

Characterization of Dust Particles Ranging in Size from 1 nm to 10 μm Collected in the LHD

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We collected dust particles ranging in size from 1 nm to 10 μm from the Large Helical Device employing two methods: an ex-situ filtered vacuum collection method and an in-situ dust collection method. The size distribution from 1 nm to 10 μm is well expressed by the Junge distribution. Dust particles are classified into three kinds: small spherical dust particles below 1 μm in size, agglomerates consisting of primary particles of 10 nm, and large dust particles above 1 μm in size and irregular in shape; this suggests three formation mechanisms of dust particles: chemical vapor deposition growth, agglomeration, and peeling from walls. In-situ collection shows that agglomeration between dust particles takes place in main discharges. The primary dust particles in agglomerates are around 10 nm in size, suggesting agglomeration between a negatively charged large agglomerate and a positively charged dust particle 10 nm in size. We have also confirmed the important fact that a large number of dust particles move during vacuum vent. Therefore, the in-situ dust collection method is needed to reveal the generation-time and -processes of dust particles and their deposition position during discharges.

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1. Introduction

Of late there has been growing concern over the formation of dust particles due to plasma-surface interaction [1–3], because dust particles pose two potential problems: first, those remaining in a fusion device are dangerous, as they can contain a large amount of tritium and can explode violently; second, they may lead to deterioration of plasma confinement. Therefore, it is important to reveal their formation mechanism and transport as well as their accumulation area. Investigation of dust in fusion plasma research devices has been carried out using ex-situ dust sampling methods [1–3]. Moreover, most such studies focused on dust particles around a μm in size. Our group at Kyushu University reported formation of carbon dust nano-particles due to interaction between electron cyclotron resonance hydrogen plasmas and carbon walls [4, 5]. This report motivates us to study small dust particles in the Large Helical Device (LHD). For this purpose, we employed two methods: one is the ex-situ filtered vacuum collection method just after an LHD campaign, and the other is an in-situ dust collection method during the campaign (the duration of main discharges or glow ones). In this paper, we report experimental results regarding characterization of dust particles ranging in size from 1 nm to over 10 μm collected from the LHD.

2. Experimental

Ex-situ collection of dust particles was carried out just after the completion of the seventh campaign of the LHD in February, 2004; of the eighth campaign in February, 2005; and the ninth campaign in February, 2006. Although ex-situ collection was performed at 20 locations on the first wall made of stainless steel (SS316) and on the divertor made of carbon (IG-430), the surface mass density, size distribution, and compositions of dust particles do not clearly depend on location. Dust particles were collected on a TEM-mesh as well as on a capillary pore membrane filter having pores 100 nm in diameter using the filtered vacuum collection method [2, 3]. At each location, dust particles on a surface area of 100 cm^2 were collected.

In-situ collection of dust particles was carried out during the ninth and tenth campaigns. For this collection, 14 pieces of Si substrate 10 \times 15 mm^2 in size were placed at a first wall using the movable sample stage [6] at the 4.5 L port of the LHD during main discharge plasmas of H₂ (600 s total duration, 247 shots, the ninth campaign in November, 2005, and 300 s total duration, 200 shots, the tenth campaign in October, 2006) or during glow discharges of H₂, He and Ne (120 h total duration, the ninth campaign in February, 2006, and 30 h total duration, the tenth campaign in February, 2007).

Then, the size and shape of dust particles were ob-

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tained with a scanning electron microscope (SEM) and a transmission electron microscope (TEM). Their mass was measured with a precision balance. Their compositions were analyzed with an energy-dispersive X-ray spectroscope (EDX).

3. Results and Discussion

The surface mass density of dust particles was obtained by the ex-situ collection method. The surface mass density after the seventh campaign is scattered in a range from 0 to 22 mg/m² and its average value is 9.8 mg/m². The average values after the eighth and ninth campaigns are 3.5 and 2.8 mg/m², respectively, which are much less than that after the seventh. The inner surface areas of the LHD chamber without and with ports are 400 m² and 700 m², respectively. Assuming an area of 400 m², the total dust inventories after the seventh, eighth, and ninth campaigns are estimated to be 3.9, 1.4, and 1.1 g, respectively. The total inventory of dust particles collected from the LHD after the fourth campaign in March 2001 is 16.2 g [2]. Therefore, the LHD becomes cleaner during its operation history and the total dust inventory after the ninth campaign is the lowest among the values for several devices such as JT-60U [3].

Ex-situ and in-situ collection reveals the existence of three kinds of dust particles: small dust particles, agglomerates of small ones, and large dust particles. The typical density ratio among them is 10⁵: 600: 5. Figure 1 displays typical images of a small dust particle, an agglomerate, and a large dust particle, respectively. Figure 2 shows the EDX spectra of (a) a small dust particle, (b) an agglomerate, and (c) a large dust particle. Au is the coating material for SEM observation. The small dust particles below 1 μm in size are spherical, and their major constituent is C, which is the divertor material. The major component of agglomerates is also C. Large dust particles above 1 μm in size are irregular in shape and consist mainly of Fe and Cr, which are the materials of the first wall.

Figure 3 shows a typical size distribution of dust particles after the eighth campaign and the cumulative percentage of surface area and volume of dust particles. There exist a large number of small dust particles less than 1 μm in size. The smaller the dust particles are, the higher their density is. The size distribution is different from the log-normal distribution of average size around 10 μm for dust particles collected from the LHD in March, 2001 (the fourth campaign) [2]. There are two possible reasons for the difference: samples were observed with a TEM applicable to dust 1-30 nm in size and a SEM applicable to dust above 30 nm in size in this study, whereas samples in March 2001 were observed with the SEM in [2]; the number of hydrogen discharge shots is larger than that in [2]. Here, the experimental size distribution is fitted using an inverse power law size distribution function (the Junge distribution, which is often employed to fit a size distribution

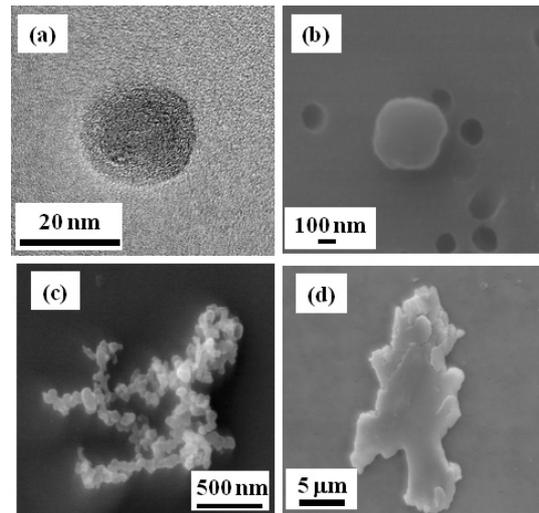


Fig. 1 (a) TEM image and (b) SEM image of a small dust particle, (c) SEM image of an agglomerate of small dust particles, and (d) SEM image of a large dust particle. All particles were collected by the vacuum-filtered collection method.

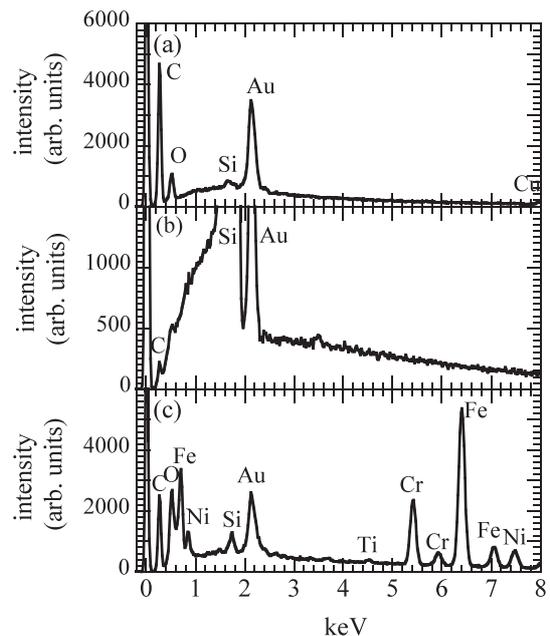


Fig. 2 EDX spectra of (a) a small dust particle, (b) an agglomerate, and (c) a large dust particle. All particles were collected by the vacuum-filtered collection method.

of atmospheric aerosols [7]) and a log-normal distribution function [8]. The Junge distribution is given by

$$\frac{dN}{dD_p} = c_j \times D_p^{-\beta}, \quad (1)$$

where, N , D_p , and c_j are the number density of dust particles, their diameter, and the proportional constant, respec-

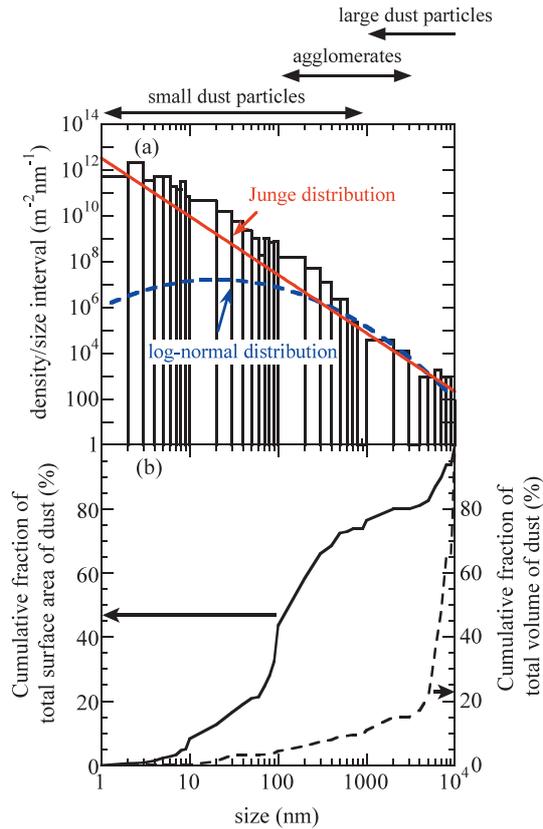


Fig. 3 (a) Typical size distribution of dust particles obtained after 8th campaign together with Junge distribution (solid line) and log-normal distribution (broken line), and (b) cumulative percentage of surface area and volume of dust particles.

tively. The log-normal distribution is obtained as

$$F(D_p) = c_1 \times \frac{1}{\sqrt{2\pi} D_p \sigma} \exp \left\{ -\frac{[\log(D_p) - \mu]^2}{2(\sigma)^2} \right\}, \quad (2)$$

where c_1 , μ , and σ are the proportional constant and the mean and geometric standard deviation of $\log(D_p)$, respectively. Using $c_1 = 1.0 \times 10^9 \text{ m}^{-2} \text{ nm}^{-1}$, and $\mu = 2$ which corresponds to $D_p = 100 \text{ nm}$, and $\sigma = 0.55$, the fitted lines of the Junge and log-normal distributions are obtained as the solid and the broken line, respectively, in Fig. 3 (a). The experimental size distribution is well expressed by the Junge distribution, suggesting that small dust particles grow into large ones in the LHD. Small dust particles of 1 nm to $1 \text{ }\mu\text{m}$ in size have 70% of the total surface area, while they have 10% of the total volume, as shown in Fig. 3 (b). These small dust particles cannot be ignored, because such particles may contain a large amount of tritium due to their wide surface area in future fusion devices such as International Thermonuclear Experimental Reactor (ITER).

The three kinds of dust particles (small dust particles, agglomerates, and large dust particles) suggest three formation mechanisms: chemical vapor deposition (CVD) growth, agglomeration, and peeling from walls. The size

ranges of these dust particles are 1 nm to $1 \text{ }\mu\text{m}$ for small dust particles, 100 nm to a few μm for agglomerates and over $1 \text{ }\mu\text{m}$ for large dust particles. The size distributions of small dust particles and agglomerates are expressed by the Junge distribution, while that of large dust particles is not. By the in-situ collection, agglomerates were collected during the main discharges and they were not during the glow discharges, indicating that results obtained by in-situ collection provide information about the generation-time and -processes of dust particles. The primary particles of agglomerates have a nearly constant size around 10 nm . The dust particles 10 nm in size have the highest probability of being positively charged, whereas those above 30 nm in size are charged negatively [9]. Agglomeration between a negative large agglomerate and a positive small dust particle takes place in the main discharges. We have also confirmed the important fact that a large number of dust particles move during vacuum vent. Therefore, the in-situ dust collection method is needed to reveal the size distribution of dust particles before vacuum vent and their generation-time and -processes, as well as their deposition position during discharges.

4. Conclusions

Dust particles in the LHD were collected using ex-situ and in-situ dust collection methods. Dust particles are classified into three kinds: small spherical dust particles below $1 \text{ }\mu\text{m}$ in size, agglomerates consisting of primary particles of 10 nm , and large dust particles above $1 \text{ }\mu\text{m}$ in size and irregular in shape; this suggests that there are three formation mechanisms: CVD growth, agglomeration, and peeling from walls. There exist a large number of small dust particles below $1 \text{ }\mu\text{m}$ in size. The smaller the dust particles are, the higher their density is. The in-situ dust collection method is needed to reveal the generation-time and -processes of dust particles and their deposition position during discharges.

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- [1] J. Winter, *Plasma Phys. Control. Fusion* **40**, 1201 (1998).
- [2] J.P. Sharpe, A. Sagara, V. Rohde, The ASDEX-Upgrade Experiment Team, H. Suzuki, A. Komori, O. Motojima and The LHD Experimental Group, *J. Nucl. Mater.* **313&316**, 455 (2003).
- [3] J.P. Sharpe, K. Masaki, A. Sagara, P.W. Humrickhouse, C.H. Skinner, the NSTX Team, T. Tanabe, N. Miya and the JT-60U Team, *J. Nucl. Mater.* **337&339**, 1000 (2005).
- [4] K. Koga, R. Uehara, M. Shiratani, Y. Watanabe and A. Komori, *Proc. ESCAMPIG16/ICRP5*, I-173 (2002).
- [5] K. Koga, R. Uehara, Y. Kitaura, M. Shiratani, Y. Watanabe and A. Komori, *IEEE Trans. Plasma Sci.* **32**, 405 (2004).
- [6] N. Ashikawa, K. Kizu, J. Yagyū, T. Nakahata, Y. Nobuta, K.

- Nishimura, A. Yoshikawa, Y. Ishimoto, Y. Oya, K. Okuno, N. Miya, T. Hino, S. Masuzaki, A. Sagara, N. Ohyabu and LHD Experimental Group, *J. Nucl. Mater.* **363&365**, 1352 (2007).
- [7] C.E. Junge, *Air Chemistry and Radioactivity* (Academic Press, 1963) pp.113.
- [8] R.M. Bethea, B.S. Duran, T.L. Boullion, *Statistical Methods for Engineers and Scientists, 3rd Ed.* (Marcel Dekker Inc., 1995) pp. 49-62.
- [9] Y. Watanabe, M. Shiratani, H. Kawasaki, S. Singh, T. Fukuzawa, Y. Ueda and H. Ohkura, *J. Vac. Sci. Technol.* **A14**, 540 (1996).