Study of the behavior of fine tungsten crystalline and amorphous particles in varying conditions

L B Begrambekov, A N Voityuk, A M Zakharov
National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), 31 Kashirskoe highway, Moscow 115409, Russia

Corresponding author e-mail: lbb@plasma.mephi.ru

Abstract. Formation of dust particles and their agglomerates with sizes ranging from tenths to hundreds of microns is observed in many modern plasma facilities. Dust accumulation can significantly affect discharge parameters in future thermonuclear facilities and lead to the accumulation of dangerously high amounts of tritium. Experiments on the generation of tungsten and tungsten-aluminium dust made of flakes formed during exfoliation of coating deposited on chamber walls from tungsten and aluminium targets sputtered by ions are described in this paper. A comparative analysis of the behavior of such dust and dust (figure 1), formed by crystal particles with simple geometric shapes (“crystal particles”) in electric field, their degassing and change occurring in temperatures of up to 2000 K is conducted.

Figure 1. SEM photograph of the tungsten dust crystal particles with simple geometric shapes.

1. Generation of amorphous tungsten dust during exfoliation of deposited layers.
Amorphous tungsten dust was formed in the “DECOR” plasma coating deposition stand [1]. Gas discharge was induced in the atmosphere of argon with hydrogen impurity. Tungsten and aluminium targets were places in the area of gas discharge, which were sputtered by plasma ions when negatively biased towards plasma potential. Collectors made of 12X18H10T grade stainless steel (closest analogue AISI 321) sheets were placed
along the plasma chamber walls, on which atoms sputtered during discharge formed the layers.

A 6A current discharge was initiated in the chamber before the target was biased to clean the surface of oxidized layers. Discharge parameters during the target sputtering were the following: working gas Ar+10% H₂, pressure 3×10⁻¹ Pa, discharge current (4-5) A, target bias -600V, current on the target (150-250) mA, sputtering duration (4-5) hours. Sputtering of aluminium target was conducted before tungsten target to generate dust particles containing aluminium.

Deposited layers were formed on stainless steel sheets during deposition which partially exfoliated and formed dust. Approx. 0.5 g of dust was collected after each discharge. The particles were in the form of flakes ranging in size from a couple of microns to 500 µm (figure 2a), and 2-6 µm in thickness (figure 2b). As such, their sizes correlated to the sizes of dust particles formed in the chambers of modern plasma facilities [2]. Particle content measured using Energy Disperision Spectroscopy (EDS) analysis, included W (up to 90% at.), Al (up to 60% at.), and O (up to 50% at.). Inclusion of a small (<10% at.) Fe, Cr and Ni impurity is apparently due to surface oxides on stainless steel exfoliating with flakes of deposited coating. The particles were calibrated by size using a 35x35 µm² cell net.

![Figure 2. Dust particles: (a) general view; (b) thickness of a dust particle.](image)

### 2. Investigation of gas desorption during heating of dust particles.
Thermal Desorption Spectroscopy (TDS) analysis of heated crystal and amorphous particles registered desorption of mostly H₂O, H₂ and –OH hydroxyl group molecules (figure 3). Total number of registered particles per unit weight of powders is shown in Table 1.

![Graphs showing desorption rates of H₂, OH, and H₂O](image)
Figure 3. TDS spectra for: (a) crystal particles; (b) amorphous particles.

Large desorption of water molecules from the surface of crystal particles can be explained by their surface area being larger than the surface area of amorphous particles at the same total mass since the average size of amorphous particles is 4-5 times larger. At the same time, hydrogen TDS spectrum form (figure 3b) for amorphous particles, as well as a higher amount of hydrogen desorbed from amorphous particles compared to crystal particles indicates that most of hydrogen came from the bulk of flake particles. The reason behind high amounts of hydrogen in amorphous particles is, supposedly, trapping of hydrogen from plasma during deposition.

Table 1. Desorption from particles of both types per 1 gram of weight.

|                  | Crystal particles | Amorphous particles |
|------------------|-------------------|---------------------|
| H$_2$O (particles/g) | 5.7×10$^{18}$    | 1.3×10$^{18}$       |
| OH (particles/g)     | 1.8×10$^{18}$    | 5.1×10$^{17}$       |
| H$_2$ (particles/g)  | 2.0×10$^{16}$    | 1.2×10$^{18}$       |

3. Study on the behavior of dust particles in electric field.

It was found that both types of particles detach from surface under applied electric field. To deduce minimal electric field strength under which the particles detach from the surface, experiments were conducted, in which dust particles were placed on a horizontal metal surface. Above the surface, parallel to it, a biased plate was located. It was found that the emission of amorphous particles with sizes ≤ 35×35 µm$^2$ began when the electric field strength between the plates was E ≈ 230 V/mm, and particles of larger size were emitted at E ≈ 150 V/mm. Crystal particles detached from the surface at E ≈ 350 V/mm. Emission of amorphous particles at lower electric field strength can be explained by lower adhesion strength with the surface of the plate, and a higher electric charge formed on particles due to larger surface area-to-volume ratio.

4. Study on the structural change of dust particles during.

During the heating experiments dust particles were placed on the box-shaped heater element made of thin tungsten sheet, which was mounted between electrodes in the vacuum chamber of a VUP-2K stand. Electric current was fed through the heater, the temperature was controlled using a tungsten-rhenium thermocouple. Dust particles were heated incrementally with a 100 K step, changes in structure and composition of particles were deduced using scanning electron microscopy and EDS.

No change in structure was observed for amorphous particles for temperatures of up to 1073 K. Starting from 1473 K, which corresponds to minimum temperature at which tungsten recrystallization occurs, metal grains and pores along grain boundaries appeared (figure 4a). With further increase of temperature, grains started growing in size, and oxygen content in flakes decreased due to decomposition of oxides. In case of amorphous particle containing high amounts (>55% at.) of aluminium, at temperatures higher than 1473 K, droplets of molten aluminium were secreted from the particle, spreading on the surface of a heater (figure 4c). For particles containing two layers, with different composition for each layer (85% at. of W on layer 1, 60% at. of Al on layer 2), cone-
shaped structure growth could be observed along the edges of a dust particle (figure 4b), which can allude to the development of internal stress in the layers. If a particle had a low Al content (<25% at.), no such phenomena occurred for temperatures specified above, and it is presumed that aluminium evaporated without forming droplets.

In case of crystal dust particle heating, larger crystals were baked together along the edges, and smaller crystals baked together into clusters (figures 4d, 4e).

5. Conclusion.
Experiments on the generation of amorphous dust particles are conducted. A comparative study of the behavior of such dust with crystal particle dust during heating and subject to electric field is conducted.

It was shown that gas desorption occurs from the surface and the bulk of dust particles during heating, as well as change in their structure: recrystallisation and metal grain growth, formation of cone-like structures due to internal stress, melting and evaporation of low melting point components (Al) in case of amorphous dust particles, baking of larger particles along the edges and baking of smaller particles into clusters in case of crystal particles.

It was shown that both types of particles are emitted from the surface under applied electric field. Minimal values of electric field strength for emission are deduced for each
type of particle, them being $E \approx 230$ V/mm for amorphous particles of size $\leq 35 \times 35$ µm$^2$, $E \approx 150$ V/mm for larger amorphous particles and $E \approx 230$ V/mm for crystal particles.

**Acknowledgments**

This work was carried out with the financial support of Ministry of Science and Education of Russian Federation under the agreement No. 14.575.21.0169 (RFMEFI57517X0169).

**References**

[1] Begrambekov L.B., Gordeev A. A., Grunin A.V., Evsin A.E., et al. The Peculiarities of hydrogen trapping and retention in zirconium with chromium coating // Materials of VII International School-Conference of young researchers and specialists IHISM’11 (24-28 October, 2011, Zvenigorod), p. 323-340

[2] S.I. Krasheninnikov et al., // Plasma Phys. Controlled Fusion 53(2011) 083001.