Thickness-dependent metal-insulator transition in epitaxial SrRuO₃ ultrathin films

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Abstract

Transport characteristics of ultrathin SrRuO₃ films, deposited epitaxially on TiO₂-terminated SrTiO₃ (001) single-crystal substrates, were studied as a function of film thickness. Evolution from a metallic to an insulating behavior is observed as the film thickness decreases from 20 to 4 unit cells. In films thicker than 4 unit cells, the transport behavior obeys the Drude low temperature conductivity with quantum corrections, which can be attributed to weak localization. Fitting the data with 2-dimensional localization model indicates that electron-phonon collisions are the main inelastic relaxation mechanism. In the film of 4 unit cells in thickness, the transport behavior follows variable range hopping model, indicating a strongly localized state. Magnetoresistance measurements reveal a likely magnetic anisotropy with the magnetic easy axis along the out-of-plane direction.

PACS: 71.30.+h, 72.15.Rn, 73.50.-h, 75.30.Gw

I. Introduction

Ferromagnetic conductive oxide SrRuO₃ (SRO) is among electron-correlated materials, which have

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attracted much attention due to intriguing electrical and magnetic properties originating from entanglement of charge, spin and orbital orders.^{1,2} SRO is also the only known ferromagnetic metal among 4d transition metal oxides with GdFeO₃-type orthorhombic crystalline structure.³ Bulk SRO exhibits a ferromagnetic Curie temperature about 160 K and a magnetic moment of 1.6 μ_B /Ru.^{4,5} Due to its high chemical stability and perovskite structure, SRO thin films can be integrated with other functional oxides. It is often used as electrodes in all-perovskite electronic devices based on heterostructures composed of superconducting, ferromagnetic, and/or ferroelectric layers.⁶⁻⁸ The electrical and magnetic properties of SRO films can be modulated by lattice strain and interface structures, as reported in ultrathin films, heterostructures and superlattices.⁹⁻¹⁴

Recently, thickness effects on electron-correlated oxides have been extensively studied in nickelates and ruthenates. The conductivity of these ultrathin films can be greatly suppressed by reducing the film thickness, with a metal-insulator transition observed below a critical thickness.¹⁵ For examples, thickness-driven metal-insulator transition was observed in LaNiO₃ ultrathin films, when film thickness was reduced to 5 unit cells (u.c.).¹⁶ In SRO ultrathin films, prominent thickness effects have also been reported. Toyato *et al.* reported a metal-insulator transition in SRO occurring at a film thickness of 4-5 u.c..⁹ Xia *et al.* reported an insulating state with an antiferromagnetic interface layer and an exchange-bias effect in ultrathin SRO film below 4 u.c. in thickness.¹⁷

It appears essential to understand the nature of electronic transport for these electron-correlated oxide films in the ultrathin limit for future investigation of SRO-based heterostructures. In this report, we present thickness-dependent transport investigations on a series of SRO ultrathin films, 4 to 20 u.c. in thickness. With decreasing film thickness, electrical transport of SRO evolves from a metallic to an insulating behavior. A transition region is observed between the insulating state and the metallic state, where the ultrathin films exhibit a temperature-driven metal-insulator transition due to weak localization originating from electron-phonon interactions. Magnetoresistance of ultrathin SRO films are also addressed.

II. Experimental

SRO ultrathin films were epitaxially deposited on (001) TiO_2 -terminated SrTiO_3 (STO) substrates (Shinkosha Co. Ltd., Japan) by pulsed laser deposition at 750 °C with an oxygen pressure of 0.05 mbar. The 248 nm ultraviolet radiation from a KrF excimer laser (CompexPro205F, Coherent) was used at a repetition rate of 4 Hz. The laser energy density on the SRO ceramic target was estimated as 2.0 J/cm². The film growth was monitored by *in situ* reflective high energy electron diffraction (RHEED). The film thickness was controlled from 2 to 20 u.c. by counting the number of RHEED intensity oscillations. After the deposition, the samples were annealed *in situ* at 750 °C in 0.5 mbar oxygen ambient for 30 minutes, and then allowed to cool naturally to the room temperature.

The structure of SRO ultrathin films were characterized by high resolution X-ray diffraction (XRD) using the BL14B1 beam line of Shanghai Synchrotron Radiation Facility ($\lambda=0.12348$ nm) and high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) in a Hitachi HD2700C microscope. An Asylum Research Cypher atomic force microscope (AFM) was used to examine the surface morphology. Resistance, magnetoresistance and Hall measurements on the SRO ultrathin films were performed using a physical property measurement system (PPMS-9, Quantum Design) at various temperatures.

III. Result and discussion

Fig. 1 (a) shows the clear RHEED intensity oscillations of the specular spot recorded *in situ* during the deposition. It has been observed that the first oscillation is much stronger than the subsequent ones. This can be attributed to the coexistence of both layer-by-layer and step-flow growth modes.¹⁸ Both of these two growth modes result in a smooth sample surface as expected. For example, Fig. 1 (b) shows the AFM image of a 10 u.c. sample, with a step-and-terrace morphology. Fig.1 (c) shows the root mean square roughness (RMS) of SRO films with various thicknesses. The RMS roughness values of all our SRO samples are around 1.5 Å, indicating atomically flat surfaces. The structure of the ultrathin SRO films was checked by XRD. Fig. 2 (a) shows, for example, the high resolution XRD ω -2 θ pattern of a 10 u.c. sample, around the (002) diffraction peak of STO. The strong diffraction from (002) plane of SRO indicates the high crystalline quality. And the presence of clear thickness fringes indicates smooth

surface and abrupt interface in our SRO/STO samples.¹⁹ The thickness (T) of the ultrathin film thus could be independently calculated from $T = \lambda / [2\Delta\theta(f)\cos\theta_B]$, where λ is the X-ray wavelength, $\Delta\theta(f)$ stands for angular distance between two adjacent fringes and θ_B means the Bragg diffraction angle.^{20,21} The thickness thus calculated, for the film with 10 periods of RHEED oscillations, is 3.98 nm, in agreement with the height of 10 u.c.. The SRO/STO interface should be coherent due to the small lattice mismatch of about 1.1%. Fig. 2 (b) shows the cross-sectional HAADF-STEM image, taking an 8 u.c. SRO film as an example. Coherent SRO/STO interface can be observed clearly. The HAADF image, formed by collecting the scattered electrons, is highly sensitive to variations of the atomic number.²² This enables identification of cations on different atomic sites, as indicated in Fig. 2 (b). The Ru cation has a larger atomic number than that of Ti, leading to a brighter contrast in the HAADF image. The integrated intensity profile of lattice spots near the SRO/STO interface is shown in Fig. 2 (c), indicating an abrupt interface. It is also revealed that the STO substrate is TiO₂-terminated.

To study the thickness dependent transport properties, sheet resistance was measured as a function of temperature for SRO films in various thicknesses. However, the transport data of films thinner than 4 u.c. are not included because the resistance is beyond the measurement capability of our facilities. As shown in Fig. 3 (a), the sheet resistance increases continuously as the film thickness decreases and three regimes can be distinguished based on the transport behaviors at low temperatures. For films more than 15 u.c. in thickness, the sheet resistance is below 0.6 k $\Omega/\square$ at room temperature and remains metallic down to 5 K. Insulating state with upturn in sheet resistance could be observed at low temperatures in SRO films from 10 to 5 u.c. in thickness. When the thickness is further reduced to 4 u.c., SRO shows an insulating characteristic in the entire temperature range studied. Toyota *et al.*⁹ and Xia *et al.*¹⁷ also reported a metal-insulator transition at ~ 4 u.c. in thickness, accompanied with a ferromagnetic-paramagnetic transition. However, Chang *et al.*¹¹ observed that SRO thin films remain metallic down to a thickness of 2 u.c.. The origin of this discrepancy is unclear at this moment. Chang *et al.*¹¹ have also compared the transport characteristics of films deposited under 3-dimensional growth to the films under step-flow growth, and proposed that the suppressed conductivity is associated with the rough surface morphology resulted from the 3-dimensional growth. However, in this work, the surface roughness is not sensitive to the film

thickness as shown in Fig. 1 (c) . Further studies are necessary to elucidate this discrepancy.

In thick SRO films, there is a kink in the sheet resistance-temperature dependence. For example, the resistance decreases more rapidly below 110 K in the 20 u.c. SRO sample, as shown in Fig. 3 (a). This transition in transport characteristics is associated with para-ferromagnetic transition in SRO, which was first observed experimentally by Fisher and Langer.²³ Fig. 3 (b) plots the derivative of resistance against temperature, in which the transition temperature, T_C for the para-ferromagnetic transition, can be easily observed. The transition temperature decreases from 110 to 90 K, as the film thickness decreases from 20 to 6 u.c., much lower than the value in bulk SRO (160 K).⁴ It has been reported that the magnetic characteristics of SRO films are highly sensitive to cell distortion or RuO_6 octahedral tilting.⁴ T_C has been found to decrease with increasing pressure due to the modulation on the π^* narrow band formed by the overlap between Ru $4d$ and O $2p$ orbitals.²⁴ The decrease of T_C in our SRO films could be ascribed to weakened ferromagnetic interaction²⁵ resulting from increased cell distortions in thinner films. The enhanced cell distortion also strengthens structural disorder in SRO films and may enhance electron-phonon correlations discussed in following paragraphs.

As reported by Allen *et al*,²⁶ bulk SRO exhibits a bad metal behavior, manifesting itself with a linear temperature dependence above T_C .^{27,28} Thicker SRO films, which are metallic in all the temperatures studied, indeed show such a bad metal behavior. As shown in Fig. 4 (a), for instance, the resistivity of 15 u.c. SRO film increases linearly with temperature up to 300 K. Below T_C , the resistivity data can be well-fitted with a quadratic temperature dependence following

$$\rho = \rho_0 + AT^2, \quad (1)$$

as demonstrated in the inset of Fig. 4 (a). The residual resistivity ρ_0 and temperature coefficient A obtained from the fitting are $182.2 \mu\Omega\cdot\text{cm}$ and $4.7\times 10^{-11} \Omega\cdot\text{cm}\cdot\text{K}^{-2}$, respectively. The T^2 dependence is well-known for a Fermi liquid and is ascribed to the electron-electron scattering.²⁹ As pointed out by Volkenshtein *et al*,³⁰ the coefficient A in the order of $\sim 10^{-11} \Omega\cdot\text{cm}\cdot\text{K}^{-2}$ corresponds to a typical metallic ferromagnet. These indicate that SRO films thicker than 15 u.c. exhibit similar transport characteristics as bulk SRO.

Upturn in the sheet resistance, indicative of a metal-insulator transition, can be observed in SRO films of

5 to 10 u.c. in thickness, at temperatures indicated by arrows in Fig. 3 (a). As previously reported in LaNiO_3 and $\text{La}_{0.75}\text{Sr}_{0.25}\text{VO}_3$ films,^{16,31} the temperature-driven metal-insulator transition in correlated thin films could be attributed to the weak localization effect. In these ultrathin films, weak localization due to microstructural disorder may play an important role in the resistivity increment at low temperatures. Electrons may experience a series of random scatterings in electronic systems with disordered impurities. The interference of electron wave functions may cause weak localization around the impurities and thus increases the resistivity. Also it has been established that the surface and interface of oxides in epitaxial heterostructures can be more complex than previously assumed.³² Disorders like lattice distortion and interface reconstruction are within atomic length scale, which cannot be easily identified.

In ultrathin SRO films, the transport characteristics can be interpreted by the competition of localization and delocalization of electron states near Fermi level,³³ which can be tuned by the degree of disorder. With increasing temperature, localization is suppressed, and the strengthened delocalization leads to a decrease in resistance. As the temperature further increases, delocalization is saturated, and electrons are scattered by electrons. This results in the presence of minima in resistance-temperature curves. As shown in Fig. 3 (a), the metal-insulator transition temperature shifts from 25 to 150 K as the film thickness decreases from 10 to 5 u.c., indicative of the strengthening of disorder with decreasing film thickness. In thinner films, higher temperature is required for the delocalization.³³

We plot the low temperature conductance of the 8 u.c. film as an example of SRO films exhibiting a metal-insulator transition. The conductance shows a metallic character above about 45 K, decreasing with the increase of temperature as shown in the inset of Fig. 4 (b). Below this temperature, the conductance decreases with decreasing temperature, showing an insulating behavior. Following Wu's work,³⁴ we use a two-dimensional (2D) model to describe the transport behavior in ultrathin SRO films, which requires that the carrier mean free path (l) be larger than the film thickness. The carriers mean free path can be estimated from,

$$l = h\tau / m^* \lambda_F = \sigma h / ne^2 \lambda_F, \quad (2)$$

where σ stands for the conductivity, e and h are the charge of an electron and the Planck's constant, respectively, n denotes the spatial density of electrons, τ is the relaxation time between two adjacent

scatterings, m^* stands for effective mass of electron, and λ_F is the electronic Fermi wavelength. For SRO thin film with different thickness from 5 to 10 u.c., λ_F is taken to be about 4.5 Å, as previously used in the literature.³⁵ Thus, the mean free path values in SRO films of 5, 6, 8, and 10 u.c. in thickness can be estimated as 2.3, 3.1, 4.5 and 5.0 nm at 50 K, respectively, which are larger than the individual film thickness. This justifies the application of a 2D weak localization model, in which the temperature dependence of conductivity is described by a renormalized logarithmic function as,³⁶

$$\sigma = \sigma_0 + p \frac{e^2}{\pi h} \ln (T/T_0), \quad (3)$$

where σ and σ_0 are the conductivity and Drude conductivity, respectively, and T_0 denotes a temperature constant related to the inelastic scattering mean free path $l_{in} \sim T^{p/2}$. If the electron-electron interactions dominate the inelastic scattering, $p = 1$; whereas, if electron-phonon interactions are the main inelastic relaxation mechanism, $p = 3$. Fig. 4 (b) shows the linear fit of the sheet conductance as a function of $\ln (T)$ for the 8 u.c. SRO film. The value of p estimated from the slope of the linear fit is about 3, indicating that the electron-phonon interactions are dominant. For the SRO films of 5, 6 and 10 u.c. in thickness, the p values are all approximately 3, indicating the same inelastic scattering mechanism. Specific heat measurement and Raman spectroscopy study on SRO thin films indicate that the phonon density of states is widely dispersed and the interaction between phonon and electron could be strong at low temperatures.^{26,37} It can be inferred from the above results that the thickness-dependent metal-insulator transition in SRO ultrathin films could be attributed to weak localization due to electron-phonon interactions.³⁴ Herranz *et al*²⁵ has reported the low temperature conductivity of SRO thin film from 4 to 320 nm in thickness. However, it shows a $T^{1/2}$ dependence, indicating the electron-electron collision as the major inelastic scattering mechanism. The strong electron-electron interaction might be ascribed to the defects induced by initial three dimensional like growth mode in thicker films.

As mentioned above, the temperature constant T_0 is related to the inelastic scattering mean free path l_{in} which denotes the strength of disorder. In principle, with higher degree of disorder, one might expect to have an increased value of T_0 . T_0 values obtained from the fitting are 1.8, 2.0, 2.4 and 2.6 K for SRO films of 10, 8, 6 and 5 u.c. in thickness, respectively. This is a direct evidence of the shortening of inelastic mean free path due to the strengthening of disorder with decreasing film thickness.

The thinnest sample, 4 u.c. in thickness, shows an insulating behavior in all the temperatures studied. We calculated the disorder parameter $k_F l$, the product of Fermi wave vector (k_F) and elastic mean free path (l),³⁸ and obtained a value of 2.55 at room temperature. The disorder parameter values of 5 and 6 u.c. films are 3.62 and 7.06, respectively. This suggests that the system disorder increases and approaches to the Ioffe-Regel limit ($k_F l \sim 1$) causing stronger localization as the thickness decreases. As reported by Scherwitzl *et al.* in LaNiO_3 ultrathin films,¹⁶ the simple thermal activation relation $\ln(\sigma) \sim 1/T$ fails to fit the temperature dependence of the conductivity in the localized insulating state. However, the transport characteristics follow the Mott variable range hopping (VRH) model³⁹ indicating that the carriers are strongly localized. The hopping conductivity is given by⁴⁰:

$$\sigma = C \exp[-(T_0/T)^{1/(d+1)}], \quad (4)$$

where T_0 is a constant associated with the density of localized states at the Fermi level, T the temperature and d the physical dimensionality of the system. Fig. 4 (c) plots the logarithm of the sheet conductance as a function of $1/T^{d/3}$. Excellent agreement with a linear dependence can be observed. With $d = 2$, the transport in this 4 u.c. SRO film is consistent with the 2D VRH mode, which implies possible correlation between the thickness-driven metal-insulator transition and a dimensional crossover from the three-dimensional (3D) metallic state to the 2D insulating state.¹⁶ In this sample, weak localization is greatly enhanced due to the increased microstructural disorder leading to a strongly localized state.

To further study the transport properties and the metal-insulator transition of ultrathin SRO films, magnetoresistance $\text{MR} = 100\% \times (R_H - R_0)/R_0$, where R_H and R_0 are resistance values measured with and without magnetic field, respectively, was measured at 5 and 50 K by applying the current within the substrate plane. It is reported that the external magnetic field will break the time-reversal symmetry and results in an increase of conductivity by destructing the weak localization.⁴¹ The negative MR is indeed observed, as shown in Fig. 5. Generally, the MR value decreases with the increase of temperature and film thickness, consistent with the characteristics of weak localization systems.⁴² This can be ascribed to the increased density of localized electrons in thinner SRO films, which can be delocalized by the applied magnetic field. The negative MR is also evidence that the dominant scattering mechanism in SRO ultrathin films at low temperatures is electron-phonon interactions. A positive MR will arise from

the spin splitting of conduction electron energies based on theoretical calculations by Altshuler and Lee.^{43,44} Butterfly-shaped MR hysteresis loops can be observed in Fig. 5 (b) and (d), when the applied magnetic field is perpendicular to the sample surface. However, the butterfly-shaped MR loops disappear and the MR values decrease slightly with the magnetic field applied in the substrate plane, indicating the magnetic anisotropy in ultrathin SRO films with the out-of-plane direction as the magnetic easy axis.⁴⁵ This is because spin alignment and thus the onset of stronger MR occur only when the applied magnetic field is strong enough to overcome the anisotropy.

In addition, for the 2D weak localization model, the magnetoconductance can be written as⁴⁴

$$\sigma(H, T) - \sigma(H=0, T) = \frac{e^2}{\pi h} [\psi(\frac{1}{2} + \frac{1}{x}) + \ln(x)], \quad (5)$$

where ψ is the digamma function and $x = l_{in}^2 \delta e H / h$. The only fitting parameter is the inelastic scattering length l_{in} , which shows a $T^{-3/2}$ dependence if the electron-phonon collisions dominate the scattering. Fig. 6 (a) shows the magnetoconductance $[\sigma(H) - \sigma(H=0)]$ in units of $e^2/\pi h$ as a function of magnetic field at low temperatures, where the data can be well fitted using equation (5). Then the l_{in} values extracted from the fitting for the films of 5, 6 and 8 u.c. in thickness, is plotted in Fig. 6 (b) as a function of temperature. A $T^{-3/2}$ dependence is indeed observed, indicative of predominant electron-phonon collisions. This is consistent with the analysis on the resistance versus temperature data.

IV. Conclusion

In summary, high quality epitaxial SRO ultrathin films have been deposited on STO single-crystal substrates by pulsed laser deposition. As the film thickness decreases, the electrical transport properties of SRO ultrathin films undergo an evolution from a metallic to a localized insulating state. With decreasing temperature, a metal-insulator transition is observed in films of 5 to 10 u.c. in thickness. This transition can be ascribed to weak localization originating probably from microstructural disorders near the interface, such as cell distortion and reconstruction. The weak localization is further identified to be dominated by electron-phonon collisions. Negative MR increasing with the reduction of film thickness also suggests the 2D weak localization originating from electron-phonon collisions. These results suggest that thickness is an important parameter to manipulate the transport and magnetic properties of

correlated ultrathin films.

V. Acknowledgement

This work was jointly sponsored by National Key Basic Research Program of China (2015CB921203), Natural Science Foundation of China (51222206 and 11374139), Jiangsu Provincial Natural Science Foundation (BK2012016), and the PAPD project of Jiangsu Province. Electron microscopy research was performed at the Center for Functional Nanomaterials, Brookhaven National Laboratory, which is supported by the U.S. Department of Energy, Office of Basic Energy Sciences, under contract no. DEAC02-98CH10886. X.S. thanks China Scholarship Council and Brookhaven National Laboratory for financial supports. The authors also acknowledge the beam time and the technical assistance offered by Shanghai Synchrotron Radiation Facility, China.

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Figure captions:

Fig. 1 (a) RHEED intensity oscillations recorded during deposition of SRO films in various thicknesses. Surface morphology over an area of $3 \times 3 \mu\text{m}^2$ of the 10 u.c. thick samples is shown in (b). (c) Root mean squared (RMS) roughness as a function of film thickness for SRO films ranging from 2 to 20 u.c..

Fig. 2 (a) ω -2 θ XRD pattern recorded around the (002) peak of the 10 u.c. thick sample. (b) Cross-sectional HAADF-STEM image of the 8 u.c. thick SRO film. Ionic positions at the interface are indicated by dots in different colors. (c) Integrated intensity profile of lattice spots near the SRO/STO interface.

Fig. 3 (a) Temperature-dependent sheet resistance of SRO films, 4 to 20 u.c. in thickness. (b) Derivative of the sheet resistance-temperature curves for 6, 8, 10, 15 and 20 u.c. thick samples.

Fig. 4 (a) Resistivity as a function of temperature for the 15 u.c. thick sample. The dash dot line is the extrapolation of the linear resistivity behavior at high temperature. The inset shows the T^2 dependence of resistivity for temperature below 120 K. (b) Logarithmic temperature-dependent sheet conductance of the 8 u.c. thick film below 45 K. The linear fit with a slope of $3.4 \times 10^{-5} \text{ S} \approx 3e^2/\pi h$ indicates $p = 3$. The grey dotted line indicates the case for $p = 1$. The inset shows the conductance of the same sample in a linear scale. (c) Logarithm of the sheet conductance as a function of $1/T^{1/3}$ for the 4 u.c. thick sample.

Fig. 5 MR as a function of magnetic field H for the 5, 6, 8 and 10 u.c. thick samples measured at 5 K (a, b) and 50 K (c, d), with the magnetic field applied in the plane of the substrate (a, c) and perpendicular to the substrate (b, d).

Fig. 6 (a) Magnetoconductance as a function of magnetic field for the 8 u.c. thick sample, at various temperatures; (b) temperature dependence of the inelastic scattering length, extracted from the fitting of magnetoconductance for samples of 5, 6 and 8 u.c. in thickness.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6