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Spherical cauliflower-like carbon dust formed by interaction between deuterium plasma and graphite target and its internal structure

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ABSTRACT

Simulated experiments to produce carbon dust particles with cauliflower structure have been performed in a liner plasma device, NAGDIS-II by exposing high density deuterium plasma to a graphite sample (IG-430U). Formation of carbon dust depends on the surface temperature and the incident ion energy. At a surface temperature 600–700 K, a lot of isolated spherical dust particles are observed on the graphite target. The internal structure of an isolated dust particle was observed with Focused Ion Beam (FIB) system and Transmission Electron Microscope (TEM) in detail. FIB analysis clearly shows there exist honeycombed cell structure with thin carbon walls in the dust particle and the dust particle grows from the graphite surface. TEM image also shows that the dust particle is made of amorphous carbon with crystallized grains with diameters of 10–50 nm.

1. Introduction

Formation of carbon dust in fusion devices becomes one of the most important subjects mainly because of safety issues associated with tritium inventory. Various kinds of dust particles have been observed in fusion devices [1–6], however, the formation mechanism has not been fully understood yet. Carbon dust particles are considered to be generated mainly from re-deposited carbon layers formed on cold remote material surfaces. On the other hand, in the previous paper [7], simulated experiments to generate carbon dust particles on a graphite surface have been conducted in a linear plasma device, NAGDIS-II [8] to find that a lot of spherical carbon dust particles can be generated at the graphite facing high density hydrogen plasmas. However, the mechanism of the carbon dust formation at plasma-facing graphite target is not clear yet.

In order to reveal how and where the isolated spherical carbon dust particles are generated, we have investigated dependence of the dust formation on the graphite surface temperature and an incident energy of deuterium ions to the graphite surface. Moreover, characterization of the internal structure of an isolated spherical carbon dust was performed with focused ion beam (FIB) system and transmission electron microscope.

2. Experimental set-up

Experiments have been carried out in NAGDIS-II [8]. High density deuterium plasma above $10^{19}\ \mathrm{m}^{-3}$ can be generated in steady state with an improved dc discharge system consisting of two anodes. Typical electron temperature measured with a single Langmuir probe is about 10 eV. The plasma density of the discharge can be controlled by changing the dc discharge current. The magnetic field strength is 0.25 T. Fig. 1 shows the experimental set-up. The diameter of the plasma column is about 20 mm. High grade isotropic graphite samples (IG430U, Toyo Tanso Co. Ltd.) of hexagonal shape with a side length of 28 mm are mounted on the water-cooled copper target located at the end of the vacuum chamber. The graphite target is inclined to a magnetic filed by 45°. The graphite target can be biased negatively with respect to the vacuum chamber to control an incident energy of deuterium ions to the graphite target. Surface temperature on the graphite target is measured with an optical pyrometer through a quartz window. Characterization of carbon dust was performed by means of a scanning electron microscope (SEM). Detailed analysis of internal structure of an isolated carbon dust particle on the graphite target was performed with focused ion beam (FIB) system and transmission electron microscope (TEM).

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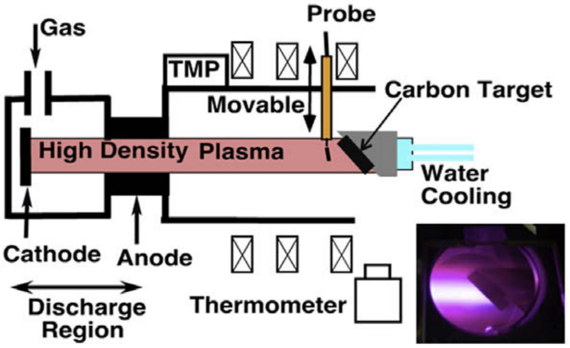

Fig. 1. Schematic arrangement of the experimental set-up.

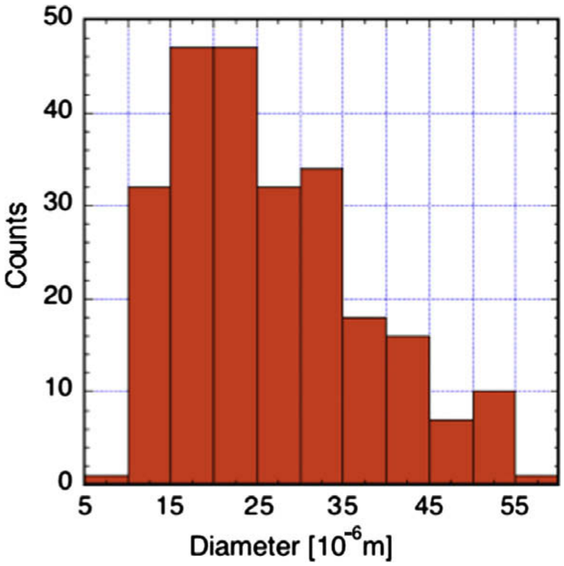

Fig. 3. Size distribution function of dust particles calculated from Fig. 2(a).

3. Experimental results and discussion

Fig. 2(a)–(f) shows SEM photographs taken at the central region of the graphite surface irradiated by high density deuterium plasma at different surface temperature. The incident ion energy is about 10 eV, where the incident ion energy to the graphite target is determined by a potential difference between the target potential and the plasma one measured with the single Langmuir probe. The exposure time is 7200 s (2 h). In the experiment corresponding to Fig. 2(a), at beginning, the surface temperature of the sample set to 600 K. However, the surface temperature was gradually increased during plasma irradiation although the sample was mounted on the water-cooled copper target, and finally reached 700 K at the end of the experiment. It is found that many spherical carbon dust particles are formed on the graphite surface with widely varying sizes, as shown in Fig. 2(a). Fig. 3 shows the size distribution function of dust particles calculated from Fig. 2(a). The average size of the dust particles and the standard deviation is 26.6 μm and 10.7 μm, respectively. The largest size is about 50 μm.

Fig. 2(b), which is a magnification of Fig. 2(a), shows that the large carbon dust particle looks like cauliflower with an agglomeration of sub-micron sized small particles. At a surface temperature of 800–1000 K, a number of the dust particles decreases dramatically as shown in Fig. 2(c) and (d), probably because chemical erosion becomes large in this temperature range. Further increase of the surface temperature around 1100 K leads to no formation of isolated dust particles, but the surface modification with agglomerated dust particles (Fig. 2(e) and (f)).

When the incident deuterium ion energy is increased to be 49.3 eV by biasing the graphite target, no carbon dust is generated as shown in Fig. 2(g) and (h) even if the surface temperature and ion fluence are almost same as those in Fig. 2(a). These experimental results suggest competition between erosion and re-deposition of carbon species, which depend on surface temperature and incident ion energy, is a key process to grow the carbon dust particles.

We made holes with a diameter of 1mm on the same graphite sample as shown in Fig. 4(e). Fig. 4(a)–(d) shows SEM photographs taken near the hole at the central region of the graphite surface. In both Fig. 4(a) and (c), spherical dust particles are generated around the hole. In Fig. 4(b), it is found that carbon dust is formed even at a sidewall inside the hole facing deuterium plasma. On the other hand, there is no dust at the opposite sidewall where no plasma is irradiated as shown in Fig. 4(d). This result indicates that carbon impurities (and/or carbon dust) are transported to the graphite surface due to plasma flow to generate carbon dust.

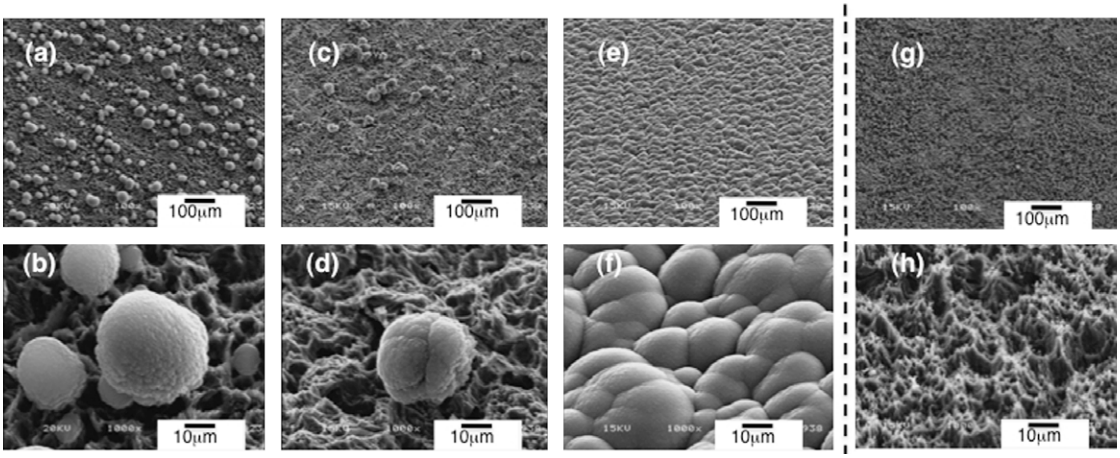

Fig. 2. SEM photograph showing the carbon dust formation on the graphite target irradiated by a deuterium plasma. Exposure time is 7200 s. (a) Surface temperature $T_s \sim 600$–700 K, incident ion energy $E_i \sim 13.4$ eV and ion fluence $\Phi \sim 3.4 \times 10^{26}$ m^{-2}, (c) $T_s \sim 800$–1000 K, $E_i \sim 8.0$ eV and $\Phi \sim 9.5 \times 10^{26}$ m^{-2}, (e) $T_s \sim 1100$–1200 K, $E_i \sim 14.8$ eV and $\Phi \sim 7.7 \times 10^{26}$ m^{-2}, (g) $T_s \sim 600$–750 K, $E_i \sim 49.3$ eV and $\Phi \sim 3.0 \times 10^{26}$ m^{-2}. Figs. (b), (d), (f) and (g) are magnifications of (a), (c), (e) and (g), respectively.

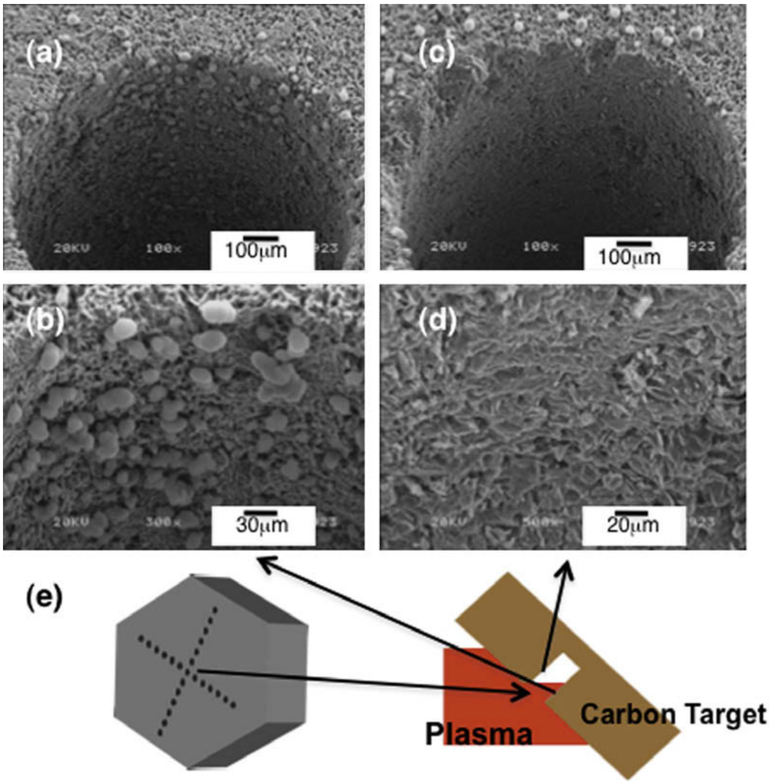

Fig. 4. SEM photographs showing the carbon dust formation near the hole ((a) and (c)). (b) at a sidewall in the hole facing plasma, and (d) at an opposite sidewall without plasma irradiation.

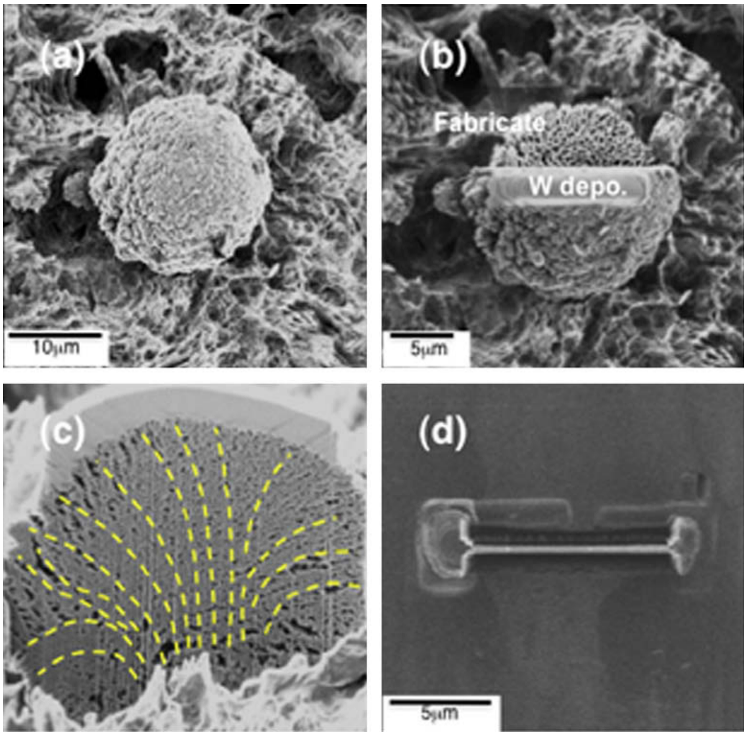

Fig. 5. SIM photographs of a carbon dust particle taken by FIB system.

Focused ion beam (FIB) system is widely used for cross-section observation of semi-conductors in industrial applications. Fig. 5 shows observation of a spherical carbon dust particle in terms of FIB system. Fig. 5(a) shows the scanning ion microscope (SIM) image of a dust particle observed in Fig. 2(a) before the FIB fabrication. After deposition of W on the dust particle, a half of the dust particle is etched by focused gallium ion beam until the equatorial plane. It is found that there exists honey-combed cell structure

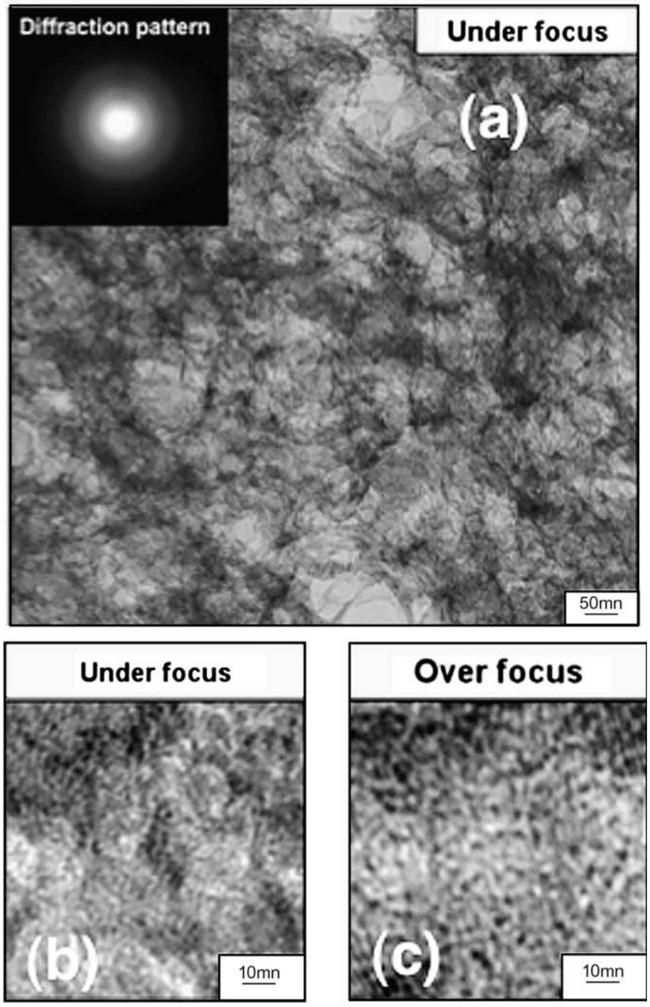

Fig. 6. TEM photographs of cross-section of the carbon dust particle in Fig. 5. Inset of Fig. (a) shows diffraction pattern of electron beam.

with thin carbon walls in the dust particle. From this image, huge amount of deuterium gas could be retained in volume of the cell structure. We are preparing to measure the deuterium retention in the dust particles by thermal desorption spectroscopy (TDS) method. After further milling of the dust particle, we observed its cross-section as shown in Fig. 5(c). This photograph clearly shows the dust particle has no concentric structure, and the dust particle radially grows upward from the graphite surface. In general, spherical dust particles are considered to be generated in gas phase, but this observation indicates that even spherical dust particles can be formed on graphite target.

More detailed observation of the cross-section of the dust particle was performed by TEM analysis. TEM sample with a thickness of 200 nm is fabricated with the FIB system as shown in Fig. 5(d). Fig. 6 shows TEM photographs of the sample. Electron beam diffraction pattern shown in the inset of Fig. 6(a) is ring shape, indicating that the thin honey-combed wall in the dust particle is fundamentally amorphous structure. However, the wall is not fully amorphous one, because the TEM image in Fig. 6(a) shows that there are crystallized grains with a size of 10–50 nm randomly oriented. Fig. 6(b) and (c) shows high spatial resolution TEM images with different focus. Both TEM images show fine structures with a size of 1–2 nm also exist in the crystallized grains. From these observations, it is concluded that honey-combed carbon walls inside of the dust particle is amorphous structure, having the 10–50 nm crystallized gains made of 1–2 nm fine structures.

4. Conclusion

Carbon dust formation on plasma-facing components has been investigated in the NAGDIS-II linear divertor plasma simulator. Formation of dust particles strongly depends on surface temperature of the graphite target and incident ion energy. Isolated spherical carbon dust particles were formed at surface temperature 600–700 K and incident energy around 10 eV. Inside structure of a carbon dust particle was observed with FIB system for a first time. SIM image shows that the dust particle has honey-combed fine structure with thin carbon walls. TEM image and electron beam diffraction pattern indicate that carbon nano-size-walls in the dust particle are made of amorphous carbon with crystallized grains with diameters of 10–50 nm.

The experimental result, indicating the possibility of carbon dust formation near the strike point on carbon divertor tiles in the fusion devices, gives an impact on the estimation of carbon dust production rate in such as ITER.

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