EUROFUSION WPJET2-PR(14) 12665

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First dust study in JET with the ITER-Like Wall: Sampling, analysis and classification

Preprint of Paper to be submitted for publication in Nuclear Fusion Letter

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.

First scanning electron microscopic study of dust particles and debris from JET with the ITER-like Wall

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Abstract
This paper reports scanning electron microscope study of the dust from the divertor surfaces of JET vessel following the first operating period of JET ITER-like wall (JET-ILW) during 2010-2012. The particles fall into three categories: clusters of amorphous-like and crystalline-like regions, individual spherical metallic droplets and debris.

keywords:

dust, JET, ITER-like wall, Tungsten, plasma facing components, erosion, deposition,

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1. Introduction

In 2010 JET implemented the ITER-like wall (JET-ILW) with plasma facing components (PFC) of beryllium (Be) in the main chamber and tungsten (W) in the divertor as foreseen in ITER [1, [**2**]. The use of W and Be as materials for PFC has been studied and their properties summarised in several reviews. Understanding dust production in tokamaks remains an important issue for machine operations PFC lifetime [3[**7**] and safety [8]. Therefore studies of the quantity, composition and structure of dust generated due to erosion of PFC and disintegrating deposits are part of ongoing tokamak scientific programs. Already a number of studies have been dedicated to understanding the production and impact of airborne and surface based dust present in various tokamaks [9[**12**]. In addition, there is a continuing program of dust collection and *post mortem* analysis of PFC in JET-ILW to build up an accurate picture of material migration and mass balance [9, [**13**-[**15**]. In 2012 dust was collected from the divertor surfaces of JET vessel following the first operating period of JET-ILW during 2010-2012. Less than 1 g of material was collected using a cyclone vacuum cleaner controlled by remote handling. These samples are currently under analysis at the IFERC Rokkasho, Japan. In addition to this large scale remote collection samples were taken by hand from smaller areas of a divertor tile which had been removed from the JET vessel by remote handling. This paper reports Scanning Electron Microscopy (SEM) studies of the particles collected in this preliminary survey and provides the first insights into the nature of the dust produced by the JET-ILW. The analysis was carried out in the Materials Research Facility (MRF) at the Culham Centre for Fusion Energy (CCFE) in the UK. This new facility has been developed to conduct materials science for the nuclear industry and is capable of handling radioactive or harmful materials; some analysis is already possible and it will be fully operational in autumn 2015.

2. Experiment

Dust samples were collected from the upper horizontal surface of Tile 1 located at the top of the inner divertor [2]. Carbon adhesive pads were used for sampling. The pads were mounted on to standard aluminium SEM specimen stubs and applied directly on the surface of the tile to collect particles. The total area sampled using the adhesives pads was ~ 5 cm^2, i.e. 2 adhesive pads. The sampling took place in the JET Beryllium Handling Facility where samples containing Be and tritium (T) can be handled safely. Subsequent to the dust collection, *post mortem* analysis has shown that the region sampled is also the region where the majority of material deposited in the divertor is located [2, 13-15]. The samples were analysed by SEM and Energy Dispersive X-ray (EDX) spectroscopy using a TESCAN Mira3 XMH FE-SEM microscope equipped with an X-Max 80 EDS detector from Oxford Instruments. The electron beam was operated at 10-30 kV. It is not possible to analyse Be with this system, however the presence of Be has been confirmed lately using ion beam analysis.

3. Results and discussion

The particles collected can be divided in three categories: clusters, individual spherical metallic droplets and debris. The secondary electron micrographs in Fig. 1 show examples of cluster particles, the most frequently observed particle type. The size of these particles varies in the range 20×40 µm (Fig.1a) to 4×4 µm (Fig 1d). Particles consist of oblong or irregular shape zones with a glassy/amorphous-like appearance, fine spherical particles, and crystalline-like agglomerates. The chemical composition of the crystalline-like and amorphous-like zones consists mainly of W, Mo, O and trace elements such as Cu, Fe and Zr.

The crystalline-like zones indicated by an arrow in Fig.1b have a higher W concentration than the amorphous regions. In general the W concentration is ~80 at.% and in some cases above 90 at.%, with the remaining constituents O <9 at.% and Mo <10 at.%. The glassy-like zone, an example of which is indicated in Fig 1b is characterised by higher O content - in some regions up to 35 at.%, and lower W concentration ~50 at.% than the crystalline-like regions. The Mo concentration is between 5-10 at.% and trace elements < 4 at.%. In Fig 1c the narrow, darker band (indicated by arrows) consist of almost 53 at.% of Mo whereas the brighter band has almost 80 at.% of W with only 5% of Mo. Fig 1d shows cluster of droplets. The smallest distinguishable droplets diameter is <50 nm. These are visible at the edges of the cluster shown in Fig. 1d. The average composition of the droplets is 15 at.% (W, Mo) and 85 at.% O. High O presence is most likely due to oxygen adsorption from the atmosphere; presence of BeO (Be easy interact and create strong bonds with O). It should be mentioned that during JET- ILW operation there was oxygen leak which might be partially responsible for relatively high O content in dust samples. Be has been identified by IBA, but will not be presented here due to limited space.

The second type of particles observed are individual spherical droplets (Fig. 2). EDX analysis of these particles shows two chemical compositions: Ni-based or W-based. The diameters of the Ni-rich droplets are <5 μm and those of the W-rich droplets are <1 μm. The average composition $Ni_{0.74}Cr_{0.16}(\text{other elements})_{0.10}$ (at.%) of the Ni-based droplets is similar to composition of Inconel. The surface of the Ni-based shown in Fig. 2 and W-based particles (not shown here) of almost 90 at.% W and 10 at.% Mo with very little O detected, have visible boundaries. These could be a larger particle with thermal cracking due to rapid cooling or alternatively smaller particles fused together.

Examples of the third type of particle shown in Fig 3 are classified as debris. Fig 3a shows clusters which reaches up to 500 μm in size. This is a typical plate/disc shaped particle of BN with an average composition of $B_{0.45}N_{0.55}$ (at.%). Erosion of a reciprocating probe located at the top of the vessel is the most likely source of BN [18]. Other types of debris include carbon fibres originating from CFC tiles and components (Fig 3b) and particles with an elemental composition which indicates its erosion from Inconel components.

4. Conclusion

In general, the particles collected consist primarily of the PFC materials used in the machine and additionally of materials used in JET for conditioning, insulation and diagnostics, e.g. C fibres, Cr and Ni from Inconel used extensively in the structural materials of JET, Mo and W used in carbon fibre composite (CFC) tile coatings in the divertor and main chamber [17] or BN which is used in in-vessel diagnostics. The JET W-coatings are 10-20μm thick and have a Mo interlayer to improve the interface to the CFC and this layer is 10-20% of the layer thickness [17] and so the observed Mo concentrations are consistent with this origin. The composition is broadly consistent with analysis of transient impurity events in JET [3]. Further detailed study of ILW dust particle morphology, Be and D content and also dust survey from other JET tokamak regions is planned. This is part of scientific activity foreseen for the next JET shut-down in 2015.

Acknowledgement

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. The CCFE Materials Research Facility is part of the UK's National Nuclear User Facility initiative.

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Figure captions

Figure 1. The secondary electron micrographs of cluster particles: (am)-amorphous-like zone, (cr)-crystalline-like zone.

Figure 2. The secondary electron micrographs of Ni-based or W-based individual droplets.
Figure 3. Particles are classified as debris: (a) boron nitride, (b) carbon fiber.

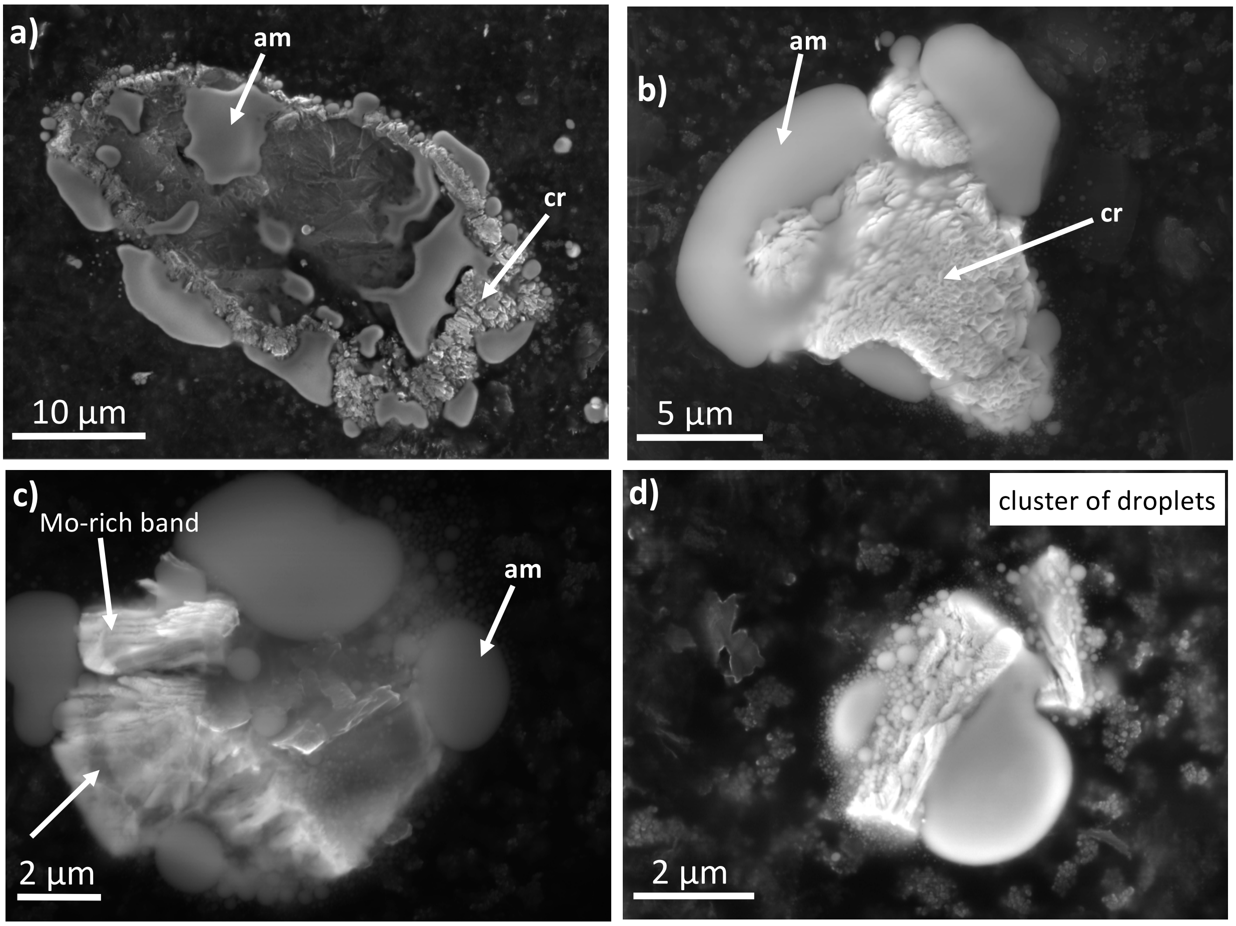
a)
am
cr
10 μm
b)
am
cr
5 μm
c)
Mo-rich band
am
2 μm
d)
cluster of droplets
2 μm

Figure 1.

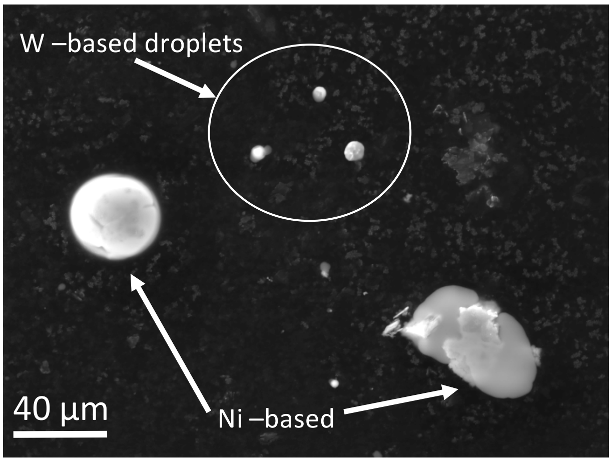

Figure 2.

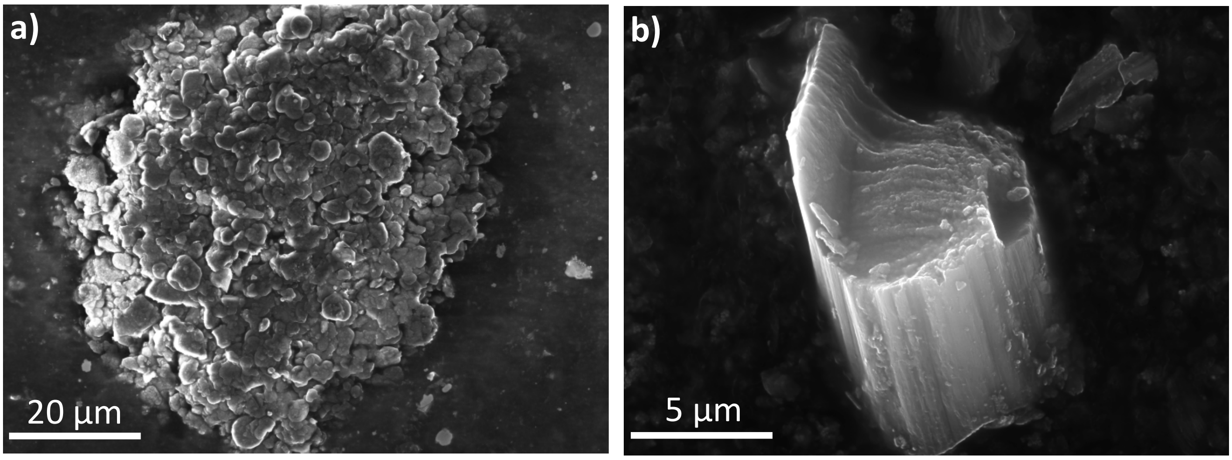

Figure 3.