STRUCTURE AND GROWTH OF CARBON NANOFIBRES BY THERMAL CHEMICAL VAPOUR DEPOSITION

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Introduction

The synthesis of carbon nanofibres (carbon filaments) by pyrolysis of carbon monoxide over an iron catalyst has been known since the early 1950's [1]. Subsequent to the first report of the arc discharge synthesis of carbon nanotubes by Iijima in 1991 [2], there have been a number of other high temperature growth techniques reported, such as laser vaporisation [3], pyrolysis [4] and plasma-enhanced chemical vapour deposition (CVD) [5]. Jose-Yacaman and co-workers [6] concluded that catalytic methods can produce carbon nanotubes with similar characteristics to that of the carbon-arc method described by Iijima [2]. CVD has recently attracted interest as the best method for growth of carbon nanotubes or nanofibres with high hydrogen-uptake capacity [7], and for atomic force microscopy tips [8].

Experimental

Carbon nanofibres have been produced in our laboratory by CVD using acetylene and Ni powder at temperatures ranging from 500 to 700 ^{o}C and at pressures of the order 10^{-2} mbar. Approximately 50 mg of nickel powder, with 4-7 μm diameter of the particles, was dispersed in an uniform thin layer on the top of a graphite plate substrate. Typically, experiments were run for 30 min. Characterisation of the material has been undertaken by both scanning electron microscopy (SEM) and transmission electron microscopy (TEM). SEM was performed using a Hitachi S 4000 microscope and TEM using Philips CM 200 microscope. Suitable TEM specimens were prepared by ultrasonic dispersion of the sample in isopropanol, and the drop of the suspension was allowed to dry on the thin amorphous carbon film supported on a copper grid.

Results and Discussion

SEM and TEM examination have shown that diameter of the carbon nanofibres is distributed in the range of 20 to 100 nm with lengths from 1 to 5 μm. Nickel catalyst particles have been observed at the end of the carbon nanofibres, as well as in the middle of the carbon nanofibre (Fig 1). Diameter of the nanofibres is approximately same as that of the nickel embedded nickel catalyst particle. The nanofibres grew with random paths forming loops, spirals and interconnecting networks.

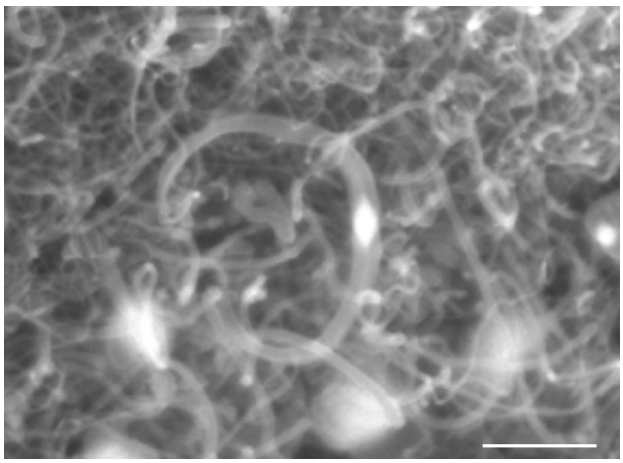
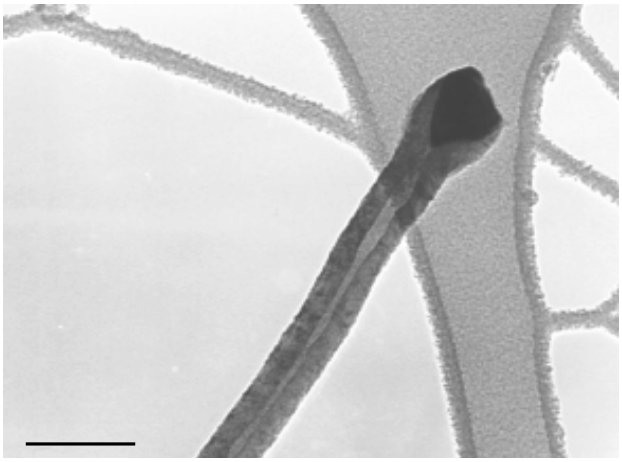


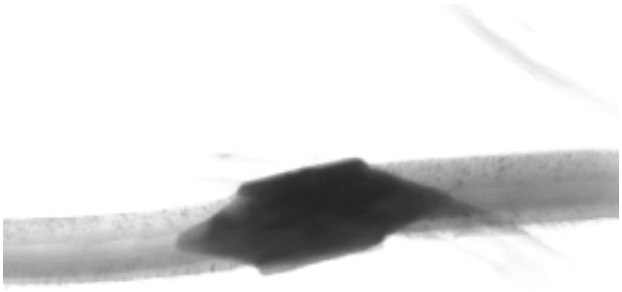
Fig. 1. SEM micrograph of carbon nanofibres grown by thermal CVD process using acetylene. Large bi-directional carbon nanofibre with diamond shape catalyst particle can be seen in the center.

Active catalyst particle is considerably smaller than those of the original metal powder, which indicates that during the reaction fragmentation of the starting material, occurred prior to carbon nanofibre formation. Convenctional whisker structure with the catalytic particle at the tip of the nanofibre is presented in the Fig 2a, and bi-directional carbon nanofibre structure in Fig 2b.

TEM studies have shown that nanofibres have a duplex structure, an outer graphitic sheet surrounding an amorphous core (Fig 2). Graphitic sheets of the carbon nanofibre are parallel to the cone shaped Ni catalyst particle forming "herring-bone" structure (Fig. 3). Towards middle and the end of the nanofibre, these graphitic sheets becoming more and more parallel to the nanofibre axis.



(a)



(b)

Fig. 2. TEM micrographs of carbon nanofibres: (a) "whisker-like" with pear shaped catalytic particle, (b) bi-directional with catalytic particle in the middle.

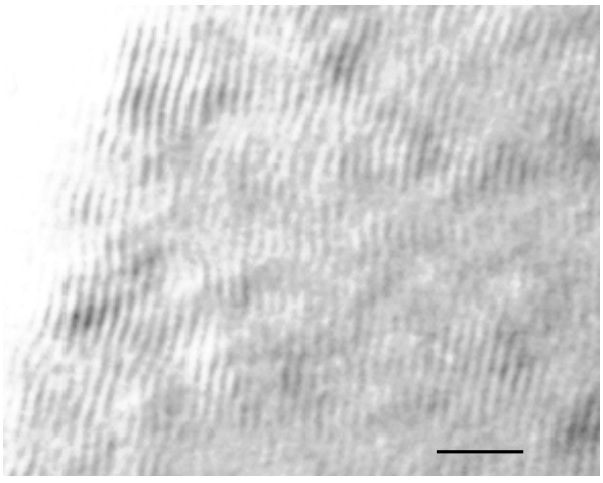


Fig. 3. Enlarged part of the carbon nanofibre showing carbon lattice fringe images almost parallel to the surface of the nanofibre.

Conclusions

The characteristics of the grown carbon nanofibres found to be mainly determined by the temperature of the reaction. Maximum thickness of nanofibres and the best graphite layers order in the carbon nanofibre skin were achieved at 600 ^{o}C. For the optimal conditions relatively high yield of the produced carbon nanofibres has been observed, based on SEM and TEM observations. Bi-directional growth from a diamond shaped particle in the middle of the carbon nanofibre has been observed more often when acetylene was used, comparing to the carbon nanofibres grown in similar conditions using methane as carbon source gas [9]. Structure and morphology of produced carbon nanofibres are very similar to the structure and morphology of the material obtained by the Rodriguez and Baker's group, which has remarkable hydrogen storage capacity [7]. The equipment needed for the CVD growth of the carbon nanofibres is very simple and can be easily modified for continuos production, with metal catalyst and hydrocarbon gas as only row materials. Therefore, CVD is very promising method for the production of carbon nanofibres on the large scale.

References

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