Carbon Dust Formation under Heavy Atomic Hydrogen Irradiation

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Dust formation mechanism in plasma-material surface interactions has been investigated by using high pressure inductively coupled plasmas (ICPs), which have a feature of high atomic particle flux (atomic hydrogen flux : $\sim 10^{24}$ m⁻²s⁻¹, ion flux : $\sim 10^{21}$ m⁻²s⁻¹). Experiments have been conducted in argon/hydrogen mixture plasma irradiation to graphite targets. In the present experiments, physical sputtering is not expected and the dominant erosion process of graphite target is chemical sputtering by atomic hydrogen irradiation. Carbon dust particles with various shapes have been observed on the graphite target irradiated by argon/hydrogen plasma. It is found that the shapes of the dust particles are strongly related to the target surface temperature, graphite spherical particle when the surface temperature is below ~1100 K and polyhedral particle like diamond when above ~1100 K. It is also shown that the carbon dust formation and growth does not choose the surface materials. The number density of dust formed decreases as the plasma input power decreases or the distance between the target and induction coil increases, while the weight loss remains almost the same even though the input power and distance between the target and induction coil are varied. The size of dust particles increases as increasing surface temperature. These experimental results indicate that the dust growth is strongly related to surface temperature.

Keywords: Carbon dust, Plasma-surface interaction, Diamond

1. Introduction

Dust particles are formed by plasma-surface interactions and transported into core and scrape off layer (SOL) plasmas in fusion experimental devices[1]. Carbon materials are used for the plasma facing components (PFCs) in fusion devices and carbon fiber reinforced composites are chosen as ITER divertor plate materials because of their superior thermo-mechanical properties. In the fusion devices using carbon PFCs, carbon materials not only are eroded significantly by hydrogen plasma but also generate many carbon dust particles. Since carbon dusts retain large amounts of hydrogen, the dust particles in fusion reactors bring several safety problems, such as tritium inventory and explosion at an accidental air leakage. The control of dust formation is an important issue in future fusion reactors. However, the mechanism of dust formation in fusion reactors has not been well understood.

In this paper, we have investigated dust formation mechanism by using high power inductively coupled plasmas (ICPs) [2].

2. Experimental setup

Experiments have been conducted by using high power ICPs [3]. High power ICPs have characteristic features, such as high particle flux (atomic hydrogen flux : $\sim 10^{24}$ m⁻²s⁻¹, ion flux : $\sim 10^{20}$ m⁻²s⁻¹), and high heat flux (~1 MW/m²). Argon gas flow rate is 60 slpm, and the injection rate of hydrogen gas into argon plasma is 0, 2, 5, 8 slpm. Although the working gas pressure is high (~4 kPa), these plasma features are very helpful to study dust formation. Figure 1 shows schematic diagram of plasma irradiation system. Three graphite targets (IG-430U, Toyo Tanso Co. Ltd.) with a diameter of 15 mm are placed at different positions. In addition, the preliminary experiments have been conducted by placing silicon crystal target at the position 2 in order to investigate the influence of the target surface material. Irradiation time is kept at 180 minutes. Axial position of the induction coil to generate ICP is movable and the distance between the target and induction coil-end is changed every from 300 mm to 420 mm with a step size of 30 mm. Figure 2 shows ion flux measured with a Langmuir probe at 10 mm above target 1. The ion flux decreases as increasing

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the injection rate of hydrogen gas and/or the distance between the target and induction coil. Surface temperature of graphite targets is not actively controlled, but can be varied by changing the plasma input power and/or position of the induction coil. Surface temperature is measured with a radiation thermometer through a quartz window. After plasma irradiation, graphite targets have been collected and their weights been measured to have the mass change of the graphite target before and argon/hydrogen plasma irradiation. Scanning after Electron Microscope (SEM) is used to observe the generated dusts on the targets.

3. Experimental results and discussion 3.1 Dust shape and particle size distributions

Since the ion impact energy at the target is lower than 4-5 eV and the ion flux to the target is $3 \times 10^{17} - 5 \times 10^{20}$ $m^{-2}s^{-1}$, which is three orders lower in magnitude than that of hydrogen atoms, the dominant erosion process of graphite target in the present experiments must be chemical sputtering by atomic hydrogen irradiation. Carbon dust particles are formed on the graphite surface even in the erosion dominant region. There are no dust particles on the graphite surface irradiated by pure argon plasma. Figure 3 shows SEM photographs of typical shapes of carbon dust particles obtained from graphite surfaces by argon/hydrogen plasma irradiation. Many dust particles with size from sub-micron to ~30 µm are

1E21 L=300 mm (a) 330 mm 1E20 360 mm on Flux [m⁻²s⁻ 390 mm 420 mm 1E19

4

1E18

1E17└ 0

2

observed on the graphite target. It is found that carbon dust particles stick strongly on the graphite surface, and are not peel off easily by some vibration and/or wind. Figure 3(a) shows that the spherical carbon dust particle has a granular surface. Figures 3(b) and (c) show agglomerated particles composed of several individual small spherical particles of 2-3µm and cluster of very small particles, respectively. The flake shown in Fig. 3(d) is probably a piece of graphite layer. The shapes of dust



Schematic diagram of plasma irradiation to graphite Fig. 1 targets







SEM photographs of carbon dust particles observed on graphite targets. The shapes of the dust are (a) ball-type, (b) and (c) Fig. 3 cluster-type, and (d) flake-type.

particles are classified into three typical shapes which are ball-type (Fig. 3(a)), cluster-type (Fig. 3(b) and (c)) and flake-type (Fig. 3(d)), where dust of cluster-type consists of two or more particles.

The size distributions of dust particles are obtained by sizing and counting dust particles with 20 SEM photographs at a magnification of 500. The dust particles with the size of submicron range cannot be counted in the present experiments. Figure 4 shows typical size distributions of dust particles on the target irradiated by argon/hydrogen plasma, where argon and hydrogen gas flow rate are 60 slpm and 2 slpm, respectively, at different distances L between the target and induction coil. Size distributions of dust particles on the target 1 and 2 irradiated by argon/hydrogen plasma at L = 300 mm are shown in Fig. 4(a) and (b). Figures 4(a) and (b) show that most of the dust particles are ball-type particles. The average size of the dust particles become small and their density decreases at the target 2, which is away from the main irradiation region. No dust particles are formed on the target 3. Size distributions of dust particles on the target 1 and 2 irradiated by argon/hydrogen plasma at L =420 mm are shown in Fig. 4(c) and (d). The plasma density and target surface temperature decreases by moving the induction coil away from the target. The shape and density of dust formed changes drastically at target position 2. On the other hand, the weight loss remains almost the same. The proportion of ball-type particles in the dust formed is low in the target of position 2 as shown in Fig. 4(d). The average values of diameter and density for ball-type particles in Fig. 4(d) are ~ 2.1 μm and $\sim 10 mm^{-2}$ respectively.

On the crystal silicon surface placed at the position 2, the dust particles, which are similar to the dust formed on graphite surface in shape and size, are observed. This result indicates that the carbon dust particles are formed not only on graphite surface, but also on silicon surface.

3.2 Surface temperature dependence of carbon dust shape, size, and number density

It is found that the shape of the dust particles is strongly related to the target surface temperature T_s , graphite spherical particles shown in Fig. 3(a) when T_s is below ~1100 K and polyhedral particles like diamond





Fig. 4 Size distribution of carbon dust particles on target surfaces. (a) and (b) ((c) and (d)) are obtained from the target 1 and 2 irradiated by argon/hydrogen plasma at the distance between the target 1 and induction coil of 300 mm (420 mm). Surface temperature T_s , weight loss M_{loss} , average diameter D_{ave} , and average density N_{ave} are shown in the figure, respectively.



Fig. 5 SEM photographs showing the carbon dusts of polyhedral shape like diamond observed on the target 1 when the target surface temperature T_s is above ~1100 K.



Fig. 6 (a) Density and (b) diameter of carbon dust particles observed on target 1 and (c) weight loss corresponding to (a) and (b) as a function of surface temperature. Surface temperature is controlled by changing the plasma input power, position of the induction coil and/or hydrogen gas flow rate.

anges from spherical shape to polyhedral shape are observed as shown in Fig. 5(b) and (c).

Size and density of dust particles are key factors in studying the mechanisms of dust formation and growth. Figures 6(a) and (b) shows the density and diameter of carbon dust particles as a function of T_s on the target 1, respectively. The weight loss corresponding to Fig. 6(a) and (b) is shown in Fig. 6(c). The number density of dust particles in Fig. 6(a) does not contain flake-type particles. Both the ion flux to the target and surface temperature decrease as the plasma input power decreases or the distance between the target and induction coil increases or hydrogen gas flow rate increases as shown in Fig. 2. The weight loss of target 1 at $T_s \sim 800$ K is almost same as that at $T_s \sim 1000$ K. However, the number of dust particles decreases significantly at $T_{\rm s} \sim 800$ K. Both the number of dust particles and weight loss decrease at $T_s > 1000$ K. On the other hand, the density of cluster-type particles d-



Fig. 7 SEM photographs of the graphite surface (b) without water cooling as shown in (a) and (c) with water cooling irradiated by argon/hydrogen plasma. Surface temperature T_s , ion flux Γ_i at 10 mm above the target, weight loss M_{loss} , and average value of number density of dust particles N_{ave} are shown in the figure.

oes not depend on the surface temperature and the proportion of cluster-type particles increases when T_s is below ~900 K. Figure 6(a) shows that almost all of the dust formed are ball-type particles at $T_s > ~900$ K. Moreover, it is found that the size of the dust increases as increasing the surface temperature. These experimental results indicate that the dust growth is strongly related to surface temperature.

The chemical erosion of graphite target depends on the surface temperature and atomic hydrogen flux rather than the hydrogen ion flux in the present experiments. The hydrocarbon particles generated by chemical erosion are dissociated by the surrounding plasma. It is considered that plasma parameters and surface temperature influence the dust formation and growth. In order to investigate which influences strongly the growth of carbon dust, plasma parameters or surface temperature, experiments have been conducted by argon/hydrogen plasma irradiation to the targets without water cooling as shown in Fig.7(a). The input power and hydrogen gas flow rate were almost the same as in the case that the surface temperature of the target 1 with water cooling is ~830 K. The experimental result is shown in Fig. 7(b). Surface temperature reached ~1050 K. The number density of carbon dust is one order larger in magnitude than that at T_s ~830 K. This result indicates that the surface temperature plays an important role in the dust formation in the present experiments.

Homogeneous nucleation and dust growth to the size of submicron may occur in the plasma[1,4]. Figure 8(b) shows that spherical dust particle consists of many small particles of submicron size. From this observation and surface temperature dependence of dust size, it can be considered that the dust growth to a large size exceeding 1 μ m occurs on the material surface.

4. Conclusions

Dust formation mechanisms have been studied by irradiating argon/hydrogen plasma to graphite targets by using high pressure ICPs. Carbon dust particles are formed on the graphite surface even in the region that chemical erosion is dominant rather than the deposition. When the surface temperature is above ~900 K, many dust particles with the size larger than 1 µm are observed on graphite surfaces. The carbon dust particles are also observed on the crystal silicon surface. The carbon dust shape depends strongly on the target surface temperature. Carbon dust shape changes from a randomly-shaped dust composed of small particles to a large spherical dust as increasing the surface temperature of the graphite target. Moreover, increasing the surface temperature up to 1100 K turns the graphite spherical particles into polyhedral particles like diamond crystal. It is found that the number density of dust particles formed decreases as the surface temperature decreases from 1000 K to 800 K by changing the plasma input power, position of the induction coil and/or hydrogen gas flow rate. These experimental results indicate that the dust growth, especially size and structural formation, is strongly related to the surface temperature.



Fig. 8 SEM photographs showing the carbon dust cluster. The surface temperature is ~900 K. Bottom picture is a magnification of (a).

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