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High pressure and high temperature induced polymerization of C_{60} quantum dots*

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We synthesized C_{60} quantum dots (QDs) with a uniform size by a modified ultrasonic process and studied its polymerization under high pressure and high temperature (HPHT). Raman spectra showed that a phase assemblage of a dimer (D) phase (62 vol%) and a one-dimensional chain orthorhombic (O) phase (38 vol%) was obtained at 1.5 GPa and 300 °C. At 2.0 GPa and 430 °C, the proportion of the O phase increased to 46 vol%, while the corresponding D phase decreased to 54 vol%. Compared with bulk and nanosized C_{60}, C_{60} QDs cannot easily form a high-dimensional polymeric structure. This fact is probably caused by the small particle size, orientation of the disordered structure of C_{60} QDs, and the barrier of oxide function groups between C_{60} molecules. Our studies enhance the understanding of the polymerization behavior of low-dimension C_{60} nanomaterials under HPHT conditions.

Keywords: C_{60} quantum dots, high pressure and high temperature, fullerene polymer

PACS: 64.70.Nd, 91.60.Gf, 61.48.–c, 64.70.km **DOI:** 10.1088/1674-1056/ab6657

1. Introduction

Fullerene C_{60}, a representative member of carbon allotropies, has gained a lot of attention in the scientific research owing to its important potential applications in the fields of superhard materials, magnetic memory, gas storage, and catalysis.[1–7] Recently, because of its unique structure and excellent physical properties, the polymerization behavior of C_{60} has been extensively studied by physicists and chemists.[8–10] Previous studies found that the double bond of C_{60} could be broken and form covalent bond with adjacent C_{60} molecules under certain high pressure and high temperature (HPHT) conditions.[11–14] Various C_{60} polymer phases have been obtained under HPHT conditions, such as C_{60} dimers (D) formed by $[2+2]$ cyclo-addition of double bonds, one-dimensional chain-like orthorhombic (O) phase, two-dimensional tetragonal (T) and rhombohedral (R) phases.[11,12,14–19] In addition, a novel quasi-three-dimensional polymeric C_{60} was successfully obtained by choosing a unique C_{60} solvate.[20]

There are significant differences between nanosized and bulk C_{60} materials due to a remarkable nano-size effect. For instance, the lattice constant of the one-dimensional C_{60} nanorods is 20% larger than that of bulk C_{60} materials.[21] Compared with the bulk C_{60} materials, the bulk modulus of two-dimensional C_{60} nanosheets increases by about 60%.[22] In this case, the polymerization behavior of C_{60} nanomaterials has attracted lots of interest. It has been found that the polymeric structure of C_{60} nanomaterials (e.g., nanosheets, nanorods, and nanotubes) requires higher pressures and temperatures than those for bulk C_{60} materials.[23–27] Moreover, there exist obvious differences in the polymerization degree and photoluminescence between C_{60} nanosheets and nanorods under identical P–T conditions, which are originated from different confinement effects in different confined dimensions.[23,25] Considering the smaller dimension of zero-dimensional C_{60} quantum dots (QDs) than other C_{60} nanomaterials, the effect of the surface and particle size on C_{60} QDs would be more pronounced. However, studies on the polymerization behavior of zero-dimensional C_{60} QDs under HPHT conditions are still open due to the difficulty in obtaining uniform size and high purity C_{60} QDs.

In this work, we successfully fabricated C_{60} QDs with a uniform size by using a modified ultrasonic process, and then explored the polymerization behavior of C_{60} QDs after HPHT treatments by Raman spectra. The results suggested that a mixed phase contained two different proportions of dimer (D) phase and one-dimensional chain like orthorhombic (O) phase was obtained at our present HPHT conditions (i.e., 1.5 GPa and 300 °C; 2.0 GPa and 430 °C).

2. Experimental details

The C_{60} QDs were prepared by using a modified sonication. In short, a saturated solution of C_{60} toluene (5 ml) was added to 50 ml deionized water, then the emulsification reaction between the C_{60} toluene solution and water occurred

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with the help of ultrasonic treatment (40 kHz, 100 W/cm^2, 1 h, 60 °C). After the ultrasonic process, the emulsion was allowed to stand for 5 h until it was stratified. The transparent and slightly yellow solution in the lower layer was C_{60} fullerene water suspension (C_{60} FWS). The synthesis process for the C_{60} FWS has been reported in details elsewhere.[28–30] The C_{60} QDs were obtained by heating dry C_{60} FWS at 200 °C in vacuum (10^{-4} Pa) for 2 h.

The HPHT experiments were performed by a piston-cylinder device, which employed the silicone oil as the pressure transmission medium to provide quasi-hydrostatic pressure conditions. In order to compare the polymerization behavior of C_{60} QDs and bulk C_{60} under HPHT conditions, the C_{60} QDs and bulk C_{60} were assembled into the same high pressure chamber to ensure the same reaction conditions. After the experiment, the residual silicone oil was cleaned with n-hexane. Earlier works found that heating before pressurization was beneficial to the formation of C_{60} polymeric phases.[23,25] In our experiments, the samples were treated under two different *P*–*T* conditions: 1.5 GPa and 300 °C (condition one) and 2 GPa and 430 °C (condition two), respectively. The detailed procedure was as following. The samples were first pressed to 0.5 GPa, then heated to 300 °C, and finally the pressure gradually increased to 1.5 GPa at the target temperature. After the samples were keep for 2 h at the final target *P*–*T* conditions, they were immediately quenched by turning off the heating power. The same procedure was adopted for the samples at 2 GPa and 430 °C.

Morphologies were characterized by field emission transmission electron microscope (TEM, JEM-2200FS JEOL). The infrared (IR) spectra were measured at ambient conditions using an infrared spectrometer (NICOLET AVATAR 370 DTGS). Thermogravimetric analysis (TGA) was performed using a Perkin-Elmer Pyris-7TGA instrument at the heating rate of 5 °C/min in N_2. Raman spectra were recorded by a Renishaw in Via Raman microscope equipped with a ZX514 nm Ar^+ laser as the excitation source. To avoid the photopolymerization of C_{60}, the output laser power was set to less than 0.2 mW. High-quality Raman spectra were collected for all samples using an integration time of 80 s.

3. Results and discussion

Typical TEM images of the C_{60} FWS and C_{60} QDs are shown in Fig. 1. The C_{60} FWS exhibits favorable dispersibility, with the particle size ranging from 15 nm to 30 nm. Compared to the normal methods for preparing C_{60} FWS,[28–30] we increased the temperature of ultrasonic water bath to 60 °C, which greatly reduces the particle size and improves the dispersibility of the particles. In addition, there is an interesting phenomenon that the particle size of C_{60} QDs is smaller than that of C_{60} FWS, which is reduced to 5–10 nm after heating, as seen in Fig. 1(b). The significant decrease in the particle size is due to the removal of water and oxygenated functional groups from C_{60} FWS by heating. The TEM results show that C_{60} QDs with a relatively uniform size and a better dispersion were successfully prepared by the above mentioned process.

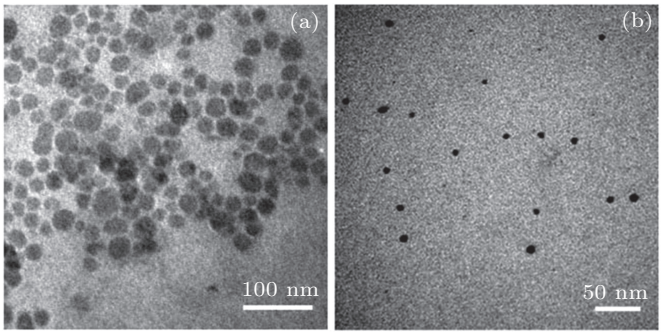


Fig. 1. TEM images of (a) C_{60} FWS and (b) C_{60} QDs.

Figure 2 presents the TGA curve (black line) and the first derivative of the TGA curve (DTG) (red line) of C_{60} FWS. There are two peaks of weight loss in the range of low temperatures. One is located at around 110 °C, which is close to the boiling point of toluene (110.6 °C) and free water, inferring that the weight loss (17%) is due to the volatilization of toluene and free water. The other one is located at around 210 °C. The weight lost (40%) here comes from the water that binds tightly to C_{60} molecules.[28,31] These two weight losses are approximately 57% of the total mass, which gives a good explanation for the remarkable reduction in the particle size after heat treatment that is observed in TEM.

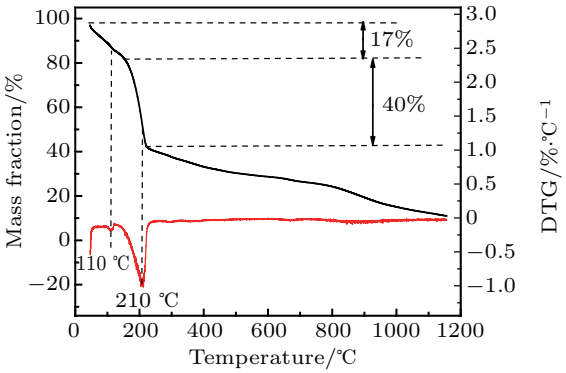


Fig. 2. TGA curve of C_{60} FWS (black line) and the DTG of C_{60} QDs (red line).

The IR absorption spectra of C_{60} FWS and C_{60} QDs are shown in Fig. 3(a). The bands at 526 cm^{-1}, 576 cm^{-1}, 1182 cm^{-1}, and 1428 cm^{-1} (marked by stars) are attributed to the intrinsic infrared absorption peaks of C_{60}.[32,33] Apart from these four vibrational modes, three additional peaks are also observed at 1080 cm^{-1}, 1384 cm^{-1}, and 1598 cm^{-1}, which are assigned to the absorption of C–OH, toluene, and C = C bond, respectively. The appearance of the C–OH and C = C bond absorption peaks suggests that $C_{60}(OH)_n$ hydrates were

formed after the ultrasonic treatment, which are in good agreement with previous studies.[28,31] After heating at 200 °C, the peak at 1348 cm^{-1} disappears, indicating that the toluene can be removed by heating, which is consistent with the result of TGA. However, the absorption peaks at 1080 cm^{-1} and 1598 cm^{-1} suggest that only part of the $C_{60}(OH)_n$ were removed and some still remained.

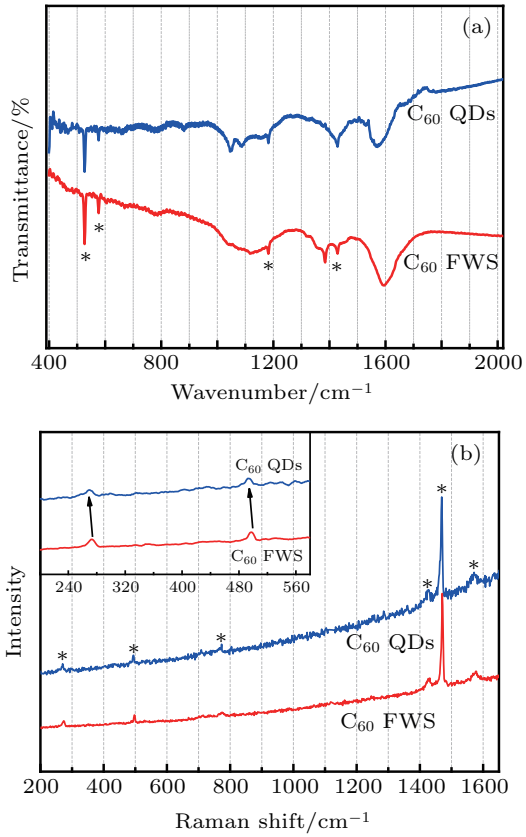


Fig. 3. (a) IR spectra of C_{60} FWS and C_{60} QDs. (b) Raman spectra of C_{60} FWS and C_{60} QDs using 514 nm laser excitation. The inset shows the linear amplification at 200–580 cm^{-1}. The marked stars represent the typical IR and Raman peaks of C_{60}.

The Raman spectra of C_{60} FWS and C_{60} QDs are shown in Fig. 3(b). Compared with ten characteristic peaks of the bulk C_{60} powder, only six characteristic peaks (marked by stars) are detected in C_{60} FWS, which are located at 273 cm^{-1} ($H_g(1)$), 498 cm^{-1} ($A_g(1)$), 773 cm^{-1} ($H_g(4)$), 1426 cm^{-1} ($H_g(7)$), 1469 cm^{-1} ($A_g(2)$), and 1573 cm^{-1} ($H_g(8)$), respectively.[23,25,34] The $H_g(1)$ and $A_g(1)$ vibration modes of C_{60} FWS, which are corresponding to the radial vibration of the C_{60} molecules, both show a blue shift of 3–4 cm^{-1}, compared to those of bulk C_{60}. Because of the hydration of C_{60} FWS, the radial vibration of the C_{60} molecules is squeezed by water, which accelerates the molecules vibration and hence results in the movement towards high frequencies.[33] After the heating treatment, the $H_g(1)$ and $A_g(1)$ vibration modes of C_{60} QDs move from 273 cm^{-1} back to 270 cm^{-1} and from 498 cm^{-1} to 494 cm^{-1}, respectively. The Raman peak of C_{60} QDs is restored to the original Raman vibration mode of bulk C_{60}, indicating the removal of most oxidation functional groups ($C_{60}(OH)_n$) and water by the heating treatment.

Raman spectroscopy has been proved to be one of the most effective methods for the exploration of the polymeric structures of C_{60}.[11,12] The $A_g(2)$ mode of the five-membered ring in the C_{60} molecule is widely used to distinguish different polymer structures, because its frequency is determined by the number of intermolecular bonds on the molecule. For example, the peak at 1464–1469 cm^{-1} corresponds to the dimer structure, the peak at 1459 cm^{-1} is the characteristic feature for the one-dimensional chain polymerization (O phase), the peak at 1447 cm^{-1} is a square structure (T phase) achieved by the nearest neighbors in the (100) plane, and the peak at 1407 cm^{-1} is the structure in which a nearest triangle is acquired in the (111) plane to form a triangle (R phase).[18,19]

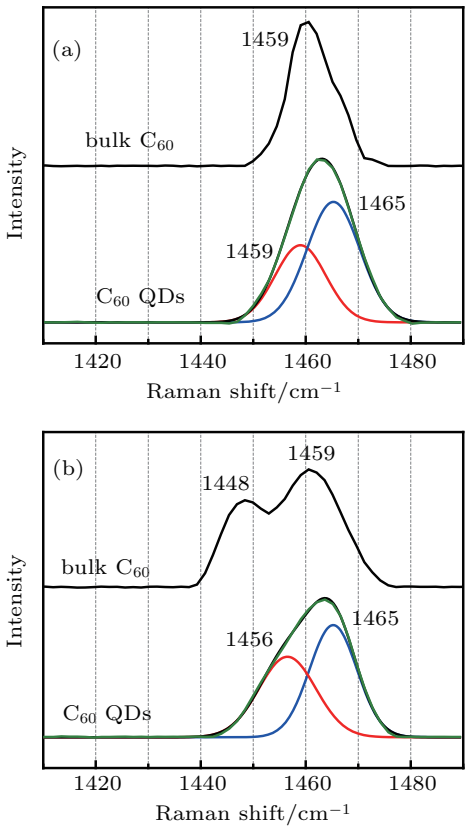


Fig. 4. Raman spectra for bulk C_{60} and C_{60} QDs polymerized at (a) condition one (1.5 GPa and 300 °C), and (b) condition two (2 GPa and 430 °C). All spectra were obtained using a 514 nm Ar+ ion excitation laser.

The $A_g(2)$ vibration modes for C_{60} QDs and bulk C_{60} treated under two different *P–T* conditions are plotted in Figs. 4(a) and 4(b), respectively. Under condition one (1.5 GPa and 300 °C), the $A_g(2)$ vibration mode shifts from 1469 cm^{-1} to 1459 cm^{-1} for bulk C_{60}, indicating that a one-dimensional chain like O phase polymerization has occurred. In contrast, for C_{60} QDs, the $A_g(2)$ vibration mode is well fitted with two Gaussian functions. The two peaks at 1465 cm^{-1} and 1459 cm^{-1} are representative of the coexistence of dimer

phase (62 vol%) and O phase (38 vol%), but not in a pure O phase like bulk C_{60}. Under condition two (2 GPa and 430 °C), for bulk C_{60}, the $A_g(2)$ vibration mode splits into two distinguishable peaks at 1459 cm^{-1} and 1448 cm^{-1}, corresponding to one-dimensional O phase and two-dimensional T-phase polymerization, respectively. Under the same condition, the $A_g(2)$ vibration mode of C_{60} QDs is still well fitted by two Gaussian functions, which are centered at 1465 cm^{-1} and 1456 cm^{-1}, representing the mixing phase of the D phase (54 vol%) and the O phase (46 vol%). It is found that, at higher temperature and pressure (2 GPa and 430 °C), the pure long chain one-dimensional O phase was not obtained for C_{60} QDs, merely accompanied with the increase of its proportion, while the two-dimensional T phase was formed for bulk C_{60} at the same condition. The above results clearly suggest that it is more difficult for C_{60} QDs to form the high dimensional polymer structure than bulk C_{60} under the same conditions.

From our group's earlier studies on the polymerization of C_{60} nanomaterials (such as nanosheets, nanorods, and nanotubes), we find that nanometer materials are more difficult to obtained polymeric structures than bulk C_{60} materials with the change of pressure and temperature.[23–27] For comparison, the polymeric structures of bulk C_{60} and C_{60} nanomaterials at two different P–T conditions are presented in Table 1. Under condition one: C_{60} nanosheets and C_{60} nanorods are transformed to the one-dimensional chain like O phase polymerization. This phenomenon is also observed for bulk C_{60}. The only difference between the nanosized and bulk C_{60} is that the Raman modes of polymeric C_{60} nanomaterials are broader than those of bulk C_{60}.[24] In the contrary, C_{60} nanotubes have been transformed to a dimer (D) phase, while our C_{60} QDs have been transformed to a mixed phase of dimer phase (62 vol%) and O phase (38 vol%). Under condition two: C_{60} nanosheets and C_{60} nanorods could form mixed phases of O phase and T phase, while C_{60} nanotubes could form an O phase. However, C_{60} QDs have been transformed to a phase assemblage of the dimer phase (54 vol%) and the O phase (46 vol%), which is similar to the polymerization process occurred at condition one, except for the different constituent ratio of the dimer and O phases. These results elucidate that it is more difficult to form the high-dimensional polymer structure for C_{60} QDs than other C_{60} nanomaterials (such as nanosheets, nanorods, and nanotubes).

Table 1. Comparison of the polymeric structures of bulk C_{60} and C_{60} nanomaterials at two different temperature–pressure conditions.

Sample	Condition one (1.5 GPa and 300 °C)	Condition two (2 GPa and 430 °C)	Reference
Bulk C_{60}	O phase	O phase + T phase	present work
C_{60} nanosheets	O phase	O phase + T phase	[24]
C_{60} nanorods	O phase	O phase + T phase	[25]
C_{60} nanotubes	D phase	O phase	[26]
C_{60} quantum dots	D phase (62 vol%) + O phase (38 vol%)	D phase (54 vol%) + O phase (46 vol%)	present work

In contrast to the bulk C_{60} materials and other C_{60} nanomaterials, it is difficult to form a high-dimensional polymeric structure in C_{60} QDs, which is mainly attributed to three reasons: (i) small particle size effect, as reported in other materials with polyhedral structure.[35,36] Based on the particle size of C_{60} QDs (less than 10 nm) and C_{60} molecules (0.71 nm), it is roughly estimated that each C_{60} QD contains only dozens of C_{60} molecules in the diameter direction. Small dimensions hinder C_{60} QDs to form a high-dimensional polymeric structure. (ii) Orientation of the disordered structure of C_{60} QDs. C_{60} polymeric structure has preferential growth directions, such as an orthorhombic structure was obtained by the one-dimensional linear chain polymerization of C_{60} molecules in the face centered cubic (fcc) lattice along the (110) direction.[11,12,14] However, C_{60} QDs exist in amorphous forms, so it is difficult to meet the requirements of orientation in forming a long chain polymerization. (iii) Residual oxygen-containing functional groups. The oxidation groups in C_{60} QDs are the big obstacle to the formation of long chain polymerization, the similar phenomenon has been observed in doped C_{60} materials under HPHT conditions.[37] Thus, we predict that it would be very challenging to obtain a high-dimensional polymeric structure even under HPHT conditions.

4. Conclusion

In summary, we fabricated uniform size C_{60} QDs using the improved ultrasonic method. The polymerization behaviors of C_{60} QDs were studied by Raman spectra at two different temperature–pressure conditions. The Raman spectra elucidated that a phase assemblage of dimer and orthorhombic phases was achieved for C_{60} QDs at 1.5 GPa and 300 °C. Moreover, this phase assemblage also existed at 2.0 GPa and 430 °C, but the content ratio of the orthorhombic phase increased from 38 vol% (at 1.5 GPa and 300 °C) to 46 vol%. Comparing to bulk and nanosized C_{60} materials, it is difficult to obtain a high-dimensional polymeric structure for C_{60} QDs at the same temperature–pressure conditions, which may be explained by the small particle size, orientation of disordered structure of C_{60} QDs, and the barrier of oxide function groups

between C_{60} molecules. It is speculated that C_{60} QDs may experience completely different bonding behavior at higher pressures and temperatures. The present study is helpful for further understanding of the polymerization behavior of C_{60}, and provides a guidance to the design of novel superhard carbon materials.

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