

# On-site preparation of one-dimensional C<sub>60</sub> polymer crystals

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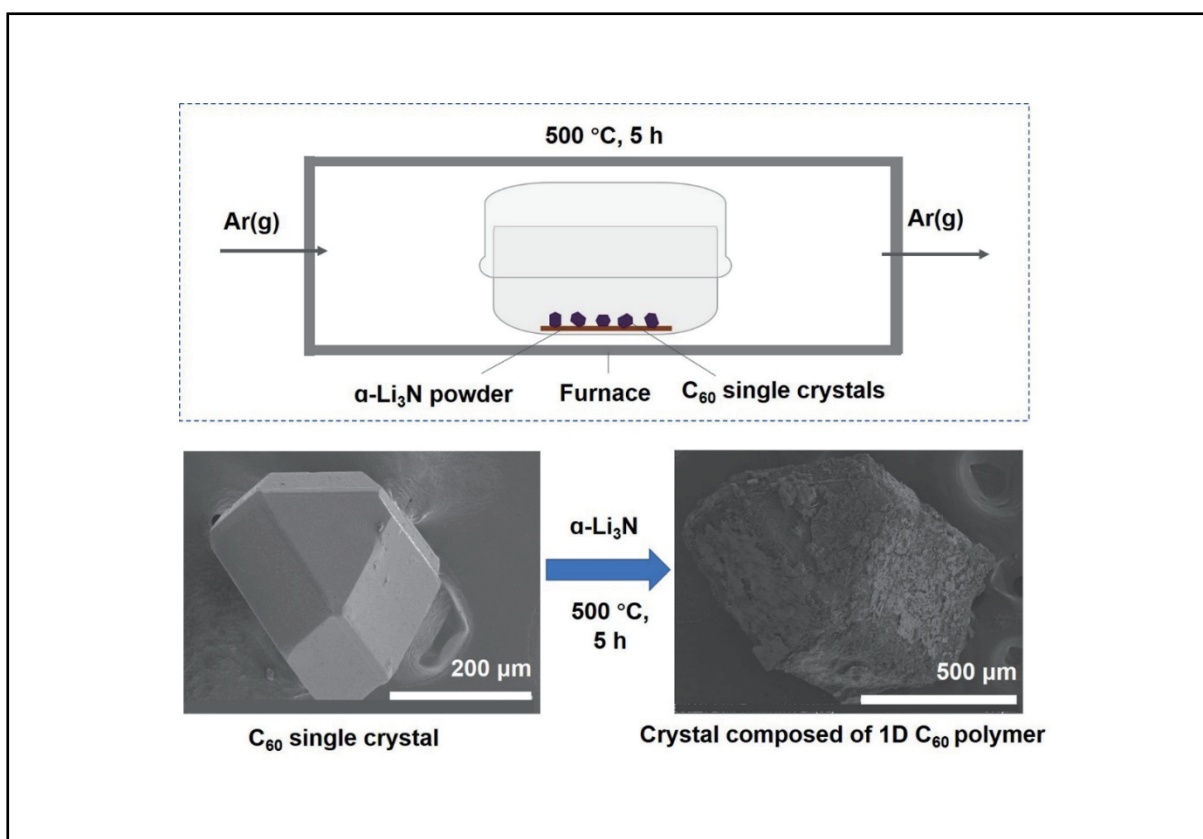
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## Graphical abstract



One-dimensional C<sub>60</sub> polymer crystals preserving the morphology were prepared by annealing with  $\alpha$ -Li<sub>3</sub>N at 500 °C for 5 h.

## Public summary

- This work proposed an on-site preparation of 1D C<sub>60</sub> polymer crystals preserving the original morphology by annealing C<sub>60</sub> molecular crystals with  $\alpha$ -Li<sub>3</sub>N or Li at 500 °C or 480 °C, respectively.
- The crystalline characteristics of C<sub>60</sub> were maintained after the formation of covalent bonds between adjacent C<sub>60</sub> molecules along the <110> direction via a [2+2] cycloaddition reaction.
- The phase transformation from C<sub>60</sub> molecules to the C<sub>60</sub> polymer occurred in the entire crystal.

# On-site preparation of one-dimensional C<sub>60</sub> polymer crystals

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Supporting Information

**Abstract:** The preparation of large crystals is highly important for the characterization and application of a newly found structure but remains a challenge for one-dimensional (1D) C<sub>60</sub> polymers. In this work, we successfully fabricated a 1D C<sub>60</sub> polymer crystal via on-site annealing of a millimeter-sized C<sub>60</sub> molecular crystal with  $\alpha$ -Li<sub>3</sub>N at 500 °C and ambient pressure. Characterizations show that the C<sub>60</sub> cages in the crystal have been efficiently connected, forming 1D chains along the <110> direction in an orthorhombic 3D structure. At the same time, the crystal maintains a morphology similar to that of the pristine C<sub>60</sub> crystal, providing opportunities for characterization of all the facets of the crystal via Raman spectroscopy and thus suggesting the formation mechanism of such crystals.

**Keywords:** fullerene C<sub>60</sub> molecular crystal; on-site preparation; 1D C<sub>60</sub> polymer crystal; covalent connection

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## 1 Introduction

First reported in 1985, fullerene C<sub>60</sub> is a cage-shaped molecule with many unique physical and chemical properties, indicating potential applications in fields such as solar cells, field-effect transistors, photocatalysis, and biomedicine<sup>[1,2]</sup>. Covalently connecting individual C<sub>60</sub> molecules can convert the molecular crystal of C<sub>60</sub> to long-range ordered C<sub>60</sub> polymers, which may lead to more novel and distinct applications. In the past few decades, high-pressure high-temperature (HPHT) treatment<sup>[3]</sup>, electron beam<sup>[4]</sup> or ultraviolet-visible light irradiation<sup>[5]</sup>, plasma radiation<sup>[6]</sup>, and metal-doping reactions<sup>[7]</sup> have been developed to prepare C<sub>60</sub> polymers. Structures of the C<sub>60</sub> dimer (C<sub>120</sub>)<sup>[8]</sup>, one-dimensional (1D) chain polymers<sup>[9]</sup>, 2D layer polymers with sixfold or fourfold symmetry<sup>[10–12]</sup>, and 3D polymers<sup>[13]</sup> have been obtained. The preparation described above either involves harsh reaction conditions, such as temperatures greater than 1000 °C and pressures greater than the GPa level in HPHT, or only induces local conversion in a small area, such as in irradiation methods, which has restricted the detailed characterization and wide exploration of potential applications.

In our recent work, we reported that the charge injection of  $\alpha$ -Li<sub>3</sub>N to C<sub>60</sub> can lower the energy barrier for bonding formation between two isolated C<sub>60</sub> cages and result in the formation of 1D C<sub>60</sub> polymers at relatively low temperatures (e.g., 480 °C) and ambient pressure<sup>[14]</sup>. This chemical preparation allows for the first-time gram-scale preparation of orthorhombic phase powders consisting of 1D C<sub>60</sub> polymers. Our preliminary characterizations have shown that the micrometer-sized polymer crystal consists of C<sub>60</sub> cages connected along the <110> direction, with two sp<sup>3</sup> bonds formed

between adjacent hexagons from two adjacent cages. Although exciting, two scientific problems remain: (i) Can a large-sized crystal of a 1D C<sub>60</sub> polymer instead of powders be prepared for more detailed studies on a facet of the crystal? (ii) What is the evolution mechanism of the C<sub>60</sub> molecular crystal to a 1D C<sub>60</sub> polymer crystal? To answer these questions, the preparation of large crystals composed of C<sub>60</sub> polymers is extremely desirable.

In this work, we first prepare large C<sub>60</sub> molecular crystals via physical vapor deposition and then directly convert the molecular crystals to polymer crystals while maintaining their features by annealing the C<sub>60</sub> molecular crystals with  $\alpha$ -Li<sub>3</sub>N crystal powder or Li metal plates. Eventually, crystals with sizes reaching ~1 mm are obtained, showing the excellent crystalline characteristics of an orthorhombic structure and consisting of 1D C<sub>60</sub> chains along the <110> direction as a result of the on-site connection of C<sub>60</sub> cages in the crystal.

## 2 Materials and methods

### 2.1 Materials

C<sub>60</sub> powder (99.5 at% purity by high-performance liquid chromatography (HPLC); Suzhou Dade Carbon Nano Technology Co., Ltd., China) and lithium nitride ( $\alpha$ -Li<sub>3</sub>N, 99.5 wt% purity, metals basis, 60 mesh; Sigma Aldrich, USA) were used. Li plates were purchased from Guangdong Canrd New Energy Technology Co., Ltd., China.

### 2.2 Preparation

#### 2.2.1 Growth of C<sub>60</sub> single crystals

C<sub>60</sub> molecular crystals were prepared via physical vapor

deposition<sup>[15]</sup>. In a typical preparation, 100 mg C<sub>60</sub> powder was loaded into a quartz tube (length: 300 mm; inner diameter: 15 mm). The tube was sealed under vacuum, placed in a two-zone horizontal furnace for 72 h and then cooled to room temperature, in which the C<sub>60</sub> powder was loaded in the temperature zone of 600 °C and the C<sub>60</sub> crystals were obtained in the temperature zone of 480 °C.

### 2.2.2 Preparation of 1D C<sub>60</sub> polymer crystals

Five milligrams of  $\alpha$ -Li<sub>3</sub>N powder and several C<sub>60</sub> crystals were mixed by manual shaking and added to a quartz dish (height: 15 mm; diameter: 20 mm) in a glove box operated in Ar (g) atmosphere. After being covered with a quartz lid, the quartz dish was transferred to a horizontal tube furnace and heated from room temperature to 500 °C at a heating rate of 5 °C·min<sup>-1</sup> in Ar atmosphere (flow rate: 100 sccm), followed by annealing at 500 °C for 5 h and naturally cooling to room temperature. To test the feasibility of using a Li plate instead of  $\alpha$ -Li<sub>3</sub>N for annealing, several C<sub>60</sub> crystals were placed on a small piece of Li plate in a quartz dish (height: 15 mm; diameter: 20 mm) in a glove box operated in Ar (g) atmosphere. After being covered with a quartz lid, the quartz dish was transferred to a horizontal tube furnace and heated from room temperature to 480 °C at a heating rate of 5 °C·min<sup>-1</sup> in Ar atmosphere with a flow rate of 100 sccm, followed by annealing at 480 °C for 5 h and naturally cooling to room temperature. After annealing, the crystal particles were carefully removed from the quartz dish without further processing for subsequent characterization.

### 2.2.3 Characterizations

X-ray diffraction (XRD) was carried out on a high-resolution X-ray diffractometer (Cu K $\alpha$  radiation with a wavelength of 0.15418 nm, operating at 40 kV and 30 mA; Rigaku SmartLab 3 kW, Japan) with a scan rate of 10 (°)·min<sup>-1</sup>. Raman spectra were collected with a Renishaw inVia Raman Microscope (UK). Two laser wavelengths of 532 nm and 785 nm were used, and the diameter of the laser spot was  $\sim$ 1  $\mu$ m. The Fourier transform infrared (FTIR) spectra were collected from 4000 cm<sup>-1</sup> to 400 cm<sup>-1</sup> with a TENSOR II (Bruker, Germany), with samples diluted in KBr powder in a tablet made by compression ( $\sim$ 20 MPa for 5 s). Scanning electron microscopy (SEM) images were obtained with a field-emission scanning electron microscope (Hitachi SU8220, Japan). Transmission electron microscopy (TEM) images were obtained with a JEOL-2100F instrument (Japan).

## 3 Results and discussion

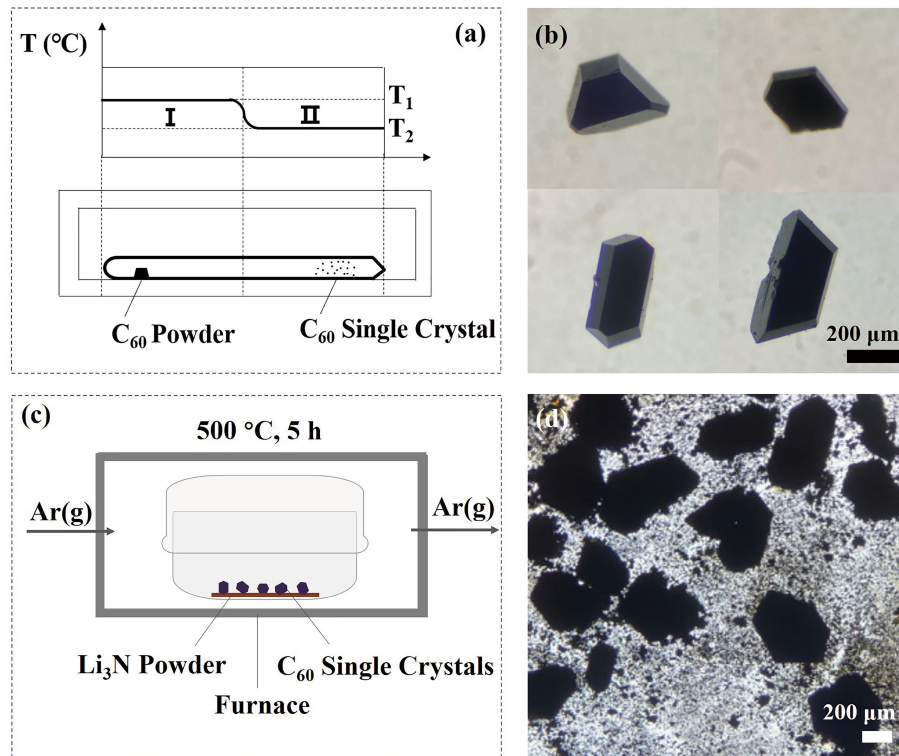
The preparation of C<sub>60</sub> single crystals by physical vapor deposition is schematically shown in Fig. 1a. After the tube loaded with C<sub>60</sub> powder was annealed for 72 h and cooled to room temperature, the C<sub>60</sub> crystals were collected in the low-temperature zone (480 °C) of the quartz tube. The optical images in Fig. 1b show crystals with distinct edges and corners, as well as smooth and glossy surfaces. Fig. 1c shows the preparation of C<sub>60</sub> polymer crystals. After annealing with  $\alpha$ -Li<sub>3</sub>N at 500 °C for 5 h, the color of the C<sub>60</sub> crystals changed from black to dark brown, and the surface of the 1D C<sub>60</sub> polymer crystals became rough, as shown in the optical images in

Fig. 1d.

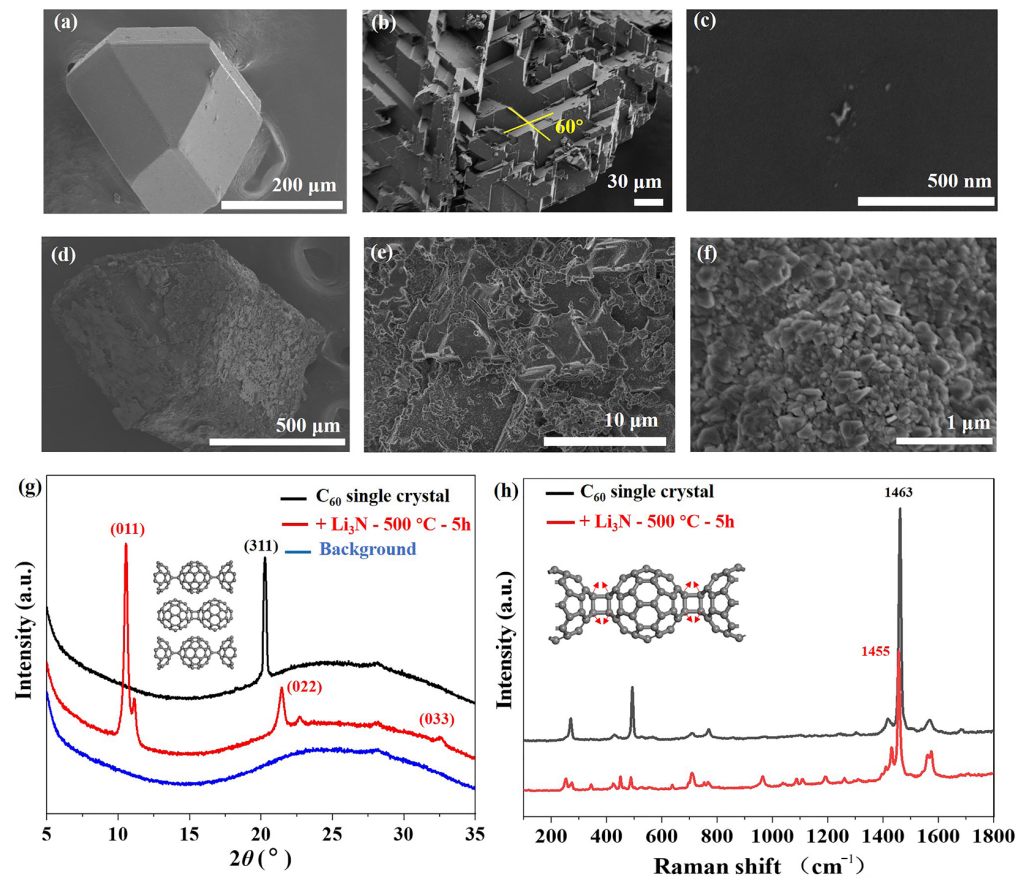
The SEM image in Fig. 2a shows a typical C<sub>60</sub> crystal with a lateral size of  $\sim$ 400  $\mu$ m and distinct edges and corners. In the magnified image of the broken internal surface made by breaking a crystal (Fig. 2b), we can see that the C<sub>60</sub> crystal exhibits distinct edges with angles of 60 and 120 degrees but all along the same directions, indicating a single-crystal feature inside the crystal. The SEM image at higher magnification (Fig. 2c) shows a uniform contrast, indicating a flat surface without features detectable by SEM under such magnification. Specifically, the size of C<sub>60</sub> crystals can be increased by prolonging the annealing time. After annealing C<sub>60</sub> crystals covered by  $\alpha$ -Li<sub>3</sub>N powder, the obtained crystals maintain their original morphology but have a rough surface, as shown in the SEM image in Fig. 2d. The higher magnification image shows that there are crystal steps on the surface (Fig. 2e), and small grains are observed at the hundred-micron scale (Fig. 2f) after annealing with  $\alpha$ -Li<sub>3</sub>N.

One C<sub>60</sub> single-crystal particle before and after annealing with  $\alpha$ -Li<sub>3</sub>N powder was tested via XRD, and the patterns are shown in Fig. 2g. Before annealing, the particle shows a strong and narrow peak derived from the (311) crystal plane of a face-centered cubic (fcc) structure. The XRD pattern (Fig. S1) from another crystal face also shows peaks at (111) and (222). After annealing with  $\alpha$ -Li<sub>3</sub>N at 500 °C for 5 h, the three strong diffraction peaks are attributed to the (011) crystal plane family of orthorhombic structures, as illustrated in the inset. That is, the connection along the  $\langle$ 110 $\rangle$  direction converted the dense stacking of C<sub>60</sub> cages in the molecular crystal to an orthorhombic phase. The Raman spectrum taken with a laser wavelength of 532 nm in Fig. 2h shows that the A<sub>g</sub>(2) peak of the original C<sub>60</sub> crystals (1463 cm<sup>-1</sup>, the stretching vibration of pentagons in C<sub>60</sub><sup>[16]</sup>) shifts to 1455 cm<sup>-1</sup> for the annealed sample, which is attributed to the formation of a bridge bond between C<sub>60</sub> molecules<sup>[17,18]</sup>, as illustrated in the inset. The broad peak at 965 cm<sup>-1</sup> from the annealed sample is attributed to the stretching vibration mode of four-membered carbon rings formed between C<sub>60</sub><sup>[19]</sup>. Owing to the reduction in the symmetry of C<sub>60</sub> polymers compared with individual C<sub>60</sub> molecules, some vibration modes in the range of 200–500 cm<sup>-1</sup> that are not observed in C<sub>60</sub> appear in the Raman spectrum of C<sub>60</sub> polymer crystals. Raman spectra taken with a laser wavelength of 785 nm (Fig. S2) also show that the A<sub>g</sub>(2) peak of the original C<sub>60</sub> crystal shifts to 1462 cm<sup>-1</sup> for the annealed sample, which is attributed to the formation of an orthorhombic structure<sup>[8]</sup>. The FTIR spectra (Fig. S3) show that more peaks are identified for the 1D C<sub>60</sub> polymer crystal because of the suppressed rotation of the C<sub>60</sub> cages, which is consistent with our recent report<sup>[14]</sup>. Peaks from oxygen- or nitrogen-related bonds (e.g., C=O stretching in carbonyl groups at 1742 cm<sup>-1</sup> [20] and the C $\equiv$ N stretching band at 2200 cm<sup>-1</sup> [21]) in the C<sub>60</sub> polymer were not observed, confirming that polymerization is the main reaction during the phase transition.

The microstructure of the pristine C<sub>60</sub> crystals was identified via high-resolution transmission electron microscopy (HRTEM). The image and the corresponding selected area electron diffraction (SAED) data are shown in Fig. 3a and b, respectively. Fig. 3a clearly shows the lattice spacing of the



**Fig. 1.** (a) Schematic of the preparation of C<sub>60</sub> crystals by physical vapor deposition ( $T_1 = 600\text{ }^\circ\text{C}$ ,  $T_2 = 480\text{ }^\circ\text{C}$ ). (b) Optical images of C<sub>60</sub> crystals. (c) Schematic of the preparation of polymer crystals by annealing C<sub>60</sub> crystals with  $\alpha$ -Li<sub>3</sub>N at 500 °C for 5 h. (d) Optical image of crystals after annealing.



**Fig. 2.** SEM images of the (a) surface, (b) internal and (c) magnified surfaces of a C<sub>60</sub> crystal. (d, e, f) SEM images of C<sub>60</sub> polymer crystals at different magnifications. (g) XRD patterns and (h) Raman spectra of C<sub>60</sub> crystals before and after annealing with  $\alpha$ -Li<sub>3</sub>N at 500 °C for 5 h taken with a laser wavelength of 532 nm.

(220) planes (~0.5 nm) of the fcc C<sub>60</sub> structure. The SAED pattern in Fig. 3b indicates the excellent crystallinity of the pristine C<sub>60</sub> crystal. The image in Fig. 3c reveals that the periodic arrangement of the carbon cages is retained and confirms the orthorhombic structure of the polymer crystal, with a measured 0.495 nm spacing corresponding to the (112) interplanar spacing. Specifically, the SAED pattern in Fig. 3d

indicates the excellent periodic characteristics of the C<sub>60</sub> polymer crystal at the nanometer scale.

To further confirm the conversion of molecular crystals to polymer crystals, Raman spectroscopy was performed with a laser wavelength of 532 nm on three facets of one particle and an internal site obtained after breaking the particle, as shown in the SEM images and Raman spectra in Fig. 4. From the

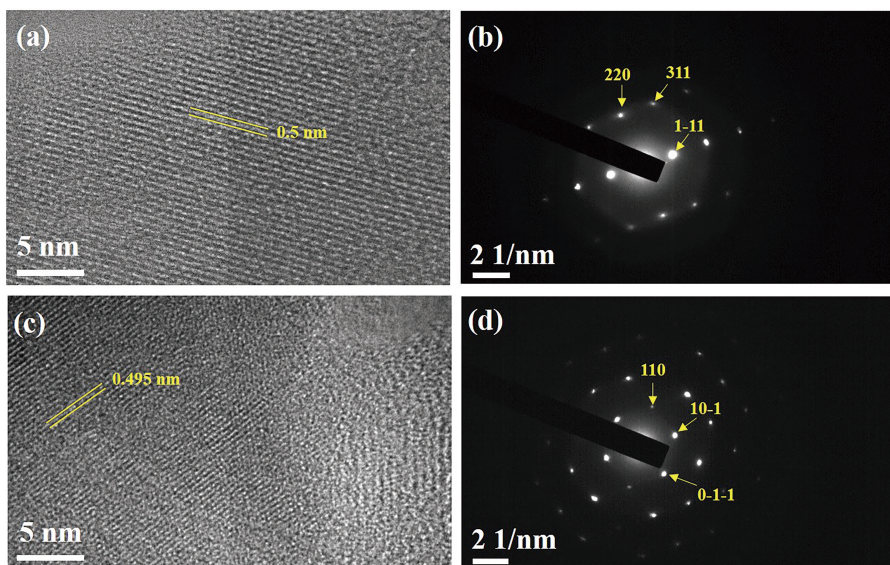


Fig. 3. HRTEM images and SAED patterns of C<sub>60</sub> crystals (a, b) before and (c, d) after annealing with α-Li<sub>3</sub>N at 500 °C for 5 h.

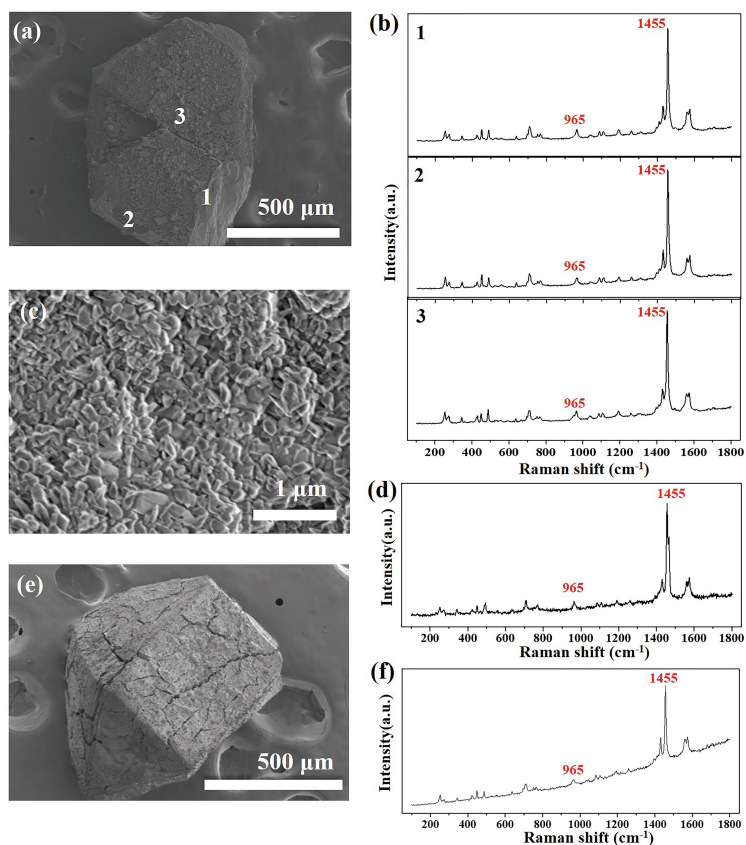


Fig. 4. (a) SEM image and (b) Raman spectra of three facets of one polymer particle. (c) SEM image and (d) Raman spectrum of the internal site of the particle. (e) SEM image and (f) Raman spectrum of a crystal obtained by annealing the C<sub>60</sub> molecular crystal with a Li metal plate at 480 °C for 5 h.

spectra, we can again see that the  $A_g(2)$  peak of the original  $C_{60}$  crystals ( $1463\text{ cm}^{-1}$ ) shifts to  $1455\text{ cm}^{-1}$ , and a broad peak at  $965\text{ cm}^{-1}$  appears, indicating that the phase transformation from  $C_{60}$  molecules to the  $C_{60}$  polymer occurs in the entire sample. This result suggests that the reaction for the connection of  $C_{60}$  cages is still valid at a depth of 1 mm into the crystal during annealing for 5 h. It can be inferred that larger  $C_{60}$  polymer crystals can be prepared by annealing larger  $C_{60}$  crystals with  $\alpha\text{-Li}_3\text{N}$  under similar conditions. To verify the role of Li, we also tested the feasibility of using a Li plate instead of  $\alpha\text{-Li}_3\text{N}$  for annealing. The SEM image in Fig. 4e shows that after annealing with a Li metal plate at  $480\text{ }^\circ\text{C}$  for 5 h, the  $C_{60}$  molecular crystal of interest maintains the crystalline morphology but has a rougher surface. The shift in the  $A_g(2)$  mode from  $1463$  to  $1455\text{ cm}^{-1}$  and the appearance of new vibration modes in the range of  $200\text{--}500\text{ cm}^{-1}$  (Fig. 4f) indicate the effective formation of 1D  $C_{60}$  polymers by connecting  $C_{60}$  cages with Li metal plates. These results indicate that Li is the active component for the reaction connecting  $C_{60}$  cages in the phase transition.

Now, we discuss the transport of Li in the crystal by assuming a short-range role of Li in the connection reaction.  $\text{Li}_4\text{C}_{60}$  is reportedly formed in a polymeric  $C_{60}$  structure<sup>[10]</sup>, roughly corresponding to a concentration of Li in the  $C_{60}$  crystal of  $\sim 10.04\text{ mol/L}$  on the basis of the structural parameters obtained from XRD and HRTEM. Assuming that the feeding of Li is sufficient, the diffusion of Li can be obtained via Fick's second law:  $\frac{C_s - C_x}{C_s - C_0} = \text{erf}\left(\frac{x}{2 \times \sqrt{D \times t}}\right)$ , where the source concentration ( $C_s$ ) of Li surrounding the  $C_{60}$  crystal is estimated as  $78.23\text{ mol/L}$  for the solid or melted Li plate,  $C_x$  is the concentration of Li at the position of  $x$  ( $\sim 10.04\text{ mol/L}$  at the center of the crystal),  $C_0$  is the concentration of Li in the pristine  $C_{60}$  crystal ( $0\text{ mol/L}$ ),  $t$  is the diffusion time of 5 h,  $x$  is the distance from the surface to the center of the crystal,  $D$  is the diffusion coefficient, and erf is the Gauss error function. On the basis of the  $x$  value of  $\sim 0.5\text{ mm}$ , we obtain a diffusion coefficient of  $\sim 2.87 \times 10^{-8}\text{ cm}^2 \cdot \text{s}^{-1}$  for Li into the  $C_{60}$  crystal. This value is greater than the diffusion coefficient of Li in solid-state Li alloy anodes ( $\sim 10^{-10}\text{ cm}^2 \cdot \text{s}^{-1}$ )<sup>[22]</sup> but lower than that in liquid Li–Pb alloys ( $\sim 10^{-4}\text{ cm}^2 \cdot \text{s}^{-1}$ )<sup>[23]</sup> or the diffusion coefficient for Li in graphite ( $\sim 7.6 \times 10^{-6}\text{ cm}^2 \cdot \text{s}^{-1}$ )<sup>[24]</sup>. On the other hand, in the present study, we cannot determine the possibility of transmittance from the cycloaddition reaction in the crystal, as indicated by the simulation results previously reported<sup>[14]</sup>. The most likely mechanism may involve the diffusion of Li across the macroscopic distance and the transmittance of the reaction at the microscopic scale around the connected cages, which needs further investigation.

## 4 Conclusions

In conclusion, this work proposes on-site preparation of 1D  $C_{60}$  polymer crystals preserving the original morphology by annealing  $C_{60}$  molecular crystals with  $\alpha\text{-Li}_3\text{N}$  or Li at  $500\text{ }^\circ\text{C}$  or  $480\text{ }^\circ\text{C}$ , respectively. Characterizations via SEM, XRD, HRTEM and SAED show that the crystalline characteristics of  $C_{60}$  are maintained after the formation of covalent bonds between adjacent  $C_{60}$  molecules along the  $\langle 110 \rangle$  direction via a [2+2] cycloaddition reaction. A detailed Raman spectrum

shows the complete phase transformation through the whole crystal. This work provides a new starting point for a deep understanding of the role of  $\alpha\text{-Li}_3\text{N}$  or Li in terms of the mechanism of  $C_{60}$  polymer crystals.

## Supporting information

The supporting information for this article can be found online at <https://doi.org/10.52396/JUSTC-2024-0071>. The supporting information includes 3 figures.

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## Conflict of interest

The authors declare that they have no conflict of interest.

## Biographies

**Xia Wang** is a Ph.D. candidate at the University of Science and Technology of China. Her research mainly focuses on microstructural regulation techniques for novel carbon materials from fullerene and graphene.

**Yanwu Zhu** is currently a Full Professor at the University of Science and Technology of China. He obtained his Ph.D. degree in Physics from the National University of Singapore in 2006. His current research interests include the synthesis of novel carbon nanomaterials and their applications for energy storage and conversion.

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