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Thickness-tuned superconductor–insulator transitions in quench-condensed Mo and MoRu films

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Abstract

The results of studies on superconductor–insulator transitions are reported for Mo and MoRu films of varying thickness deposited on SiO underlayers and on bare glass. The film samples were quench-condensed on glass substrates held at liquid He temperatures. MoRu films on SiO, which offer the most homogeneous film morphology, showed a critical sheet resistance of transition, R_c , of ~ 6.7 k Ω . Both series of Mo films on SiO and MoRu films on bare glass had $R_c \simeq 13$ k Ω . These values are larger than those previously reported for MoGe, MoSi, and MoC films. For the above films in the insulating region far from the transition, the exponent of the thermally activated resistance ranged from 0.5 to 1.0.

1. Introduction

Despite two decades of continuing interest in the transition from superconducting to insulating behavior of the transport properties of quasi-two-dimensional (2D) systems at low temperatures, a full understanding is still elusive. A variety of systems, such as high- T_c cuprate superconductors with large anisotropy, 2D tunnel junction arrays, and thin metal films, have been used for studies of the superconductor–insulator (SI) transition [1–10]. The previous work has shown that when film thickness, composition of composites, disorder, carrier concentration, or magnetic field is gradually varied for a series of samples or a single sample, the temperature dependence of the sheet resistance R , i.e., the resistance per square, transforms from insulating to superconducting behavior; that is, the system changes from one with a negative temperature coefficient of resistance to one with a positive coefficient in the lowest temperature region. A model of quantum phase transition in the 2D limit predicts the critical sheet resistance separating superconductors and insulators at zero temperature to be a universal value, independent of the microscopic details of the sample. This value would not differ much from the quantum resistance for electron pairs, $R_q = h/4e^2 = 6.45$ k Ω , which the dirty boson model predicts for the very special case of self-duality [11, 12]. In this model, the phase of the order parameter associated with superconductivity plays an

important role, and Cooper pairs remain even on the insulating side of the SI transition. The phase-only model with nonzero amplitude would be appropriate for granular films of ‘high quality’, which are composed of superconducting particles of uniform size having a well-defined energy gap and the same distance between neighboring particles. Experiments with homogeneous films have been reported which indicate that Cooper pairs remain on the insulating side [13]. On the other hand, electron tunneling experiments with amorphous Bi films near the SI transition indicate that the superconducting energy gap is zero or extremely small at the SI transition point [14]: this suggests that fluctuations in the amplitude of the order parameter play an important role and the localization effects of unpaired single electrons characterizing the transport properties on the insulating side. It has been reported that the mean-field superconducting transition temperature T_c of homogeneous films decreases greatly as the normal-state sheet resistance, R_n , increases with decreasing film thickness. This T_c suppression has been ascribed to the enhanced electron–electron interaction due to the diffusive character of the electron motion in 2D disordered systems [15, 16]. The R_c estimated from the depression of superconductivity based on this theory is presumed to have non-universal values dependent on the material.

The electrical properties of ultrathin films near the SI transition seem to depend greatly on the morphology of the

film. The temperature dependence of the sheet resistance, $R(T)$, of nominally homogeneous films shows a monotonic decrease with increasing temperature on the insulating side, and a monotonic increase on the superconducting side, unlike the quasi-reentrant resistive transitions of granular films. The homogeneous film, which contains neither Josephson junctions nor quasi-particle tunneling junctions, would be a simple and interesting system for the study of the SI transition. Experiments in SI transitions using a wide variety of ‘homogeneous’ films have measured values of critical sheet resistance, R_c , ranging from ~ 2 to ~ 20 k Ω [4–9]. This may suggest the intrinsic absence of a universal aspect of R_c of the SI transition. Nevertheless, it should be noted that the $R(T)$ seems to be highly sensitive to the preparation conditions of the sample, even when prepared as an amorphous film with a microscopically homogeneous structure. Quench condensation onto a substrate cooled to liquid He temperatures produces amorphous films of metals such as Bi and Ga [17]. These films were considered to be microscopically homogeneous. However, quench-condensed (q-c) Bi films formed without an underlayer on a glass substrate, for example, exhibit $R(T)$ curves near the SI transition that are significantly different from those produced on an insulating SiO, Ge, or Sb underlayer [18]. Material, thickness, and preparation process of the insulating underlayer also change the $R(T)$ near the SI transition. Moreover, the $R(T)$ curves of ultrathin q-c Bi films appear to depend upon differences in the experimental conditions between research groups, even though the different studies used similar film preparation processes [7, 14, 19, 20].

Homogeneous molybdenum composite films for experiments in the SI transition have been prepared on substrates held at room temperature or higher by evaporating or sputtering Mo together with a non-metallic and nonsuperconducting material such as Ge, C, or Si, and the transport properties have been measured *ex situ* at low temperatures [4, 5, 21, 22]. Studies of the field-tuned SI transition in MoGe films have shown the remarkable result that the scaling behavior corresponds to the prediction of the dirty boson model with R_c near the R_n of the film, but at the lowest temperatures a metallic phase appears in the intermediate field region [21, 22]. As for the disorder-tuned SI transition in zero magnetic field, however, MoGe, MoC, and MoSi films have considerably lower R_c 's (2–3.5 k Ω) than R_q , unlike a variety of q-c metal films [4, 5, 8]. Quench-condensed Mo has been considered to be amorphous [23–25]. Adding a second metal, Ru, to Mo would stabilize the amorphous structure of q-c films, as shown previously for other materials [17]. In this work, we report the results of SI transitions in q-c Mo and MoRu films, which are composed of only superconducting elements.

2. Experimental details

We prepared, using an evaporator-cryostat composite, a series of disordered Mo or MoRu films on an insulating SiO underlayer or bare glass and measured *in situ* the evolution of $R(T)$ at low temperatures. First, a glass substrate as thin as ~ 0.15 mm was attached to a Cu plate with liquid indium to ensure good thermal contact. Care was taken to produce

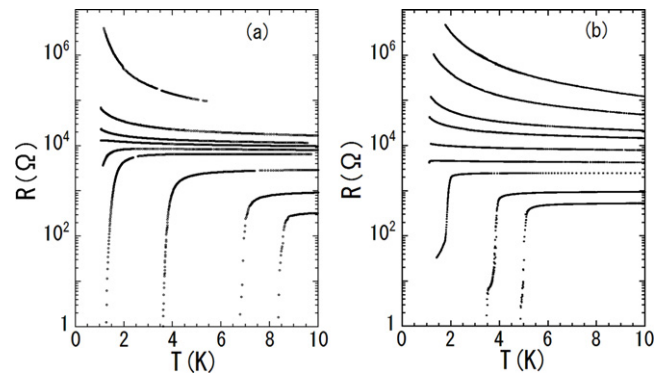


Figure 1. (a) Temperature dependence of the sheet resistance of Mo films on a SiO underlayer. The film thickness was 6.2 (top), 7.0, 7.6, 8.0, 8.5, 9.1, 12.3, 24.7, and 62.4 (bottom) Å. (b) Temperature dependence of the sheet resistance of MoRu films on a SiO underlayer. The film thickness was 16.9 (top), 18.0, 19.4, 20.6, 23.1, 27.4, 32.5, 49.1, and 69.4 (bottom) Å.

a thin, void-free indium layer in the proximity of a sample film which would be subsequently quench-condensed. Four lead wires were affixed in advance on the glass substrate with indium solder, and a thin Cu shadow mask was placed just in front of the substrate; the mask had a slit with shape similar to the Greek letter π , of 1.00 mm in width and 4.7 mm in length between voltage terminals for electrical measurements using a four-probe dc method. Then, the Cu plate was mounted on a Cu sample holder, which was shielded from thermal radiation by two sets of Cu plates with a shutter thermally anchored to liquid ^4He and liquid N_2 baths. After the chamber was evacuated and the sample holder was cooled using liquid ^3He , a SiO layer about 30 Å thick was quench-condensed, as an underlayer if necessary, onto the glass substrate by thermal evaporation from a Ta boat. This was followed by the evaporation with an electron beam gun of Mo (99.95%) at a rate of 0.02 to 0.7 Å s^{-1} or $\text{Mo}_{75}\text{Ru}_{25}$ at a rate of 0.06 to 0.6 Å s^{-1} . The pressure near the evaporation sources was approximately 10^{-8} to 10^{-7} Torr during deposition. The evolution of $R(T)$ for a series of Mo or MoRu films with various thicknesses was obtained by repeating the sequence of additional deposition of Mo or MoRu and *in situ* electrical measurement. During the experiment the temperature of the sample holder was kept below 20 K to prevent the amorphous films from crystallizing. The thicknesses of q-c films were evaluated with a quartz-crystal monitor, which was placed outside radiation shields at liquid N_2 temperature.

3. Results and discussion

Figures 1(a) and (b) show the $R(T)$ of Mo and MoRu films, respectively, with various thicknesses on SiO underlayers. The figures indicate that both series of films are nominally homogeneous because there is no indication of the quasi-reentrant behavior characteristic of granular ultrathin superconducting films, and the T_c increases greatly with increasing film thickness. We also prepared a series of Mo films on a 15 Å-thick underlayer of Ge, which had been used to acquire the homogeneous q-c films of metals such as Bi and Pb. The

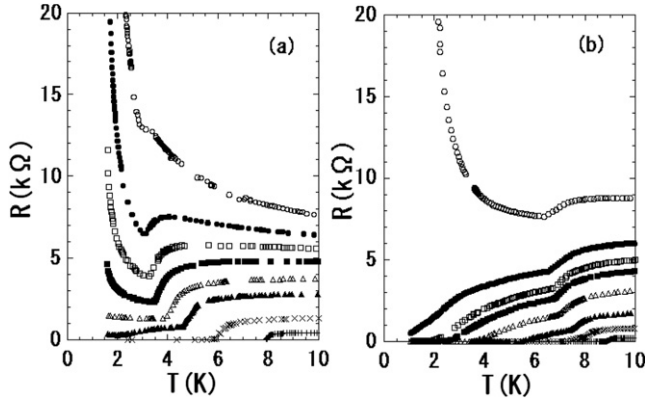


Figure 2. (a) Sheet resistance versus temperature of Mo films (10.2–55.6 Å) on a 15 Å-thick Ge underlayer. (b) Sheet resistance versus temperature of Mo films (18.3–88.3 Å) on bare glass.

Mo films on Ge showed a considerable decrease in T_c with increasing R_n as observed for ‘homogeneous’ superconducting films. However, the samples exhibited quasi-reentrant resistive transitions characteristic of granular films, as shown in figure 2(a). This may be ascribed to the inhomogeneity due to intermixing between Mo and Ge, which would occur even at liquid He temperatures. Moreover, quasi-reentrant resistive transitions peculiar to ultrathin granular superconducting films composed of weak links and single-electron tunnel junctions were observed also for the Mo films quench-condensed onto a glass substrate without an underlayer, as shown in figure 2(b). The T_c of Mo films on bare glass was less depressed with increasing R_n than that of Mo films on Ge. We believe that amorphous Mo forms in our q-c Mo films. Firstly, the superconducting transition temperature at the thick limit, T_{c0} , which was estimated by extrapolation to infinite film thickness, was 9.8 K for q-c Mo films; this value is substantially higher than 0.92 K of bulk crystalline Mo [23–25]. Secondly, as the temperature was raised at the end of the sequence of *in situ*

measurements, the R of the 62.4 Å-thick Mo film exhibited a slight decrease ($dR/dT < 0$), suggesting localization and interaction or annealing effects, and a sharp decrease to about one third around 180 K. This sharp decrease in resistance can be interpreted as the crystallization of the amorphous Mo film.

The $R(T)$ of Mo films on SiO with various thicknesses near the SI transition are magnified in figure 3. The film 8.49 Å thick exhibited signs of a resistive transition to the superconducting state at lowest temperatures. The film 8.44 Å thick, however, showed no inclination toward a positive temperature coefficient of resistance. The inset of figure 3 shows the dependence of R on thickness at temperatures 1.1, 1.2, 1.3, and 1.4 K for the films near a critical point. The crossing point identifies the critical sheet resistance R_c and the critical film thickness d_c . Thus, the R_c and d_c of Mo films on SiO were evaluated to be 12.8 kΩ and 8.5 Å, respectively, at temperatures down to 1.1 K. We had very nearly reproducible results for three series of Mo films on SiO prepared under almost the same conditions. Two series of MoRu films on SiO had almost the same values of $R_c = 6.7$ and 6.8 kΩ. However, they showed $d_c \simeq 25.4$ Å and 38.8 Å, and $T_{c0} = 6.5$ K and 6.0 K, respectively. The significantly different values of d_c and T_{c0} were likely due to the difference in composition of Mo and Ru between two series of q-c MoRu films, because an ingot of $\text{Mo}_{0.75}\text{Ru}_{0.25}$ was used as the evaporation source for the electron beam gun, and the vapor pressures of Mo and Ru have different temperature dependence around the evaporation temperature, ~ 2500 °C.

Strongly insulating films exhibit a thermally activated hopping law:

$$R(T) = R_0 \exp[(T_0/T)^x], \quad (1)$$

where R_0 , T_0 , and x are constants which depend on film disorder, the details of the interactions, and the dimensionality of the system. Here, in the case of $x = 1$, equation (1) results in the Arrhenius form, whereas in the case of $x = 1/2$, it

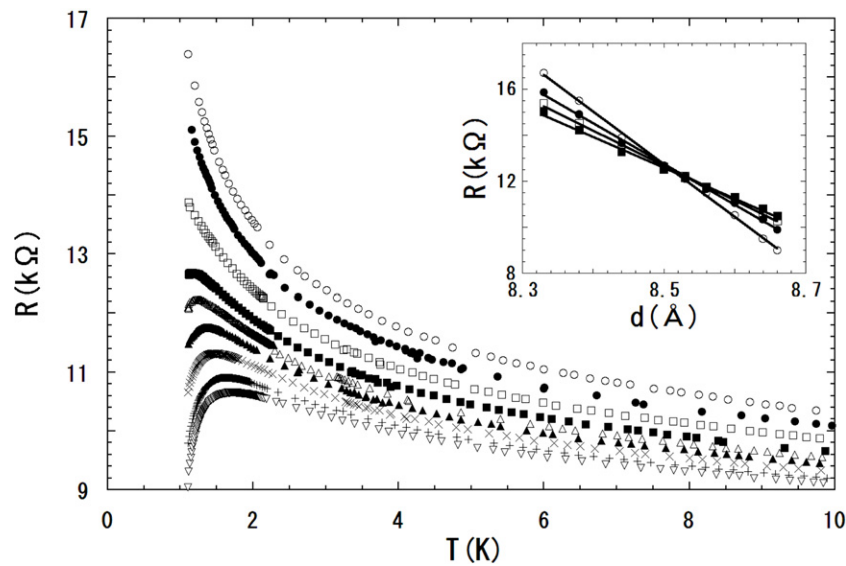


Figure 3. Sheet resistance versus temperature of Mo films (8.3–8.7 Å) on SiO near the critical sheet resistance. Inset: sheet resistance versus film thickness at 1.1 (○), 1.2, 1.3, and 1.4 (■) K.

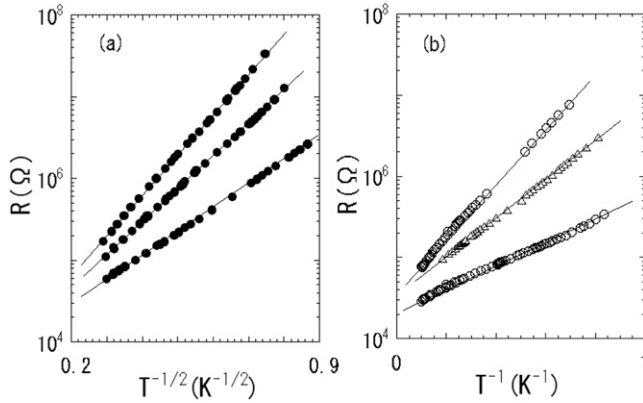


Figure 4. (a) Semi-logarithmic plot of R versus $T^{-1/2}$ for MoRu films (15.5–18.0 Å) on SiO. (b) Semi-logarithmic plot of R versus T^{-1} for Mo films (5.9–6.5 Å) on SiO.

results in the form of the variable range hopping resistance in the presence of Coulomb interactions between electrons [26]. There are a number of reports on the value of x of q-c films in the literature. For homogeneous q-c Bi films, it has been shown that x has a value of unity for the thinnest films and drops to 1/2 with increasing thickness, and the temperature dependence of conductance for the films near the SI transition is close to logarithmic [27]. Later studies by Marković *et al.* indicated that for Ag, Bi, Pb, and Pd films, $x = 0.75$ in the insulating deep region [28]. For all series of ‘homogeneous’ MoRu and Mo films in the present work, the exponent x increased with increasing $R_{10\text{ K}}$ up to $\sim 30\text{ k}\Omega$. In the insulating region far from the SI transition, the exponent x was observed to be about unity for both Mo films on SiO and MoRu films on bare glass, and about 0.5 for MoRu films on SiO. For MoRu films on SiO with R at 10 K, $R_{10\text{ K}}$, of 230, 130, and 65 kΩ, the logarithm of R is plotted as a function of T^{-x} with $x = 0.5$ in figure 4(a), and for Mo films on SiO, it is plotted as a function of T^{-x} with $x = 1$ in figure 4(b): both figures show straight lines. Here, the value of the exponent x was determined for $\log R$ versus T^{-x} to show the best fit to a straight line with x being varied as a fitting parameter in steps of 0.05. Near the SI transition on the insulating side, the sheet conductance, i.e., $1/R$, of both the Mo and MoRu films on SiO showed a slight deviation from the logarithmic temperature dependence; this dependence has been reported for normal-metal films in the weakly localized regime.

In the superconducting region, the fluctuation-enhanced conductance of Mo and MoRu films above T_c has been given by the sum of the Aslamazov–Larkin (AL) term and the Maki–Thompson (MT) term, unlike q-c Bi films [18]. It would be a good guess that the MT term makes a considerable contribution to the fluctuation-enhanced conductance of Mo and MoRu films, because the intrinsic pair breaking parameter proportional to $(T/\Theta_D)^2$ is not large [29]: the values of the Debye temperature Θ_D of crystalline bulk Mo and Ru are as high as 450 K and 600 K, respectively, while the value of Bi is as low as 119 K. Whether this difference in $R(T)$ between Bi and MoRu films on the superconducting side influences the characteristics of the SI transition remains unclear in our work.

Table 1. Sample parameters for several series of MoRe and Mo films in this work. T_{c0} is the bulk superconducting transition temperature, R_c is the critical sheet resistance, and x is the exponent of the thermally activated hopping. A value of $x \sim 2.5$ for Mo films on bare glass was determined in the lowest temperature region, which showed the quasi-reentrant resistive transitions.

	T_{c0}	R_c (kΩ)	x
MoRu on SiO	6.0–6.5	6.7–6.8	0.5
MoRu on glass	6.4	13	1.0
Mo on SiO	9.8	12.2–12.8	1.0
Mo on glass	9.8	Reentrant	(~ 2.5)

Table 1 summarizes the values of T_{c0} , R_c , and x determined in this study for Mo and MoRu films deposited with and without a SiO underlayer. The bulk superconducting transition temperature, T_{c0} , was determined by extrapolation to $R_{10\text{ K}} = 0$ from a plot of T_c versus $R_{10\text{ K}}$ in the low $R_{10\text{ K}}$ region. For the exponent x , the values in the insulating deep region are listed. Note that the R_c of MoRu films on SiO was roughly equal to R_q , but that of Mo films on SiO was about twice as large as R_q . There is a report that MoRu films prepared by quench condensation onto glass without an underlayer showed $T_{c0} = 10.5\text{ K}$, $R_c = 10\text{--}20\text{ k}\Omega$, and $x = 1$ [25]. The latter measurements of R_c and x correspond to those of our MoRu films without an underlayer. The value of T_{c0} is, however, different. Our MoRu films might have contained more or less Ru than the sample films used in the previous study, or may have had very small amounts of magnetic impurities which depressed superconductivity. The morphology of the q-c films in this work was probably changed somewhat by using an insulating underlayer or by adding another transition metal (Ru) to Mo. This presumably led to a variety of values of R_c and x as shown in table 1. As mentioned above, Mo and MoRu films on SiO show the $R(T)$ curves characteristic of ‘homogeneous’ films, while the Mo films formed on a glass substrate without an underlayer seem to be granular because they show the quasi-reentrant behavior, and the T_c is less depressed than that shown in figure 1(a) with increasing R_n . Furthermore, the dependence of the resistivity on thickness also indicates that Mo films on SiO are fairly homogeneous, as follows. The resistance of the films on SiO became measurable at an average thickness of about 6 Å. However, the resistance of Mo films quench-condensed directly onto glass could not be measured up to 18 Å. Moreover, the resistivity of Mo films deposited on SiO drastically dropped with increasing thickness up to 12 Å and remained constant above $\sim 20\text{ Å}$. On the other hand, the $R(T)$ of MoRu films on bare glass showed no signs of granular morphology, despite the absence of the SiO underlayer. We believe therefore that the MoRu films on a SiO underlayer are the least granular of the various sets of films examined in this work: the downward convexity in the low temperature region of resistive transitions in figure 1(b) may show the existence of slight inhomogeneity due to different values of T_c of some materials, which alloying of Mo and Ru causes, though resistive transitions only in zero magnetic field give less information on homogeneity of sample films. The value of R_c for MoRu films on a SiO underlayer is

roughly equal to R_q . If lower temperatures are available for the electrical measurement, the R_c will become somewhat larger because one or several insulating curves nearest to the SI transition probably change to superconducting curve at lower temperatures. According to our experiments under similar conditions, the R_c of amorphous Bi films on a SiO underlayer was 5.2 k Ω , which is a little smaller than R_q [20]. The most salient feature of our results in the present paper is that q-c homogeneous Mo composite films have values of R_c of about R_q : they are larger than the values previously reported for MoGe, MoC, and MoSi films whose experimental results have been considered to be one of the grounds for the absence of a universal aspect of R_c of the SI transition in microscopically homogeneous systems in zero magnetic field. The addition to Mo of large amounts of nonsuperconducting material such as Ge, C, or Si in order to stabilize the amorphous state may harm superconductivity in the ultrathin Mo composite films with large R near the SI transition. This may reduce the experimental values of R_c of MoGe, MoC, and MoSi films.

Figure 5 shows the dependence of normalized T_c on $R_{10\text{K}}$ for Mo and MoRu films. For nominally homogeneous films deposited on underlayers and sufficiently thick films on bare glass having monotonic sharp resistive transitions, the T_c was defined as the temperature at which R is one-half that of $R_{10\text{K}}$. For Mo films with high $R_{10\text{K}}$ on bare glass, it was defined as the upper temperature of a plateau reached during the broad resistive transition to the superconducting state. In figure 5, the T_c of MoRu films deposited on a SiO underlayer is seen to decrease more sharply than others with increasing $R_{10\text{K}}$. The lowering of T_c from the bulk value T_{c0} caused by enhanced Coulomb interactions in two-dimensional systems was theoretically given by Finkel'stein as follows:

$$\frac{T_c}{T_{c0}} = \exp\left(-\frac{1}{\gamma}\right) \cdot \left[\frac{\left(1 + \frac{(r/2)^{1/2}}{\gamma - r/4}\right)}{\left(1 - \frac{(r/2)^{1/2}}{\gamma - r/4}\right)} \right]^{1/\sqrt{2r}}, \quad (2)$$

where $r = R_n e^2 / 2\pi^2 \hbar$ and $\gamma = 1 / \ln(k_B T_{c0} \tau / 2\pi \hbar)$ [15]. Three solid curves are given in figure 5 which represent the theoretical results with γ being -0.15 , -0.17 , and -0.20 . Data on Mo films deposited without an underlayer, which would be of granular morphology, deviate greatly from Finkel'stein's formula. Data obtained from MoRu films deposited on a SiO underlayer, which are considered to be the most homogeneous, show a comparatively good agreement with the theoretical curve. In the high $R_{10\text{K}}$ region, however, the data deviate from theoretical prediction. This deviation may be mainly due to the films being outside the region in which the above theoretical result would apply. Although a similar deviation has also been seen in the plots for highly homogeneous q-c Bi films on underlayers, it is hard to verify that this deviation has no relation to inhomogeneity [20]. Even though there is some granularity in the q-c film deposited on SiO, the mechanism of the transition to global superconductivity may be different from that in the usual granular films as follows. Here, it is plausible that at an early stage of the quench condensation of metals such

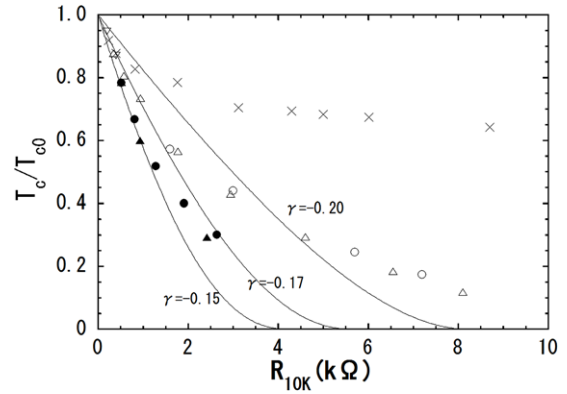


Figure 5. Normalized T_c versus $R_{10\text{K}}$ of Mo films on SiO (open symbols), MoRu films on SiO (closed symbols), and Mo films on bare glass (cross symbols). The circles and the triangles represent different series of films. Solid lines show Finkel'stein's formula.

as Mo and MoRu onto a SiO underlayer, the ultrathin film formed is a 2D random distribution of fine clusters. When a single cluster of diameter less than ~ 2 nm is placed at an isolated position where electrons cannot tunnel through either vacuum or insulating materials to the neighboring clusters, it would not show superconductivity even at zero temperature as a result of the large energy level spacing in the cluster. Further condensation of the material decreases the distance between clusters to strengthen the correlation between them. Consequently, even if a single cluster is still a non-superconductor owing to a sufficiently small diameter, Cooper pairs could be formed in the 2D system, and the phase coherence would be retained over a long distance across clusters. For this type of film, the formation of Cooper pairs may be not independent of retaining the long-range phase coherence, unlike the usual case for ultrathin granular films composed of relatively large islands such as q-c Pb and Sn films on bare glass [30]. It seems probable that the high temperatures ($\sim 2500^\circ\text{C}$) used by the Mo and Ru evaporation source caused some granularity even in the q-c MoRu films deposited on SiO. This may have resulted in a larger value of R_c than our previous result, ~ 5.2 k Ω , found for amorphous Bi films deposited on SiO or Ge, considering that a set of granular films has a tendency to show a larger R_c for the SI transition with increasing R . For q-c 'homogeneous' films near the SI transition, *in situ* measurements of the morphology with an atomic force microscope or a scanning tunneling microscope, together with the corresponding $R(T)$ curves, will give us valuable information, though they will be accompanied by serious difficulties [31].

It is important to note that 'inhomogeneous' films seem to have a larger value of x . Our MoRu films on SiO in the insulating region far from the SI transition indicated that $x \simeq 0.5$. MoRu films without an underlayer as well as Mo films on SiO, however, exhibited $x \simeq 1.0$; this value for MoRu films on bare glass was also reported in [24]. Mo films on bare glass exhibiting the quasi-reentrant resistive transition showed a value of x as large as ~ 2.5 in the region below the temperature at which the $R(T)$ has a minimum. These results indicate that the film morphology may affect the hopping

exponent x of strong insulating films. It was shown that for a series of q-c Be films on bare glass, the $R(T)$ of the insulating films cannot be fitted to a simple hopping law of equation (1) with $x = 1/4, 1/3, 1/2$, or 1 [32]. We consider that this is probably due to the granularity in the Be films because the $R(T)$ of the films with $R_{10\text{ K}} \sim 10\text{ k}\Omega$ shows the quasi-reentrant behavior.

4. Summary

In summary, we have measured *in situ* the evolution of the temperature dependence of the sheet resistance $R(T)$ with increasing thickness of Mo and MoRu films on SiO underlayers and bare glass which were quench-condensed onto cryogenic glass substrates. MoRu films on SiO, which present the most homogeneous morphology in these films, show the transition from superconducting to insulating behavior at the critical sheet resistance $R_c \simeq 6.7\text{ k}\Omega$ in the temperature region down to 1.1 K. Both series of Mo films on SiO and MoRu films on bare glass have $R_c \simeq 13\text{ k}\Omega$. These values are markedly larger than 2–3 k Ω which was previously reported for amorphous Mo composite films such as MoGe, MoSi, and MoC films. For the above Mo and MoRu films in the insulating region far from the SI transition, the exponent of the thermally activated resistance ranges from 0.5 to 1. This suggests that the exponent depends on the morphology, which would be sensitive to the preparation conditions of q-c films.

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