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Electrical properties of K-doped superfulleride thin films

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K-doped superfulleride films (K_xC_{60}, $x > 6$) of an average homogeneous composition of $K_{11 \pm 0.3}C_{60}$ and a strong [111] texture were prepared by depositing C_{60} on K multilayers at 200 K, in a proportion such that $K:C_{60} > 12:1$, and then annealing these layers to 300 K. This $K_{11}C_{60}$ superfulleride film was doped with C_{60} to form another superfulleride phase of average composition K_8C_{60} and then doped further with C_{60} to form homogeneous K_6C_{60} and K_3C_{60} fulleride phases. The electrical properties of these superfulleride films are compared to those of alkali metal fulleride and alkaline earth metal fulleride films. The doping-resistivity profile showed a resistivity minimum, $\rho_{min1} = 4.0 \times 10^{-3}$ Ω cm due to the half filled t_{1u} band in K_3C_{60} and another dip in resistivity to 9.5×10^{-3} Ω cm at higher K-doping levels of $K_{11}C_{60}$, presumably due to the unfilled t_{1g} band, as in the alkaline earth metal fullerides. The resistivity of the K_3C_{60} and $K_{11}C_{60}$ films showed only minimal variations with film thickness, probably because the films prepared in this study had large grain sizes, and hence, resistivity was invariant with film thickness. The temperature dependence of the resistivity for 300 Å films of K_3C_{60}, K_8C_{60}, and $K_{11}C_{60}$ was also studied in the 200–350 K temperature range. $K_{11}C_{60}$ films showed a metallic behavior like K_3C_{60} films, while conduction in K_8C_{60} films exhibited an activated behavior that could be described by a granular metal model for resistivity in the dielectric region. © *1999 American Institute of Physics.*
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I. INTRODUCTION

The electrical properties of thin films of C_{60},[1] K-doped fullerides (K_xC_{60}, $0 < x < 6$),[2–6] and C_{60}-polymer composites[7] have been of great interest, and applications as transistors,[8] rectifying diodes,[9] and C_{60}-polymer heterojunctions[7] have been proposed. The synthesis of K-doped superfulleride phases (K_xC_{60}, $x > 6$)[10–13] presents a new class of materials with potentially interesting electrical properties.[14] Thin films (from a few monolayers to 300 Å thick films) have been grown on Au(110),[10,13] Rh(111)[11,12] and inert substrates[14] by low temperature deposition of C_{60} on K multilayers. The occupation of the t_{1g} state for the superfulleride films was confirmed by ultraviolet (UV) photoemission spectroscopy[10–13] and from bleaching of the $h_u \rightarrow t_{1g}$ transition in UV–visible absorption spectra.[14] The objective of the current study is to present measurements of electrical properties of thin films of K-doped superfullerides and compare them to those of K-doped fullerides.

II. EXPERIMENTAL METHODS

The experimental apparatus and methods used in the preparation and characterization of the superfulleride thin films have been described in a previous publication.[14] A high vacuum chamber with $P = 5 \times 10^{-7}$ Torr was used for the growth of the fulleride and superfulleride films. *In situ* characterization of these films was performed by electrical resistivity measurements and UV–visible absorption spectra, while *ex situ* x-ray diffraction (XRD) was used to characterize the crystal structure and grain size of the films. The substrate for all the depositions was single crystal quartz with four 1000 Å silver pads utilized in four point resistivity measurements. Deposition rates were determined by a quartz crystal microbalance and film thicknesses were calibrated by ellipsometry. Potassium from a SAES getter was deposited on the substrate maintained at 200 K or lower, followed by C_{60} (MER Corp., 99.5% pure) deposition, in a proportion such that $K:C_{60} > 12:1$ on an atom basis. The high conductivity of metallic potassium ($\rho \ll 10^{-3}$ Ω cm) was the major component of electrical measurements after C_{60} deposition at $T < 200$ K, but on annealing to 300 K for about 2 h the resistivity of the film gradually increased to about $10^{-2} – 10^{-3}$ Ω cm, suggesting the interdiffusion of K and C_{60} to form fulleride and superfulleride compounds along with desorption of excess potassium from the surface. This picture is based on photoemission studies of C_{60} deposition on K multilayer at 100 K, which suggest that the three-dimensional K film spreads evenly over the substrate on C_{60} deposition[13] and interdiffuses with C_{60} to form superfulleride phases.[11–13] At low temperatures, excess potassium could form a continuous film on the substrate and cause a low resistivity, but annealing to 300 K should desorb metallic potassium, and hence, the resistivity values reported here are assumed to be solely due to the fulleride or superfulleride phases.

III. RESULTS AND DISCUSSION

Figure 1 shows resistivity measurements obtained upon deposition of C_{60} on a 170 Å K film at 200 K, followed by annealing to 300 K. We note that the fulleride phases are formed in the opposite sequence from those obtained by de-

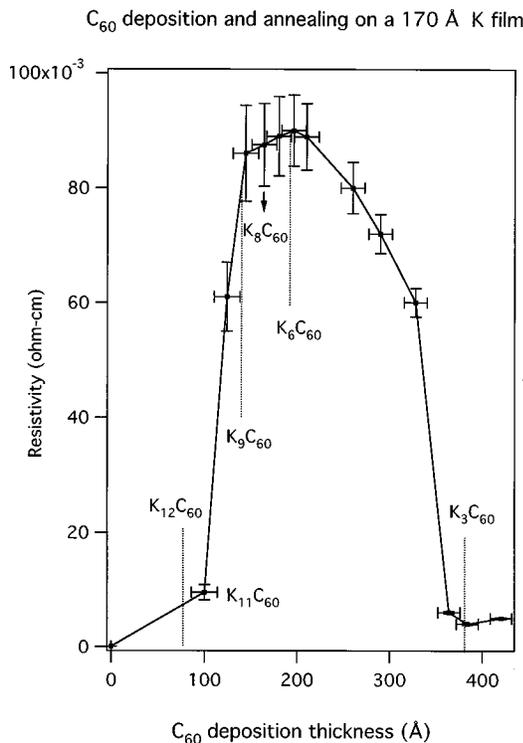[a)]Electronic mail: koel@chem.1.usc.edu

FIG. 1. Doping-resistivity profile for K-doped superfulleride and fulleride phases. The $K_{11}C_{60}$ film was formed by C_{60} deposition on K-multilayers (approximately 170 Å) at 200 K followed by annealing to 300 K until the resistance stabilized. All other points were generated by C_{60} deposition on the $K_{11}C_{60}$ film at 200 K followed by annealing to 300 K for points between $K_{11}C_{60}$ and K_6C_{60} and annealing to 500 K for points after the K_6C_{60} phase.

positing K on polycrystalline C_{60} films, as has been performed by earlier researchers.[2–6] The horizontal distance in Fig. 1 between the resistivity minimum at 4×10^{-3} Ω cm corresponding to 380 Å K_3C_{60} and the resistivity maximum at 95×10^{-3} Ω cm corresponding to 190 Å K_6C_{60} was used to assign an average stoichiometry to the K_xC_{60} phases as indicated by the dotted lines at K_9C_{60} and $K_{12}C_{60}$. In this assignment, the change in lattice parameter on formation of the fulleride phases is assumed to be minimal in comparison to the C_{60} lattice parameter.[15] The error bars are estimated from measurements using independent techniques for resistivity and C_{60} film thickness (ellipsometry), and hence, represent maximum possible errors. A film of 100 Å C_{60} deposited on a 170 Å K film at 200 K yielded a superfulleride phase with an average composition of $K_{11\pm0.3}C_{60}$ (henceforth referred to as $K_{11}C_{60}$) after annealing to 300 K. This superfulleride phase was subsequently doped by C_{60} deposition at 200 K, followed by annealing to 500 K (over a two hour period) to successively form fulleride phases K_6C_{60} and K_3C_{60}.

The resistivity of K_3C_{60} films prepared by the above method with a thickness of 380 Å was 4×10^{-3} Ω cm and this compares well with that observed for [111]-oriented (also called textured) K_3C_{60} films.[2–4] This suggests that the method followed in the current study for the growth of superfulleride and fulleride phases probably results in films of homogeneous composition. In addition, UV–visible absorption spectra[14] of the phases assigned to K_3C_{60} and K_6C_{60} in

Fig. 1 match those for films composed entirely of K_3C_{60} and K_6C_{60}, respectively, where the composition was determined by Rutherford backscattering (RBS).[16] Furthermore, it has been suggested that low temperature film doping produces ''metastable'' homogeneous compositions based on resistivity measurements of fullerides.[15] Also based on the narrow widths of the features in photoemission spectra and $C(1s)$ x-ray absorption spectra (XAS),[13] it was concluded that a uniform charge state was reached for the superfulleride films prepared by low temperature deposition of C_{60} on K multilayers (as done in the current study). All these previous interpretations support the assignment of the superfulleride film in Fig. 1 to a single homogeneous phase that may be continuously doped with C_{60} to form relatively homogeneous phases of lower superfullerides and fullerides.

In Fig. 1, there is one resistivity minimum $\rho_{min1} = 4.0 \times 10^{-3}$ Ω cm assigned to a 380 Å film of K_3C_{60} and another dip in resistivity to 9.5×10^{-3} Ω cm at higher K-doping levels than K_6C_{60}, which is assigned to a 100 Å film of $K_{11}C_{60}$. Only one resistivity maximum is observed at $\rho_{max} = 95.0 \times 10^{-3}$ Ω cm corresponding to a 190 Å film of K_6C_{60}. The presence of two dips in resistivity, one due to the half filled t_{1u} band in K_3C_{60} and the other presumably due to the unfilled t_{1g} band in $K_{11}C_{60}$, with a resistivity maximum in between due to the filled t_{1u} band in K_6C_{60} is quite similar to the resistivity profile for alkaline earth doped fullerides.[17,18] While the resistivity values for K_3C_{60} films are much lower than those for alkaline earth fullerides with a half filled t_{1u} band, the resistivity due to the unfilled t_{1g} band in $K_{11}C_{60}$ is comparable to that for the alkaline earth fullerides Sr_5C_{60} ($\rho_{min2} = 1.1 \times 10^{-2}$ Ω cm, Ca_5C_{60} ($\rho_{min2} = 6.0 \times 10^{-3}$ Ω cm), and Ba_5C_{60} ($\rho_{min2} = 3.0 \times 10^{-3}$ Ω cm). If the bonding in these phases were purely ionic, then at ρ_{min2} for the alkaline fulleride phases, 10 electrons are transferred to each C_{60} molecule. For the superfulleride phase $K_{11}C_{60}$ prepared in this study, 11 electrons are transferred to each C_{60} molecule, if the bonding is purely ionic. However, for alkaline earth fullerides hybridization between t_{1g} bands of C_{60} and $4s$ bands of the alkaline earth dopants has been suggested.[19] In view of the similarities discussed above, it is likely that hybridization also should occur in superfullerides and $K_{11}C_{60}$ would have less than 11 electrons transferred per C_{60} molecule, such that it exhibits a high electrical conductivity similar to K_3C_{60}. For the case of the alkaline earth fullerides, a second resistivity maximum ρ_{max2} was observed for Sr_xC_{60} and Ca_xC_{60} at $x = 6.6$ and 7.1, respectively, but not for Ba_xC_{60} (much like the case for K-doped superfullerides studied here). From the photoemission and $C(1s)$ XAS spectra of superfulleride films,[13] it was determined that even at maximum charge transfer, the t_{1g} band was not completely filled. Possible reasons for incomplete filling of the t_{1g} band have been discussed in the literature.[13,20] Upon C_{60} doping of $K_{11}C_{60}$, we find that resistivity rises, but plateaus out much before the formation of the characteristic cusp for K_6C_{60}. Because the UV–visible absorption spectra of K_xC_{60} ($x \sim 8$), where the plateauing begins, shows features corresponding to filling of the t_{1g} band, which differs from K_6C_{60} (discussed later), we label this composition as a different phase.
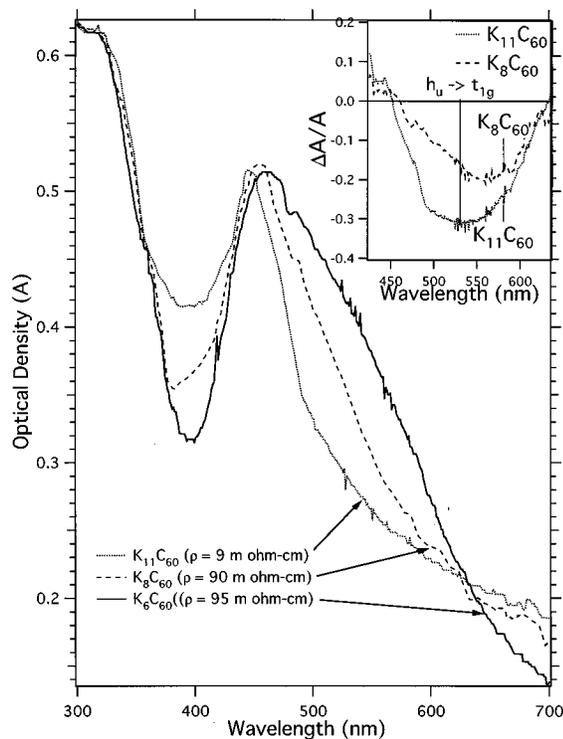
FIG. 2. Comparison of the UV–visible absorption spectra of 300 Å $K_{11}C_{60}$, K_8C_{60} and K_6C_{60} films. The inset shows the difference spectrum obtained from the difference of the optical densities of the respective superfulleride films from that of K_6C_{60} normalized to the optical density of K_6C_{60}. The bleach of the $h_u \rightarrow t_{1g}$ transition is seen at 530 nm (2.35 eV) in the difference spectrum.

Figure 2 shows a comparison of the UV–visible absorption spectra for 300 Å films of K_6C_{60}, K_8C_{60}, and $K_{11}C_{60}$. The peak at 460 nm for both the superfulleride phases is narrowed and blue-shifted in comparison to K_6C_{60}. This probably occurs because the peak arising from $h_u \rightarrow t_{1g}$ transitions is bleached for superfullerides upon filling of the t_{1g} band, as has been argued for the bleaching of the peak arising from $g_g + h_g \rightarrow t_{1u}$ transitions for fullerides on filling of the t_{1u} band.[16] The bleaching of $h_u \rightarrow t_{1g}$ transitions is readily visible in the difference spectra of the superfullerides from K_6C_{60} films, as shown in the inset of Fig. 2. A negative peak is seen at about 530 nm (2.35 eV) that is consistent with the energy band assignment for this transition from photoemission spectra of superfullerides.[13] Prolonged annealing of 300 Å-thick K_8C_{60} and $K_{11}C_{60}$ films to $T > 325$ K causes conversion of these phases to K_6C_{60}, as determined from UV–visible absorption spectra and resistivity measurements. The K_8C_{60} phase could not be obtained by annealing $K_{11}C_{60}$ films because it is converted directly to K_6C_{60}. Hence, we suggest that the desorption of potassium multilayers drives the decomposition of the metastable superfulleride phases to the more stable K_6C_{60} phase as follows:

$$K_8C_{60(s)}/K_{11}C_{60(s)} \xrightarrow{325-350\ K} K_6C_{60(s)} + K_{(g)}. \qquad (1)$$

Figure 3 shows a comparison of the x-ray diffraction (XRD) results for polycrystalline C_{60}, [111]-textured C_{60}, and $K_{11}C_{60}$ films. The polycrystalline C_{60} film was grown by
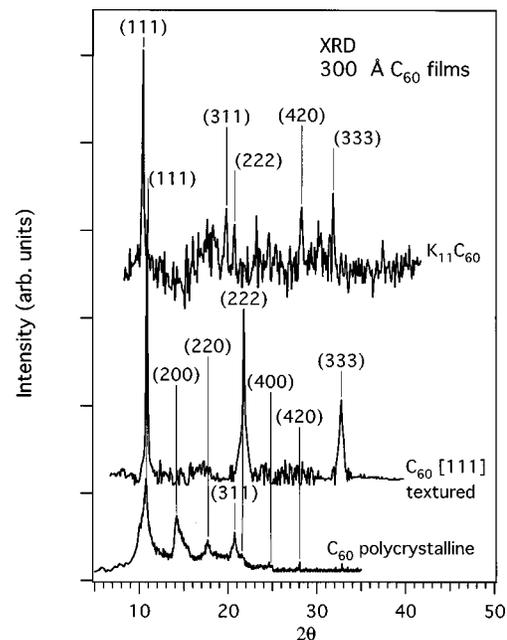

FIG. 3. XRD scan of 300 Å of randomly oriented polycrystalline C_{60}, [111]-textured C_{60}, and $K_{11}C_{60}$ films. The *ex situ* XRD of the $K_{11}C_{60}$ film was obtained after exposing the superfulleride film to the ambient atmosphere.

depositing C_{60} on a quartz substrate at room temperature. Figure 3 shows face-centered-cubic (fcc) polycrystalline peaks of C_{60} with a lattice parameter of 14.2 Å and a grain size of about 80 Å. The growth of textured C_{60} films may be accomplished by many methods.[5,21,22] A typical XRD pattern of [111]-textured C_{60} films grown on a H-terminated Si(111) substrate using the method described by Hebard *et al.*[21] is also shown in Fig. 3. The grain size for this case is close to 1000 Å and the absence of reflections from the (200), (220), (311), (400), and (420) planes shows the textured [111] orientation of the film. The topmost spectrum in Fig. 3 shows the XRD pattern of a 300 Å-$K_{11}C_{60}$ film, that was exposed to atmospheric pressure for *ex situ* XRD measurements following the growth and *in situ* characterization described above. K-doped C_{60} compounds are known to be extremely air sensitive[23–25] and easily form potassium oxides[23] or carbonates[25] upon exposure to water vapor or oxygen by the diffusion of potassium out of the C_{60} lattice. The topmost spectrum of Fig. 3 has sharp peaks at positions expected for reflections from C_{60} fcc planes. However, a noisy background probably arising from potassium oxides is also seen. If the $K_{11}C_{60}$ lattice in the bulk of the film is intact following diffusion of potassium out of the lattice near the surface to form an oxide layer, then the XRD spectrum in Fig. 3 is relevant to the structure of the $K_{11}C_{60}$ films. The XRD of the $K_{11}C_{60}$ film shows a fcc structure with a lattice parameter of about 14.8 Å, slightly larger than that of pristine C_{60}. The grain size of these films is about 425 Å, which is larger than the grain size of 80 Å seen for polycrystalline C_{60} films and comparable to the grain size of 1000 Å seen for textured C_{60} films. The $K_{11}C_{60}$ films in Fig. 3 are characterized by weak intensities for reflections from (200), (220), (311), and (400) planes, compared to that from the (111) plane, and so show a high [111]-texture. Thus, the K_xC_{60} films grown by the
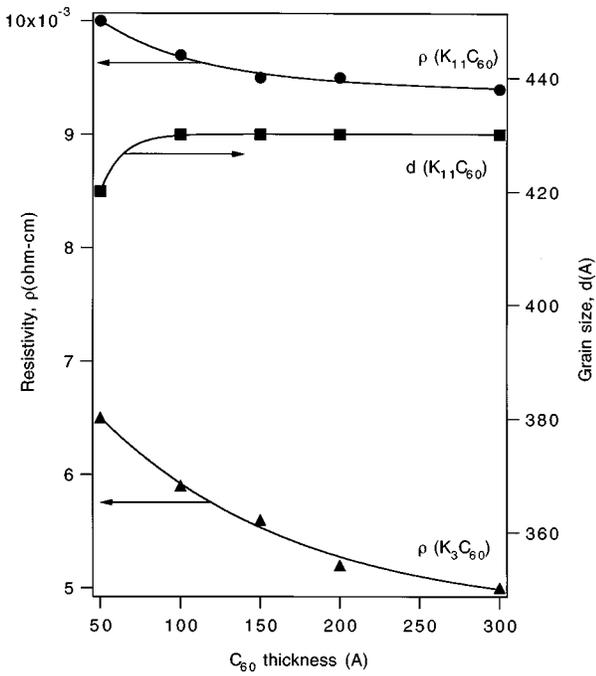
FIG. 4. Variation of resistivity for K_3C_{60} and $K_{11}C_{60}$ films with film thickness and the variation of grain size for $K_{11}C_{60}$ films with film thickness.

FIG. 5. Temperature dependence of resistivity (ρ) for 300 Å K_3C_{60}, K_8C_{60}, and $K_{11}C_{60}$ films plotted as $\log(\rho)$ vs ($1000/T$) showing the metallic behavior of K_3C_{60} and $K_{11}C_{60}$ films and the activated behavior of K_8C_{60} films.

method described in this study result in textured films with relatively large grain size. Previously, Hebard et al.[5] obtained [111]-textured K_3C_{60} and K_6C_{60} films with large grain size by annealing K and C_{60} films deposited at 300 K to temperatures just below the decomposition temperature of the respective phases. The method used in the current study is similar, since K and C_{60} films are both deposited at low temperatures (200 K) and then annealed to 300 K, which is just below the decomposition temperature of the superfullerides. Growth of textured films with large grain size is important in view of potential applications, particularly since wide variations in the electrical properties of fulleride films of the same composition[3] were attributed to variations in texture, grain size, and thickness that arise from the different preparation procedures followed.[26]
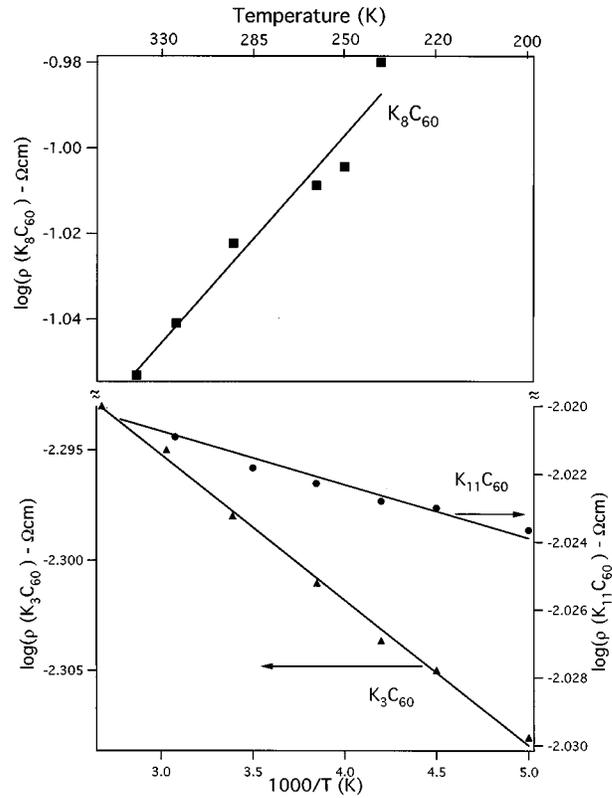
The polycrystalline K_3C_{60} films of Kochanski et al.[2] prepared by depositing K on C_{60} films at room temperature show a large drop in resistivity for an increase in film thickness. These variations were attributed to impurity scattering[6] or grain size effects.[2] Because the thickness of the films utilized in Fig. 1 vary from 100 to 380 Å, it is necessary to demonstrate that the variations in resistivity were due to phase changes and not due to changes in film thickness. Figure 4 shows measurements of the resistivity of K_3C_{60} and $K_{11}C_{60}$ films and grain size of the $K_{11}C_{60}$ films for 50–300 Å
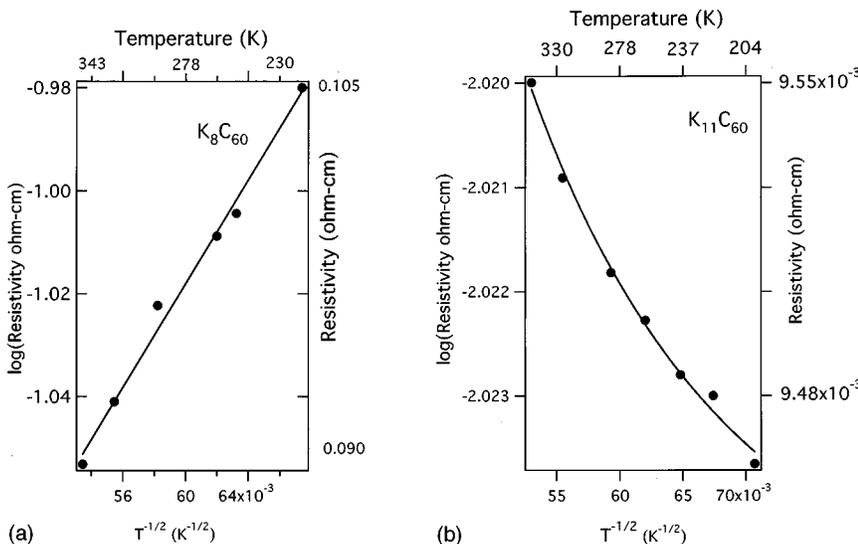

FIG. 6. Granular metal model of resistivity applied to 300 Å of the superfulleride films: $\log(\rho)$ vs $T^{-1/2}$ plotted for (a) K_8C_{60} and (b) $K_{11}C_{60}$ films.

film thickness. The variations in resistivity and grain size with thickness for both the K_3C_{60} and $K_{11}C_{60}$ films is minimal. Hence, thickness variations do not adversely effect the resistivity measurements reported here. It is likely that the large grain size and strong [111] texture of the fulleride and superfulleride films prepared as described herein reduces the variation of grain size and resistivity with film thickness, in contrast to polycrystalline films of smaller grain size and poor texture.[2]

Figure 5 shows resistivity variations with temperature between 200–350 K for 300 Å films of K_3C_{60}, K_8C_{60}, and $K_{11}C_{60}$. K_3C_{60} films show metallic behavior, as was seen for [111]-textured films[2,3] and differ from randomly oriented granular K_3C_{60} films that exhibit an activated dependence of resistivity on temperature. This again demonstrates the high quality of films grown in this study. $K_{11}C_{60}$ films showed a positive temperature coefficient of resistivity (i.e., negative slope in Fig. 5) much like K_3C_{60} films. This indicates that the $K_{11}C_{60}$ phase produced was also metallic. K_8C_{60} films showed a negative temperature coefficient of resistivity (i.e., positive slope in Fig. 5) like alkali metal fullerides, A_xC_{60}, $x<3$.[6] This insulating behavior is consistent with photoemission spectra that show no emission at E_F for a superfulleride phase with an approximate composition of K_8C_{60}.[10,13] The temperature dependence of resistivity for the superfulleride films differ considerably from that of alkaline earth fullerides. In those cases, conduction is activated at all compositions.

Figure 6 shows the results of the granular metal model for resistivity[27] applied to 300 Å films of K_8C_{60} and $K_{11}C_{60}$, following that done by Stepnaik et al.[6] for $Rb_{1.4}C_{60}$ and $Rb_{2.8}C_{60}$ films. The logarithm of resistivity is proportional to $T^{1/2}$ for a film in the dielectric region.[28] This was seen for K_8C_{60} films, but not for the metallic $K_{11}C_{60}$ films. Thus, the K_8C_{60} films consist of phase-separated grains of metallic and nonmetallic constituents with electrical conduction occurring by tunneling between the neutral grains. This leads to charging and contributes to the negative temperature coefficient of resistivity for these films. $K_{11}C_{60}$ films, on the other hand, consist predominantly of grains of metallic constituents. Upon heating the metastable $K_{11}C_{60}$ and K_8C_{60} films to temperatures above 350 K, grains of the nonmetallic constituents grow at the cost of the metallic constituents to form an insulating film composed of the more stable K_6C_{60} phase.

IV. CONCLUSIONS

K-doped superfulleride films of an average homogeneous composition of $K_{11\pm0.3}C_{60}$ and a strong [111] texture have been prepared by deposition of C_{60} on K multilayers at 200 K and subsequent annealing to 300 K. This $K_{11}C_{60}$ superfulleride film can then be doped with C_{60} to form another superfulleride phase of composition K_8C_{60}. Subsequent C_{60} doping leads to homogeneous fulleride phases K_6C_{60} and K_3C_{60}. The doping-resistivity profile showed two dips in resistivity, one that arises from the half filled t_{1u} band in K_3C_{60} with a $\rho_{min1}=4.0\times10^{-3}\,\Omega\,cm$, and another to $\rho=9.5\times10^{-3}\,\Omega\,cm$ presumably due to the unfilled t_{1g} band in $K_{11}C_{60}$. A resistivity maximum occurred between these at

$95\times10^{-3}\,\Omega\,cm$ due to the filled t_{1g} band in K_6C_{60}. The resistivity of the K_3C_{60} and $K_{11}C_{60}$ films showed only minimal variations with film thickness, probably because the films prepared in this study had a large grain size. The temperature dependence of the resistivity for 300 Å films of K_3C_{60}, K_8C_{60} and $K_{11}C_{60}$ in the 200–350 K temperature range showed that $K_{11}C_{60}$ films were metallic, like K_3C_{60} films, while conduction in K_8C_{60} films was activated. This latter behavior could be described by a granular metal model for resistivity in the dielectric region.

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