text{Cu}_x\text{O}_6\).

In this paper, we show the thermoelectric properties in polycrystalline samples of \((\text{Sr}_1-x\text{La}_x)_2\text{ErRuO}_6\), in which partial substitution of \(\text{La}\) for \(\text{Sr}\) supplies electrons to let \(\text{Sr}\) is in the low spin state, which implies that the doped electron occupies the upper Hubbard \(t_{2g}\) manifolds. On the basis of the measured data, the electronic states and the conduction mechanism are discussed.

II. EXPERIMENTAL
Polycrystalline samples of \((\text{Sr}_1-x\text{La}_x)_2\text{ErRuO}_6\) \((x=0, 0.05, 0.1, 0.2, \text{ and } 0.3)\) and \((\text{Sr}_1-x\text{La}_x)_2\text{YRuO}_6\) \((x=0, 0.1, \text{ and } 0.2)\) were prepared by solid-state reaction. Stoichiometric amounts of \(\text{SrCO}_3\), \(\text{La}_2\text{O}_3\), \(\text{Er}_2\text{O}_3\), \(\text{Y}_2\text{O}_3\),
and RuO$_2$ were mixed, and the mixture was calcined at 900°C for 12 h in air. The calcined powder was ground, pressed into a pellet, and sintered at 1250°C for 60 h in air. X-ray diffraction was measured with a Rigaku RAD-IIC (Cu Kα radiation), and no impurity phases were detected in the prepared samples.

The X-ray absorption spectra were measured at BL-11B KEK-PF, Japan. All the Ru L edge spectra were measured at room temperature in the fluorescence yield mode using a photodiode detector. The base pressure in the chamber was 10$^{-7}$ Torr. The electrical resistivity $\rho$ was measured using a four-probe method with a constant current of 1 mA from room temperature to 800 K in air with a home-made measurement probe inserted in a cylinder furnace. The Seebeck coefficient $S$ was measured with a quasi-steady-state method from room temperature to 800 K in air with a home-made measurement probe in a cylinder furnace; the edges of a bar-shaped sample was pasted to two ceramic plates working as heat batheres, one of which was heated by a nichrome heater. The temperature difference was monitored with a differential thermocouple made of Pt-PtRh. The thermoelectric voltage from the voltage leads was carefully subtracted. For some of the samples, the Seebeck coefficient was measured with a steady-state method using a copper-constantan differential thermocouple from room temperature down to 100 K in a liquid Helium cryostat. The thermal conductivity was evaluated from the thermal diffusivity measured from room temperature to 800 K in air with a laser flash method (ULVAC-Riko TC2000) and the heat capacity measured with differential scanning calorimetry (Netzsch DSC404F3) in Ar flow. The magnetization in field cooling (FC) and zero field cooling (ZFC) processes was measured using a superconducting quantum interference device magnetometer (Quantum Design MPMS) from 5 to 300 K in an applied field of 1 T.

### III. Results and Discussion

First of all, let us examine the valence state of the Ru ion in the title compound. Figure 1 shows the Ru L$_3$ edge spectra of (Sr$_{1-x}$La$_x$)$_2$ErRuO$_6$ and (Sr$_{1-x}$La$_x$)$_2$YRuO$_6$. For $x=0$, two peaks are observed around 2840 and 2843 eV, which evidences the existence of the pentavalent Ru ion as was already reported. The peak positions and intensities, and accordingly the valence state of the Ru ion, are essentially identical between the Er- and Y-based compounds. For Ru$^{3+}$ oxides such as SrRuO$_3$ and RuO$_2$, the L edge spectra show broad peaks at 2838 and 2841 eV$^{23}$ and by using the L edge spectra we can roughly evaluate the valence state from the peak energies. For the La substituted samples, the peaks are shifted to lower energy by around 0.3 eV, indicating that the valence state of the Ru ion shifts to a lower valence, which is consistent with a naive picture that the La substitution for Sr supplies electrons to the system to create a tetravalent Ru ion per La. Comparing the leading edge on the lower-energy side, we find that the Ru ions in the $x=0$ samples are in a similar valence state between Er and Y. These data thus warrant that the species of the rare-earth ion in the B site do not affect the valence state of the Ru ions.

Figure 2(a) shows the electrical resistivity of (Sr$_{1-x}$La$_x$)$_2$ErRuO$_6$. All the resistivities decrease with increasing temperature, indicating that the samples are nonmetallic. The magnitude systematically decreases with increasing $x$, indicating that the carrier concentration increases with increasing La content. The resistivity decreases roughly by two orders of magnitude from $x=0$ to 0.2, but seems saturated near 0.3, suggesting the solubility limit of La substitution.

Figure 2(b) shows the Seebeck coefficient of (Sr$_{1-x}$La$_x$)$_2$ErRuO$_6$. The sign for all the samples is negative, and the magnitude systematically decreases with increasing $x$ except for $x=0$. These results show that the substituted La ion acts as a donor to supply electrons to the system. The Seebeck coefficient for $x=0$ is close to zero at 300 K, and the magnitude increases with increasing temperature, possibly because small amounts of electrons and holes inevitably doped through unwanted nonstoichiometry and/or impurities show complicated temperature dependence. The Seebeck coefficient for $x=0$ is different from that previously reported by Aguirre et al.$^{1}$ The magnitude of the Seebeck coefficient of their sample was much larger, and decreased with increasing temperature, possibly owing to a smaller amount of carriers introduced in their sample. The Seebeck coefficients for $x > 0$, on the other hand, are essentially independent of temperature above room temperature.

The Seebeck coefficient below room temperature decreases with decreasing temperature, suggesting a $T$-linear behavior, although the low-temperature measurements were seriously affected by the high resistance of the samples. Since the $T$-linear Seebeck coefficient is a hallmark of metals having a finite Fermi energy in the
valence band, the data clearly indicate that the doped samples are essentially metallic in the sense that there is a Fermi surface. Then the nonmetallic resistivity is ascribed to the scattering time, which is reasonable because the RuO$_6$ octahedra are well separated by the ErO$_6$ octahedra to make transfer integrals small.\textsuperscript{22}

Now we will evaluate the carrier concentration and the effective mass by following the method applied to Nd$_{1.9}$Ce$_{0.1}$PdO$_4$ by Shibasaki and Terasaki.\textsuperscript{22} The $T$−linear Seebeck coefficient can be associated with the diffusive term of the Seebeck coefficient for a single parabolic band given by

$$S = -\frac{\pi^2 k_B^2 T}{2 eE_F},$$

(1)

where $E_F$ is the Fermi energy. From the $T$−linear slope of the measured data, the Fermi energy is obtained as listed in Table I.

On the other hand, the temperature-independent Seebeck coefficient can be analyzed with the Heikes formula, an asymptotic expression of the Seebeck coefficient in the high temperature limit, where the thermal energy of $k_B T$ is much larger than the band width or the transfer energy.\textsuperscript{24} We examined various forms for the Heikes formula, and find that the observed Seebeck coefficient is well explained by the expression given by

$$S = -\frac{k_B}{e} \ln \frac{2 - p}{p},$$

(2)

where $p$ is the carrier number per Ru. From the constant Seebeck coefficient at high temperature, the carrier concentration $n$ is evaluated as listed in Table I. Using $n$ and $E_F$, we further obtain the effective mass $m^*$ through the following expression for a single parabolic band given by

$$E_F = \frac{\hbar^2}{2m^*} (3\pi^2 n)^{\frac{2}{3}},$$

(3)

as listed in Table I. A crossover temperature around which the Seebeck coefficient changes from $T$−linear to temperature-independent can be read off from Fig. 2(b) to be 200-300 K, which should correspond to the energy scale for the transfer energy. Mazin and Singh calculated the band structure of Sr$_2$YRuO$_6$, and evaluated the transfer energy to be 0.14 eV, which is in fact several times larger than the thermal energy of 300 K. The correlation effects may further reduce the transfer energy, because the valence bands consist of the lower Hubbard $t_{2g}$ manifolds, which include the spin-dependent hopping.

The carrier concentration in Table I is roughly proportional to the La concentration $x$, and the magnitude of 10$^{21}$ cm$^{-3}$ is the same order of the carrier concentration estimated by the assumption that one substituted La ion supplies one electron. This clearly indicates that the substituted La acts as a donor in a simplest approximation. The effective mass is evaluated to be nearly $x$−independent value of 3$m_0$, which suggests that the electron doping in this system is rigid-band-like. The mobility is formally calculated as $\mu = 1/ne\rho \sim 10^{-4}$ cm$^2$/Vs at 800 K, which is too small for the Boltzmann transport where the mean free path must be longer than the lattice spacing. This is understandable because the doped carriers are well localized in a RuO$_6$ octahedron, and the electrical conduction occurs via hopping from one RuO$_6$ octahedron to another.\textsuperscript{22} The hopping process accompanies a finite activation energy which appears in the temperature dependence of the resistivity shown in Fig. 2 (a). Nevertheless we find that the mobility is almost independent of the La content, indicating that La substitution changes only the carrier concentration like a rigid-band picture.

Figure 3 shows the temperature dependence of the thermal conductivity of (Sr$_{1-x}$La$_x$)$_2$ErRuO$_6$. The ther-
minal conductivity slightly decreases with increasing temperature, with a small magnitude of 3-9 mW/cmK. The value for x = 0 is slightly higher than the previously reported value by Aguirre et al., but their value may come from porosity of the sample (the sample density of 78-81%). In contrast, the density of our samples is larger than 90%, and we think that this low thermal conductivity observed here is intrinsic. The value for x = 0 is indeed anomalously low, and is close to the minimum thermal conductivity proposed by Cahill et al. Recently Wan et al. have reported that an oxygen deficient aluminium oxide Ba$_2$DyAlO$_6$ exhibits a low value of close to 10 mW/cmK at 1000°C. They associated this low value with oxygen deficiency, but the present data imply that a different mechanism does exist, for the title compound includes no significant oxygen vacancies. The doped sample of x = 0.1 is more disordered, and thus the thermal conductivity is expected to be reduced from x = 0, which is seriously incompatible with the observation. We note that the electron contribution of the thermal conductivity is evaluated to be 0.02 mW/cmK for x = 0.1 at 800 K using the Wiedemann-Franz law, which cannot be a reason of the increase in thermal conductivity from x = 0 to 0.1. We suggest that the double perovskite structure of A$_2$BB$'$O$_6$ may be a key ingredient; Aguirre et al found characteristic micro-domain structures in the transmission electron microscope. Ohtaki et al. reported that the double perovskite oxide Sr$_2$FeMoO$_6$ also shows a low thermal conductivity. In spite of such low thermal conductivity, the dimensionless figure of merit ($ZT = S^2σT/κ$) of the present ruthenate remains low ($ZT ~ 10^{-3}$ at 800 K) because of the high resistivity.

Let us discuss the electronic properties of the doped electron through the spin state of the Ru$^{4+}$ ions by measuring the susceptibility. In order to avoid a large magnetization arising from Er ions, we used (Sr$_{1-x}$La$_x$)$_2$YRuO$_6$. Figure 3 shows the temperature dependence of magnetic susceptibility for (Sr$_{1-x}$La$_x$)$_2$ErRuO$_6$ (x = 0 and 0.1).

The magnetic phase transition is visible at $T_N = 34$ K for x = 0, below which the magnetic susceptibility shows temperature hysteresis, which is consistent with previous measurements. We analyze the susceptibility from 50 to 300 K using the Curie-Weiss law given by

$$\chi = \frac{\mu_{\text{eff}}^2 \mu_B^2}{3k_B(T + \theta)},$$

and evaluate the effective magnetic moment $\mu_{\text{eff}}$ and the Weiss temperature $\theta$. The effective magnetic moment is 3.87 $\mu_B$/Ru for x = 0, which is close to a theoretical value of $S = 3/2$ and also consistent with the previous work. For x = 0.1 and 0.2, the spin number obtained experimentally ($S_{\text{exp}}$) is given by an average of Ru$^{5+}$ and Ru$^{4+}$ in a simplest approximation as

$$S_{\text{exp}} = (1 - x)S_{\text{Ru}^{5+}} + xS_{\text{Ru}^{4+}},$$

where $S_{\text{Ru}^{5+}}$ ($=3/2$), $S_{\text{Ru}^{4+}}$, and x are the spin number of Ru$^{5+}$, the spin number of Ru$^{4+}$, and the fraction of Ru$^{4+}$, respectively. As listed in table 1 $S_{\text{Ru}^{4+}}$ is calculated from x and $S_{\text{exp}}$ to be close to unity, indicating that Ru$^{4+}$ is in the low-spin state. Accordingly, we conclude that the conduction band for the doped electrons in this system is composed of the upper Hubbard $t_{2g}$ manifolds.

We notice that the transition temperature $T_N$ is much smaller than the Weiss temperature $\theta$, which has been associated with frustration effects. We also notice that $\theta$ is anomalously reduced with x, while $T_N$ does not change much. Even if some frustration effects may appear in the title compound, the disorder effects on $T_N$ and $\theta$ are highly difficult to explain; The induced Ru$^{4+}$ seems to weaken the spin-spin interaction (x $\theta$) but seems to
TABLE II. Various parameter for (Sr$_{1-x}$La$_x$)$_2$YRuO$_6$. $\mu_{\text{eff}}$, $\theta$, $S_{\text{exp}}$, and $S_{\text{Ru}^{4+}}$ are effective Bohr magnetic moment, Curie-Weiss temperature, spin of experimental value, and spin of Ru$^{4+}$ calculated Eqs. (4).

| $x$ | $\mu_{\text{eff}}$ ($\mu_B$/Ru) | $\theta$ (K) | $S_{\text{exp}}$ | $S_{\text{Ru}^{4+}}$ |
|-----|-------------------------------|-------------|------------------|------------------|
| 0   | 3.87                          | 338         | 1.5              | -                |
| 0.1 | 3.73                          | 240         | 1.43             | 1.16             |
| 0.2 | 3.65                          | 196         | 1.39             | 1.23             |

leave the ordered energy gain ($\propto T_N$) intact. In addition to the charge transport, the magnetic properties of this compound is not trivial, which should be clarified by further investigations. Singh and Tomy found two-step magnetic transition (27 and 32 K) in Sr$_2$YRuO$_6$ from a careful magnetization measurement, which implies an existence of two components.

Here we will discuss the electronic states and the conduction mechanism of the doped Sr$_2$ErRuO$_6$. Mazin and Singh calculated the electronic band structure of Sr$_2$YRuO$_6$. Despite the complicated structure, the electronic states can be quite simply understood; the RuO$_6$ octahedra are responsible for the valence band and the electrical conduction, which are isolated from each other by the YO$_6$ octahedra. Thus the electronic states are essentially understood from the energy levels of the RuO$_6$ cluster broadened by a small intercluster hopping. Then the highest occupied bands for the undoped compound are the lower Hubbard bands of the three $t_{2g}$ character, which are fully occupied. Thus this material is a Mott insulator in the sense that a charge gap is open between the upper and lower Hubbard bands. When electrons are doped, the upper Hubbard bands are partially occupied, which dominate the charge transport to give the negative Seebeck coefficient. We expect that the doped electrons are easily localized partially because of the small intercluster hopping energy of 0.14 eV. In addition, the electrons feel Hund’s coupling to the three electrons in the lower Hubbard bands in hopping from one site to another, which may further reduce the effective bandwidth and cause the activation energy in the mobility. This doped Mott insulator is therefore difficult to be metallic, which is a reason for the high resistivity and the Heikes-formula-type Seebeck coefficient.

Finally, we will make brief comments on the chemical properties of this double perovskite ruthenate. (i) The chemical substitution for the $B$ and $B'$ sites can also supply electrons to some extent. We made polycrystalline samples of Sr$_2$Er$_{1-x}$Ce$_x$RuO$_6$ and Sr$_2$ErRu$_{1-x}$Mo$_x$O$_6$, and measured the resistivity and Seebeck coefficient. We found that the high-temperature Seebeck coefficient is roughly independent of temperature, and the magnitude is determined by the formal valence of the Ru ions. The resistivity of Sr$_2$ErRu$_{1-x}$Mo$_x$O$_6$ is much higher than the other series, indicating that the Ru-O network is a conduction path. (ii) The title compound is highly stable in air up to 1000 K. We fabricated a trial product of the thermoelectric module consisting of Ca$_3$Co$_9$O$_{19}$ and (Sr,La)$_2$ErRuO$_6$, and examined the high-temperature stability. The module is highly stable up to 1000 K both mechanically and electronically, indicating that the title compound could be a candidate for an n-type thermoelectric oxide if the resistivity could be reduced substantially. (iii) We examined the substitution of 3d elements for Ru, and found that Cu and Zn ions were partially substituted for Ru to decrease resistivity. However, no trace of superconductivity was detected in our experiment.

IV. SUMMARY

We have prepared polycrystalline samples of (Sr$_{1-x}$La$_x$)$_2$ErRuO$_6$ and (Sr$_{1-x}$La$_x$)$_2$YRuO$_6$. The x-ray absorption and susceptibility measurements have clarified that the La substitution for Sr creates Ru$^{4+}$ in the low spin state, and indicates that the conduction bands are the upper Hubbard $t_{2g}$ manifolds. For $x=0.1$ at 800 K in air, the Seebeck coefficient is negative and large ($-160$ $\mu$V/K), and the thermal conductivity shows a low value of 7 mW/cmK. These two values are quite favorable as a thermoelectric material, and are one of the best data among the thermoelectric oxides. One last drawback is its high resistivity, which comes from a small transfer hopping between the RuO$_6$ clusters. If the transfer hopping were improved significantly by properly substituting the Sr or Y sites, the ordered ruthenates could be promising candidates for an n-type thermoelectric oxide.

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