Dust generation and accumulation in JET-ILW: morphology and stability of co-deposits on main plasma-facing components and wall probes

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Abstract
Dust particles and co-deposits were sampled for the first time from beryllium limiters and bulk tungsten divertor (both after ILW-3), and test mirrors from the main chamber after ILW-2 and ILW-3. The focus was on the morphology of molten particles and metal outgrowths. In parallel, the stability of beryllium layers under the impact of hot water was examined on limiters and Be coatings. The study performed by several microscopy methods including SEM, AFM, FIB, TEM and Be-sensitive EDX has revealed: (i) an asymmetric distribution of Be particles with the largest objects (125–550 μm) on side surfaces of the Be tile; (ii) the presence of highly porous particles, resembling blisters; (iii) very few thin flakes of co-deposits on the divertor, on the shadowed edge of lamella; (iv) the elemental composition and internal structure of the needle-shaped outgrowths on the mirrors: MoO; (v) no detectable impact of water on the beryllium morphology.

1. Introduction
The JET tokamak with the ITER-like wall (JET-ILW) has been operated since 2011 [1]. Limiters are made of solid beryllium (Be), while other plasma-facing surfaces in the main chamber are coated either with Be or with tungsten (W). Inner and outer divertor tiles are made of carbon fibre composites (CFC) with W coatings, while the load bearing plate in the divertor base is made of over 9000 bulk W lamellae. With these features, JET-ILW is for ITER the most relevant controlled fusion device.

Three ILW campaigns have been fully completed: ILW-1 (2011–2012), ILW-2 (2013–2014) and ILW-3 (2015–2016). Regular retrieval of plasma-facing components (PFC) and erosion-deposition probes (EDP, also called wall probes) during shutdowns after each campaign has allowed for detailed ex situ analyses to understand and quantify material migration and fuel retention, thus providing essential information regarding safety aspects and development of ITER scenarios. Dust generation and accumulation by erosion-deposition processes and, in water-leak accidents is considered by ITER as particularly serious safety hazard. In a system with kilometres of pipes there is a risk of cooling agent leakage. Systematic studies are missing to address a question whether hot moisture/liquid would enhance the layer detachment and dust generation? If ‘yes’, will it happen directly under exposure to water or upon drying the tiles? Therefore, material study program at JET has also emphasised the determination of dust formation mechanisms, the quantity and morphology of particles collected by three methods: (i) remotely controlled vacuum cleaning of the divertor; (ii) local sampling of loosely bound matter from PFC by sticky pads and (iii) collection of mobilized dust on various erosion-deposition probes located in the divertor and in the main chamber [2]. This line of studies has also comprised the search for dust accumulation on metallic mirrors, both used for plasma diagnosis [3] and those tested in JET for ITER [3–6].

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The overall objective of this work was to complete all aspects in dust studies following JET-ILW operation with deuterium fuel. For that reason it is focused on topics never reported studied before: the analyses of particles collected for the first time ever directly from the bulk metal PFC: beryllium limiters and tungsten lamellae in the divertor, Tile 5. Two other topics dealt with the stability assessment of co-deposits and Be coatings under the impact of moisture and, the qualitative characterisation of specific outgrowths formed on the test mirrors from the main chamber.

2. Experimental

Studies were carried out on materials originating from four distinct regions in the reactor: inner wall limiters and cladding tiles, test mirrors from the outer wall and bulk tungsten divertor. Carbon sticky pads attached to microscopy tabs were used to sample dust particles and co-deposits from: (i) Be mid-plane inner wall guard limiter (IWGL) tile 2XR11 facing the plasma in all three ILW campaigns and (ii) bulk tungsten divertor (Tile 5, Module 14N, Stacks A-D) after ILW1-3.

The high field side of JET-ILW comprises 16 beam of inner wall guard limiters (IWGL) separated by inner wall cladding (IWC) recessed by 6 cm with respect to the limiter top surface defining the last closed flux surface in limiter operation. Ten limiter beams are composed of 19 castellated Be blocks 320 mm wide. The other six beams and also tiles of IWC are made either of Be-coated Inconel or W-coated CFC. Due to the complex 3D first wall structure and the magnetic field components, shadowing effects occur leading to asymmetric distribution of heat loads onto limiters in the poloidal direction which was presented in detail in [7–10]. Beryllium migration in JET-ILW is described in [11]. The selected IWGL tile 2XR11 is from the region of the most intense plasma contact with the inner wall in the limiter phase: around 19 100 s in total, as inferred from [16]. Due to the curvature of the tile, the tile wings are progressively deeper into the scrape-off layer (SOL) and the region changes from net erosion to net deposition. Images in figure 1 show the castellated Be tile with marked 13 locations of sampling on the plasma-facing surface (PFS, Samples 8, 10–18), the side surfaces of the shaded wings of the tile (Samples 7 and 19) and on the side surface located in the 2 mm wide gap between the tiles in the beam, Sample 20. The right (R) and left (L) hand side of the IWGL tile can be denoted as the ion- and electron-drift side.

A module of bulk tungsten Tile 5 contains four stacks (A)–(D), each with 24 individually shaped lamellae oriented in the poloidal location. In total, in 96 modules there are 9216 lamellae. The lamellae were produced using electro-discharge machining without subsequent surface treatment. Their surface is therefore characterized by a micro-crack network. The total plasma-facing area of the lamellae is 3.3 m². The outer strike point in JET is located on Stacks C and D, and rather seldom on (B), while Stack (A) was used for experiments on deliberate tungsten melting and study of melt layer motion [12]. It is also pointed out that the power load to lamellae in a given stack is not uniform; pieces 1–3 are in the shadow of the toroidally adjacent tile, while pieces 22–24 receive the greatest load [13, 14]. For the present study, six dust samples were collected from Stacks (B), (C), (D) and one from the Inconel structure (module carrier) under Stack (A). The description of the samples, designated as 100–105 is given in table 1.

The study was also carried out to determine the character of needle-shaped outgrowths [5] formed on test polycrystalline molybdenum (Mo) mirrors exposed on the main chamber wall in special cassettes with the so-called baffled and tubed channels of the mirror carriers. The baffled channels were tested as it was thought they could provide an improved protection of mirrors against erosion and deposition [15]. In this contributions
The impact of hot water and water vapour on Be dust generation was studied for two specimens from: the IWGL central area with a 1 mm wide belt of co-deposit containing up to $4 \times 10^{18}$ D atoms cm$^{-2}$, exposed to ILW-1 [16]; (ii) Be-coated Inconel IWC tile located above the midplane during two campaigns ILW1-2. The treatment had four stages. Before the exposure to water the samples were dried first by heating to 95 °C for 1 h, then to 110 °C for 1 h and, afterwards weighed. After the samples had been cooled down any loose dust on the surface was washed away into the water collection. Then the IWGL sample was placed in boiling water so that part of the surface was in the water and the other part was in the steam formed by the water. The water level kept constant by adding water when needed but naturally there were some small variation during the experiment. The IWC tile was heated to 200 °C to make sure to get a faster boiling and greater thermal shock when drops of room temperature water were dropped on it. The exposure was followed by a search for any loose dust in water and on the surfaces. Finally the samples were dried again in the same manner and the collected water was evaporated. Both the sample and the any remains from the water was again weighted.

The dust particles and deposits were investigated in terms of their size/morphology, composition, surface topography and internal features using scanning electron microscopy (Hitachi SU-8000 FE-SEM) combined with energy-dispersive X-ray spectroscopy (EDX - Thermo Scientific Ultra Dry, type SDD, enabling Be detection), focused ion beam (FIB/SEM, Hitachi NB5000) and scanning transmission electron microscopy (STEM, Aberration Corrected Dedicated STEM Hitachi HD-2700).

### 3. Results and discussion

#### 3.1. Dust sampled from the Be inner wall limiter

The examinations performed at the limiter tile show an asymmetric surface damage pattern. The largest number of particles being fragments of co-deposited layer formed on the limiter were present at samples designated as 10 and 11, collected from the right side of the tile, in a dark-colour area, relatively close to the edge of the plate. The images of the particles along with the EDX chemical spectrogram are presented in figure 2. The particles were relatively large: 125–550 μm. One may infer that this is re-solidified matter after partial or complete melting. In majority of them porosity was observed. As shown in figure 2(b) heavily porous particles, resembling blister were found. Concerning morphology diversity of structures was observed: solid, porous or built of small particles. Locally stratified structures were revealed. It seems that at least at the part of particles there is a kind of skin. The examined particles were also inhomogeneous in terms of their chemical composition, as exemplified in figure 2(e). Referring to the microstructure of the material it is characterized by a multiphase structure, locally resembling dendrites or cellular forms.

Observations carried out in the backscattered electron (BSE) mode show multiphase structure with areas rich in light and heavy elements. The EDX elemental composition spectra clearly show the presence of beryllium. The particles contain also O, C, Ni, N, W and Cr, figure 2(f). Such composition indicates intermetallic phases. The medium- and high-Z impurities like Ni and W are seen at the surface of all limiter tiles around the vessels. Ni, Fe and Cr come from erosion of the Inconel outer vessel wall by charge exchange (CX) neutrals. W has been eroded from the main chamber and divertor tiles and material migration in the SOL [17]. Numerous EDX measurements have been made on different particles. In part of the re-solidified regions mainly Be has been detected. This result may suggest that not only the re-deposited layer but also the surface layer of the limiter tile has been re-melted.

The second area where a large number of Be particles were accumulated was the side surface located in the 2 mm wide gap between the tiles in the beam. Sample 20, where overheated and porous objects reaching 600 μm have been identified, i.e. similar as on Samples 10 and 11. At other samples collected on the right side of the tile,

| Table 1. Description of samples collected from Tile 5. |
|-----------------|--------------|-----------------|
| Name            | Stack        | Description     |
| Sample 100      | B            | selected debris/particles across stack B |
| Sample 101      | A            | Inconel structure under stack A          |
| Sample 102      | D            | lamella 8, shadowed edge of the D8 lamella |
| Sample 103      | D            | lamella 19, shadowed edge of the D19 lamella |
| Sample 104      | C            | lamella 7       |
| Sample 105      | B            | lamella 7       |
only several particles were found (Sample 12), while no Be particles were collected on the curved part of the tile, right at the edge of the plate, Samples 7 and 8.

On the left-hand side of the plate, no Be particles or only single ones were found. The particles collected on samples 17 and 19 have the same morphology (large, re-solidified fragment of co-deposit of multiphase structure), with distinct spherical particles formed at their surface, figure 3. This result can be interpreted in the way that, since the particles attached to the tape on samples taken from neighbouring locations are almost identical, they correspond to tile surface structure, which would indicate that there is a deposit on the surface in this region of the tile, which has been severely overheated and re-solidified. In the case of Sample 15 (‘mirror’ position to 12), only a cluster of fine particles, up to 20 μm, was collected. These are debris of co-deposits rich in beryllium. This result proves the presence of a co-deposit in that region. For sample 16, corresponding to 11 in terms of sampling, one 135 μm large porous Be particle of a layered structure was revealed. Single particles were present on Sample 13 originating from the limiter centre. In this case, however, they showed no signs of overheating, whereas the layered structure is clearly visible, as shown in figure 4. Such structure is typical for strata formed on the divertor or beryllium plates [10, 18, 19]. No loose Be particles stucked to the pad of Sample 14 taken also from the tile central region.

The study has also revealed the presence of several other classes of particles on the plate, such as carbon-based and ceramic ones, Fe, Al, Si, W-, Ni-rich as well as boron nitride.

The structures of the overheated particles are similar to those observed for the spherical Be objects present on the silicon dust collectors located above the outer and inner divertor during ILW-3 and, in co-deposits on the
Identification of spherical Be droplets (3–8 μm in diameter) has been documented after ILW-2 campaign [2, 21]. Such objects (12–20 μm in diameter), of spherical shape have been found also on the monitors retrieved from the machine after ILW-3 campaign. They are characterized by the same size range as the spherical particles present at the flakes revealed on Samples 17 and 19.

A detailed insight into the internal structure of spherical particles from the silicon detectors located in the machine during ILW-3 is provided in figure 5. Results in figures 5(b) and (c) show that the surface layer constitutes a ‘skin’ which composition is different than the interior. In some cases, the inner structure resembles dendrites, as shown in figure 5(c). The phase present in ‘dark areas’ is rich in Ni. It contains also Fe and Be. The phase present in ‘white areas’ is rich in W and Cr. Additionally regions rich in C were found. The mapping of W, Ni and Cr shown in figure 6 from the particle present in figure 5(b), clearly illustrates differentiation of W and Ni distributions. W and Cr are present in the same areas.

In summary, the study conducted both at particles’ surfaces, FIB-produced cross-sections and thin foils studied by STEM clearly indicates complexity of structures present in the overheated particles.

The results obtained show that the surface modification of mid-plane inner limiter tile 2XR11 is dominating by melting on the right-hand side of the tile and by no or extremely low material delamination in its central part and left side. The results also prove that the re-deposition is not restricted to the plasma facing sides. The large fragments of deposits (up to 600 μm) were collected from the top side of the tile. The melting in the deposition zone on the right-hand side of the IWGL has been already observed after ILW-1 and ILW-2 [22]. The observed pattern correlates also with the fuel retention [10].

### 3.2. Dust on bulk W divertor lamellae

The sample collected from the Inconel structure under Stack A contained mainly C-based fibres, up to 1 mm in length, and other C-based particles, of the size above 100 μm. Additionally there were small (<20 μm) aluminosilicates along with Fe- and Ni-based particles. Small W particles (around 10 μm) were also found: flakes and ball-type empty spheres formed from the tungsten coatings on the CFC divertor tiles; as reported in [2, 23].

The matter collected from Stack B contained several large objects and also smaller particles were analyzed. The results can be summarised as follows: (i) one Be chunk (300 μm) and two spherical Ni-based particles of 15…
μm and 180 μm, (ii) a large number of BN debris of up to 1 mm; (iii) four C-based observed; (iv) small Fe-based, Al and W particles were identified.

Two samples from stack D, from the shadowed edge of lamella 8 and 19 (Samples 102 and 103, respectively) were examined. On Sample 102 a large number of small fragments of disintegrated co-deposits was found, size of a single object not exceeding 20 μm. EDX has clearly proven high W content thus indicating that these were co-deposited layers formed locally in the shadowed area of Stack D thus proving that the eroded W is transported into gaps between the lamellae where it becomes re-deposited. It is stressed that among all studied divertor samples that was the only place where the sampling indicated the presence co-deposits. The examined flakes also contained C, N, O and Na. Another interesting observation is presence of a large Be-flake with a size of 800 μm with the surface covered by a thin layer of cracked W-rich deposit. A similar result has been presented in [24] for vacuum-cleaned dust from the divertor after ILW-3. Aluminium-rich particles have also been detected, but their presence is related to the remote handling operation during the shutdowns [25]. The type of particles present at Samples 104 (lamella C7) and 105 (lamella B7) is the same. In summary, the bulk tungsten divertor is known to be a net tungsten erosion area [26]. However, the most important is that no single W droplet had been detected thus indicating no melting and splashing of the divertor material. These data are consistent with microscopic observations which revealed that lamella C23 (ILW-1) originating from the highest loading part showed erosion and only near-surface melting, in particular along crack edges [22, 27]. Concerning lamellae A23 and C14 no evidence of surface modification was observed. The examinations of side surface of lamella C22 revealed presence of a thin (300–600 nm), stratified deposition layer composed predominantly of tungsten with some oxygen content [27]. This layer was related to the deposition of tungsten atoms eroded from PFS of the lamellae, i.e. from the local surface of the divertor.

### 3.3. Outgrowths on test mirrors from the main chamber

In contrast to test mirrors coated with thick deposits following the exposure in the diverter, the thickness of the co-implanted or modified surface layer on the main chamber mirrors was of the order of 20–40 nm [4, 5]. However, on mirrors from specially design baffled and tubed channels (as described in [5] and Experimental) characteristic outgrowths (precipitate like) needle-shaped objects have been formed. These have been either circularly-agglomerated clusters 5–10 μm in diameter, figure 7(a) and (b) or individual 1–3 μm long and 0.1–0.6 μm wide forms, figure 7(c). In all cases EDX has identified Mo and O as main constituents, as documented in figure 7(d). Contrary to the needle area, the oxygen signal recorded from the mirror surface is either low or absent in the spectra.

In order to characterize the internal structure of outgrowths, a FIB-produced thin foil for STEM observations was examined. As seen in figure 8 the cluster of ‘needles’ is highly porous: individual particles are visible. Their thickness ranges from 25 to 40 nm. It should be noted that the structure of such objects is unstable and easily degraded under the impact of the gallium ion beam during FIB milling. This fact may explain the altered structure of some clusters. Linear oxygen and molybdenum distributions along the cross-section of the cluster shown in figure 9 indicate the presence of molybdenum oxide(s): MoO/MoO₂.
The exact origin and the formation mechanism of the structures remains unknown. One can hypothesize or speculate (even if this formulation is not appreciated by the community) that they were formed as a result of local arcing or parasitic discharge between the mirror and the protective structure (tube or baffle). No further or better evidence can be given at the present stage even with the cutting edge technology. The fact remains undisputable: no such forms had been detected in standard mirrors holders used either for the First Mirror Test program [4, 5] or in the ITER-like mirror test assembly [6].

Figure 7. SEM images of needles clusters (a), (b), (c) individual needles and (e) EDS spectrum.

Figure 8. (a) SEM image of the cluster of needles cross-section, FIB and STEM images of its structure revealed at the tin foil: (b), (e), (f) bright field image, (c) secondary electron and (d) Z-contrast mod.

Figure 9. Line scans of O, Mo and W distribution across the cluster of needles cross-section, thin foil. W deposition is used to produce a protective coating on a sample prior to the FIB cutting.
3.4. Impact of moisture

Exposures of Be wall components to hot moisture (water and vapour) have been done. These were the first experiments of that character because until recently only the impact of ambient atmosphere (including moisture) on the layer detachment was briefly discussed for carbon-rich deposits \[28\]. For the bulk Be from IWGL and the Be-coated sample cut from an IWC tile (preheated to 200 °C) it was not possible to see any formation of dust either optically or from weight measurements. No dust was found after the water was evaporated. In other words: no impact of hot water and hot vapour has been observed. Results of microscopy and EDX studies are shown in figures 10. No major difference in surface topography is perceived in SEM micrographs (figures 10(b) and (c)) between the unexposed and water-treated surface. Also the qualitative sample composition is the same in both cases, as documented by EDX (figures 10(d) and (e)): Be and O being the main constituents accompanied by co-deposited C and Ni. These results are encouraging from the point of view of reactor safety, but it must be stressed that no far-reaching conclusions should be drawn from these early results.

4. Concluding remarks

This work concludes dust studies after three ILW campaigns. For the first time particles sampled from the bulk metal PFCs (Be limiters and W divertor) have been examined. The study of IWGL has shown an asymmetric pattern of particle distribution with the largest number of particles (fragments of co-deposited layer) only on one, right side of the tile: 125-550 μm objects re-solidified after partial or complete melting, including highly porous, resembling blisters particles and only single particles present in samples originating from the centre of the tile. Large fragments of deposits (up to 600 μm) were also on surfaces located in gaps between the limiter tiles. The morphology—from surface topography to internal microstructure—of overheated particles has shown their complexity, indicating that the formation occurred in a multistep process.

From the point of view of material erosion, dust generation and reactor safety three results are listed. First, a small amount of matter isolated by sticky pads once again proves and confirms good adherence of co-deposits to the underlying substrates. Secondly, two exposures of Be wall components to hot moisture (water and vapour) have been done. Early results of this work do not give indication of dust generation and alteration of surface structure under the water impact. These results allow for some optimism regarding the Be layer stability, but it is definitely to early for any far-reaching conclusions. The third point and statement are based in the entire studies of dust from ILW: no single solid W droplet has been found thus indicating that the local surface melting of lamellae \[22, 27\] has not led to metal splashing.

The fourth outcome of this work is related to detailed structural and elemental analyses of outgrowths on the test mirrors. It is difficult to assess the general weight of this result. Those needle-shaped forms appeared only on
m為了 the specifically designed with baffled channels which are now not considered by ITER. However, if the underlying mechanism of ‘needles’ formation was related to a local parasitic discharge (arching), such phenomena should be taken into account in the construction of mirror holders. This is because the presence of such artefacts would create additional difficulties in the regeneration of mirror optical properties by any cleaning method.

In summary, the dust study programme in JET-ILW has comprised all aspects of dust generation, release, accumulation and collection. It has been an integral part of material migration and fuel retention studies from material erosion [29] to fuel content in individual particles [24, 30, 31]. Besides ex situ analyses of the retrieved loose matter and particles sticking to PFC and wall probes, as detailed as possible in situ assessment of metal splashing and dust/droplet migration have been done using high-speed and high-resolution cameras, e.g. [29, 32]. All these data, in turn, have helped and served modelling: prediction and validation [33–36].

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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