

Production Process of Carbon Nanotube Coagulates

カーボンナノチューブ微粒子のプラズマ合成過程

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Control of production process of carbon nanotubes is urgent and important theme. Here, production process of carbon nanotubes by the arc-discharge method is in-situ monitored by the laser Mie-scattering method. As a result we can measure specio-temporal distribution of size and density of nanotube coagulates in the arc reactor. In this process the strong heat convection modifies the flow and cooling rates of the carbon coagulates. In case of arc production of carbon clusters, in-situ monitoring would be important.

1. Introduction

Carbon nanotubes (CNTs) have big potential of applications as new functional material. However, production of long defect-free nanotubes, control of chirality and selective production of metal-nanotubes are still challenging themes. In order to develop the production method, in-situ monitoring of the production processes is necessary, though the cluster size in the reaction gas phase is in the range of nm. Figure 1 shows a model figure of the production process of single-walled carbon nanotubes by the arc discharge method, in which CNTs grow in the hot gas phase. [1]

Not only CNTs, metallo-fullerenes, which has potential application to contrast-media of MRI, copper doped CNTs for the use of nano-scale cables, [2] metal-capsulated carbons for the use

of nano-sized metal powders. Nano-sized diamond particles have been developed by Osawa group of Shinsyu Univ. and started to sell. [3]

Watanabe and Shiratani have developed the in-situ monitoring method of silicon nanoparticles during plasma processings by silane gas, so called the Mie scattering method. [4, 5] They successfully obtained specio-temporal distributions of silicon particles in the discharge space, and control the production and diffusion of silicon particles.

Here by the similar method, production process and diffusion process of nanotube coagulates in the arc plasma reactor are investigated. [1, 6, 7] Scattered laser light by particles at two scattering angles are simultaneously measured and compared with the theoretical values. [8] Then we can confirm average size and density of the carbon nanotubes coagulates.

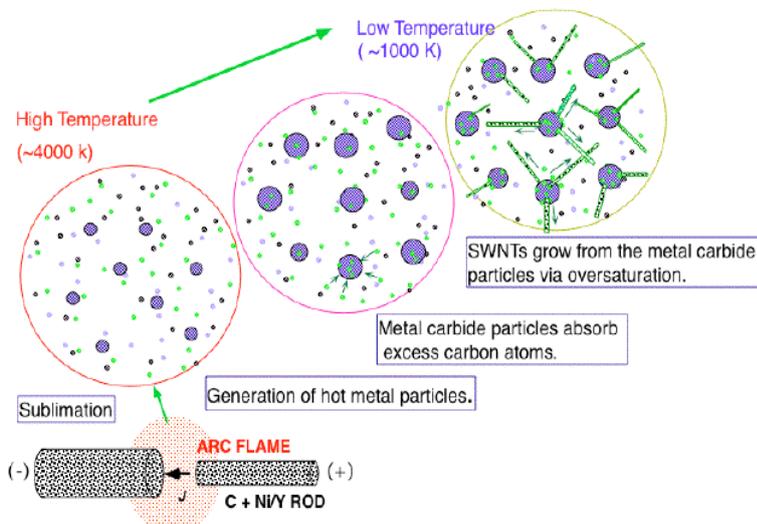


Fig. 1 A model figure of production process of CNTs by the arc discharge method.

2. Experimental

Schematic of the reactor is shown in Fig. 2, where a 6.5 inch diam., 270 mm high (volume of 1.8 L) metal chamber is used. A square-type carbon anode and a column-type cathode are set under 0.5 atm of He gas. A pulse-modulated green laser ($\lambda = 532$ nm) is penetrated from the bottom side to the top side through the center of the arc plasma. Along the laser axis (z axis), scattered lights with scattered angles of 15 deg. and 90 deg. are detected using optical fibers, and averaged by using lock'in amplifiers.

3. Results and discussion

Figure 3 shows vertical distribution

of estimated cluster size and density over the arc center under the gravity-free condition. The cluster size d starts to grow and the cluster size increases from $z=1$ cm (above the arc plasma), where discharge current $I_d=40$ A and He pressure $p=40$ kPa.

Figure 4 shows pressure dependence of estimated cluster size and density at $z=2$ cm for the normal-gravity condition. By the convection effect, the cluster size decreases by increasing He pressure, though the cluster size is kept almost constant.

Figure 5 shows discharge current dependence of estimated cluster size and density at $z=2$ cm for the normal-gravity condition. The cluster size decrease by increasing the current, while the cluster density gradually increases with the current.

The cluster size increases under the zero-gravity, higher-pressure, higher-discharge current or at the large z position. Under the normal gravity, the cluster size becomes smaller, and the heat-convection strongly influences the cluster growth. These results are consistent with the theoretical prediction. Development of the laser Mie-scattering method and precise control of the arc discharge method are under investigation.

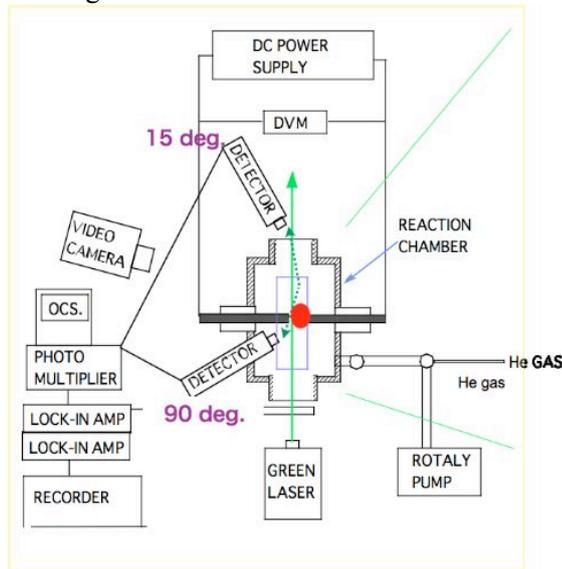


Fig. 2 Schematic of the experimental setup.

Acknowledgment

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References

- [1] T. Mieno & M. Takeguchi, New Diamond & Frontier Carbon Technol. **16** (2006) 139.
- [2] A. Koshio, Converttech **37**, No. 4 (2009) 102 (in

Japanese).

- [3] Please visit “<http://nano-carbon.jp/>”.
- [4] M. Shiratani, S. Matuo, Y. Watanabe, Jpn. J. Appl. Phys. **30** (1991) 1887.
- [5] Y. Watanabe, J. Phys. D: Appl. Phys. **39** (2006) R329.
- [6] T. Mieno & M. Takeguchi, J. Appl. Phys. **99** (2006) 104301.
- [7] T. Mieno, J. Jpn. Soc. Microgravity **26** (2009) 72 (in Japanese).
- [8] C. F. Bohren & D. R. Huffman, “Absorption and Scattering of Light by Small Particle”, John Wiley Sons, 1983.

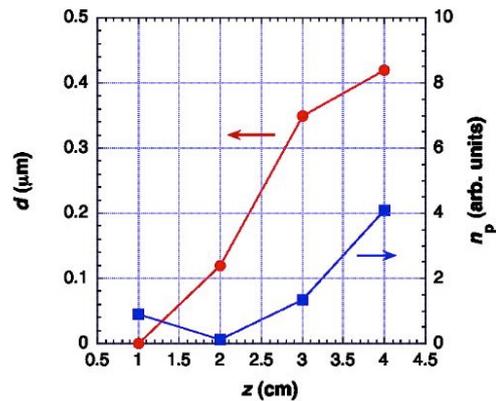


Fig. 3 Estimated cluster diameter d & cluster density n_p under gravity-free condition vs. scattering position over the arc center. $I_d=40$ A $p=40$ kPa.

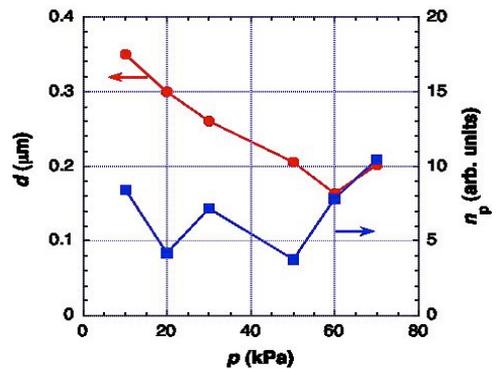


Fig. 4 Estimated cluster size & density vs. He pressure. Under normal gravity condition. $I_d=40$ A, $z=2$ cm.

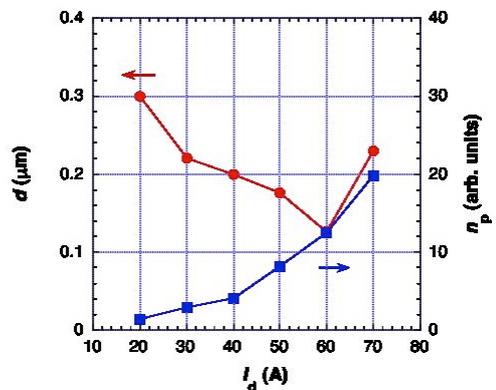


Fig. 5 Estimated cluster size & density vs. discharge current under normal gravity condition. $p(\text{He})=40$ kPa, $z=2$ cm.