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Observation of Characteristic Assembled Structure of C60

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Several kinds of assembled structures of C60 have been synthesized from solution and were observed by an electron microscopy. Metal doped C60 particles were deposited as a film on silver plates by electrolysis. Microscopic needle-like structures were observed in the films and were about 0.5 μ m in the length. The length of the particles increased as the temperature of electrolysis solution increasing. On the other hand, macroscopic needle-like structures of C60 have been assembled by an extraction from nonelectrolysis supersaturated solution. Most of samples were few mm in the length, where layered microstructures with thickness of about 30nm were observed by TEM. In a huge needle-like sample obtained by very slow extraction, ultrafine fibers with the diameter of about 50nm were also observed.
Key word: C60, needle-like structure, ultrafine fiber

1. Introduction

Since the epochal discovery of C60[1] and the development of mass production of Fullerenes[2], they have been widely studied in the fields of materials science because of high feasibilities as a new class of functional materials. Properties of Fullerenes often depended on syntheses processes as shown in the results of single crystals or thin films[3]. We have taken notice of a solution process. The purposes of this work are to observe several characteristic structures of C60 assembled from solution and to investigate their common structures. The specimens were prepared by two kinds of solution processes. The one of the processes was an electrolysis and the other an extraction from supersaturati on solution. The obtained samples were observed by an electron microscopy, SEM and/or TEM.

2. Assembled Metal Doped C60 by Electrolysis

2-1 Sample Preparation

The schematic process of the used electrolysis is shown in Fig. 1. The cell was put in a glovebox filled with N2 gas. The cell had two rooms divided by an ion-exchange membrane. Solution in the working electrode cell was 1.5mmol/l-C60 toluene. Solution in the counter electrode cell was 0.1mmol/l-RbClO4 N, N-Dimethylformamide (DMF). A black film was deposited on a silver plate of the working electrode. The temperature of electrolysis solution was changed from 10℃ to 60 ℃. The details of electrolysis conditions appeared in elsewhere[4].

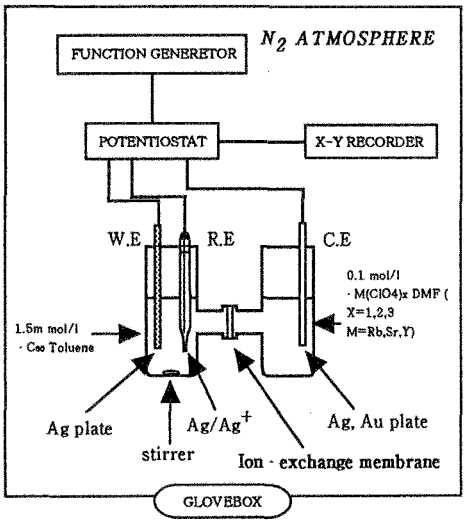

Fig. 1 The schematic process of electrolysis.

2-2 Results

Figure 2 shows the typical SEM photographs of RbC60 compounds obtained by changing the temperature of electrolysis solution Tele.

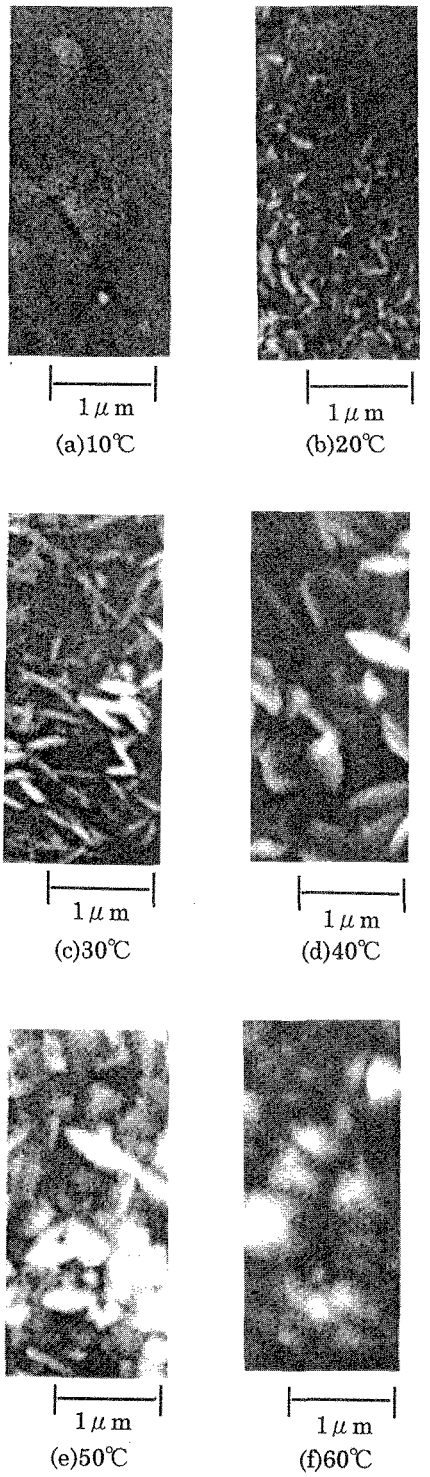

Fig.2 The SEM photographs of RbC60 compounds obtained by changing the temperature of electrolysis solution.

At comparatively low Tele's, characteristic needle-like particles were observed as a microstructure. The length of the particles was less than 0.5 μ m and increased as increasing Tele. At Tele's above 50℃ a growing mode changed and the particles became round and fat.

3. C60 Extracted from Nonelectrolysis Solution

3-1 Sample Preparation

Needle-like assembled structures of C60 were extracted from toluene solution of C60. When the solution of 50ml was dried in a day, the length of the needle was typically about few mm and the diameter was about 1 μ m as shown in Fig.3

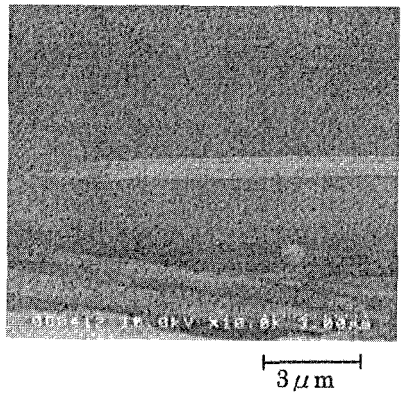

Fig.3 needle-like sample extracted by evaporation of toluene solution of C60 in a day.

Figure 4 shows the huge needle-like sample that was obtained as a result of very slow evaporation of toluene solution in a closed tube. For about six months the needle gradually grew at the inner wall of the tube near the surface of solution. The length and the diameter of the sample attained up to about 20mm and 100 μ m, respectively. The samples obtained were observed by an electron microscopy. The needle-like sample was cut into a very thin piece by a focussed ion beam etching technique and the cross section of the sample was also observed by TEM.

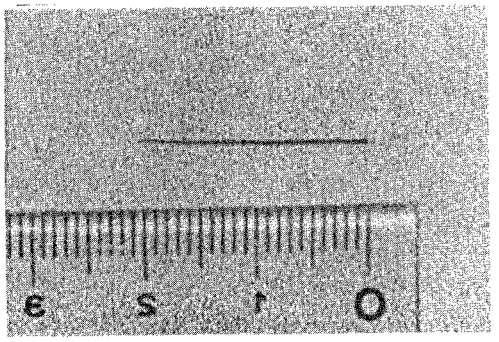

Fig. 4 The huge needle-like sample that was obtained as a result of very slow evaporation of toluene solution.

3-2 Results

Figure 5 shows the TEM photograph of the cross section of the needle-like particle. Layered structures with a thickness of about 30nm were observed. The assembled C60 was amorphous judging from a halo electron diffraction pattern. The SEM photograph of the surface of the huge needle-like sample is shown in Fig. 6. It suggested that an assembly of C60 molecules accompanying with rolled layers took place in a growth of a needle. Furthermore the smallest structure in the needle was an ultrafine fiber with the diameter of about 50nm as shown in Fig. 7. This result directly demonstrates that an extraction process from solution has a high feasibility in order to synthesize ultrafine fibers of C60 with the diameter of few tens nm.

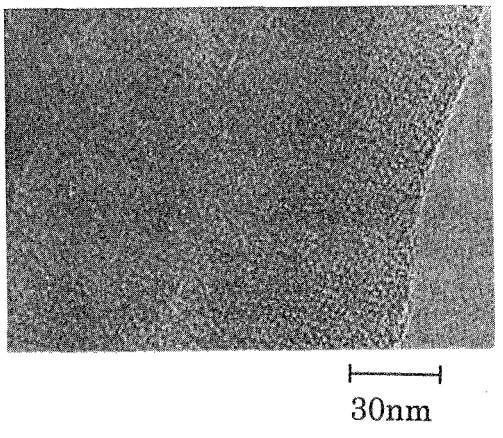

Fig. 5 The TEM photograph of the cross section of the needle-like particle.

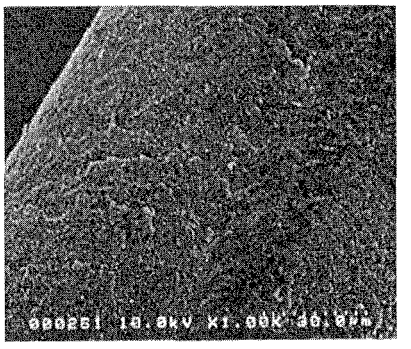

Fig.6 The SEM photograph of the surface of the huge needle-like sample.

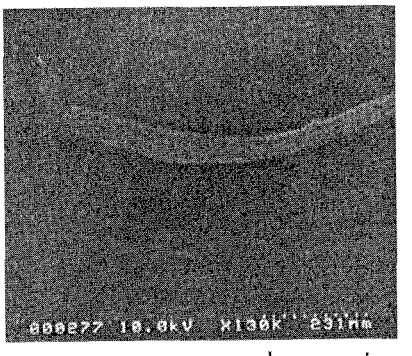

Fig. 7 Ultrafine fiber in the huge needle-like sample.

4. Summary

We have prepared assembled structures of C60 by several solution processes and observed their microstructures. As a common mode a growth of needle-like structures appeared though the size was quite different for each the synthesis conditions. A dimension of fundamental assembled C60 structures from solution was about few tens nm. Especially we observed first ultrafine fibers of C60 with the diameter of about 50nm. The result suggests that an extraction from solution is expected to be a noble process in order to obtain assembled C60 fibers with diameters of few tens nm.

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