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Pressure-induced insulator-metal transition in Ca₂Ru_{0.92}Fe_{0.08}O₄ investigated by infrared microspectroscopy

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ABSTRACT

We investigated pressure-induced insulator-metal transition in $Ca_2Ru_{0.92}Fe_{0.08}O_4$ by using infrared microspectroscopy. As the pressure is increased up to 1.7 GPa, we observed a large increment of the reflectivity in the entire mid-infrared range. Accompanied by such a clear signature of the insulator-metal transition, we found an evidence of the structural transition from the frequency shift of the Ru-O stretch phonon mode which is attributed to the shortening of the in-plane Ru-O bond length. When we compared these pressure-dependent changes with the corresponding temperature-dependent results, we found that the pressure-induced metallic state has a higher reflectivity as well as the higher phonon frequency. Indeed, it turns out that the pressure-induced metallic state of $Ca_2Ru_{0.92}Fe_{0.08}O_4$ looks very similar with the metallic state of Sr-substituted Ca_2RuO_4 not only in the reflectivity level but also in the phonon frequency. This suggests that the electronic properties are closely related to the structural degree of freedom, and the pressure can be a useful parameter to induce the transitions from the Mott-insulator to the metal and further to the superconductor as observed for Sr_2RuO_4 .

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1. Introduction

Whereas chemical substitutions have often been used to influence the electronic state through the controls of a band-filling and/ or a bandwidth using ions of different valences or ionic radii, respectively [1], there is sometimes a clear limitation in this method, for example, in growing high-quality samples with a large amount of the chemical substitution. Instead of disturbing the materials by chemical methods, one can adopt other static perturbations, such as pressure [2–4], electric field [5], magnetic field [6], and so on. Pressure, in particular, is able to control the electronelectron correlation effect by reducing the intersite distance and hence increasing the electron transfer integral or the bandwidth, and a number of studies have been done to investigate the pressure-induced insulator-to-metal transition (IMT) [7,8].

 Ca_2RuO_4 is an antiferromagnetic Mott insulator. Transition to the metallic state can be driven in various routes, namely, by an increase of temperature above 357 K and a chemical substitution in a form of $Ca_{2-x}Sr_xRuO_4$ [9–11]. Interestingly, applying a pressure *P*

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https://doi.org/10.1016/j.cap.2017.10.015 1567-1739/© 2017 Published by Elsevier B.V. only up to about 0.5 GPa can induce the similar transition to the metallic state [12,13]. Whereas the structural changes are confirmed to occur accompanied by the insulator-metal transition from the x-ray scattering experiment [14], it is also suggested that the superconducting state can appear at P > 9 GPa below 0.4 K similarly for Sr₂RuO₄ below 1.4 K under the ambient pressure [14,15].

In this paper, we investigated the pressure-induced insulatormetal transition in Ca₂Ru_{0.92}Fe_{0.08}O₄ by using infrared microspectroscopy. A slight Fe doping in Ca₂RuO₄ can increase the mechanical stability during the insulator-metal transition whereas the material itself shares most of the basic properties with Ca_2RuO_4 including the IMT temperature T_{IM} , i.e., 380 K and 357 K for Ca₂Ru_{0.92}Fe_{0.08}O₄ and Ca₂RuO₄, respectively [16]. As we applied the pressure at room temperature, we found a clear signature of the insulator-metal transition at about 1 GPa; the reflectivity in the mid-infrared region shows a large increase from about 5% to about 60%. Also, we observed a strong blue shift of a Ru-O stretch phonon mode. Based on these observations, we discuss pressure-dependent changes in electronic and structural phases, and compare them with the corresponding changes obtained upon the temperature increase and the chemical substitution.

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2. Experimental

A high-quality Ca₂Ru_{0.92}Fe_{0.08}O₄ single crystal is grown by floating zone method [16]. For the experiment under the application of the hydrostatic pressure using a diamond anvil cell (Diacell CryoDAC-Mega, Almax easyLab Ltd.), the sample is prepared to have a size about 120 um in diameter, and then it is contained in a chamber of the 530 um wide Cu gasket which is filled with a pressure medium KBr. For the pressure calibration, the ruby powders are put next to the sample. From the ruby fluorescence at several places, we confirmed that the pressure is applied homogeneously throughout the sample chamber. For the reflectivity measurements in the infrared region, we used the commercial Fourier-transform spectrometer (Bruker 80v) combined with the infrared microscope (Hyperion 2000). With variations of temperature and pressure, we obtained reflectance spectra between 280 and 7500 cm^{-1} and between 550 and 7500 cm^{-1} , respectively. We mounted the gold film close to the sample inside the diamond anvil cell, and obtained the reflectivity spectra with the reference correction assuming that the reflectivity from the diamond-Au interface is 100%. In this procedure, it would be possible to have the reflectivity values underestimated due to a roughness of the sample surface.

3. Results and discussion

Before examining the pressure-dependent results, we investigate the temperature (*T*)-dependent reflectivity changes. Fig. 1(a) shows reflectivity spectra of $Ca_2Ru_{0.92}Fe_{0.08}O_4$ obtained by varying temperature from 300 K to 460 K. At 300 K, the frequency dependence of the spectrum behaves like a typical insulator; the reflectivity in the mid-infrared region is flat and small-valued. The sharp



Fig. 1. Temperature-dependent infrared reflectivity spectra of $Ca_2Ru_{0.92}Fe_{0.08}O_4$. (a) Reflectivity spectra in a full spectral range. (b) Far-infrared reflectivity spectra showing the response of a transverse-optic phonon mode. Symbol indicates fitting results using a Drude-Lorentz model. (c) Reflectivity at 1500 cm⁻¹ (red square) and transverse-optic phonon mode frequency (blue circle) as functions of temperature. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

spikes in the low frequency region is attributed to the optical phonon modes. With an increase of temperature, the reflectivity spectra exhibit dramatic variations. First, the reflectivity increases gradually, and shows an abrupt jump between 360 K and 380 K. The high reflectivity at T > 380 K and its frequency dependence clearly suggest that the material is in the metallic state at the high temperature in good agreement with the *dc*-transport measurement [16]. Second, the phonon modes also exhibit intriguing *T*-dependent changes. With an increase of temperature at $T < T_{IM}$, the low frequency modes located below 500 cm^{-1} show red-shifts, but the higher frequency mode located around 600 cm⁻¹ exhibits a blue shift. With a further increase of temperature above T_{IM} , most of the phonons become smeared out due to the screening effect by free carriers, but the highest frequency mode still remains visible with its frequency shifted further to the higher frequency side. Several tiny dip or spike structures seen below 400 cm⁻¹ originate from the imperfect reference correction due to the atmospheric absorptions.

To trace the *T*-dependent changes in the phonon responses, we fit the reflectivity spectra by using a Drude-Lorentz model for the dielectric constants,

$$\tilde{\epsilon}(\omega) = \epsilon_{\infty} + \frac{\omega_p^2}{\omega^2 + i\gamma\omega} + \sum_j \frac{S_j}{\omega_j^2 - \omega^2 + i\Gamma_j\omega}$$

Here, ε_{∞} is the high-frequency dielectric constant. The second term in the right corresponds to the Drude response of the free carriers with the plasma frequency $\omega_{\rm p}$ and the scattering rate γ . And, the third term accounts for the phonon contributions where S_j , ω_j , and Γ_j represent the strength, the frequency, and the damping constant of the j-th phonon mode, respectively. As complex-valued refractive index is given as $\tilde{N} = n + ik = \sqrt{\tilde{\varepsilon}}$, the reflectivity from the airsample interface can be calculated as

$$R_{as} = \frac{(n-1)^2 + k^2}{(n+1)^2 + k^2}.$$

One of the fitting curves is shown with open circles in Fig. 1(a)and (b) which could reproduce the experimental result obtained at 300 K. Note that all the results at different temperatures could be fit reasonably well. Fig. 1(c) displays the T-dependent variation of the mode frequency ω_{TO} of the highest frequency phonon mode. As T increases, ω_{TO} increases gradually, and it jumps to have the higher value at T_{IM} . Actually, these behaviors are quite similar with the Tdependent changes of infrared reflectivity; the reflectivity at 1500 cm^{-1} is displayed by closed symbols in Fig. 1(c). These behaviors clearly indicate that the insulator-metal transition with an increase of temperature is closely related to the structural transition. A factor-group analysis yields a total of 33 $[11(B_{1u} + B_{2u} + B_{3u})]$ infrared-active modes for Ca₂RuO₄ with an orthorhombic crystal structure (space group *Pbca*- D_{2h}^{15}) [17], and the highest frequency mode corresponds to the Ru-O stretch mode. Therefore, the strong blue shift of this mode is attributable to the shortening of the inplane Ru-O bond length in good agreement with the general behaviors observed for the layered ruthenates upon their transitions to the metallic state [18,19].

Let us now turn our interest to the pressure-dependent results. Fig. 2(a) displays the reflectivity spectra obtained with the application of the hydrostatic pressure from 0.2 GPa to 1.7 GPa. Note that the reflectivity here is defined at the diamond-sample interface, and called R_{ds} . With an increase of the pressure, R_{ds} of Ca₂Ru_{0.92}Fe_{0.08}O₄ exhibits large changes. In particular, when the pressure is increased to about 0.9 GPa and 1.2 GPa, the overall reflectivity shows a strong enhancement by about 60%. This manifests that the insulator-metal phase transition occurs at P~1 GPa.

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