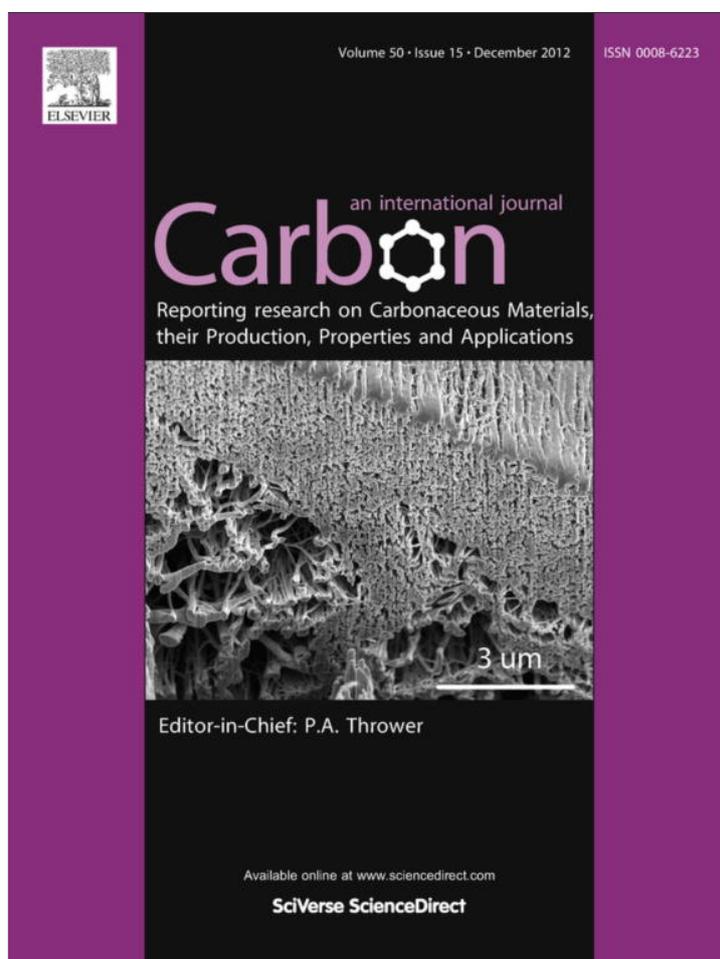


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## Phase transitions in single crystal tubes formed from C<sub>60</sub> molecules under high pressure

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### ABSTRACT

Pure single crystal tubes formed from C<sub>60</sub> molecules, with a face-centered cubic (fcc) structure were fabricated by a liquid–liquid interfacial precipitation method using C<sub>60</sub> powder. A bulk transition from fcc to a simple cubic structure and a surface transition from (1 × 1) to (2 × 2) have been observed around 246 K (bulk transition temperature T<sub>B</sub>) and 214 K (surface transition temperature T<sub>S</sub>), respectively, during the measurement of the temperature dependence of electrical resistance. The initiation of the two transitions under pressure was investigated using a piston cylinder high pressure apparatus and it was found that both T<sub>B</sub> and T<sub>S</sub> increase with increasing pressure. And the C<sub>60</sub> molecules at the surface of the tube exhibit the same behavior of that in the bulk at a pressure of about 2.1 GPa.

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## 1. Introduction

Due to its unique physical and chemical characteristics, C<sub>60</sub> has been extensively studied since its discovery [1]. At room temperature, the C<sub>60</sub> crystal has a face-centered cubic (fcc) structure (*Fm* $\bar{3}$ *m*) with  $a = 14.2 \text{ \AA}$  and undergoes a first order phase transition to a simple cubic (sc) structure (*Pa* $\bar{3}$ ) at around 250 K [2–5]. This transition is called bulk transition and it is characterized that the fcc lattice has an orientational disorder while the sc has an orientational order. The sc phase is realized by the forming of inequivalent orientations of the four molecules in the fcc cell without any lattice distortion or molecular displacements [2,3]. Using the electron energy-loss spectroscopy (EELS) and low-energy electron diffraction (LEED), Goldoni et al. [6] reported that this bulk phase transition is associated with a surface counterpart, a transition from (1 × 1) to (2 × 2) in symmetry of the (111) surface at around

225 K, about 30 K lower than the temperature of the bulk transition. The calculations by Laforge et al. [7] suggest that there is a two-stage rotational disordering in C<sub>60</sub> molecular crystal surface. One molecule in the (2 × 2) surface unit cell has significant different orientation from the other three molecules, making it more frustrated, and its thermal-activated rotational behavior differs from that of the others. So the surface thermal rotational disorder undergoes an intermediate regime that the more frustrated molecule becomes disordered much earlier than the other three molecules, which also lose their rotational order at 230 K. And Goldoni et al. [8] provided strong experimental evidences for this partial disordered intermediate regime by high resolution core level photoemission. In addition, when temperature decreases to about 90 K, a phase transition called as glass transition happens [9]. These phase transitions were confirmed by Kumar et al. [10] in C<sub>60</sub> thin films by temperature dependent electrical transport measurements.

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Recently, fullerene nanomaterials have attracted attention for their importance in both fundamental research and potential applications in nanoscale devices. Wang et al. reported the synthesis of  $C_{60}$  sheets and suggested that  $C_{60}$  crystals in even small size will be a potential candidate of superhard materials [11].  $C_{60}$  rods with nano- and submicro-meter dimensions were polymerized under high pressure and high temperature conditions and were indexed to a rhombohedral polymeric structure [12]. Liu et al. fabricated nanowires and nanotubes from  $C_{60}$  by the dipping porous alumina templates into a solution containing  $C_{60}$  [13], but the tubes were polycrystalline and had a mixture of face-centered cubic and hexagonal close-packed phases. Later, a modified liquid–liquid interfacial precipitation (LLIP) method was used to fabricate pure fullerene nanotubes (pure fullerene nanowhiskers with hollow structures) [14–17], which are single crystal and have an fcc structure after being fully dried at room temperature. These tubes, known as  $C_{60}$  nanotubes, with diameters smaller than 1000 nm and several millimeters in length, could be re-dissolved in appropriate solvents, so they may be useful not only as adsorbents and catalysts but also as templates for the various forms of materials such as fibers and membranes [17].

To design and optimize the properties of the nanodevices made of nanotubes and nanowires, it is essential to study the electronic properties of their building blocks: individual nanotube and nanowire. Recently, the intrinsic electronic transport properties of individual nanowires have been explored by using focused ion beam (FIB) technique and four-probe electrical measurement [18,19]. As well known, the physical properties of a material are determined by its structure. High pressure, as a new dimension, can tune the crystal structures and electronic structures of materials and further their physical properties. Molecular crystals such as  $C_{60}$  materials are very sensitive to high pressure since the intermolecular bonds in them are easily compressed and the molecules themselves may also be modified under pressure [20,21]. In our previous work [22], we have reported the amorphization and isostructural phase transition of  $C_{60}$  nanotubes induced by high pressure. Here, we report the temperature dependence of electrical resistivity in a single  $C_{60}$  nanotube and the evolution of phase transition temperature under pressure up to 2.4 GPa.

## 2. Experiments

The  $C_{60}$  nanotubes are fabricated by the LLIP method. As reported elsewhere [14–16,22], the pristine  $C_{60}$  powder (MTR Ltd. 99.5%) was dissolved in pyridine, and then the pyridine solution of  $C_{60}$  was added to isopropyl alcohol in a proper mixture ratio. One minute ultrasonic dispersion was taken to obtain suitable diffusion at the interface. In order to promote the growth of the  $C_{60}$  nanotubes, the solutions were exposed to visible light such as blue light with a center wavelength of 468 nm. The hollow structure of the  $C_{60}$  nanotubes was characterized using transmission electron microscope (TEM, JEM-2000EX). In the TEM experiments, the specimens were placed on a copper micro grid with carbon film.

The  $C_{60}$  nanotubes were spread on a silicon oxide surface and an appropriately isolated one was identified by a scan-

ning electron microscope. Then two pairs of Pt microleads typically 500 nm in width and 100 nm in thickness were fabricated by FIB deposition (Dual-Beam 235 FIB System from FEI Company, the working voltage of the system was 5 kV for the electron beam and 30 kV for the focused ion beam, respectively, and the current of the focused ion beam was 50 ~ 500 pA), as shown in Fig. 1(b). Finally, the electrical connection between the Pt microleads and the sample holder was made by highly conductive silver paste and Pt wires.

The measurements of temperature-dependent resistance curves  $R(T)$  under hydrostatic pressures up to 2.4 GPa were carried out by using the four-probe technique in a piston cylinder pressure cell, with a 1:1 mixture of silicon oil and coal oil as pressure transmitting medium. The pressures were loaded at room temperature.

## 3. Results and discussion

Fig. 1(a) shows a typical hollow image of the  $C_{60}$  nanotube, the tubular structure is clearly revealed, and the diameters of the sample are smaller than 1000 nm with the lengths exceeding several micrometers. Most of the nanotubes have a width of 200–500 nm, and those with diameters smaller than 200 nm seldom show tubular structure, this means a moderately large diameter is needed for the formation of  $C_{60}$  fullerene nanotubes fabricated by LLIP [14]. A typical SEM image of four probes on an individual nanotube is shown in Fig. 1(b), the platinum microleads fabricated by FIB deposition have a good contact with the nanotube. The nanotubes have a fcc structure with a space group of Fm3m and cell constant of  $a = 1.424$  nm at room temperature [23].

Fig. 2 shows the electrical resistivity ( $R$ ) as a function of temperature ( $T$ ) of a single  $C_{60}$  nanotube. The measured temperature range is 120 ~ 290 K. The features at temperatures 246 and 214 K as seen in Fig. 2 are attributed to two different transitions in  $C_{60}$  nanotube, these transitions are supposed to be designated as fcc to sc phase transition (bulk transition temperature  $T_B$ ) and surface transition from  $(1 \times 1)$  to  $(2 \times 2)$  reconstruction (surface transition temperature  $T_S$ ), respectively. As temperature decreases, the resistivity increases, while it decreases between 246 and 251 K. We suppose this change is related to the bulk transition similar to the case in a pure  $C_{60}$  single crystal where the transition happens

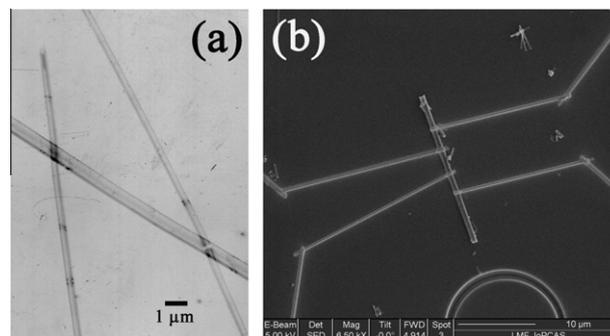
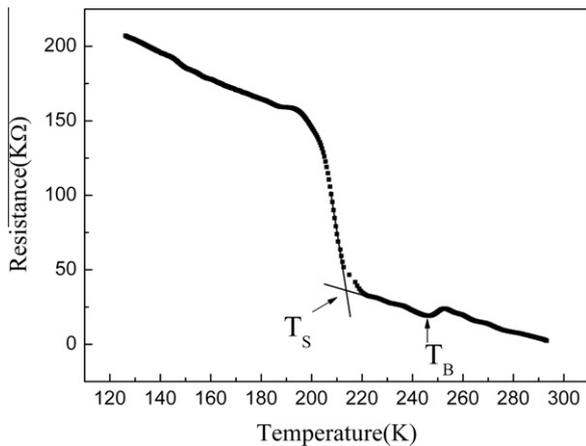


Fig. 1 – (a) TEM image of the  $C_{60}$  nanotubes, (b) typical SEM images of a single  $C_{60}$  nanotube and the attached four Pt microleads.



**Fig. 2 – Temperature dependence of resistance  $R(T)$  for a single  $C_{60}$  nanotube.**

around 250 K [2–4] accompanying a sharp increase in the conductivity when the crystal is cooled down through the bulk transition [4]. The bulk transition temperature in  $C_{60}$  nanotubes is a little lower compared with the  $C_{60}$  single crystal and this may be due to the nano-size effect. The  $C_{60}$  nanotube could exhibit both bulk and surface characteristics just because its hollow structure and the nanoscale effect, we think. In  $C_{60}$  thin film, it was reported that a surface transition from  $(1 \times 1)$  to  $(2 \times 2)$  happens at the temperature around 225 K [6–8]. In the  $C_{60}$  nanotube, as the temperature decreases to about 214 K, 32 K lower than  $T_B$ , a huge increase of resistance happens. This is supposed to be correlated with the surface transition, and the  $T_S$  is a bit lower than that in  $C_{60}$  thin film. When the surface rearrangement from  $(1 \times 1)$  to  $(2 \times 2)$  happens, the number of molecules in a surface will be less and intermolecular distances become larger, which leads to anomaly in resistivity [10].

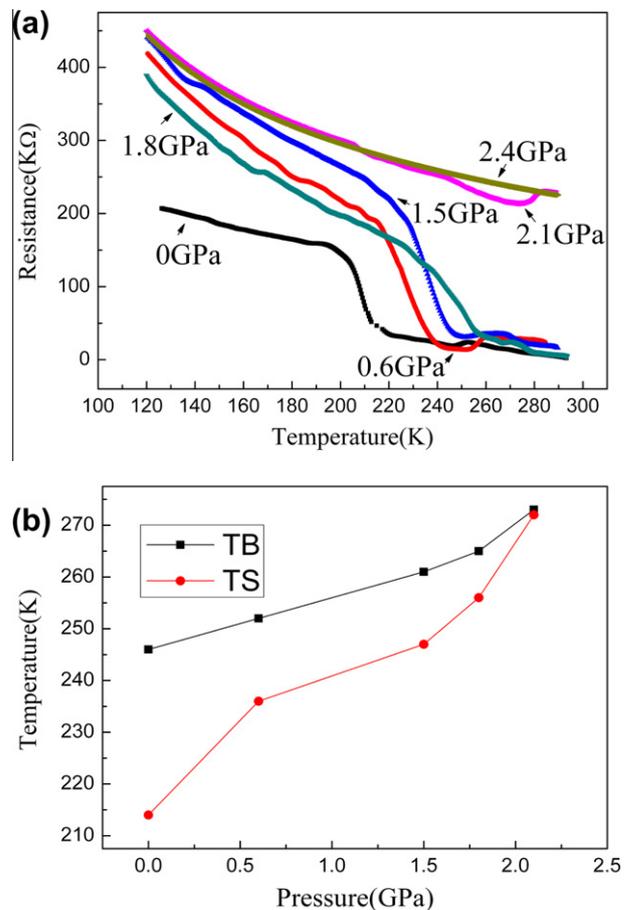
The thermally activated conductivity of the  $C_{60}$  nanotubes can be written as [24]:

$$\sigma = \sigma_0 \exp(-E/K_B T)$$

where  $\sigma$  is the conductivity of the  $C_{60}$  nanotube,  $\sigma_0$  is the conductivity prefactor,  $K_B$  is the Boltzmann constant,  $T$  is the absolute temperature, and  $E$  is the activation energy. The activation energies of the carriers motion can be more or less accurately estimated from  $(\ln \sigma^{-1})$  versus  $(1/T)$ . Through fitting the temperature dependence of the resistance curve, the activation energies are obtained as 0.21 and 0.17 eV, respectively, above and below the bulk phase transition temperature  $T_B = 246$  K. In the literatures, the band-gap values of  $C_{60}$  crystals obtained by optical or photoemission measurements were reported to be about 1.9–2.3 eV [25,26]. Our calculated activation energies are much lower than half of the energy gap, but they are comparable to the published data [4,5,18,27,28]. The activation energies in the  $C_{60}$  bulk crystals were reported to be 0.66–0.58 and 0.35–0.38 eV above and below  $T_B$  [4,5] calculated through temperature dependence of electrical resistance, respectively. Wen et al. [5] supposed that the obviously low conduction activation energy may indicate the presence of similar midgap states in  $C_{60}$  crystal as the case of  $BaBiO_3$ . For  $BaBiO_3$ , the optical measurement indicates

a gap of 2.0 eV while the electrical conductivity measurement gives the activation energy of 0.24 eV [29], the inconformity is attributed to the formation of midgap states related to the local charge density. In doped  $C_{60}$  with K, Takahashi et al. [26] observed the formation of midgap states. Moreover, Arai et al. [27] reported that the activation energy of  $C_{60}$  single crystal is 0.26 and 0.15 eV above and below  $T_B$ , respectively, which is ascribed to an impurity level. In our case, the FIB process may induce some defects to the  $C_{60}$  nanotubes at the junction, these defects could decrease the conduction activation energy. In addition, different from the bulk  $C_{60}$ , since the  $C_{60}$  nanotube has some bending structure characteristic (tubular structure), which would cause some changes of band structure, this is also one possible reason for its having different activation energies. And our results are close to that of  $C_{60}$  nanorods reported by Ji et al. [18], in which the activation energies are 0.16 eV above  $T_B$  and 0.12 eV below  $T_B$ . The lower activation energies of  $C_{60}$  nanotubes than that of bulk  $C_{60}$  indicate the easier carrier injection of  $C_{60}$  nanotube, which will be invaluable for future nanodevice fabrication.

The temperature dependences of electrical resistivity for a single  $C_{60}$  nanotube under different pressures are shown in Fig. 3(a). With increasing pressure, both  $T_B$  and  $T_S$  move to higher temperatures, and the temperature range  $\Delta T = T_B - T_S$



**Fig. 3 – (a) The temperature-dependent resistance curves  $R(T)$  of  $C_{60}$  nanotube measured under different pressures, (b) the bulk transition temperature  $T_B$  and surface transition  $T_S$  versus pressure  $P$ .**

becomes small (Fig. 3(b)). The two phase transition temperatures almost overlap to one under 2.1 GPa. When the pressure reaches 2.4 GPa,  $T_B$  and  $T_S$  could not be measurable in our experimental temperature range. It was reported that the fcc structure of bulk  $C_{60}$  crystals transforms into a sc structure as the pressure reaches 0.23 GPa [30], and Wang et al. reported that the  $C_{60}$  sheets undergo a similar transformation at a pressure of about 1.3 GPa, about 1 GPa higher than that for bulk  $C_{60}$  [11]. The pressure could facilitate the phase transition from the fcc to the sc structure in some  $C_{60}$  crystalline materials, this is consistent with the  $T_B$  increases as pressure increases. We all know that the surface properties of a solid are influenced to a large extent by the bulk properties, the surface rearrangement from  $(1 \times 1)$  to  $(2 \times 2)$  could be resulted from the fcc to sc bulk phase transition [6]. So the  $T_S$  exhibits a similar behavior as  $T_B$ .

Based on classical Monte Carlo simulation, Laforge et al. [7] reported that there are altogether four phases or regimes of  $C_{60}$  in the whole temperature range: a fully ordered state ( $Pa\bar{3}$ ) below 150 K; an intermediate phase with 1/4 molecules disordered and 3/4 molecules ordered surface on a ordered bulk ( $Pa\bar{3}$ ) between 150 and 230 K; a disordered surface layer on an ordered bulk ( $Pa\bar{3}$ ) between 230 and 250 K; a fully rotationally disordered state ( $Fm\bar{3}m$ ) above 250 K. In our experiments,  $T_S$  was determined as 214 K, which corresponds to the state that 3/4 molecules at the surface are ordered while 1/4 disordered. Crystal fields and intermolecular interactions compose the rotational ordering forces, and both of them at surface are different from that in bulk. As reported by Laforge et al., in fullerene, the crystal field is quantitatively very important in the bulk and its change at the surface may lead to an early thermal disordering taking place [7]. With increasing pressure, the crystal cell of  $C_{60}$  nanotube is compressed and the distance among the molecules becomes shorter, and consequently, the crystal field and intermolecular interactions are both enhanced, which favor orientational order. Hence, higher temperature would be needed to break the ordered state under pressure. In addition, in  $C_{60}$  single crystal, the sc phase with lattice constant of  $a = 14.04 \pm 0.01 \text{ \AA}$  is denser than the fcc phase with lattice constant of  $a = 14.17 \pm 0.01 \text{ \AA}$  [2], so the sc structure could be favored under high pressure. Based on above reasons, it would be reasonable that the  $T_B$  increases with increasing pressure, just as observed in our experiments. Under pressure,  $C_{60}$  molecules at surface would lose their mobility and specific surface effect, so that their behavior would trend to the bulk characteristics. In this case, the  $T_S$  would increase with pressure. It is expected that at a certain pressure all the  $C_{60}$  molecules at the surface would exhibit the same behavior of that in the bulk. In our study, this happens at a pressure of about 2.1 GPa, where  $T_S$  and  $T_B$  almost overlap to one, indicating the uniform property in the  $C_{60}$  nanotube.

Our studies show that the pressure could affect surface physical properties of functional materials through changing their structures. The individual surface property may be declined under pressure. As well known, many devices are designed based on the specific surface properties of functional materials. So high pressure effect must be considered in the practical applications of the devices in some particular environments, such as undersea operations and mining works, etc.

## 4. Conclusions

Pure single crystalline  $C_{60}$  nanotubes with fcc structure were fabricated by LLIP method. Two phase transitions, bulk transition and surface transition, have been observed at  $T_B = 246 \text{ K}$  (a little lower than that in bulk  $C_{60}$  single crystal and  $C_{60}$  thin film) and  $T_S = 214 \text{ K}$ , respectively. Calculated activation energies are 0.21 eV above  $T_B$  and 0.17 eV below  $T_B$  and lower than that of  $C_{60}$  bulk material, indicating the easier carrier injection in  $C_{60}$  nanotubes. It is found that both  $T_B$  and  $T_S$  increase with increasing pressure and they overlap to one at about 2.1 GPa, indicating the uniform property in the  $C_{60}$  nanotube.

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