Morphology of Heat-Treated Benzene-Derived Carbon Fiber

by
A.Yoshida and Y.Hishiyama
Musashi Institute of Technology, 1-28-1 Tamazutumi, Setagaya-ku, Tokyo,
and
M.Endo
Faculty of Engineering, Shinshu University, 500, Wakasato, Nagano,
Japan

Benzene-derived carbon fiber (BVCF) has been grown by pyrolysing a mixture of benzene and hydrogen at about 1100°C (1). The diameter of the fiber is typically about 10 μm and the length is generally in the range 10-25 cm. The most characteristic structural features of the fibers are an annular ring-like structure of carbon layer planes concentrically set around the fiber axis and a high degree of graphitization (2,3). Morphology study was carried out by a scanning electron microscope (SEM) as a function of heat treatment temperature (HTT) (4). It was found that the annual ring-like structure of the cross section of the as-prepared fibers is retained throughout the heat treatments up to 3000°C, and the external morphology is subjected to significant change with the heat treatment above 2200°C. For the fibers heat treated above 2800°C, characteristic polygonal appearances, unlike the cylindrical forms of the fibers heat-treated to lower temperatures, were observed(4,5).

Recently, a high resolution SEM which operates at lowest acceleration voltage of 200 V for the primary electron beam and the acceleration voltage can be adjusted stepwise up to 50 kV has been developed commercially. This kind of SEM is convenient for observation of the surface of carbon materials, because the diffusion depth of the primary electron beam for carbon materials is very deep. The depth is estimated to be 3 μm at the acceleration voltage of 15 kV (6). The deep diffusion depth reduces substantially the resolving power determined from the acceleration voltage.

The proper acceleration voltage for carbon materials determined experimentally ranges between 2 and 5 kV. Using this new type of SEM, morphology of BVCF has been reexamined in the present study. In addition to the specimens heat-treated between 1100°C and 3000°C, the specimens heat-treated to 3500 °C have been newly studied, which was done kindly by Dr. A . W. Moore of UCC.

Good cross sections of the filaments of BVCF heat-treated above 2600°C are hardly obtained by conventional cutting. Cutting of the bundles of these fibers were made in liquid nitrogen by a sharp razor blade. SEM used in the present study was JEOL JCXA 840. Observations were made with the acceleration voltage of 2 kV for the primary electron beam. No pre-treatment was made for the observations.

SEM photographs for the BVCF are shown in Photos. 1-18. The bald top and the annual ring-like structure of the as-grown fiber are retained throughout the heat treatments. Well developed graphite layers are seen for the specimens heat-treated to temperature above 2200°C. The heat treatments cause modifications of the annular ring-like structure. The specimens heat-treated to 2200°C show long hollows inside and a rope-like texture on the surface, formed by the abrupt decrease in the average interlayer spacing d_{002} , i.e. the d_{002} value decreases from 3.420 Å to 3.387 Å with increase of HTT from 2000°C to 2200°C (2). The specimen heat-treated to 2800°C has a polygonal appearance. The polygonization occurs drastically as heat treatment progresses. The specimen heat-treated to 3000°C shows polygons in deep inside of the fiber. Polygons develop further by heat treatment to 3500°C. The core of the fiber formed in the as-grown stage remains even after the heat treatment to 3500°C. The core may be a cylindrical hollow tube as observed in the early stage of the fiber growth (3).

Acknowledgements

The authors would like to thank to Prof. M.S.Dresselhaus of MIT and Dr. A.W.Moore for their kind discussions and high temperature heat treatment.

References

1. T.Koyama, Carbon 10, 757 (1972).
2. T.Koyama, M. Endo and Y. Hishiyama, Japan. J. Appl. Phys. 13, 1933 (1974).
3. A. Oberlin, M. Endo and T. Koyama, J. Cryst. Growth, 32, 335 (1976).
4. M. Endo, Y. Hishiyama and T.koyama, J. Phys. D: Appl. Phys. 15, 353 (1982).
5. T.C.Chieu, G.Timp, M.S.Dresselhaus, M.Endo and A.W.Moore, Phys. Rev. B,27,3686(1983).
6. R. Castaing, Adv. Electronics and Electron Phys.

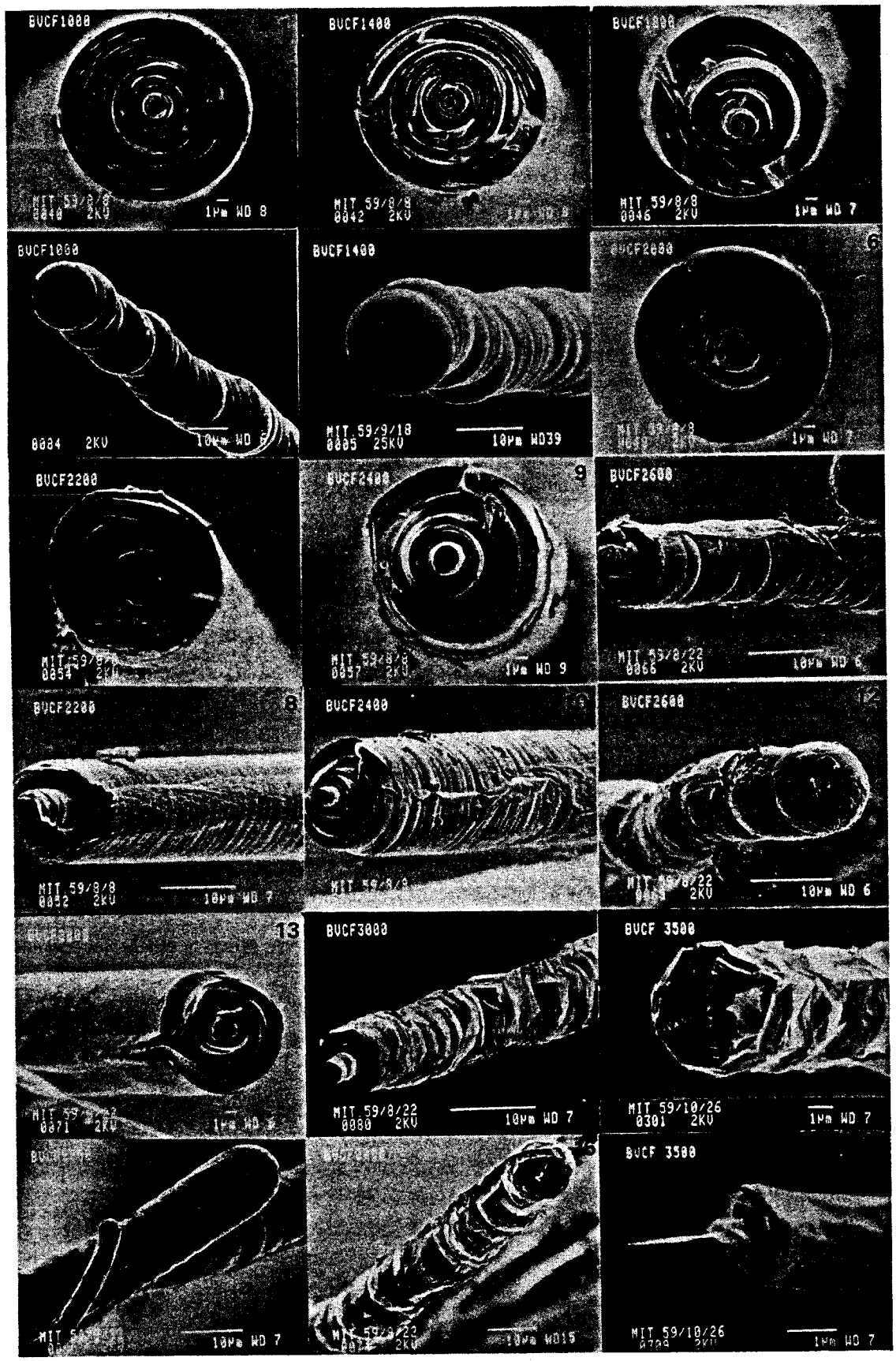

Fig. 1 SEM photographs showing the morphology change on the surface and the cross-section of the benzene-derived vapor-grown carbon fibers heat treated to various temperatures; (1)(2)as-prepared,(3)(4)1400°C,(5)1800°C,(6)2000°C (7)(8)2200°C,(9)(10) 2400°C, (11)(12) 2600°C, (13)(14)2800°C, (15)(16) 3000°C,(17)(18) 3500°C.