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Plasma influence on tungsten powder

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Abstract

Modifications of tungsten powder comprised of micro particles with dimensions: 1 ± 0.2 µm and 5 ± 1.5 µm (“small“ and «large” particles) under the influence of heating, electric field and hydrogen- and argon ion irradiation are investigated in this work. The processes in irradiated powder are described and discussed. Among them there are powder outgassing, particle emission from the powder surface in the electric field, pasting of small particles all over the large ones, integration of the adhered small particles and formation of the uniform layer around the groups of large particles, cone growth on uniform layers, formation of volumetric chains of sticking together tungsten particles and their transformations. Driving forces and processes providing different types of powder modifications and the role of each of them in the specific phenomena are discussed.

Keywords: dust particles; thermal release; tungsten.

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1. Introduction

Here introduce the paper, and put a nomenclature if necessary, in a box with the same font size as the rest of the paper. The material eroded due to plasma irradiation from the surface of the plasma facing components of fusion devices is finally transformed into dust. Appearance of heavy atoms in the core plasma increases the irradiative losses thus negatively influencing plasma parameters. Dust accumulation in the vessel is considered as a critical issue for ITER. The accumulation of tritium in the dust will lead to the increase in the tritium in-vessel inventory. A

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number of papers were devoted to the investigations of dust formation and their behavior in fusion devices. Features and mechanisms of dust formation were considered by Makhlaj et al. (2013), Krasheninnikov et al. (2013), Rosanvallon(2009) and others, dust migration and the influence of dust on fusion device performance were analyzed by Krasheninnikov et al. (2011), Litnovsky et al. (2013), Coenen et al. (2015) and others, dust levitation and behavior in plasma by Hong et al. (2013), and so on. The metal dusts (Ca, Na, Mg, etc.) behavior in some technological processes was described by Jaworek et al. (2007).

At the same time detailed investigation of plasma interaction with dust layers, has not been fulfilled yet. Evaluation of peculiarities and mechanisms of plasma-dust interaction can help to overcome the negative consequences of dust presence in plasma and fusion devices. The paper investigates behavior of tungsten powder under various influences, including peculiarities of powder outgasing during heating, powder particles emission in electric field, powder modifications under hydrogen- and argon ion irradiation. Driving forces and processes providing observed phenomena are discussed. The powder particle dimensions, the ranges of powder temperature, electric field, ion irradiation were selected to be comparable to corresponding parameters of plasma-dust interaction in fusion devices described by Kurnaev et al. (1995), Phillips et al. (2003), Balden et al. (2013), Rohde et al. (2013), Denkevits et al. (2005), Pitts et al. (2013) and others.

2. Experimental

Experiments were performed in the device schematically shown in Fig.1 (a). Residual gas pressure in the device did not exceed $P \leq 1.5\times10^{-4}$ Pa, pressure of the working gas was $P \leq 1$ Pa (hydrogen) and $P \leq 10^{-1}$ Pa (argon). Hydrogen or argon plasma was ignited in the cylindrical plasma chamber between the heated tungsten cathode and anode. Plasma ions were drawn off from plasma through a ring shaped opening in the anode and were accelerated towards thin tantalum plate (substrate) aligned parallel to anode and biased negatively. The measurements showed that hydrogen plasma consisted mainly of H_2^+ ions (82-85%), and the remaining part was represented by H_1^+ and H_3^+. Thus, the experimental results were analyzed assuming sample irradiation solely with H_2^+ ions. For powder irradiation with hydrogen atoms the effect of hydrogen molecule dissociation on the heated cathode was used.

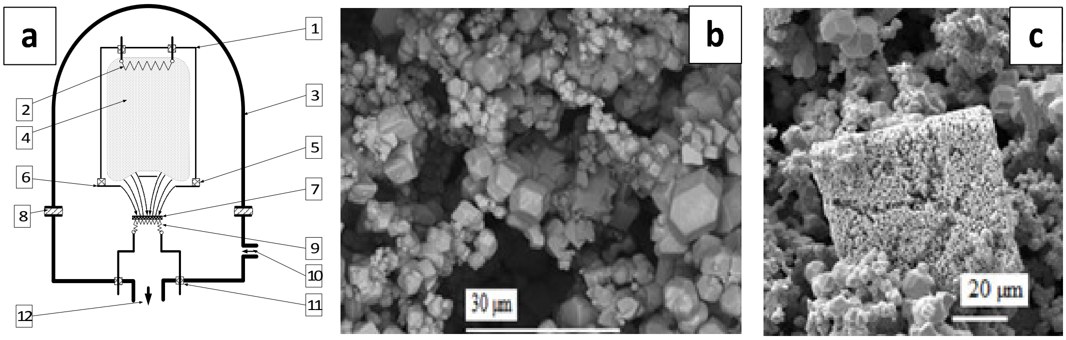

Fig. 1. (a) The scheme of experimental device: 1-Plasma chamber; 2-heated cathode; 3-vacuum chamber; 4-gas discharge area; 5,11-insulator; 6-anode; 7-irradiated substrate with powder layer; 8-vacuum sealing; 9 – heater; 10-gas puff line; 12- pump line. The agglomerations of the particles of the virgin powder: (b) three dimensional chains of the large particles; (c) the clusters with geometrical forms of the small particles.

For the experiments a tungsten powder (≥ 99.5 W; 0.2 Mo; 0.12 ($O+H_2O$) and 0.18 others) was used. The powder was obtained by reduction of tungsten anhydride in a hydrogen atmosphere and consisted of two main fractions of crystalline micro particles with mean dimensions 1 ± 0.2 µm and 5 ± 1.5 µm (hereafter: “small” and “large” particles). The dimensions of powder particles well correlate with those of dust particles (3 ± 1 µm) in fusion devices. The ratio of the amount of “large” particles to that of the “small” ones equals ≈ 1/4.

The first measurements have shown that the thickness of the powder layer including more than 5-10 layers of big particles has no influence on its behavior. Thus, experiments were performed with 2÷3 mm-thick powder layer placed on the substrate. It was convenient to operate with massive powder layers and to observe the details of investigated phenomena. The powder was heated indirectly by heating substrate with direct current. According to Pitts et al. (2013) the irradiation power density of the ITER divertor tiles will reach ≈10 MW/m^2 and will heat its surface up to ≈ 1400 K. The temperature of dust particles on the tile surface could be higher due to low thermal conductivity between the tile and the dust layer. The temperature range of investigation was selected so that to be in relation to the powder condition in the ITER. The gases released during powder heating were analyzed by thermal desorption spectrometry (TDS) in the MICMA device described by Airapetov et al. (2011).

3. Results and discussion

Scanning electron microscope images revealed certain agglomerations in the virgin powder. Large particles assembled as three-dimensional chains with some amount of small particles around them (Fig.1b). In some areas small particles organized clusters shaped geometrically with distinctive dimensions 40-60 µm (Fig.1c). Sometimes, they surrounded the parts of the chains. Appearance of these clusters may be assigned to the action of interatomic forces providing attraction of contacting particles.

If the powder was heated slowly (≈ 5 K/s), the powder outgassing took place in the temperature range 550-850 K. The TDS measurements revealed that emitted gas consisted predominantly of H_2O molecules. Comparative analysis of their TDS spectra from both tungsten powder and bulk tungsten showed that during powder heating H_2O molecules were released from the layer of the surface sorption. It was approximately 5.3×10^{18}mol./g corresponding to ≈ 5×10^{19} mol./m^2. If the temperature ramp rate was ≥100 K/s, powder did not have enough time for outgasing in the temperature range mentioned above. Thus, pulse desorption occurred at higher temperatures pushing out upper layers of powder.

Electric field exceeding 350 V/mm, caused vacillation of powder surface; the particles left the surface and flew toward the anode. The emission continued practically with constant rate, and 2 mm thick powder layer was removed from the substrate within 8-10 minutes. Intensity of the particle emission was approximately equal for outgassed and not outgassed powder and was independent of electric field direction. The rate of emission increased with the increase of electric field. Emission was stopped above temperature ≈1300 K.

If the electric field influence was accompanied by hydrogen atom irradiation particle emission started at about 300 V/mm. Under ion (hydrogen or argon) irradiation, particle emission was observed at lower electric field and depended on ion current density. Emission started at 290, 260, 220 V/mm, for 10, 30, 50 A/m^2 accordingly.

Emission of the powder particles and their movement occurred as a result of particle charging in the electric field. Calculation shows that to overcome gravity the small particles must have the electric charge ~10^{-18} C in the field 300 V/mm. In fact, the electric charge leading to emission seems to be higher because of adhesion of the neighboring particles. Atom- and ion irradiation activated formation of sorbed layers and oxidation of the particle surfaces. As a result, the adhesion between the neighboring particles weakened, and the threshold electric field for particle emission decreased. Termination of the particle emission at the temperature ≥ 1300 K points out to reinforcement of the bonds between the powder particles at high temperatures. It caused formation of big groups of particles. Electric forces appeared to be unable to abstract them from the surface.

Heating of the powder up to 1800 K did not change the shape of agglomerations (chains and clusters) existing in the virgin powder. At the same time hydrogen ion irradiation stimulated the chain of modifications even under lower temperatures. Under irradiation with power density of 0.1 MW/m^2 ($E = 2$ keV, $j = 5$ mA/cm^2) small particles pasted all over the large ones, if the powder temperature was ≈1300 K. The temperature increase up to 1500 K led to integration of small particles. At 1700 K individual small particles on the surface of the large ones could not be distinguished. They formed a uniform layer on the chains of large particles (Fig.2a). When temperature approached 1800 K the adhering of small particles and formation of uniform layers occurred on the sides of the clusters (Fig. 2b).

Under higher power density irradiation (0.91 MW/m^2, $E = 7$ keV, $j = 13$ mA/cm^2) similar powder modifications took place at lower temperatures (1200÷1650 K). In particular, small particles adhered to the large ones at 1200 K, and the uniform layer on their chains was formed at 1100 K. At the temperature 1800 K shapeless clusters appeared and grew up to macroscopic dimensions joining both small and large particles (Fig. 2c).

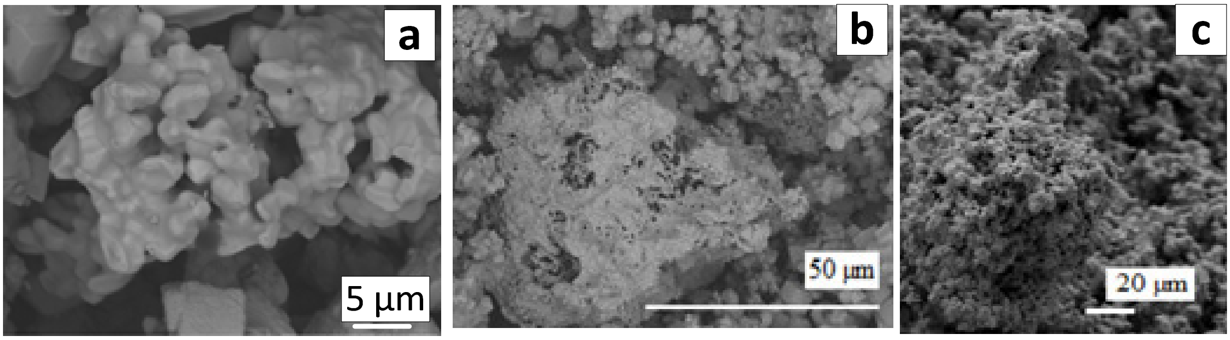

Fig. 2. The tungsten powder modifications under hydrogen ion irradiation (E = 1 keV/at, j ≈ 1×102 A/m2): (a) T = 1300 K, (b) T = 1450 K, (c) T=1800 K.

Irradiation of tungsten powder with argon ions was performed in the similar ranges of irradiation conditions (2 keV $\leq E \leq$ 7 keV, $j \leq 5$ mA/cm^2, T = 300÷1850 K). Beside the phenomena described above cone structures were formed on geometrical clusters, when they were covered with uniform layers (Fig. 3a). Irradiation with higher power density led to creation of volumetric structure consisted of partially melted and sticking together particles (Fig. 3b).

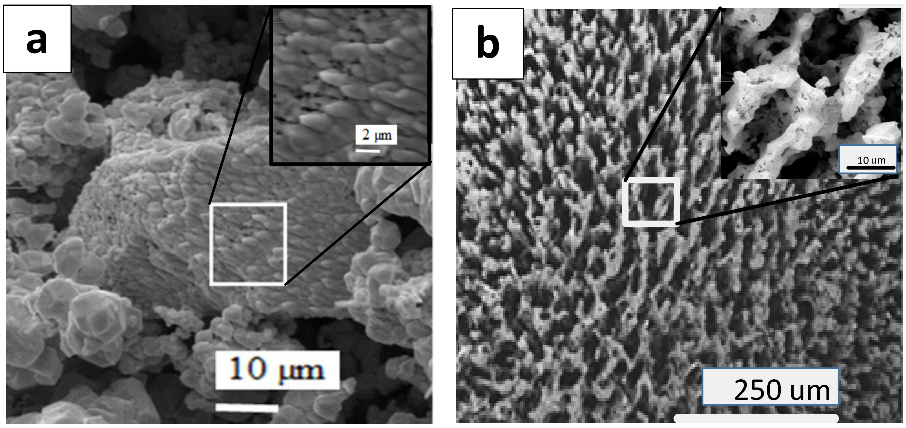

Fig. 3. (a)Cone structure formation on the sides of geometrical agglomerations under argon ion irradiation. (T = 1300 K, E =1 keV/at, $j \approx 1\times10^2$ A/m^2); (b) Volumetric structure formed in tungsten powder under argon ion irradiation (T =1850 K, E = 7 keV/at, $j \approx 3\times10^2$ A/m^2).

Experimental results allow revelation of driving forces and processes of the observed powder modifications. Electric forces caused particle mobility leading finally to small particle collection on the large ones. Adhering of small particles to the surface of the large ones occurred due to the influence of interatomic forces. Ion irradiation and powder heating activated destruction of the adhered particles and surface diffusion facilitating their transformation to uniform layer under surface tension. Comparably big flat area of large particle surfaces assisted in formation of the uniform layers on their chains at lower temperatures than that of geometrical clusters. Investigations of cone growth on tungsten surface under argon ion irradiation described by Begrambekov et al. (1996), suggest that cone structure on geometrical clusters appeared under action of ion sputtering, and tensions and crystallization processes in the uniform layer stimulated by ion irradiation. Formation of shapeless clusters occurred as a result of further activation of particle destruction and surface diffusion under increasing powder temperature and irradiation power density.

4. Conclusion

Modifications of the tungsten powder comprised of micro particles with dimensions: 1 ± 0.2 μm and 5 ± 1.5 μm under influence of heating, electric field and hydrogen- and argon ion irradiation have been investigated. Large particles of the virgin powder were shown to be assembling as three dimensional chains collecting some amount of small particles. At the same time, the fraction of small particles united into clusters having geometrical forms and distinctive dimensions 40-60 μm.

The powder outgasing took place in the temperature range 550-850 K. If the temperature ramp rate was high (≥ 100 K/s), powder outgasing occurred at higher temperature, and pulse release of water molecules pushed out significant part of the upper powder layer.

The particles emitted from the powder surface when electric field directed normally to the surface approached 350 V/mm. Simultaneous influence of electric field, hydrogen- or argon ions and hydrogen atom irradiation led to particle emission at lower electric field.

Heating of the powder up to 1800 K did not change the shape of agglomerations (chains and clusters) existing in the virgin powder. Hydrogen plasma ion irradiation ($E = 1$ keV/at, $j = 10$ mA/cm^2) stimulated the chain of modifications along with the increase of powder temperature in the range 1300-1800 K. It is pasting small particles all over the large ones, integration of the adhered small particles, formation of uniform layers covering both chains of the large particles and clusters of the small ones. Under higher power density irradiation ($E = 7$ keV/at, $j = 26$ mA/cm^2) similar powder modifications occurred in the lower temperature range 1200-1650 K.

Besides the phenomena described above argon ion irradiation initiated cone growth on geometrical clusters cowered with uniform layers and creation of volumetric structures consisted of partially melted and sticking together powder particles.

Driving forces and processes providing different powder modifications were found to be interatomic forces, surface tension, electric field forces, sputtering by ion irradiation, irradiation enhanced surface diffusion, crystallization, tensions in the near surface layer.

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