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Mechanical Properties of Nanometer-sized Fullerene C_{60} Whiskers Studied by *In situ* High-Resolution Transmission Electron Microscopy

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1. Introduction

Various types of structure have been reported concerning fullerenes: C_{60}, multi- and single-walled nanotubes, onion-like shells, and corns [1-7]. Mechanical properties of these fullerenes, e.g. strength and elasticity, are expected to be useful for elements of nano electromechanical systems, as addressed by simulations and experiments of collision [8-10]. Recently, Miyazawa *et al*. synthesized a different type of one-dimensional fullerene crystals; single crystalline whiskers consisting of C_{60} molecules [11-13]. By transmission electron microscopy, the authors observed the curved C_{60} whiskers, suggesting high flexibility. However, the whiskers had been curved during specimen preparation before the observation. Thus it seems to be difficult to identify origins of curved structures: it is unclear whether the curved structure is introduced as structural features during syntheses or by deformation at specimen handling. In this letter, in order to investigate the mechanical nature of the C_{60} whiskers, we observed *in situ* deformation and fracture of C_{60} whiskers by transmission electron microscopy and estimated their elastic limit.

2. Experiment

We synthesized C_{60} whiskers by a liquid-liquid interfacial precipitation method using a saturated solution of C_{60} molecules in toluene and isopropyl alcohol [11-13]. After precipitation, we dispersed the whiskers on a plate and mounted it on a specimen holder of a transmission electron microscope equipped with a piezo manipulation system [14]. A nanometer-sized silicon tip of a microcantilever, as used for atomic force microscopes, was coated with a gold film of 5 ~ 10 nm thickness. The microcantilever was then fixed on another specimen holder. Both specimen holders were inserted into the microscope. The individual C_{60} whiskers protruded from the edges of the plate were deformed with the cantilever-tip. The deformation was observed *in situ* using a TV rate system with a time-resolution of 17 ms.

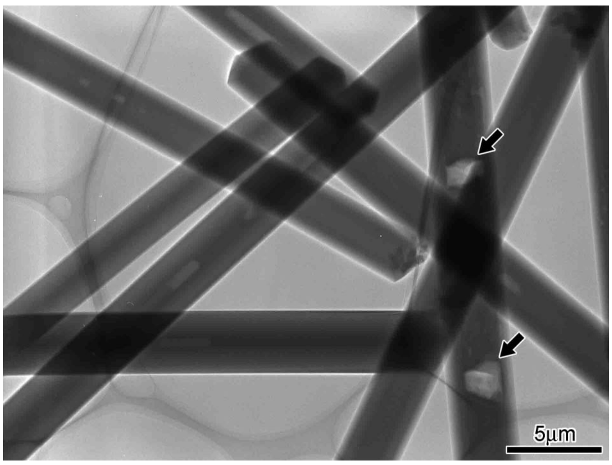

Fig. 1 Bright field image of C_{60} whiskers on a microgrid. Arrows indicate void-type defects.

3. Results and Discussion

Figure 1 shows a bright field image of C_{60} whiskers dispersed on a microgrid as used for transmission electron microscopy. The C_{60} whiskers show well-defined crystal habits: homogeneous diameters along their longer growth axes, and truncations with flat surfaces. By selected-area electron diffraction, we found that C_{60} whiskers have a body-centered tetragonal structure with lattice constants of a = 0.98 nm and c = 1.60 nm. The longer growth axes are parallel to the <100>, and the truncation surfaces are the {100} and {101}. The diameter of C_{60} whiskers ranged from 200 to 1000 nm. The length extended to submillimeters.

For analyses of deformation, we selected C_{60} whiskers

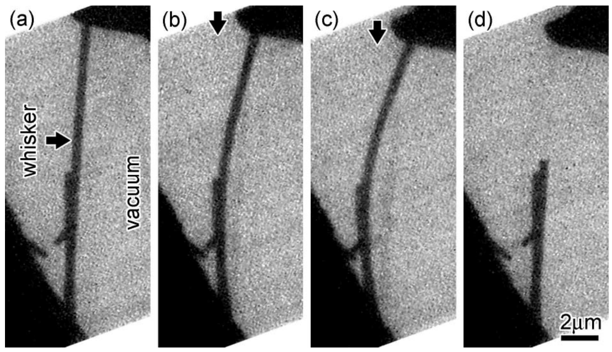

Fig. 2 (a)-(d) Time-sequential images of the deformation and the fracture of a C_{60} whisker. The diameter of the whisker is 540 nm and the effective length for deformation is 17±1 μm.

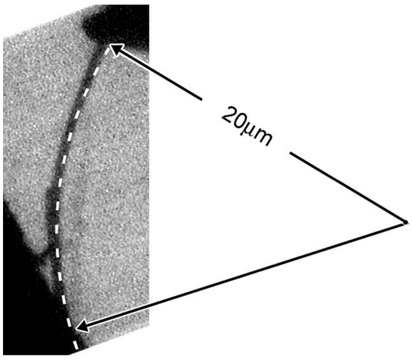

Fig. 3 Bright field images of the C_{60} whisker in Fig. 2(c).

of typical diameters around highest-counted regions from 500 to 700 nm. We also confirmed their homogeneous diameters and crystallinity: C_{60} whiskers containing defects, e.g. as indicated by arrows in Fig. 1, were not selected in this experiment.

Figure 2 shows time-sequential bright-field images of the deformation of a C_{60} whisker of 540 nm in diameter. The length of a deformed part was 17±1 μm. The dark triangular region on the top of Fig. 2 is the tip of the cantilever and the dark region at the bottom is the plate. First the whisker is supported between the cantilever-tip and the plate (Fig. 2(a)). Bight regions around the whisker are a vacuum. The cantilever-tip is displaced along the direction indicated by a bold arrow, and the whisker arches (Figs. 2(b) and 2(c)). Then we added the displacement. As a result, the whisker broke as shown in Fig. 2(d).

The minimum curvature of radius during the bending in Fig.2(c) is 20 μm as indicated in Fig. 3. We estimated strain according to the fomula,

$$\varepsilon = \frac{d}{2\rho} \qquad (1)$$

where d is the diameter of the whiskers, and ρ is the curvature of the radius. The curvature, ρ=20 μm, corresponds to a strain of 0.013.

The fracture surface was the {100} perpendicular to the growth axis. Fracture occurred promptly to analyze the procedure by the time resolution ~ 17 ms. After fracture, pieces dispersed as shown in Fig. 2(d). These results show that this fracture is brittle.

According to formula (1), the strain decreases for smaller diameter with the same curvature of radius, although formula (1) is based on continuum model and can not be applied to the diameters of a few molecules. In silicon wires, which are also one-dimensional semiconductor, similar variation in strain was reported. The fracture strain of silicon wires with micrometer width increased to 0.006-0.03 [15,16]. When the width of the silicon wires reduces to a few nanometers, the fracture strain increases up to ~ 0.3 [17]. We may expect similar improvement of the fracture strain for the C_{60} whisker due to the reduction of their diameter.

4. Conclusions

We deformed individual C_{60} whiskers and observed their arched structures. The fracture planes and nature were identified; the whisker bent first elastically, then fractured brittlely on the {100}. We estimated the fracture strain ~ 0.013 based on the continuum model.

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