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Studies of Dust from JET with the ITER-Like Wall: Composition and Internal Structure

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Abstract

Results are presented for the dust survey performed at JET after the second experimental campaign with the ITER-Like Wall: 2013-2014. Samples were collected on adhesive stickers from several different positions in the divertor both on the tiles and on the divertor carrier. Brittle dust-forming deposits on test mirrors from the inner divertor wall were also studied. Comprehensive characterization accomplished by a wide range of high-resolution microscopy techniques, including focused ion beam, has led to the identification of several classes of particles: (i) beryllium flakes originating either from the Be coatings from the inner wall cladding or Be-rich mixed co-deposits resulting from material migration; (ii) beryllium droplets and splashes; (iii) tungsten and nickel-rich (from Inconel) droplets; (iv) mixed material layers with a various content of small (8 - 200 nm) W-Mo and Ni-based debris. A significant content of nitrogen from plasma edge cooling has been identified in all types of co-deposits. A comparison between particles collected after the first and second experimental campaign is also presented and discussed.

Key words: dust, JET, ITER-like wall, beryllium, tungsten, erosion-deposition, material mixing

Introduction

Processes of dust generation and transport have been studied in present-day tokamaks for the last two decades. The main driving forces in research have been related to a whole spectrum of safety aspects and then licensing of a future reactor-class device, e.g. ITER. This has also included the assessment of deposition and dust formation on the reliability of in-vessel diagnostic components, especially metallic first mirrors. Therefore, detailed dust surveys have been carried out regularly in all medium- and small-size tokamaks and in several simulators of plasma-wall interaction (PWI). The bibliography of works performed until year 2010 is in [1], while results of recent examination in several machines are also available: TEXTOR [2,3], Tore Supra [2,4], ASDEX-Upgrade [5,6].

Operation of JET with the ITER-Like Wall (JET-ILW) has brought a spectrum of new research aims and results connected to the new wall structure and composition: beryllium in the main chamber and tungsten in the divertor [7-9]. It has created new challenges related to the analysis of materials retrieved from the torus: marker tiles, wall probes [10,11] and dust. The aim in studying dust from JET-ILW is to provide a robust data base for the predition of dust generation in ITER, particularly categories and major formation mechanisms. Though JET-ILW has the best possible proximity to ITER, in PWI studies both similarities and differences in the structure of plasma-facing components (PFC) between two machines are thoroughly taken into account, e.g. solid Be and W components in ITER, while in some areas of JET-ILW tiles with either tungsten or beryllium coatings are used, as detailed in [7].

Dust retrieval from 22 out of 24 divertor modules was performed by vacuum cleaning during major in-vessel interventions (shut-downs) after two ILW campaigns. The key result and positive message for ITER was that very small quantities of loose matter were found: around 1.4 g after 2011-2012 and 1.8 g after 2013-2014 operation [12], thus corresponding to about 0.06 g m⁻² and 0.08 g m⁻² of the divertor surface area, respectively. These amounts are over two orders of magnitude smaller (scaled to plasma operation time) than those retrieved after the 2007-2009 campaign in JET with the carbon wall (JET-C) [13], when over 200 g was removed. After the first ILW campaign, besides vacuum cleaning, dust was also sampled by a carbon sticker from a single location in the inner divertor, as reported in [14]. A widespread collection was performed after the second campaign.

This paper is focused on detailed studies of material sampled from many points on the divertor tiles and on the test mirrors located in the main chamber. The main goal is to relate the morphology of dust from various locations with the overall deposition pattern and with the operation history. This also includes a comparison of internal structure of co-deposits from the inner divertor after the two campaigns in JET-ILW, in the following called ILW-1 and ILW-2. The emphasis is on particles relevant from the ITER point of view: Be-rich deposits and metal droplets, while debris from W-coated carbon fibre composite (W/CFC) tiles will be only briefly addressed, as not relevant because no such coatings are foreseen for a reactor-class machine.

Experimental

The study was carried out for samples collected on tiles from the divertor modules which were not vacuum cleaned after the 2013-2014 campaign. Two complete modules equipped with erosion-deposition diagnostics [10] were transferred from the torus to the Beryllium Handling Facility (BeHF). Dust sampling was done manually at several different positions using adhesive carbon pads of 2.54 cm (1 inch) in diameter. This method enables a direct correlation between the location and the type of dust particles. However, the procedure has one disadvantage: only the bottom part and some side surfaces of the collected matter can be examined by optical or scanning electron microscopy (SEM), because the top layer is embedded in the glue of a pad. This problem is partly overcome by using focused ion beam (FIB) technique to obtain crosssections of particles, but even this approach still prevents direct studies of the top layers. It is also limited to objects not thicker than 20 micrometers; above that value the quality of results is degraded. The information on the sampling is detailed in Table 1, while images in Figure 1 show the divertor poloidal cross-section and sampling locations on Tile 0, respectively.

Dust found under Tile 6 on the frame of the divertor carrier was collected by wiping with a swab and then the matter was transferred to a sticky pad. In addition, brittle and dust-forming co-deposits on a metallic test mirror (First Mirror Test, FMT [15,16]) was examined. The mirror was located the inner divertor in the shadow of Tile 3. No special dust sampling was required in this case.

The composition, size, surface topography and internal features of dust and deposits were examined using a large set of electron and ion beam methods. The analyses performed at the Warsaw University of Technology comprised: SEM (Hitachi SU 8000) combined with energy-dispersive X-ray spectroscopy (EDX, Thermo Scientific Ultra Dry, type SDD enabling Be analysis), focused ion beam (FIB/SEM, Hitachi NB5000) and scanning transmission electron microscopy (STEM, Aberration Corrected Dedicated STEM Hitachi HD-2700). Depth

profiling (Be, C, N, O, W) was determined by time-of-flight heavy ion elastic recoil detection analysis (ToF-ERDA) with a 36 MeV ¹²⁷I⁸⁺ beam. The method gives good depth resolution of a few nm and it is particularly suited for studying smooth surfaces, e.g. mirrors. The information depth is limited to a few hundreds of micrometers because of a low incidence angle (22°). Global fuel retention studies (i.e. deuterium content analysis) could not be performed on samples collected by sticky pads. Thermal desorption, for obvious reasons, is out of question. However, detailed information on fuel retention in deposits determined by ion beam analysis (IBA) can be found [12].

Results and discussion

The presentation of dust survey follows the divertor poloidal cross-section starting from the inner divertor Tile 0 which acts as the High Field Gap Closure (HFGC) plate. A comparison of dust after the consecutive campaigns will be made for apron on Tile 1. The description of dust collected from plasma-facing surfaces will be complemented by the characterisation of particles taken from the divertor carrier below Tile 6. Finally, dust-forming deposits on the test mirror will be presented.

Dust on divertor tiles

Tile 0 has two distinct regions which can be perceived in Fig. 1(b). A sample collected outside the "blackish" deposition zone is characterised by a small density of particles: thin though fairly large (up to $300 \ \mu m$) flakes rich in beryllium. It is documented by the image of a flakes in Fig. 2(a). The corresponding X-ray spectrum, Fig 2(b) clearly proves that Be is the major constituent, around 90 at. %. The structure of the flakes has features of Be coatings from the inner wall cladding tiles, as observed in secondary electron images. The structure of some areas suggests that the material was molten.

Images in Fig. 3 show particles of mixed columnar and stratified structure detached from the deposition zone: thick and cracked Be-based layers. There are regions rich in tungsten and nickel: bright areas in Fig. 3(b) recorded in the backscattered electron (BSE) mode and analysed with EDX. While the presence of tungsten may be attributed to the detachment of underlying W coating on the CFC tile, the presence of nickel can only be related to the deposition of that element eroded from the in-vessel Inconel components: torus wall or a grill of the antenna for ion or electron resonance heating, or from damaged tie rods in Tile 7. The studied region on Tile 0 is known to be the major deposition zone in the divertor

with the deposit thickness reaching 50 μ m (cumulative after two campaigns) and the deuterium content of up to 1×10^{19} cm⁻² [12].

For Tile 1 the analyses have been concentrated on the apron area which may be perceived as the extension of Tile 0, also from the point of view of deposition: layers up to 20 µm thick [12,17-19]. Dust from the apron was sampled after each campaign. This allows for a meaningful comparison because the operation time was similar in both cases: around 19.5 h including approximately 6.5 h of limiter and 13 h of X-point plasmas. After the first campaign the most important result was the identification of Be dust in the form of droplets and flakes detached from co-deposited layers. The internal structure of such flakes has been recently examined. STEM images in Fig. 4(a) and (b) show, respectively, the bottom and top layer of a 4 µm thick flake formed during ILW-1. Bottom of the layer corresponds to the initial phase of operation, while the top is related to end of the 2011-2012 ILW campaign. A crystalline columnar structure suggests quiescent growth of the deposit. The layer contains also tiny tungsten particles: very small amount at the beginning, Fig 4(a). Significantly increased deposition of 8-15 nm W particles occurred at the final stages of operation; it is shown in Fig. 4(b). The presence of W inclusions is most probably attributed to the erosion of W-coated carbon fibre composite (W/CFC) tiles in the divertor and some places on the main chamber wall. As a consequence, the composition of deposits is clearly correlated with the history of operation: L-mode in the initial phase and a greater input power for H-mode discharges towards the end. Disruptions at high power shots additionally contributed to the generation of tiny metal particles.

Examples of dust collected from the Tile 1 apron after the second campaign with most discharges in the H-mode are in Fig. 5 (a)-(d), showing a 200 µm object, EDX spectra from two regions (marked 1 and 2) and STEM images for cross-sections from two different areas of that object. Already an overview BSE image in Fig. 5(a) indicates a complex mix of light and heavy elements. A cross section in Fig. 5(c) recorded in the high-angle-annular dark field (HAADF) mode shows fairly uniform co-deposit composed of low-Z elements (Be, C, O, N) with only one remarkable tungsten layer visible as a bright stripe. Another cross-section, Fig. 5(d), is a sandwich of low-Z (beryllium-carbon) strata and regions with 100-200 nm inclusions of heavy metals (tungsten and nickel) in the low-Z matrix. Tungsten can originate both from the bulk metal (Tile 5) and coated tiles, but nickel must come from Inconel or steel components. There are no such Ni-rich parts in the vicinity of Tile 1. One may suggest that the particle was formed

in another location (e.g. Tile 6 where Ni droplets appeared because of damaged Inconel tie rods of Tile 7) and then it was mobilised by a disruption. This statement is justified by the fact that two cross-sections described above are only 100 μ m apart. Therefore, such big differences in morphology cannot be attributed to not uniform deposition. A big particle is not a regular flake but an agglomerate of small particulates formed originally on the apron and in other places. It should be stressed that all types of co-deposits after the 2013-2014 campaign (dominated either by light or heavy elements) contain significant amounts of nitrogen retained after plasma edge cooling by gas injection. Nitrogen retention in JET co-deposits has already been reported [16,18,20], but the dust studies show that nitrogen is present in all types of re-deposited materials. Corresponding X-ray spectra deposit are in Fig. 5(b).

Rounded beryllium objects, 8-10 μ m in diameter and smaller, have been detected on the apron. A small spherical Be droplet shown in Fig. 6 originates most probably from a melt layer on overheated limiters. The volume of a sphere of 10 μ m in diameter is 5.25×10^{-10} cm³, what corresponds to less than 1 ng in weight and 6.5×10^{13} atoms, at atomic density of 1.24×10^{23} cm⁻³. In a single sphere there is only a tiny amount of matter. However, from ex-situ analyses it is not possible to conclude on the amount of material ablated when the droplet was passing plasma from the place of origin. Based on the photographic survey, events of limiter melting related to disruptions were not unique and some of them could cause certain material losses. Elongated beryllium splashes (approx. 100 μ m in diameter, 5 μ m thick, i.e. 1.25×10^{-8} cm³) have been found on test mirrors exposed in the main chamber [16].

Matter collected from Tile 3 contains only small carbon-based and ceramic particles. Fairly clean and dust-free tile surfaces could be expected given the fact that the strike point was on that tile during majority of pulses in 2013-2014 [12].

A sloping part of Tile 6 is a deposition zone [12] and this is reflected by character of particles which are similar to what has been detected in the deposition zone of Tile 0 and on the apron of Tile 1, i.e. these are mainly fragments of co-deposits. The detached flakes are relatively large – up to 200 μ m in size. They have a layered structure, although granular forms are detected as well. Both composition and internal structure are diversified. The regions rich in either light (Be, C, N, O) and or heavy elements are present (W or/and Ni). Although a distinct Be peak was visible only in one spectrum, a high oxygen peak registered in other EDX spectra taken from the areas rich in light elements indicates that Be is a major constituent of these particles.

Particles/layers rich in tungsten (originating from the W coating) and nickel are detected at the bottom part of particles being small fragments of co-deposit. They have different forms: the smallest objects observed with SEM are fine nanoparticles embedded in the matrix composed of light elements (Fig. 7) whereas the largest observed pieces are several microns in size.

Dust on the divertor carrier and on test mirrors

Two main groups of particles have been found on the sample collected by brushing the diveror carrier under Tile 6: (i) detached fragments of W-Mo coatings and (ii) beryllium-rich flakes. The size of W or W-Mo debris ranges from several hundred of μ m to a few mm. Their surface was overheated, locally revealing melt zones. The largest Be-rich particle was approximately 2x2 mm in size. Its general appearance and internal structure are presented in Figures 8(a) and (b), respectively. The surface is uniform: morphology and elemental composition are changed within the individual flake limits. The deposit is composed of fine particles/grains, Fig. 8(b), thus making the structure very porous. This form of beryllium deposit has been observed for the first time. Other particles composed of different constituents have been also detected: carbon, ceramics and metals, namely steel, Inconel and copper.

A general appearance and details of a stratified structure deposited on a molybdenum test mirror from the inner divertor are shown in Fig. 9(a) and (b), respectively. The layer is smooth, but in several places it is broken most probably due to internal stresses. As a result, a number of sublayers are displayed. A cross-section obtained by FIB, Fig. 9(c), reveals the layer thickness (1 μ m) and an internal structure. It also clearly proves that the deposit adheres poorly to the Mo substrate. Depth profiles obtained by ToF-HIERDA show beryllium as the main constituent of the layers, see Fig. 9(d). Based on microscopy and HIERDA data one may conclude that the stratified layers were formed in a series of events in which beryllium, originally transported to the divertor and deposited there, was evaporated and then re-deposited in the shadowed zone of the divertor where mirrors are located. Such processes are to be taken into account in the design of diagnostic systems in a reactor-class machine. It should also be stressed that tungsten and nitrogen are in noticeable amounts especially in the near surface layer (30-70 nm), i.e. the region corresponding to the final period in the campaign. The amount of carbon in the same region is relatively small thus indicating that the operation did not lead to a significant damage of the W coatings despite H-mode discharges.

Concluding remarks

The most important result of dust surveys is the small amount of loose matter (1.5 g level) retrieved after ILW campaigns. Examination of dust sampled from various regions of the divertor has identified a range of particles. Besides not ITER-relevant debris from tungsten coatings on CFC tiles there are two major classes of importance to ITER: (i) mixed deposits rich in beryllium and (ii) metal droplets (Be, W, Ni) born in melting events of wall materials. Further split into sub-categories is not intended here for a single reason: the comparison of deposits from the two campaigns clearly proves the increased complexity of particles due to agglomeration of particles formed originally under different conditions and, most probably, in different places. This indicates mobilisation and transport of fine dust, for instance, as a consequence of off-normal events. Therefore, conclusions regarding the size range should be rather restricted to metal droplets and splashes (from 3 µm for droplets to 100 µm for splashes), but certainly not to agglomerates or even co-deposits, which are brittle and easily disintegrate into smaller objects.

In summary, tile analyses [12] and locally-specific dust survey provide very detailed data on the overall morphology of particles. High-resolution dust studies allow, to fair extent, for conclusions on dust generation, its migration and agglomeration. An important contribution of this work to PWI studies in a metal-wall tokamak is a detailed insight into properties of deposits formed on diagnostic components, i.e. mirrors. Transport of light (Be) and heavy (W) metals to the shadowed region in the divertor is well documented once again [20] and it is also shown that such metal layers brake and form dust. In this sense test mirrors serve as long-term deposition monitors. One may consider this type of probes being placed even in a reactor-class machine, especially that the application of removable samples in ITER is under planning [21].

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Table 1. List of studied materials with details on their origin and sampling location.

Tile / sample	Region	S-coordinate (mm)
0 (35)	Outside deposition zone	50
0 (31)	Deposition zone	137
1(24)	Apron (horizontal part of Tile 1)	210
3 (1)	Lower part below	590
6 (11)	Deposition zone in shadow of Tile 7	1415
Under Tile 6	Divertor carrier	n/a
Mirror (119)	Inner divertor, first mirror in the cassette	n/a

Figure captions

Fig. 1. (a) Divertor cross-section in JET-ILW and (b) marked positions of sampling on Tile 0 after ILW-2.

Fig. 2. (c) Beryllium flakes from the inner wall cladding on Tile 0 in the area without deposits and (b) corresponding EDX spectrum.

Fig. 3. Thick co-deposits collected in the deposition zone of Tile 0 after ILW-2..

Fig. 4. STEM images of: (a) the bottom and (b) top layer of beryllium flake on apron of Tile 1 after the ILW-1 campaign in 2011-2012.

Fig. 5.

Deposits on apron of Tile 1 after ILW-2 campaign in 2013-2014: (a) a typical agglomerated particle, (b) EDX spectra from two regions, (c) stratified Be-rich deposit; (d) highly porous mixed deposit with light and heavy elements.

Fig. 6. A spherical beryllium particle on apron of Tile 1 after the first ILW campaign.

Fig. 7. Particles rich in tungsten present in deposits originating from Tile 6, BSE mode.

Fig. 8. Be-rich co-deposit on the divertor carrier under Tile 6 after ILW-2: a) SEM image of fractured edge, b) STEM image.of internal structure,

Fig. 9. Deposition on a test mirror from the inner divertor after ILW-2: (a) broken dust-forming layer, (b) stratified structure of the broken deposit; (c) deposit cross-section obtained by FIB and (d) ToF-HIERDA depth profiles of elements in the deposit

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