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One-step synthesis of C_{60} nano-assemblies at different temperatures

Dedi Liu [a,b,*], Dongping Liu [a], Dapeng Dong [a], Yangyang He [a], Jia Liu [a], Bingbing Liu [b]

[a] School of Physics and Materials Engineering, Dalian Nationalities University, Dalian 116600, PR China
[b] State Key Laboratory of Superhard Materials, Jilin University, Changchun 130012, PR China

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ABSTRACT

Nanoassemblies (microtubes, microrods, nanoflowers and nanorod arrays) composed of C_{60} nanorods, were fabricated with a solution evaporation method. The structures and the growth mechanism of C_{60} nano-assemblies were investigated. The C_{60} microtubes obtained at room temperature had a hexagonal structure, C_{60} microrods and nanoflowers synthesized at 0 °C and −15 °C had a similar mixed hexagonal structure, and the C_{60} nanorod arrays had a structure indexed as an orthorhombic structure. The morphology of the C_{60} nano-assemblies depended on the shape and size of the seed crystals, which were closely related to the precipitation process from the saturated solution at different temperatures.

1. Introduction

Recently, much research has focused on the synthesis of nanocrystals with predetermined morphologies, which are important for a new generation of optical, electronic, and other devices. C_{60} is a useful zero-dimension material with many novel physical and chemical properties [1,2]. Very recently much attention has been given to the fabrication of nanocrystals with different morphologies, using C_{60} molecules as building blocks [3–10]. Several methods have been found to obtain single C_{60} nanocrystals with various shapes and dimensions, such as the solution evaporation method [4–7], the liquid–liquid interface method [8,9] and the template method [3,10]. However, dispersed nanocrystals could not meet the requirements for applications as nanodevices with advanced functions. Therefore, the production of nano-assemblies of single C_{60} nanocrystals with pre-determined shapes became a new research focus.

The current approaches to get C_{60} nano-assemblies tend to be based on physical methods, such as patterning and template-based methods [3,10]. However, it is difficult to assemble the friable C_{60} nanocrystals safely with physical methods, and it is also difficult to remove the template from the as-grown samples. Therefore, finding a single-step method to synthesize assembled C_{60} nanocrystals without any templates has become a key challenge in the design of integrated materials as functional devices.

In this work, C_{60} nano-assemblies with microtube, microrod, nanoflower and parallel nanorod array morphologies were synthesized with a single-step modified solution evaporation method. Different morphologies were obtained by varying the evaporation temperature. All nana-assemblies were assembled from C_{60} nanorods, but different C_{60} nano-assemblies had different crystal structures. The mechanism of formation of these C_{60} nano-assemblies was tentatively explored. The very simple method of synthesis should make this method very useful in applications, such as the production of nano-devices and functional materials.

2. Experiment section

C_{60} (purity > 99.9%) was purchased from Wuda Sanwei Carbon Cluster Corporation, China, toluene was purchased from Beijing Chemical Plant, China, and m-xylene (purity > 99.0%) was purchased from Phentex Corporation, USA.

A saturated toluene solution of C_{60} was deposited as small drops on a substrate (Si, glass, aluminum foil, etc.) and the same volume of m-xylene was placed in the same way about 1 cm away, but not on the same substrate. The two drops of solutions were covered with an inverted glass beaker about 80 ml in volume. After slow evaporation of the C_{60} toluene solution in the m-xylene atmosphere under the cover at different reaction temperatures, including room temperature, 0 °C, −15 °C and −40 °C, we obtained C_{60} nano-assemblies on the substrate at the original position of the toluene solution.

The samples on the substrate were examined at room temperature by means of scanning electron microscopy (SEM, SSX-550 SHIMADZU, Japan), X-ray powder diffraction (Rigaku D/max-RA, using $CuK_{\alpha}1$

* Corresponding author at: School of Physics and Materials Engineering, Dalian Nationalities University, Dalian 116600, PR China.
E-mail address: liudedi@dlnu.edu.cn (D. Liu).

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radiation with λ = 1.5406 Å) and Raman spectroscopy (Renishaw inVia, UK with an 830 nm laser as excitation to avoid photo-polymerization).

3. Results and discussion

Fig. 1a-h shows typical SEM images of the nano-assemblies of C_{60} nano-rods, synthesized at different temperatures. Four kinds of morphologies were observed for the samples: microtubes, microrods, nanoflowers and nanorod parallel arrays. Fig. 1a and b show SEM images of samples produced at room temperature. The images reveal that the C_{60} crystals had microtube morphologies with outer diameters in the range of 2–7 μm. A single C_{60} microtube is depicted in Fig. 1b. A detailed examination of this picture shows that the microtubes had a hexagonal cross section. Also, an incomplete microtube was found among our samples, as shown in the insert of Fig. 1b. The SEM image of this incomplete microtube appears as a bundle of nanorods with diameters of several hundred nanometers, from which we deduced that the microtubes were assembled from parallel C_{60} nanorods. For comparison, Fig. 1c and d showed C_{60} crystals obtained at 0 °C, C_{60} microrods having diameters in the range of 1 to 2 μm were fabricated at this temperature. The tips of these C_{60} microrods indicated that also the microrods consisted of several C_{60} nanorods assembled together. Fig. 1e and f show SEM images of C_{60} crystals obtained at −15 °C. Beautiful flower-like morphologies were observed for the C_{60} crystals synthesized at this temperature. Again, all the C_{60} flowers were assembled from C_{60} nanorods. In this case, the diameters of the C_{60} nanorods were about 500 nm, and their lengths were 40 to 50 μm. The diameters of the C_{60} flowers were in the range of 80–100 μm. Fig. 1 g and h, finally, show that when the reaction temperature was −40 °C, C_{60} nano-arrays consisting of C_{60} nanorods arranged in parallel were formed. The C_{60} nanorods growing in a same direction along the substrate surface had diameters ranging from 200 nm to 500 nm.

To investigate the crystal structures of the as-grown C_{60} nano-assemblies fabricated at different temperatures, XRD experiments were carried out. We show in Fig. 2 diffraction patterns obtained for samples synthesized at room temperature, 0 °C, −15 °C and −40 °C,

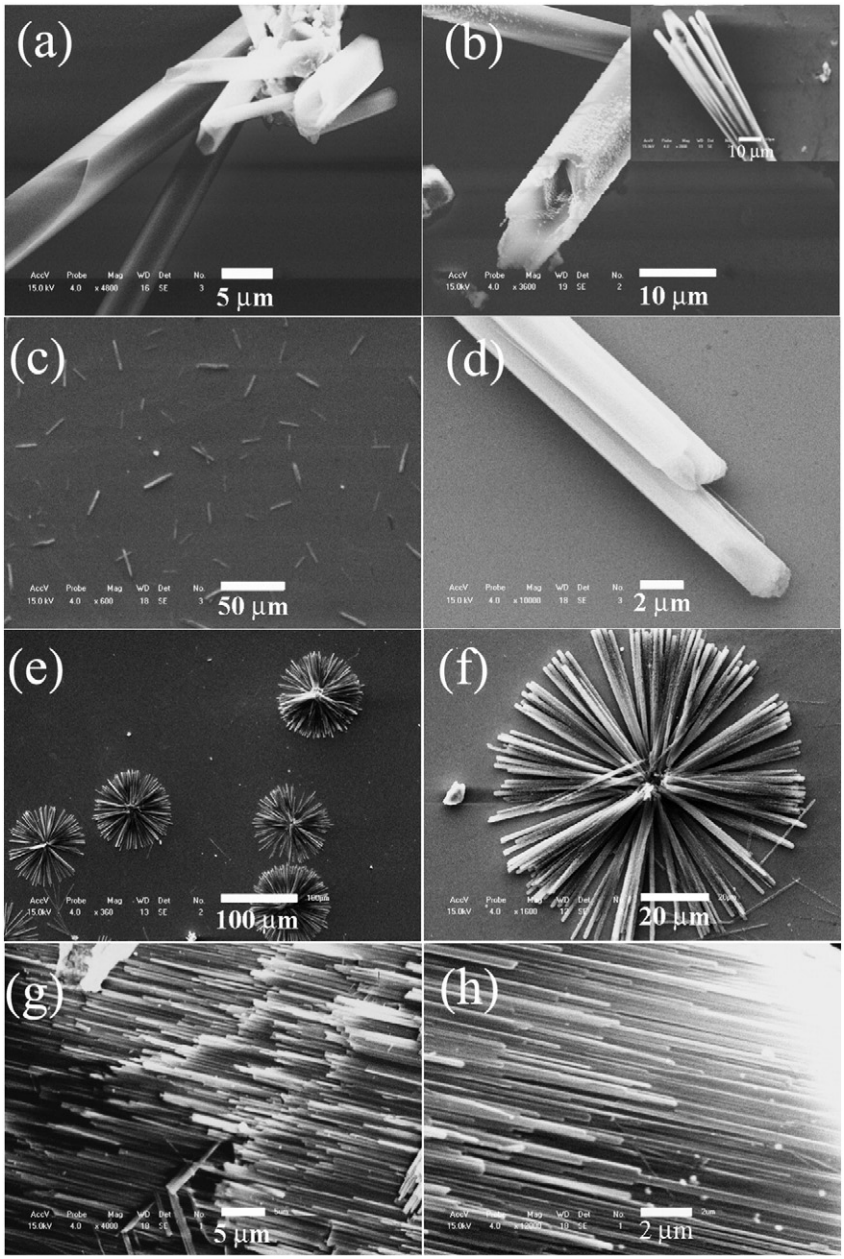

Fig. 1. SEM images of C_{60} nano-assemblies synthesized at room temperature (a, b), 0 °C (c, d), −15 °C (e, f) and −40 °C (g, h), respectively.