X-ray Absorption Spectroscopy Study of the Effect of Rh doping in Sr₂IrO₄

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We investigate the effect of Rh doping in Sr₂IrO₄ using X-ray absorption spectroscopy (XAS). We observed appearance of new electron-addition states with increasing Rh concentration (x in Sr₂Irₓ−ᵧRhₓO₄) in accordance with the concept of hole doping. The intensity of the hole-induced state is however weak, suggesting weakness of charge transfer (CT) effect and Mott insulating ground states. Also, Ir J_{eff} = 1/2 upper Hubbard band shifts to lower energy as x increases up to x = 0.23. Combined with optical spectroscopy, these results suggest a hybridisation-related mechanism, in which Rh doping can weaken the (Ir J_{eff} = 1/2)–(O 2p) orbital hybridisation in the in-planar Rh-O-Ir bond networks.

Novel phenomena in 5d transition metal oxides (TMOs) have been a central subject in condensed matter physics. The large spin–orbit (SO) coupling transforms the 5d electron states from crystal-field-driven orbital states to certain SO-entangled J_{eff} states (J_{eff}: effective total angular momentum)¹². Consequently, a variety of exotic phases, including a relativistic Mott insulator¹–⁶, a quantum spin liquid⁷–¹¹, and a topological insulator¹²–¹⁴, are shown and expected in 5d TMOs. In the case of the most well-known 5d TMO, Sr₁IrO₄, even a superconductivity is expected because its crystal structure¹⁵, low energy Hamiltonian¹⁶,¹⁷, and magnetic excitation¹⁸,¹⁹ are similar to those of cuprates, La₂CuO₄. Indeed, recent angle-resolved photoemission spectroscopy studies showed the cuprate-like Fermi arc band and the d-wave gap in K doped Sr₂IrO₄²⁰,²¹.

One of the important issues to be resolved for 5d TMOs is how electron/hole doping affects their ground state. It is because the doping behaviours will provide us the clues for understanding emergent phenomena including superconductivity. Studies on La₂−ₓSrₓCuO₄, for instance, manifested a charge transfer ground state where doped holes reside in the ligand O orbitals²²,²³. Such hole states in O 2p orbitals form the Zhang-Rice singlet state²⁴, which has been known as the key component of high Tc superconductivity. Therefore, studies on carrier doping effects on 5d TMOs are necessary.

This article focuses on the effect of Rh doping in Sr₂IrO₄. Our Rh L-edge X-ray absorption spectroscopy (XAS) study revealed that the valence of Rh at low Rh concentrations is mainly +3 rather than +4, consistent to recent studies²⁵,²⁶. Therefore, Sr₁Ir₁−ₓRhₓO₄ can be viewed as hole-doped iridates analogous to hole-doped cuprates. With O K-edge XAS, we particularly report two distinct features. One is that a new electron-addition state emerges near the chemical potential as a result of Rh doping. The intensity of the hole-induced states is, however, rather weak compared to the case of cuprates or nickelates. This suggests that the charge transfer (CT) effect is weak and doped holes mainly reside in the Ir 5d orbitals, being consistent with known Mott insulating ground state of Sr₁IrO₄. The other feature is that the 5d upper Hubbard band (UHB) hybridised with in-plane oxygen (Oₖ) 2p orbitals undergoes a strong redshift whereas the same UHB but hybridised with apical oxygen (O₅) 2p orbitals hardly does. We propose that Rh doping can decrease the hybridisation between Ir and O via charge reconstruction in Oₖ ions and/or via the less extended nature of Rh wave function. Optical spectroscopy study further showed that the onset energy of CT transition from Oₖ 2p to Ir 5d indeed decreases with increasing Rh concentration, supporting the interpretation of the XAS results.

Results

Rh L₁-edge XAS was performed to estimate the hole concentration. Figure 1(a) shows the spectra of the Sr₂Ir₁−ₓRhₓO₄ crystals for x = 0.10, 0.23 and 0.41. The peak near 3006 eV corresponds to the L₁-edge white line, which

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The peaks highlighted by filled triangles in Fig. 3 are the Ir 5d states hybridised with Ir ions while the peaks marked as open triangles correspond to the Ir 5p states hybridised with Rh ions. Thus, we attribute the split (of roughly 0.6 eV) as a chemical shift due to different chemistry between O atoms in Sr2IrO4 and Sr2RhO4. Therefore, peaks A and B correspond to the electron-addition states hybridised with O or O2− orbitals, respectively. The energy difference between peaks A and B (~0.6 eV) in Fig. 3(b) reflects the difference in energy between O2− and O2− orbitals.

With increasing x, we observe appearance of lower-energy peaks, A and B, as highlighted in Fig. 3. The energies of peaks A and B are clearly distinct from those of the Sr2IrO4 and Sr2RhO4 peaks. Such lower-energy peaks are generally shown in hole-doped systems including nickelates20 or cuprates22,23, because new electron-addition states appear due to hole doping. Since the energy of such states should be close to that of original occupied states, the difference in energy between UHB and the hole-induced states should roughly reflect the charge gap. We see that the energy difference between peak A and Ir UHBs (also, between peak B and Ir UHBs) is roughly 0.5 eV, similar to the known value of the charge gap in Sr2IrO4. Therefore, peaks A and B correspond to the electron-addition states hybridised with O or O2− orbitals, respectively. The energy difference between peaks A and B (~0.6 eV) in Fig. 3(b) reflects the difference in chemistry between O2− and O2− similar to the cases of UHBs and UHBs.

Apart from the low-energy peaks (A and B), it is clearly shown in both Fig. 3(a,b) that the UHBs peaks shift to lower energy with increasing x. The amount of the UHB, redshift with respect to the case of undoped iridate is approximately 0.25 eV at x = 0.23. Interestingly, the redshift is much less predominant in the case of UHBs. Such an anisotropic peak shift in the two peaks is not easy to understand because they share identical Ir 5d orbitals (Jsat = 1/2 UHB). As will be discussed in the next section, the redshifts are the signatures of reduced O 2p–Ir 5d hybridisation strength upon the hole doping.
To conclude this section, we observe two distinct features due to Rh doping in the O $K$-edge spectra: One is the emergence of new electron-addition states ($A$ and $B$) at low energy and the other is a concomitant redshift of Ir UHB$_p$. These will be further analysed in Discussion.

**Discussion**

To describe the spectral evolution upon Rh doping quantitatively, we fitted the O $K$-edge spectra with $E$/$c$ using Lorentz-Gaussian models. For $x = 0.04$–$0.23$ samples, only Rh$^{3+}$ $(d^6)$ prevails so that there could exist three unoccupied states near the chemical potential: the hole-induced states (peak $A$), Ir UHB$_p$, Rh eg band with increasing order of energy. Figure 4(a) shows the best fitting result of $x = 0.23$ spectra using the three peaks with a higher energy background for Ir eg band (dashed line). Meanwhile, for highly doped samples ($x \geq 0.41$), it was difficult to obtain reliable fitting results because of the mixed contributions of Rh$^{3+}$ and Rh$^{4+}$ states.

We note that the intensity of peak $A$ in Rh doped Sr$_2$IrO$_4$ is much smaller than those in cuprates and nickelates. Figure 4(b) shows the areal intensity of peak $A$ normalised by that of UHB$_p$, namely $I_A/I_{UHB}$ of the Rh-doped iridates as a function of $y$. Small error bars account for the uncertainty in determining the peak intensity. For comparison, we appended the $I_A/I_{UHB}$ values in (La$_{1-x}$Sr$_x$)$_2$CuO$_4$ as a function of the number of holes per Cu as well. For the case of the cuprates, $I_A/I_{UHB}$ exceeds 1 even at $y = 0.1$, representatively showing the strong nature of CT insulator. In contrast, $I_A/I_{UHB}$ in Rh doped Sr$_2$IrO$_4$ at $y = 0.11$ (or $x = 0.10$), is only approximately 0.14. This indicates that the CT effect in iridates is much weaker compared to that in the cuprates. This observation is consistent with the Mott insulating ground state in Sr$_2$IrO$_4$, in which doped holes should reside mainly in metal d orbitals rather than ligand orbitals.

Another important finding in the O $K$-edge XAS spectra is that only UHB$_p$ undergoes a redshift whereas UHB$_a$ does not despite they represent the same UHB. Figure 5(a) shows the amount of redshifts ($\Delta E$) of UHB$_p$ and UHB$_a$ with respect to those of undoped Sr$_2$IrO$_4$ as a function of $y$. Only the data for $x \leq 0.23$, which exhibit little secondary phase (Rh$^{4+}$), are collected. The error bars in the figure account for the uncertainty in determining the peak position through the fitting processes. In order to understand the origin of the redshifts, we first suspect the consistency with the structural evolution. It has been reported that with increasing $x$, the Ir-O bond is slightly shortened in average and the Ir-O-Ir bonding angle increases in average. This moderate structural distortion would tend to increase the hybridisation strength of the Ir $J_{\alpha\beta} = 1/2$ UHB–O 2p orbitals, which should have resulted in a blueshift of UHB$_p$ or UHB$_a$. Therefore, structural change upon Rh doping cannot explain the redshifts of UHB.

The plausible mechanism is the decrease of orbital hybridisation in the Ir-O bonds. The energy of UHB can be significantly influenced by the orbital hybridisation of bare Ir $J_{\alpha\beta} = 1/2$ state and O 2p state. The schematic for the orbital hybridisation is illustrated in Fig. 5(b). As a result of the hybridisation, formed are an antibonding state $\pi^*$, which is commonly called “Ir UHB”, above the chemical potential and a bonding state $\pi$, commonly called “O 2p”,

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**Figure 2.** Polarization-dependent O $K$-edge XAS spectra of Sr$_{2-}Rh_x$O$_4$ crystals. The solid and dashed curves show the spectra taken with $\sigma$- and $\pi$-polarised light, respectively. The inset shows the measurement geometry.
What is observed in the O $K$-edge XAS data is the lowering of $\pi^*$ (UHB) hybridised with O $p$. Therefore, the redshift of UHB can be easily understood if Rh doping can effectively reduce the

**Figure 3.** Low energy parts in the O $K$-edge XAS spectra with (a) $E//c$ and (b) $E\perp c$, where $c$ is the surface normal axis. The spectra of Sr$_2$RhO$_4$ were taken from ref. 27. The $E\perp c$ spectra are reproduced from the $\sigma$-polarisation data, and the $E//c$ spectra are deduced from the relation: $f_{E//c} = t_{E//c} - \cos^2 60^\circ t_{E\perp c}$. Solid (open) triangles highlight the peak positions of the Ir $j_e = 1/2$ UHB hybridised with the in-plane (apical) O 2p.

**Figure 4.** (a) The $E//c$ spectra of $x = 0.23$ sample. The open circles are the experimental data and the black solid line is the fitting result. The red, blue, purple, and dashed curves are for Ir UHB, peak A, Rh $e_g$, and the Ir $e_g$ background, respectively. (b) $I_A/I_{UHB}$ as functions of $y$ (concentration ratio of dopants to host cations). The data of La$_2$CuO$_4$ is taken from ref. 23 for the comparison.

below the chemical potential. What is observed in the O $K$-edge XAS data is the lowering of $\pi^*$ (UHB) hybridised with $O_p$ (UHB). Therefore, the redshift of UHB can be easily understood if Rh doping can effectively reduce the
orbital hybridisation. This mechanism is more effective in the in-planar directions because the influence of Rh can reach Ir only via the in-plane O, namely, through Rh-O\(^p\)-Ir bond chains due to quasi-2-dimensional crystal structure of Sr\(_2\)IrO\(_4\). This explains why UHB\(^p\) suffers much more intense redshift compared to UHB\(^a\) (see Fig. 5(a)).

We propose two possible microscopic origins how Rh doping can decrease the orbital hybridisation between Ir\(^d\) and O\(^{2p}\) orbitals. One is related to the difference of valence of ions. When the iridate is doped with Rh\(^{3+}\) ions, charge redistribution in O\(^p\) ions occurs: Since Rh can supply only 3 electrons to the O\(^p\) ions, O\(^p\) tend to attract more electrons from neighbouring Ir ions. It effectively increases the electronegativity of O\(^p\) in Ir-O\(^p\) bond, thereby making the Ir-O\(^p\) bond ionic. Similar mechanism but opposite trend has been discussed in H\(^d\) doped VO\(_2\)\(^{30}\). The enhanced ionic character of the Ir-O\(^p\) bond can decrease hybridisation between two ions. The other is related to the less extended nature of Rh wave functions. According to the results of previous dynamical mean-field theory calculations, hybridisation between Ir and O ions is much stronger than that between Rh and O ions\(^{31}\). Because the valence \(d\) orbitals comprise the admixture of Ir and Rh\(^d\) orbitals, Rh doping can effectively decrease the hybridisation between Ir\(^5d\) and O\(^{2p}\) orbitals.

To support the hybridisation mechanism discussed above, we measured the difference in energy between \(\pi\) and \(\pi^*\) using optical spectroscopy. Figure 5(c) shows the in-plane real part of optical conductivity \(\sigma_1(\omega)\) of the \(x = 0, 0.10, \) and 0.23 samples. According to a previous study\(^{32}\), the broad features above photon energy of 2.5 eV can be attributed to a charge excitation from \(\pi\) (O\(^p\)) to \(\pi^*\) (Ir UHB). Meanwhile, the broad peak near 1 eV and the bump near 2.3 eV can be attributed to Ir \(d-d\) transition, which is less relevant to the main interest in this work. It is clearly observed that the onset energy of the high energy bump decreases with increasing \(x\). This suggests that the Ir UHB–O\(^{2p}\) hybridisation becomes weaker as the hole doping is promoted, consistent with our XAS observations and interpretations.

The energy differences between \(\pi\) and \(\pi^*\) bands can be roughly estimated from the extrapolations toward the abscissa. Compared to the case of undoped Sr\(_2\)IrO\(_4\), the onset energy of \(x = 0.23\) (\(x = 0.10\)) decreased by approximately 0.5 eV (0.3 eV), which is about two times larger than the amount of the UHB\(^p\) redshift (shown in Fig. 5(a)). This is consistent with the hybridisation mechanism for weakened hybridisation not only lowers the \(\pi^*\) band but also lifts up the \(\pi\) band by similar magnitude. Therefore, \(\sigma_1(\omega)\) data supports the hybridisation mechanism for the UHB redshifts.

It is worthwhile to consider the consequence of reduced Ir UHB–O\(^{2p}\) hybridisation due to Rh doping on the low-energy physics in Sr\(_2\)IrO\(_4\). It is well known that the low energy physics in strongly correlated electron systems can be largely determined by the ratio of on-site Coulomb interaction (\(U\)) to the single electron bandwidth (\(W\).
According to the results of a recent ab initio calculation, the value of $U$ would decrease due to enhanced ligand screening so that $U/W$ should decrease altering the electronic structure near the bandgap. However, the results of recent angle-resolved photoemission experiment show that Rh doping induces only a rigid shift of bands without an appreciable change in band dispersions. This controversy can be resolved by noting that the value of $W$ should decrease as well, because the decrease in hybridisation strength (by Rh doping) will reduce the bandwidth of the UHB. Therefore, the value of $U/W$ should not change substantially.

In conclusion, Rh doped Sr$_2$IrO$_4$ exhibit peculiar evolution in electronic structure: i) a charge transfer state emerges near the chemical potential but the feature is not that strong as in the hole-doped cuprate, consistent to the Mott insulating ground state, and ii) the Ir UHB energy is lowered by a few tenths eV due to reduced Ir $J_{\text{eff}} = 1/2$ UHB–O 2p hybridisation strength upon the hole doping.

**Methods**

**Materials.** High-quality single crystals Sr$_{1-x}$Rh$_x$O$_3$ (0 ≤ x ≤ 0.71) were synthesized off-stoichiometric quantities of SrCl$_2$, SrCO$_3$, Ir$_2$O$_3$, and RhO$_2$ using self-flux method. Detailed methods are described elsewhere.

**X-ray absorption spectroscopy.** Polarization-dependent O K-edge X-ray absorption spectroscopy (XAS) was performed at the 2A beamline of the Pohang Light Source in total electron yield mode. To obtain a clean surface, we cleaved samples in situ in an ultra-high vacuum (~6 × 10$^{-8}$ Pa). We fixed the angle of the incident beam at 60 degrees, and changed the direction of polarisation to resolve the in- and out-of-plane O responses. Rh L-edge XAS was performed at the 16A1 beamline of the National Synchrotron Radiation Research Center in Taiwan in fluorescence yield mode. All the XAS spectra were collected at room temperature.

**Optical spectroscopy.** We performed ellipsometry measurement to obtain the real part of optical conductivity between 0.74 and 4 eV at room temperature using a V-VASE ellipsometer (J. A. Woollam Co.).

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Author Contributions
C.H.S. and D.-Y.C. conceived the experiments, and C.H.S., D.-Y.C. and C.-T.K. performed the XAS measurements. L.J.S. performed the optical measurement. T.F.Q. and G.C. fabricated the crystals. D.-Y.C. and T.W.N. supervised the research. All authors contributed to the writing of the manuscript.

Additional Information
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