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In Situ Transmission Electron Microscopy of Bending Process of Crystalline C_{60} Nanowhiskers

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1. Introduction

A new form of C_{60} single crystals, i.e., nanowhiskers (NWs), was discovered by Miyazawa *et al.* in 2001 [1]. They show a face-centered-cubic structure and have high aspect ratios of length to diameter [1-4]. Mechanical properties of the NWs have been investigated for the application to nanometer-scale functional and structural devices [5-8]. Asaka *et al.* performed buckling tests of C_{60} NWs by *in situ* transmission electron microscopy (TEM) and estimated the Young's modulus of C_{60} NWs: 28-54 GPa [6, 7]. Tubular crystalline C_{60} NWs (C_{60} nanotubes) were also examined and the Young's modulus was estimated to be 62-107 GPa [8]. For weaker impressed force, Young's modulus can be estimated accurately by bending test rather than by buckling test. In this paper, we performed the bending tests of individual C_{60} NWs by *in situ* TEM.

2. Experiment

We synthesized C_{60} NWs by a liquid-liquid interfacial precipitation method using a saturated solution of C_{60} molecules in pyridine and isopropyl alcohol [1-4]. The solution including precipitated NWs was dropped on a semicircular microgrid for TEM. The microgrid was mounted on a specimen holder of a transmission electron microscope equipped with a piezomanipulation system. A silicon microcantilever with a nanometer-sized tip was fixed onto a cantilever holder. Both specimen and cantilever holders were inserted into the microscope. Then, we deformed individual C_{60} NWs using the cantilever tip at room temperature in a vacuum of 1×10^{-5} Pa. The deformation process was observed *in situ* using a television rate system. Simultaneously, the force applied on the NWs was measured by an optical detection of the cantilever deflection.

3. Result and Discussion

Figure 1 shows a bright filed image of a C_{60} NW fixed on the edges of the microgrid. The diameter of the NW is 123 nm. The two triangles in Fig. 1 indicate the fulcra during bending, and the effective length for the bending is 1.41±0.07 μm. From selected-area electron diffraction, we found that the crystal structure of the NW is a body-centered tetragonal structure with lattice constants of a=0.910 nm and c=1.66 nm. The longer growth axis of C_{60} NWs is aligned parallel to <100>.

Figure 2 shows time-sequential bright field images of the deformation process of the NW seen in Fig. 1. The dark triangular regions in the upper part of each frame of Fig. 2 are the cantilever tip, and the dark region in the bottom is the microgrid. The brighter region is a vacuum. First, cantilever tip was placed to contact with the NW, as shown in Fig. 2(a). Then, the NW was pressed along the direction indicated by the arrow in Fig. 2(a). The NW was bent, as

Fig. 1 Bright filed image of C_{60} NW. The outer diameter is 123 nm. The two triangles indicate the fulcra during deformation.

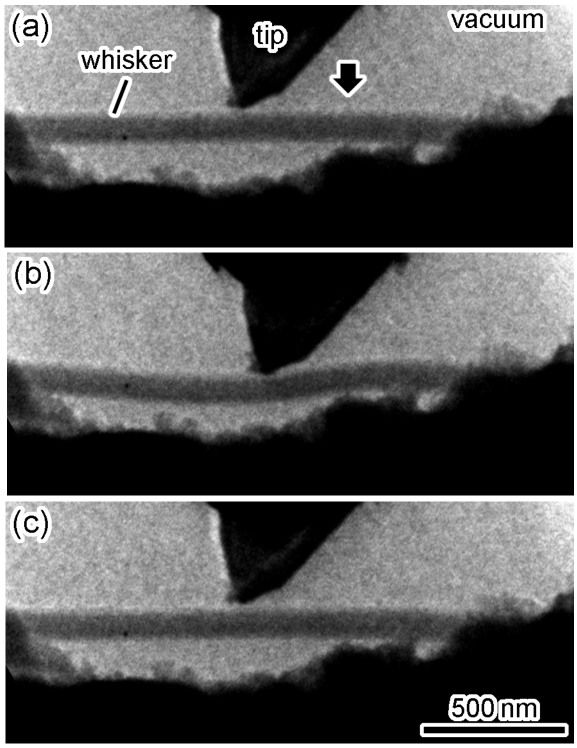

Fig. 2 Time-sequential bright filed images of bending of C_{60} NW presented in Fig. 1. The bending is an elastic deformation.

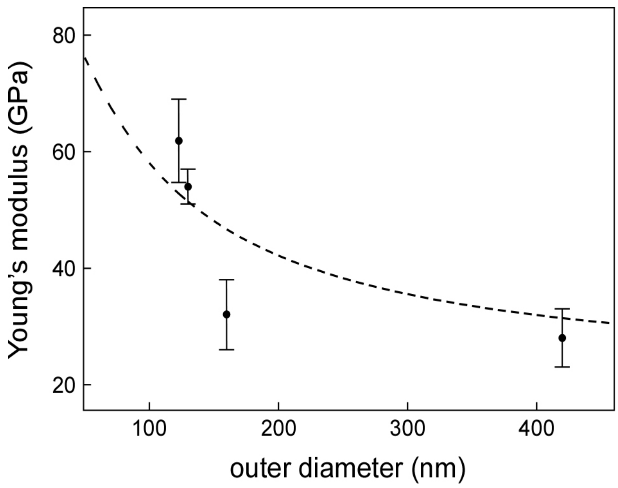

Fig. 3 Young's modulus of C_{60} NW as function of outer diameter. The dashed line represents the calculated Young modulus on the basis of a core-shell model of NW.

shown in Fig. 2(b). Subsequently, the tip was retracted, and the NW recovered its initial straight shape, as shown in Fig. 2(c). Thus, this observation indicates that the bending is an elastic deformation. This process repeated several times for the same NW.

From the *in situ* bending tests, we estimated the Young's modulus of the NW to be 53-69 GPa. This value is 305-610 % larger than that of C_{60} bulk crystals [9-12] and 72 % smaller than that of C_{60} nanotubes [8]. In Fig. 3, we plotted the Young modulus of the present NW with previously reported values [6, 7] as a function of outer diameter. In Fig. 3, the Young modulus increases as the outer diameter decreases. Miyazawa *et al*. proposed that C_{60} NWs have core–shell structures; the structure of the regions near the side surfaces is different from the inner region along the long axis [13]. Therefore, on the assumption that the Young moduli of the inner and outer regions of a NW are different, we calculated the total Young modulus of NWs as a function of the outer diameter. The calculated values are well-fitted when the wall thickness is selected to be a constant value, 10 nm, as represented with the dashed line in Fig. 3.

4. Conclusions

We performed bending tests for individual C_{60} NWs by *in situ* TEM. The Young modulus of the C_{60} NWs was estimated to be 53-69 GPa. The present result supports the core–shell structural model for C_{60} NWs.

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