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## Electrical resistivity and stoichiometry of $K_xC_{60}$ , $Rb_xC_{60}$ , and $Cs_xC_{60}$ films

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### Abstract

Electrical resistance measurements as a function of stoichiometry have been carried out on  $K_xC_{60}$ ,  $Rb_xC_{60}$  and  $Cs_xC_{60}$  thin films for  $0 < x < 6$ . The annealed films show global resistance minima at  $K_3C_{60}$ ,  $\rho_{\min} = 4 \times 10^{-3} \Omega \text{ cm}$  ( $60^\circ\text{C}$ ),  $Rb_3C_{60}$ ,  $\rho_{\min} = 4 \times 10^{-3} \Omega \text{ cm}$ , and  $Cs_1C_{60}$ ,  $\rho_{\min} = 7 \times 10^{-2} \Omega \text{ cm}$ . All of the annealed films show additional features in the vicinity of  $x=4$ , but the manifestation of the  $A_4C_{60}$  phase ( $A=K, Rb, Cs$ ), in transport studies is dependent on the metal and the annealing conditions. The  $A_6C_{60}$  phase is apparent for all of the metals studied and shows a relatively high resistivity. The activation energies of the conduction process show well defined stationary points at  $K_3C_{60}$  (not activated),  $Rb_3C_{60}$  (not activated),  $Cs_1C_{60}$  (minimum),  $Cs_4C_{60}$  (maximum), with less distinct features between  $4 < x < 6$  in all cases.

### 1. Introduction

$C_{60}$  undergoes doping with alkali metals and these compositions include insulators, conductors and superconductors [1–6]. In this Letter, we present resistivity measurements of  $K_xC_{60}$ ,  $Rb_xC_{60}$ , and  $Cs_xC_{60}$  films, as a function of temperature, stoichiometry and annealing conditions for  $0 < x < 6$ . There have been a number of reports of transport studies on these systems, including two studies of conductivity as a function of composition ( $x$ ) [7,8].

It is known that  $K_3C_{60}$  and  $Rb_3C_{60}$  are superconductors and the transport properties of these compositions have been studied in the form of granular thin films [9,10], crystalline thin films [11] and doped single crystals [12,13]. These phases are readily identified in transport studies as they give rise to resistivity minima and show a positive temperature coefficient of resistivity. The  $A_3C_{60}$  compositions ( $A=K, Rb$ ), are formed from the pristine solid by

occupancy of the interstitial sites of the fcc lattice of crystalline  $C_{60}$  and lead to a half-filling of the  $t_{1u}$  LUMO of  $C_{60}$  [6]. The other compositions do not have the characteristic signature of these phases and have received less attention although photoemission experiments can clearly identify  $A_6C_{60}$  in which the  $t_{1u}$  level is fully occupied and the material rendered insulating [14–16]. In the present work we focus on the doping profile of these films in the range  $0 < x < 6$ , in an effort to further the understanding of the doping process in granular thin films and in order to assess the reproducibility and degree of control which can be routinely achieved in vacuum doping experiments.

### 2. Experimental

We followed the procedure adopted in our previous studies of  $Ca_xC_{60}$ ,  $Sr_xC_{60}$ , and  $Ba_xC_{60}$  films in

UHV [17,18]. The UHV chamber used in this work is equipped with a Radak I  $C_{60}$  source so that the  $C_{60}$  films were grown in situ on substrates held at room temperature and were not exposed to oxygen prior to doping. The  $C_{60}$  films were grown from material produced in the spark erosion process [18] and purified by liquid chromatography [20]. Films produced in this way consist of random polycrystalline grains of dimension  $\approx 60 \text{ \AA}$  [21]. The alkali metals were deposited by thermal evaporation from an SAES getter source. The UHV chamber also contains a quartz crystal microbalance (QCM) so that we could directly monitor the amount of  $C_{60}$  and metal which were deposited. The system was baked prior to deposition and our experiments were conducted at pressures between  $10^{-8}$  and  $2 \times 10^{-9}$  Torr.

We used thin  $C_{60}$  films ( $\approx 200 \text{ \AA}$ ), deposited on sapphire, quartz or pyrex glass substrates. The electrical measurements were carried out using substrates with evaporated aluminum and silver multilayers for contact pads. The quartz crystal microbalance was calibrated for the determination of the  $C_{60}$  film thickness by RBS measurements on a  $C_{60}$  film grown on clean hydrogen-terminated Si. Ex situ RBS analysis of the alkali-metal peak in the doped  $C_{60}$  films when combined with the measurements obtained from the microbalance during the experiment allowed us to obtain the film composition. We experienced some difficulties with the RBS metal determinations on sapphire due to peak asymmetry. For this reason we also investigated other substrates, and most of our experiments were carried out on glass. Apart from the difficulties with the RBS determination, however, the sapphire, quartz and pyrex glass substrates gave identical doping profiles within the errors of our experiment (based on the QCM measurements taken for each point). We estimate the uncertainty in our derived values of  $x$  at  $\pm 0.5$ , mainly due to the error in the RBS measurements on the thin metal/ $C_{60}$  films.

In the Ca and Sr doping study a one-minute anneal at  $180^\circ\text{C}$  after each addition of metal to the film was necessary to equilibrate the system and obtain meaningful resistivities [16], whereas in the experiments on  $Ba_xC_{60}$  films we annealed for 1 min at temperatures of  $180^\circ\text{C}$ ,  $200^\circ\text{C}$ , and  $220^\circ\text{C}$  without fully equilibrating the film composition at doping levels beyond  $x=3$  [17]. In the present study we report two

sets of resistivity measurements; the first were determined on films held at room temperature ( $32^\circ\text{C}$  in our UHV chamber) throughout the experiment (Fig. 1), while the second set (Fig. 2), were obtained with films which were subjected to the  $180^\circ\text{C}$  one-minute anneals used in the previous work on alkaline earths (hereafter we refer to these as room temperature and annealed film experiments). After each addition of metal we measured the resistivity of the room temperature films at 5 min intervals in order to follow the equilibration of the metal throughout the  $C_{60}$  film. We considered the film to be equilibrated when successive readings agreed to within 10%. Based on other experiments it seems that in the case of granular thin films, annealing temperatures in excess of  $100^\circ\text{C}$  are adequate for the equilibration of  $K_xC_{60}$ ,  $Rb_xC_{60}$ , and  $Cs_xC_{60}$  films, although these conditions do not produce well-ordered films. The resistivity values reported herein were determined at either  $32^\circ\text{C}$  (Fig. 1), or  $60^\circ\text{C}$  (Fig. 2), and the activation energies were obtained from resistivity data measured in the range  $60^\circ\text{C} < T < 160^\circ\text{C}$  (Fig. 3). During the course of our studies we carried out many experiments and as far as possible we report representative results, but there remains a degree of variability in the doping profiles which we are unable to entirely eliminate. The more obvious variations are discussed below.

### 3. Results

#### 3.1. $K_xC_{60}$ films

The resistivity as a function of doping of a  $K_xC_{60}$  room temperature film is shown in fig. 1. The resistivity shows a well-defined minimum at  $x=3.1$  where  $\rho_{\min}=3.1 \times 10^{-3} \Omega \text{ cm}$  ( $32^\circ\text{C}$ ) and the last point on Fig. 1 is taken at  $x=6.0$  with  $\rho=88 \Omega \text{ cm}$ . The increase in resistivity beyond  $x=5.5$  is pronounced.

The resistivity of a  $K_xC_{60}$  annealed film as a function of doping is shown in Fig. 2. The resistivity shows a well-defined minimum at  $x=2.8$  where  $\rho_{\min}=4.6 \times 10^{-3} \Omega \text{ cm}$  ( $60^\circ\text{C}$ ) and the last point on Fig. 2 is taken at  $x=6.3$  with  $\rho=51 \Omega \text{ cm}$ . The shoulder in the resistivity curve occurs at  $x \approx 3.8$  where  $\rho=6 \times 10^{-2} \Omega \text{ cm}$  ( $60^\circ\text{C}$ ). The shape of the  $K_3C_{60}$  minimum was observed to vary from the sharp dip seen in Fig. 1a to the more rounded feature of Fig.

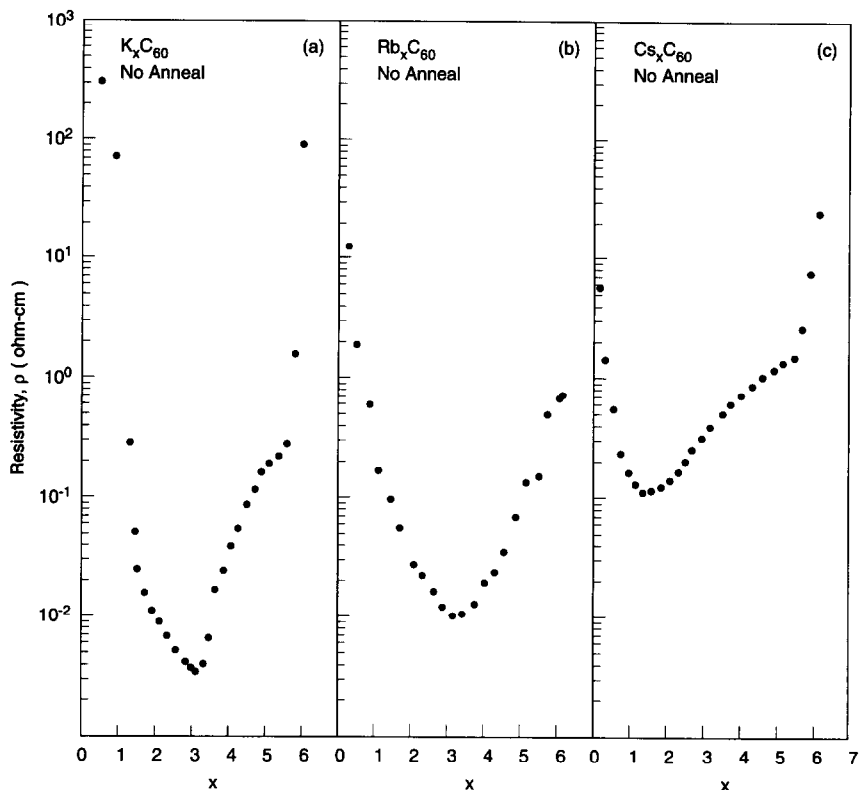


Fig. 1. Resistivity measured in UHV at 32°C as a function of  $x$ .

2a. The shoulder near  $x=4$  was less pronounced in other experiments.

The conductivity shows activated behavior (Fig. 3), except for the compositions  $x=2.5$  and 2.8, where the temperature coefficient of the resistivity is positive. Under the conditions of our experiment the  $K_xC_{60}$  annealed films showed metallic behavior somewhere in the range  $2.5 < x < 3.5$ , typically over a spread in  $x$  values of 0.5. There is a maximum at  $x=4$  and a minimum at  $x=5$  in the activation energies with values of 0.12 and 0.08 eV, respectively. In general these maxima and minima were observed at  $x$  values of 3.8–4.4 and 4.8–5.6, respectively.

### 3.2. $Rb_xC_{60}$ films

The resistivity as a function of doping of a  $Rb_xC_{60}$  room temperature film is shown in Fig. 1. The resistivity shows a broad minimum at  $x=3.1$  where  $\rho_{\min} = 1.0 \times 10^{-2} \Omega \text{ cm}$  (32°C) and the last point on Fig. 1 is taken at  $x=5.9$  with  $\rho=0.7 \Omega \text{ cm}$ .

The resistivity of a  $Rb_xC_{60}$  annealed film as a function of doping is shown in Fig. 2. The resistivity shows a minimum between  $x=2.8$  and  $x=3.0$  where  $\rho_{\min} = 3.8 \times 10^{-3} \Omega \text{ cm}$  (60°C) and the last point on Fig. 2 is taken at  $x=6.0$  with  $\rho=6 \Omega \text{ cm}$ . The shape of the minimum shown in Fig. 2b is typical, with a clear asymmetry on either side of  $x=3$ . The approach to the minimum is gradual for  $x < 3$ , but quite sharp for  $x > 3$ .  $K_xC_{60}$  annealed films exhibited slightly sharper minima than  $Rb_xC_{60}$  annealed films.

The conductivity shows activated behavior (Fig. 3), except for compositions between  $x=2.5$  and 3.4. In this range (Fig. 2b) there are seven points with a positive temperature coefficient of resistivity. Under the conditions of our experiment  $Rb_xC_{60}$  annealed films showed metallic behavior somewhere in the range  $2.5 < x < 3.5$ , over a spread in  $x$  values slightly greater than observed for  $K_xC_{60}$  annealed films. There is a maximum at  $x=4.9$  and a minimum at  $x=5.6$  in the activation energies with values of 0.12 and 0.09 eV, respectively. In general these maxima and min-

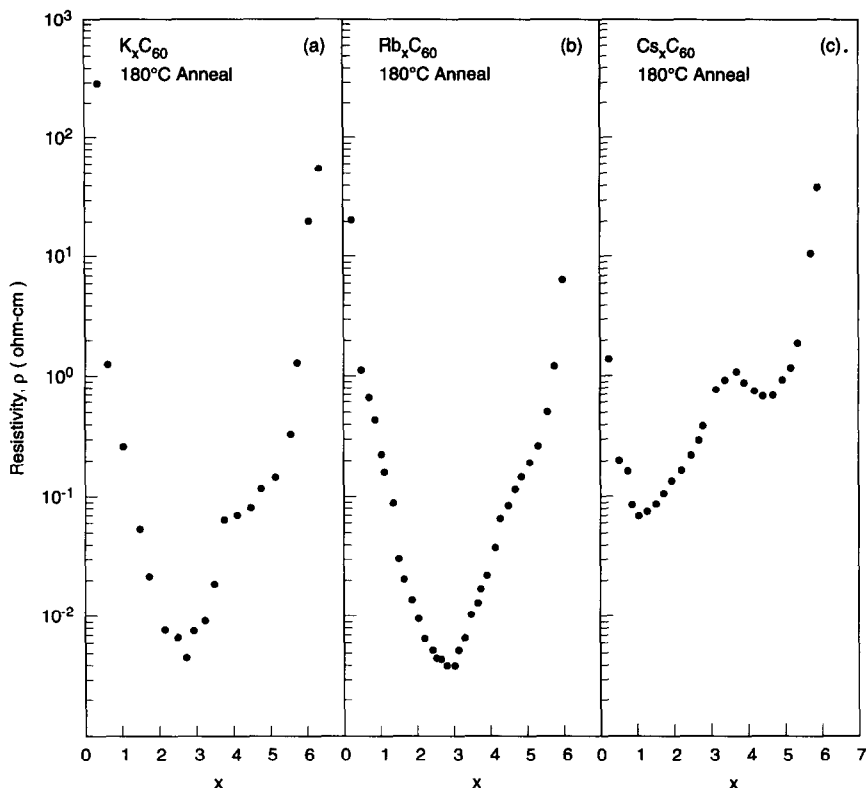


Fig. 2. Resistivity measured in UHV at 60°C as a function of  $x$  after 180°C anneals.

ima were observed at  $x$  values of 4.5–5.0 and 5.5–5.9, respectively. The minimum in the activation energy at  $x=1.0$  was not present in all experiments.

### 3.3. $Cs_xC_{60}$ films

The resistivity as a function of doping of a  $Cs_xC_{60}$  room temperature film is shown in Fig. 1. The resistivity shows a broad minimum at  $x \approx 1.4$  where  $\rho_{\min} = 1.1 \times 10^{-1} \Omega \text{ cm}$  (32°C) and the last point on Fig. 1 is taken at  $x=6.1$  with  $\rho=44 \Omega \text{ cm}$ . In general the position of the minimum occurred between  $1.0 < x < 1.6$ .

The resistivity of a  $Cs_xC_{60}$  annealed film as a function of doping is shown in Fig. 2. The first minimum in the resistivity occurs at  $x=1.0$  where  $\rho_{\min_1} = 7.0 \times 10^{-2} \Omega \text{ cm}$  (60°C). There is then a resistivity maximum at  $x=3.6$  where  $\rho_{\max} = 1.1 \Omega \text{ cm}$  followed by a second minimum at  $x=4.5$  where  $\rho_{\min_2} = 0.7 \Omega \text{ cm}$ . The last point on Fig. 2 is taken at  $x=5.8$  with  $\rho=68 \Omega \text{ cm}$ . The double minimum shown in Fig. 2 is typi-

cal, and in some room temperature runs a suggestion of the second feature is also apparent. In general the position of the first minimum occurred between  $1.0 < x < 1.4$ . Within the accuracy of our experiment the point of inflection between the maximum and minimum associated with the second feature occurs at  $x \approx 4$ .

The  $Cs_xC_{60}$  films never show metallic behavior (Fig. 3). The  $Cs_xC_{60}$  annealed film shows minima at  $x=1.0$  and  $x=5.7$  in the activation energies with values of 0.08 and 0.14 eV, respectively. There is a well-defined maximum in the activation energy of the conductivity at  $x=4.1$  with a value of 0.2 eV. In general the second feature gave rise to maxima and minima at  $x$  values of 4.0–4.4 and 5.5–5.9, respectively.

## 4. Discussion

Since the report of conductivity in alkali-metal-doped  $C_{60}$  and  $C_{70}$  films the chemistry and physics of

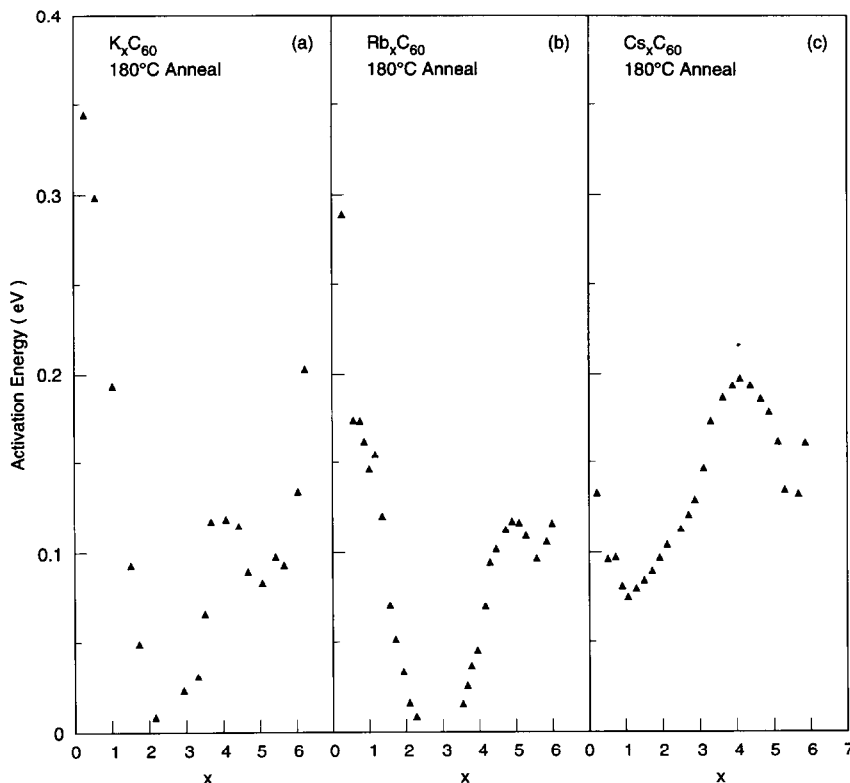


Fig. 3. Activation energy of the conductivity of the films shown in Fig. 2.

these materials has shown remarkable development. In the case of  $A=K$  and  $Rb$ ,  $A_1C_{60}$  [22],  $A_3C_{60}$  [23],  $A_4C_{60}$  [24], and  $A_6C_{60}$  [25] bulk phases have been definitely identified [26,27]. The first two phases are formed by population of the interstitial sites of the face centered cubic lattice of  $C_{60}$  [22,23,28], whereas the latter two are body centered tetragonal [24] and body centered cubic [25] structures, respectively. With the exception of  $Cs_3C_{60}$ , the same phases have been characterized for  $Cs$ . Similar compositions have been observed in doped films by stoichiometric resistivity measurements [7,8] photoemission studies [14–16] and Raman spectroscopy [29–32].

Our results for  $K$ - and  $Rb$ -doped  $C_{60}$  films are in good agreement with two previous studies of the electrical resistivity as a function of stoichiometry [7,8]. The minimum in resistivity for  $K$  and  $Rb$  doping was found to occur at  $x=3$ , where the films showed metallic behavior. For  $K_3C_{60}$ , the resistivities were  $\rho_{\min}=5.5 \times 10^{-3} \Omega \text{ cm}$  (46°C, 400 Å thick film) [7] and  $\rho_{\min}=3.1 \times 10^{-3} \Omega \text{ cm}$  (27°C, 500–4000 Å film)

[8], which may be compared with the value of  $\rho_{\min}=4.6 \times 10^{-3} \Omega \text{ cm}$  (60°C, 200 Å film) found in the present study. For  $Rb_3C_{60}$   $\rho_{\min}=2.8 \times 10^{-3} \Omega \text{ cm}$  (27°C, 500–4000 Å film) was reported [8], compared with the value of  $\rho_{\min}=3.8 \times 10^{-3} \Omega \text{ cm}$  (60°C, 200 Å film) found in the present study. Rather similar values have been reported for doped single crystal, granular and crystalline film  $K_3C_{60}$  and  $Rb_3C_{60}$  [9–13].

The resistivity of the  $K_xC_{60}$  film exhibits the least variation with annealing procedure, and it is apparent that potassium readily diffuses through the  $C_{60}$  film. The value of  $\rho_{\min}=3 \times 10^{-3}$  found in the room temperature experiment is in agreement with the result from the annealed film and with the previous determination of  $\rho_{\min}=2 \times 10^{-3} \Omega \text{ cm}$  [1]. The room temperature resistivities of  $\rho_{\min}=1 \times 10^{-2}$  found for  $Rb_3C_{60}$  and  $\rho_{\min}=1 \times 10^{-1} \Omega \text{ cm}$  found for  $Cs_1C_{60}$  are significantly higher than those determined for the annealed films, but are in reasonable agreement with the previous minimum resistivity measurements [1]

of  $1 \times 10^{-2}$  and  $2.5 \times 10^{-1} \Omega \text{ cm}$ , respectively.

The resistivity values observed for annealed films of  $\text{K}_3\text{C}_{60}$  and  $\text{Rb}_3\text{C}_{60}$  are comparable to those found for the second minimum in the resistivity of annealed alkaline-earth-doped  $\text{C}_{60}$  films:  $\rho_{\text{min}2} = 5 \times 10^{-3}$  ( $\text{Ca}_5\text{C}_{60}$ ),  $10 \times 10^{-3}$  ( $\text{Sr}_5\text{C}_{60}$ ) and  $3 \times 10^{-3} \Omega \text{ cm}$  ( $\text{Ba}_5\text{C}_{60}$ ) [17,18]. In this case the conductivity is associated with population of the  $t_{1g}$  band of  $\text{C}_{60}$  [17,18,33–36] and some of these compositions are superconductors [35,36].

In our experience the other features in the resistivity data exhibit some sample dependence. Although the rise in resistivity in the vicinity of  $x=6$  is seen by all workers, its magnitude is quite variable, perhaps because of residual and uncontrolled disorder in the films under the conditions of these experiments. In general we find for  $\text{A}_6\text{C}_{60}$  films ( $\text{A}=\text{K}, \text{Rb}, \text{Cs}$ ),  $\rho_{\text{max}} \approx 1 \Omega \text{ cm}$  for room temperature films and  $\rho_{\text{max}} \approx 100 \Omega \text{ cm}$  for annealed films. The resistivities of some annealed  $\text{A}_6\text{C}_{60}$  films have shown  $\rho_{\text{max}} > 1000 \Omega \text{ cm}$ . In bulk studies it has been shown that the bcc  $\text{A}_6\text{C}_{60}$  phase is not a line compound and therefore may form as a substoichiometric composition with alkali-metal vacancies [27]. The very rapid rise in resistivity for  $5.5 < x$  therefore can be interpreted as a band filling effect which occurs as the composition of this phase becomes stoichiometric.

We find the global resistivity minimum for Cs doping at  $\text{Cs}_1\text{C}_{60}$  (Fig. 2c, although some films indicate a minimum slightly beyond this point), whereas a previous study found the resistivity minimum at  $\text{Cs}_{3.5}\text{C}_{60}$  [8]. The shoulder in the resistivity data shown in Fig. 2b, provides an indication of the presence of  $\text{Rb}_1\text{C}_{60}$  [8].

The  $\text{A}_4\text{C}_{60}$  composition seems to be present in all of the doping experiments, but the signature of this phase is less than characteristic in our work. In our previous experiments on  $\text{Ca}_x\text{C}_{60}$ ,  $\text{Sr}_x\text{C}_{60}$  and  $\text{Ba}_x\text{C}_{60}$  films the activation energies of the conductivity provided a useful indicator of the underlying phases [16,17]. The same holds true here for  $\text{Rb}_1\text{C}_{60}$  (weak minimum),  $\text{Cs}_1\text{C}_{60}$  (minimum),  $\text{K}_3\text{C}_{60}$  (not activated),  $\text{Rb}_3\text{C}_{60}$  (not activated),  $\text{K}_4\text{C}_{60}$  (weak maximum), and  $\text{Cs}_4\text{C}_{60}$  (maximum). The maximum for the  $\text{Rb}_x\text{C}_{60}$  films is closer to  $x=5$ . However the second minimum in the activation energies between  $x=5.0$  and  $5.9$  which is seen for all metals is difficult to explain unless the  $\text{A}_4\text{C}_{60}$  phase is taken to

be insulating. The activation energies do not change very much for  $4 < x < 6$ , and the sharp changes in conductivity apparently reflect the population of vacancies in substoichiometric  $\text{A}_6\text{C}_{60}$ .

## 5. Conclusions

Our results show that it is possible to dope  $\text{C}_{60}$  films to sharp minima which are associated with  $\text{Cs}_1\text{C}_{60}$ ,  $\text{K}_3\text{C}_{60}$  and  $\text{Rb}_3\text{C}_{60}$ . Thus in addition to  $\text{K}_3\text{C}_{60}$  and  $\text{Rb}_3\text{C}_{60}$ , which have been extensively studied, it is possible to prepare  $\text{Cs}_1\text{C}_{60}$  by monitoring the resistivity as a function of doping. In contrast to potassium, however, cesium and rubidium doping require annealing of the  $\text{A}_x\text{C}_{60}$  films in order to obtain equilibrated compositions with optimum transport properties.

The activation energies of the conductivity of the  $\text{A}_x\text{C}_{60}$  films strongly support the insulating nature of the  $\text{A}_4\text{C}_{60}$  phase. In the case of  $\text{Cs}_4\text{C}_{60}$  this phase exhibits an activation energy of  $\approx 0.2 \text{ eV}$ , which is higher than that shown for any of the doped  $\text{C}_{60}$  films with incompletely filled bands, and is even higher than that seen for the  $(\text{Ae})_3\text{C}_{60}$  films (where  $\text{Ae}=\text{Ca}, \text{Sr}$  and  $\text{Ba}$ ), which have nominally filled  $t_{1u}$  bands [17,18,33,34]. The energy gap in the  $\text{A}_4\text{C}_{60}$  phase cannot be explained within a simple one-electron band picture [6,16,37] and the electronic structure of this composition remains unresolved.

## 6. Acknowledgement

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