EUROFUSION WPJET2-CP(16) 15362

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Preprint of Paper to be submitted for publication in Proceedings of 29th Symposium on Fusion Technology (SOFT 2016)

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

Micro-/nano-characterization of the surface structures on the divertor tiles from JET ITER-like wall

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Micro-/nano-characterization of the surface structures on the divertor tiles used in the first campaign (2011-2012) of the JET tokamak with the ITER-like wall (JET ILW) were studied. The analyzed tiles were a single poloidal section of the tile numbers of 1, 3 and 4, i.e., upper, vertical and horizontal targets, respectively. A sample from the apron of Tile 1 was deposition-dominated. Stratified mixed-material layers composed of Be, W, Ni, O and C were deposited on the original W-coating. Their total thickness was ~200-400 nm. By means of transmission electron microscopy, nano-size bubble-like structures with a size of more than 100 nm were identified in that layer. They could be related to deuterium retention in the layer dominated by Be. The surface microstructure of the sample from Tile 4 also showed deposition: a stratified mixed-material layer with the total thickness of ~200 nm. The electron diffraction pattern obtained with transmission electron microscope indicated Be was included in the layer. No bubble-like structures have been identified. The surface of Tile 3, originally coated by Mo, was identified as the erosion zone. This is consistent with the fact that the strike point was often located on that tile during the plasma operation. The study revealed the micro- and nano-scale modification of the inner tile surface of the JET ILW. In particular, a complex mixed-material deposition layer could affect hydrogen isotope retention and dust formation.

Keywords: JET ILW divertor; TEM observation; Deposition; Erosion

1. Introduction

Plasma-facing components (PFCs) in fusion devices are heavily bombarded by energetic particles such as hydrogen isotopes and several kinds of impurities. Hence, some part of the PFCs are eroded and a certain amount of the eroded materials are deposited, and form a deposition layer on the specific part of the PFCs through the plasma wall interaction (PWI). When a deposition layer is formed on the PFCs, its hydrogen recycling properties could be changed from the initial conditions. In particular, since carbon related materials cause high sputtering erosion and make a thick deposition layer, accumulation of hydrogen isotopes in such a deposition layer lead to the rapid increase of fuel inventories inside of the vacuum vessel. Generally, such a deposition layer has unstable and brittle properties and would become a possible source of undesired impurities such as dust against the fusion plasma. If tritium is used as a fueling gas, tritium content dusts will be continuously formed. That is a problem when seen from the perspective of the radiological safety. A combination of the tungsten (W) divertor and the beryllium (Be) first wall is supposed to be used in ITER PFCs. It has been predicted that this combination will strongly reduce tritium build-up compared to the initial material choice of CFC strike point tiles, W divertor and Be first wall [1].

The integrated test of the ITER PFCs of the W divertor and the Be first wall in the large sized tokamak JET, the JET ITER-like wall (ILW) experiment [2], has been launched for demonstrating an ability to operate a large high power tokamak and investigation of the material erosion, deposition and fuel retention [3,4]. The first ILW campaign was conducted from 2011 to 2012. Many kinds of post-mortem analyses for the extracted PFCs, the first wall bulk/coated Be samples and the divertor W-coated carbon fiber composite (CFC) tiles, have been carried out to date [5,6]. Studies with ion beam analysis (IBA), thermal desorption spectroscopy (TDS) and secondary ion mass spectrometry (SIMS) revealed the unique feature of the long-term fuel retention in JET ILW components. The deuterium (D) retention occurs by implantation and co-deposition with the impurity elements of Be, C, O and W, and the highest retention values were found to correlate with the thickness of the deposited impurity layers [6,7]. Over half of the total amount of the retained D fuel were retained in the divertor region, and the highest retention values were measured from the regions with the highest deposition [6]. Such thick deposition layers had a complex multilayered structure, and every layer was dominated by Be with some W and O content [8]. Understanding the mechanical properties of deposits is important as brittle layers may exfoliate and contribute to flake and dust production, as observed in the JET carbon

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**See the Appendix of F. Romanelli et al., Proc. of the 25th IAEA FEC 2014, Saint Petersburg, Russia* wall [9]. However, to date no flaking of deposits in the divertor have been observed for the JET-ILW. Whereas in the erosion dominant surface like a strike point of the divertor plasma would be created the microscopic point defects such as dislocation loops and bubble-like structures. These types of defects would change the fuel retention properties of the surface. Thus, understanding of the retention amount of D as a function of surface morphologies in the divertor region is quite important for assessment of both fuel inventory and behavior of dusts, including a certain amount of the hydrogen isotopes, in the JET ILW. Poloidal distribution of the thickness of the deposition layer and its chemical composition and surface morphologies in the W-coated CFC divertor tiles have been investigated with the retention amount of the D and T [4,6,7,10,11]. However, characterization of the surface structures on the divertor tiles in nanometer level have never been performed to date. Since nanostructure directly affects the trapping and detraining of the hydrogen isotopes (D, T) and mechanical property of the deposition layer itself, nanoscale information should be obtained.

In the present work, therefore, micro-/nano-characterization of the surface structures on the divertor tiles used in the first campaign (2011-2012) of the JET tokamak with the ILW was studied by means of focused ion beam fabrication (FIB) and transmission electron microscope (TEM) observation, etc. All analyses in this study have been carried out at International Fusion Energy Research Centre (IFERC), QST Rokkasho.

2. Experimental procedures

Fig. 1 shows the poloidal cross-section of the JET ILW divertor with three representative plasma geometries [12], and positions of the surface samples (17 mm in diameter) which were transferred to IFERC, QST Rokkasho. This figure was schematically drawn based on Fig. 1 in Ref. [12]. In this study, 1-10c, 3-2a and 4-10b from the inner divertor tiles of upper, vertical and horizontal were selected for analysis. Photographs of those samples are also shown in the left-hand side column in Fig. 1. The selection of samples for this experiment was based on the result of an imaging plate (IP) analysis conducted by other collaboration experiments. All JET divertor tiles except tile 5 consist of W coated CFC with the thickness of 10 to 20 μm [4,13]. A set of special marker tiles composed by tiles 1, 4, 6, 7 and 8 were further

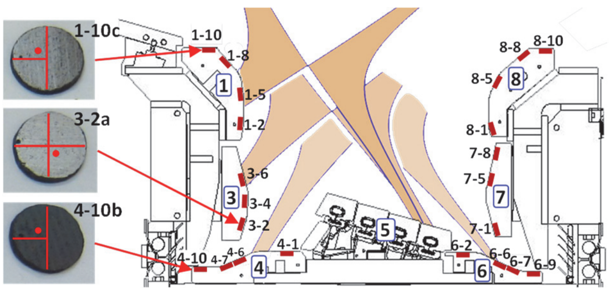

Fig. 1 Poloidal cross-section of the JET ILW divertor with three representative plasma geometries [12], and positions of all samples. Photographs of three analyzed sample are shown in a left side row. Lines and symbols in photographs are the cutting tracks and the analyzed positions, respectively.

coated with a W marker layer with a thickness of about 3 μm with a 3 μm thick Mo interlayer between W marker layer and the thick W coating [4,14]. Tile 3 was without the W marker layer and only had the Mo layer on the top surface [4]. Prior to micro/nanoscopic analysis, disc samples were cut to small pieces by low speed cutter. Red lines drawn on the sample photographs in Fig. 1 correspond to the cutting tracks. Red small symbols on the same photographs are the analyzed area in this study. Then, micro- and nano-scale modification of the tile surface were analyzed by means of scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDS) analysis, electron probe micro analyzer (EPMA), focused ion beam (FIB) and transmission electron microscope (TEM).

3. Results and discussions

3.1 Apron part of the upper tile: Tile 1 (1-10c)

Prior studies reported that a very thick deposition layer composed by 70-80 at.% of Be, 7-10 at.% of O, 10-15 at.% of C, about 3 at.% of W and 1-5 at.% of D was confirmed at the position of the 1-10c sample, i.e., the apron part of the upper tile (tile 1) [4]. The thick deposition layer had a complex multilayer structure with a complex multilayer [8]. Fig. 2 shows SEM images of all three tile surfaces in this study. The 1-10c sample showed typical deposition structure. Complementary analysis of EDS and EPMA revealed that the deposition layer was composed by the mixture of W, C, O, Ni and Be elements. These results are consistent with previous work mentioned above, except with Ni [4]. Although the detailed discussion of Ni we would like to avoid in this paper, it might be derived from the Inconel components used at many parts of the JET PFCs [3]. It is interesting that the mixed-material deposition layer has obvious directivity in the growth. Possible incident direction of the deposited elements were depicted on the SEM image. Since the deposited impurities were transferred by plasma flow to the surface, oblique deposition pattern might have been confirmed. Indeed, the angle of the incident direction is parallel to the direction of the toroidal magnetic field. Fig. 3 shows cross-sectional SEM images and corresponding element profile of W, C, O, Mo and Be detected by EPMA along with a dashed line in each SEM image of 1-10c and 3-2a samples. The left side of each image corresponds to the top side (plasma facing side) of the samples. The W protection layers were artificially created on the top surface of each sample before FIB fabrication for protecting the top surface of the samples against the Ga fabrication beam in FIB. In the case of the 1-10c sample, thickness of the deposition layer was estimated to be ~1.5 μm. This value is much thinner than

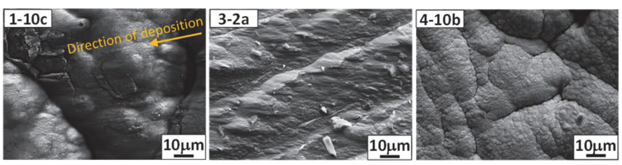

Fig. 2 SEM images of 1-10c, 3-2a, and 4-10b samples. Incident direction of the deposited elements in 1-10c shown by an arrow in the image.

that of the previous work, e.g., over 10 μm [8]. The reason for the difference can be considered as follows: flux of the deposited elements of 1-10c sample might be lower than that of the analyzed position of Ref. [8], because of the shadow effects by the neighbor tile. Since the deposited element has a strong directivity at the apron part of the upper tile (1-10c sample), the growth rate of the deposition layer on the shadow area could be slower than the open area. In general, fairly high Be content is detected through the entire thickness of the deposit. There is some decrease of the signal measured in the top layer, i.e., the region corresponding to the end of the experimental campaign. It may be related to local deposition effects, caused for instance by disruptions, during that phase of JET-ILW operation.

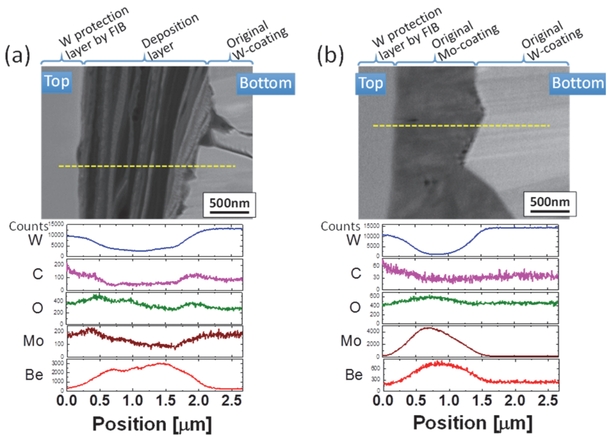

Fig. 3 Cross-sectional SEM images of (a) 1-10c and (b) 3-2a sample fabricated by FIB, and corresponding element profile of W, C, O, Mo and Be along with a dashed line in each SEM image. W protection layers were artificially created on the top surface of the each sample by FIB.

The cross-sectional bright field (BF) TEM image of the mixed-material deposition layer and corresponding electron diffraction (ED) pattern of 1-10c sample is shown in Fig. 4. The deposition layer has a stratified, and a geological-like layer structure. This layer structure must be caused by the discharge and operational history of JET. The dark image layers are mainly composed by relatively high-Z element such as W, and the bright layers would be low-Z element such as Be, O and C. Diffused ED pattern indicates that the layer has the amorphous-like structure. Many nano-size bubble-like structures were observed in the layer. The largest size was over 100 nm. Judging from the size and density of the bubbles, this might be related to D retention in the layer. Indeed, it has been reported earlier that the amount of D on the apron part of the upper tile (tile 1) was highest (~10^{23} D/m^2) among the three tiles [6,7]. The large-size bubble formation would cause undesired dust formation due to the exfoliation of the deposition layer. In the case of Fig. 4, the nano-size

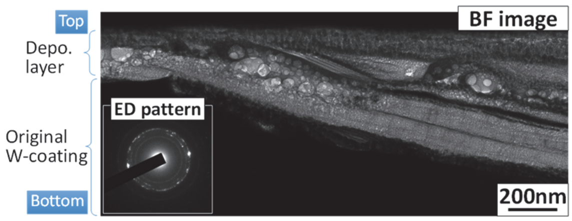

Fig. 4 Cross-sectional BF-TEM image of 1-10c sample and corresponding ED pattern from the deposition layer.

bubble-like structures were formed in a concentrated manner in a certain depth in the mixed-material deposition layer. When the bubble-like structures are further grown or some thermal stresses are induced on the layer, a specific layer containing a large amount of bubbles might be fractured, and then the entire layer will be exfoliated and act as a dust in the vacuum vessel.

3.2 Vertical target tile: Tile 3 (3-2a)

The inner divertor strike points in many discharges were placed in Tile 3. The SEM image of 3-2a in Fig. 2 shows strong erosion features caused by high plasma flux. Indeed, cross-sectional SEM image and EPMA profile in Fig. 3-(b) indicates that there are no deposited elements on the original Mo-coating. A notable point in the EPMA spectra is that not only Mo but also Be showed high-counts in entire depth direction of the original Mo-coating. Fig. 5 shows the cross-sectional BF-TEM image and corresponding ED pattern from the original Mo-coating of the 3-2a sample. The original Mo-coating layer was composed by fine crystal grain with size of 200-500 nm. One of the possible reasons of the high counts of Be in EPMA analysis can be considered as follows. A certain amount of the eroded Be elements form the first wall was transferred to the divertor plasma and injected into the original Mo-coating. Since the divertor strike points in many discharges were located on the 3-2a tile, the surface temperature of the original Mo-coating could have been relatively higher than other tiles. Therefore, injected Be elements might have been diffused to the deeper region in the original Mo-coating. A certain amount of Be in the 3-2a sample area was also detected in previous research by means of Rutherford backscattering spectroscopy (RBS) analysis [6]. On the other hand, D retention of this area has been reported to be ~10^{22} D/m^2 which is lower than that of the deposition dominant area such as the apron part of the upper tile (Tile 1) as discussed in section 3.1 [6,7].

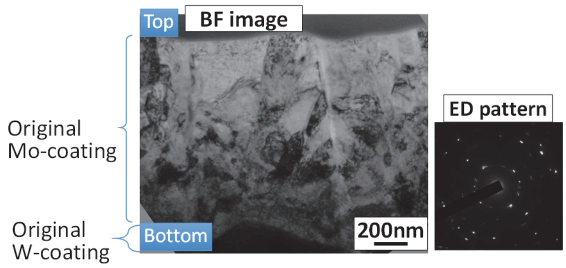

Fig. 5 Cross-sectional BF-TEM image of 3-2a sample and corresponding ED pattern from the original Mo-coating.

3.3 Horizontal target tile: Tile 4 (4-10b)

Previous studies have reported that tile 4 showed a deposition dominant feature, and main components of the deposition layer were Be, C and W [4]. In particular, special feature of this tile was that carbon was enriched in the deposits [4]. In the case of this study, mixed-material deposition layer composed by C, Be, W and O were also confirmed. C, W and O were detected by means of EDS, and Be was detected by analysis of Debye ring from ED pattern of TEM image. Judging from a surface morphology of this tile (4-10b sample) in SEM image of Fig. 1, a thick deposition layer was formed without directivity of the growth. Fig. 6-(a) shows the cross-sectional BF-TEM image of the 4-10b sample. A very fine stratified layer structure with the thickness of 200-300 nm was formed on the original W-coating. The dark layers in the BF-TEM image are mainly composed by a relatively high-Z element such as W, and the bright layers are low-Z element such as C, O and Be. Large size cavities, e.g., nano-size bubble-like structures confirmed in the 1-10c sample were not observed in the deposition layer. Since this sample was located at the shadowed area from a divertor plasma, plasma flux could not have been directly injected into the surface. Therefore, a static environment for the growth of the deposition layer seems to have been established around 4-10b sample. Fig. 6-(b) shows the ED pattern of the mixed-material deposition layer, and the diffused pattern indicates that the layer was composed by very fine grains. The dark field (DF) TEM image shown in Fig. 6-(b) was obtained from a part of the Debye ring as drawn in the white circle region in the ED pattern. The fine bright spots correspond to the individual grain of the deposited impurities. It should be noted that the grain size of the deposited impurities varied from an initial growth phase (bottom side of the DF-TEM image) to a latter phase (top side of the DF-TEM image). The grain size of the initial phase, within the thickness of up to around 50 nm from the original W-coating, is estimated to be ~10 nm in maximum, and it is larger than that of the latter phase with size of several nm. This means that history of the PWI seems to be different even in the growth period of the single deposition layer.

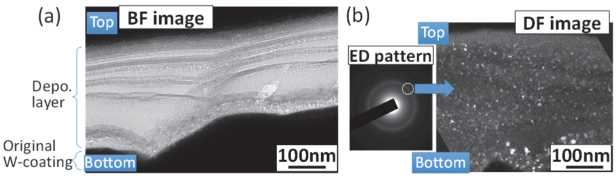

Fig. 6 (a) Cross-sectional BF-TEM image of 4-10b sample. (b) ED pattern of the mixed-material deposition layer and DF-TEM image obtained from a part of the Debye ring as drawn in the white circle region in the ED pattern.

Previous studies indicated that retention amount of D in this sample area was not largely different from the position of the 3-2a sample (~10^{22} D/m^2) [7]. In the case of the shadowed area like the 4-10b sample, D retention would be continuously and statically increased with increasing the discharge time. This might be lead to the increasing of the T retention. On the other hand, possibility of the exfoliation of the entire deposition layer might be lower than that of the other divertor area because nanostructure of the mixed-material deposition layer has very fine structure without any large size cavities.

4. Summary

Micro-/nano-characterization of surface of the divertor tiles from JET ILW of 1-10c (tile 1), 3-2a (tile 3) and 4-10b (tile 4) were conducted. The stratified and geological like mixed-material deposition layer composed by W, C, O, Mo and Be with the thickness of ~1.5 µm was formed on the original W coating of the 1-10c sample. Majority of the deposited elements was Be, and they had a strong incoming directivity to the surface. Many nano-size bubble like structures were observed in the deposition layer. These structure may lead to a high amount of the D retention in the tile 1 [6,7]. Such bubble-like structures could cause undesired dust formation due to the exfoliation of the deposition layer, although to date no exfoliation of divertor deposits has been observed. The surface of the 3-2a sample, originally coated by Mo, was identified as an erosion zone. This is consistent with the fact that the strike point was often located on that tile during the plasma operation. The deposited Be seemed to be diffused to the deeper region in the original Mo-coating. The diffused Be may change the property of a hydrogen isotope retention in the bulk Mo, but further fundamental investigation of the effects of the Be content in the bulk Mo should be conducted. The surface microstructure of the 4-10b sample also showed deposition dominant. A stratified mixed-material layer structure with the thickness of 200-300 nm was confirmed on the original W-coating. In the case of the shadowed area like the 4-10b sample, D retention would be continuously and statically increased with increasing the discharge time. This might lead to the increasing of the T retention, but the possibility of the exfoliation of the entire deposition layer might be lower than that of the other area of the divertor because the nanostructure of the mixed-material deposition layer has very fine structure without any large-size cavities.

In summary, the study revealed the micro- and nano-scale modification of the inner tile surface of the JET ILW. In particular, complex mixed-material deposition layer could affect the hydrogen isotope retention and dust formation.

Acknowledgments

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training program 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission. This work has been supported by the ITER Broader approach Activity.

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