Comprehensive Analysis of Metal Dust Particles in JET-ILW, and Impact on Fusion Reactor

N. Ashikawa[1,2*], N. Asakura[3], M. Oyaidzu[3], K. Isobe[3], A. Widdowson[4], J. Grzonka[5,6], M. Rubel[7], M. Hara[8], T. Otsuka[9], D. Hamaguchi[3], H. Kurotaki[3], S. Nakano[3], J.H. Kim[3], M. Miyamoto[10], Y. Hatano[8], R. Sakamoto[1,2], K. Heinola[11], A. Baron-Wiechec[4], H. Tanigawa[3], M. Nakamichi[3], T. Yamanishi[3] and JET Contributors**

[1]*National Institute for Fusion Science, Toki, 509-5292 Japan*
[2]*SOKENDAI, Toki, 509-5292 Japan*
[3]*National Institute for Quantum and Radiological Science and Technology, Rokkasho, 039-3212 Japan*
[4]*CCFE, Culham Science Centre, Abingdon, OX14 3DB UK*
[5]*Warsaw University of Technology, Warsaw, 01-152 Poland*
[6] *Institute of Electronic Materials Technology Warsaw, 01-919 Poland*
[7]*Royal Institute of Technology (KTH), Stockholm, SE-10044 Sweden*
[8]*University of Toyama, Toyama, 930-8555 Japan*
[9]*Kindai University, Higashi-Osaka, 577-8502 Japan*
[10]*Shimane University, Matsue, 690-8504 Japan*
[11]*University of Helsinki, Helsinki, FI-00560 Finland*
*** See the author list of "Overview of the JET results in support to ITER" by X. Litaudon et al. to be published in Nuclear Fusion Special issue: overview and summary reports from the 26th Fusion Energy Conference (Kyoto, Japan, 17-22 October 2016)*

[*] *E-mail contact of main author: ashikawa@lhd.nifs.ac.jp*

Abstract: A comprehensive analysis of collected dust particles in the Joint European Torus ITER-like Wall (JET-ILW) after the first campaign in 2011-2012[1] has been carried out at the International Fusion Energy Research Centre (IFERC) in order to identify dust characteristics such as structures, material components and hydrogen isotope retention. Analysis of the cross-section structure as well as surface one began with from a large-size dust particle (~100 μm). For the flake-type beryllium (Be)-based dust, it was found that the damaged Be crystal structure contained a larger oxygen component near the surface (~2 μm). The specific dust flake of beryllium oxide in the experiment device was shown for the first time. Deuterium retention in small weight of dust particles (4.4 mg) was evaluated to be 1.2 x 10^{21} atoms/g by thermal desorption spectrometry (TDS), which estimated to be 8.2 x 10^{20} atoms for entire dust particles collected from the inner divertor (0.7 g). This result firstly suggests that a contribution of dust particles to the total retention in the experiment was small, i.e. less than 1% of the total retention in deposition layers of the inner divertor target.

1. Introduction

In Demo reactor designs, tritium retention and materials/dust depositions on plasma facing materials (PFMs) are influenced by boundary plasma conditions and temperatures on surfaces of PFMs [2]. Hence, it is important to understand retentions of hydrogen isotopes, characteristics of material/dust particles depositions and their amounts. However, analytical data of metal dust particles in actual fusion devices are not sufficient due to limited number of metal wall machines [3-6].

Joint European Torus (JET) started operation with the ITER-like Wall (ILW) in 2011[1] using tungsten (W)-coated CFC/bulk-tungsten divertor tiles and beryllium first wall, respectively. The main aims are: development of an integrated operation scenario for ITER, power handling and material behavior with metal walls, assessment of fuel inventory and development of ITER-oriented engineering solutions, e.g. remote handling.

After the first JET-ILW campaign (2011-2012) dust particles were collected from the vacuum vessel [7]. Dust particles in JET contain tritium and beryllium. For analyses of JET-ILW dust particles, so-called controlled areas with access restrictions are necessary. A R&D building in JAEA Rokkasho Fusion Research Centre fulfils these requirements and new JA-EU collaboration work [2] in Rokkasho center started in 2013. Dust particles from JET with carbon (JET-C) and metal wall (JET-ILW) were transported to Japan; materials arrived at the Rokkasho center in August, 2014. Following the first JET-ILW operation, dust particles were collected from the tiles in 22 divertor modules, i.e. from 92% of the divertor area. In total nearly 1 gram was collected: 0.7 g from the inner and 0.3 g from the outer divertor [7, 8]. It should be stressed that these quantities are over two orders of magnitude smaller than carbon phase dust particles of 188 g.

The determination of dust characteristics, such as structures, material composition and hydrogen isotope retention are important issues. In particular, observations of tritium amounts and their relationship between tritium and compositions are required. Therefore, this study was focused on material composition and hydrogen isotope retention in dust from JET-ILW.

2. Experimental

2.1 Dust collection

The dust collections were done using a cyclone type vacuum cleaner after the vacuum vent in JET. Collected dust particles were accumulated in a pot located below a cyclone on installed on the vacuum cleaner. Dust particles were collected separately from the inner (Tiles HFGC, 1, 3 and 4) and the outer (Tiles 5, 6, 7 and 8) regions in the divertor. In Fig. 1 they are marked in red and blue, respectively. Access to the remote areas of the divertor was not

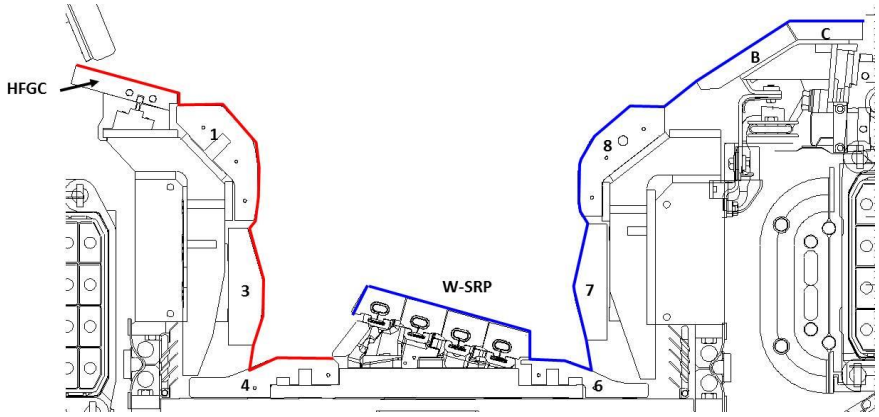

Figure.1 Schematic drawing of cross section of the divertor surface areas for dust vacuuming shown at the inner divertor in red and the outer divertor in blue. The masses of dust collected from the inner and outer divertor are 0.7 and 0.3 g, respectively [3].

possible during the shutdown in 2012. The amounts of dust particles collected from the inner and outer divertors were 0.7 and 0.3 g, respectively [3].

ILW dust particles from these two locations were transported from Culham in UK to Rokkasho Center in Japan. Dust particles from JET-C campaign 2009 were also sent to Rokkasho for comparison. Dust particles at each locations were stored in different glass pots and sealed in aluminum foil bags to reduce tritium off-gas from the package for the shipment from the UK to Rokkasho Center. For a collection of released tritium from dust particles, we inserted into aluminum packages in a glove bag, and opened these packages. The maximum count of released tritium in the glove bag was 98Bq/cm^3 measured by environmental tritium monitor systems. High level tritium was remained on surfaces of glass pots and surface cleaning was necessary before taking out from the glove bag. Afterwards the weight of each pot was measured by a microbalance in order to check future history. All bottles are kept in stainless steel containers; JET-ILW and JET-C dust particles are separated.

2.2 Surface analysis

Surface morphology was determined by an optical microscope and a scanning electron microscope (SEM; JEOL JXA-8530F) with an energy dispersive X-ray spectroscopy (EDX). The atomic concentrations on the surface of dust particles were measured using X-ray photoelectron spectroscopy (XPS; ULVAC-PHI Inc., PHI500 Versa Probe II). A 25 W Mg source was used with a 100 μm spot size. An argon ion gun of 4 keV was used to etch the surfaces of dust particles to remove surface contaminants originated from the sample exposure to air. Nano-scale observations were conducted by the transmission electron microscope (TEM). An atomic concentration on cross-section image was measured by Electron probe micro analyzer (EPMA; JEOL JXA-8530F). Signals of EPMA are very sensitive to surface roughness, hence a dust particle was treated by a focused ion beam (FIB) to make a cross-section and smooth surface for this analysis.

Retention and trapping characteristics of hydrogen isotopes in a small amount of dust particles were evaluated by TDS measurements. The thermal desorption of molecules was measured under a heating rate of 0.5 K/s up to 1273 K by quadrupole mass spectroscopy (QMS). For the TDS analysis, dust particles could not be placed directly in the TDS vacuum chamber. Therefore, a few milligrams of dust particles was placed on a tantalum tray with an inner diameter of 8 mm and a depth of 1 mm, which was set on the TDS system. This method of TDS was operated for dust particles of JT-60U in QST, Naka [3]. The quantitative tritium was analyzed using a liquid scintillation counting (LSC). In this analysis for ILW dust particles, limited dust particles less than 1g could be used. To catch dust particles located on the inner wall of glass pot connected by the static electricity, polycarbonate membrane filter, which wipes off dust particles, was used. Using the combination between the standard cocktail of Hionic Fluor and the toluene base solution, namely Soluene-350, polycarbonate membrane filters were dissolved before LSC analysis. Dust particles were put on copper tapes to fix these dust particles on base plates, in the case of SEM, TEM and XPS, so on.

3. Results and discussion

3.1. Surface morphology and composition

Surface morphologies of overall dust particles at the inner divertor are shown in Fig.2 by SEM/backscattering detector and a size of these dust particles is less than 100 μm. Main elements of dust particles are identified: Be and W with admixtures of oxygen (O), carbon (C), nickel, nitrogen, molybdenum and some aluminum measured by EDX and EPMA. In Fig.2, 10% of dust particles is identified as W, and remaining dust particles are assessed to be Be. The analysis started from a large-size flake dust, i.e. 40-120 μm, to determine material components both on the surface and in the cross-section by cutting the dust (using FIB) as shown in Fig.3. A scanning ion microscope (SIM) image on the cross-section and a spatial profile of an atomic concentration measured by EPMA are shown in Fig. 4 (a) and (b), respectively. A cross-section of dust flake is the middle region with the width of 8 μm, and the upper and lower layers corresponds to W-coat for protection of FIB etching and a part of a copper tape, respectively. Analytical results show that main component is Be and that a higher oxide concentration of 10% is observed only at the bottom of the sample (equivalent to the top surface of the Be layer). At the bottom

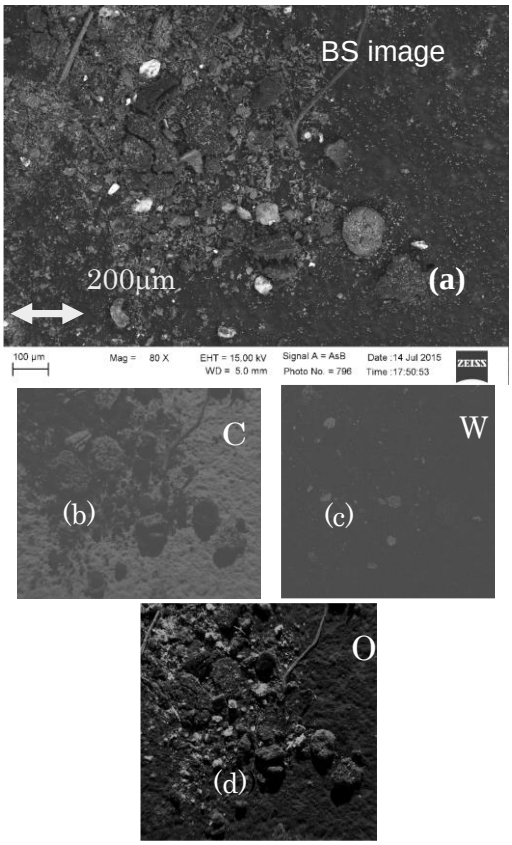

Figure.2 (a) Surface morphologies of overall dust particles measured by SEM / backscattering detector. (b)-(d) 2-D mapping images of C, W, O compositions measured by EDX. The same scale was used in SEM and EDX images.

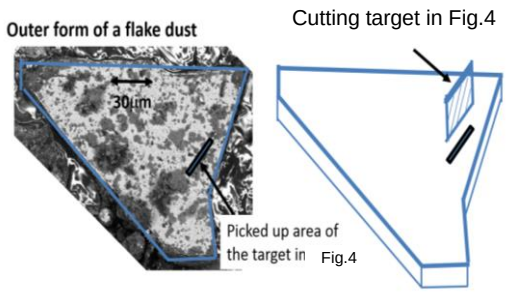

Figure.3 Surface morphology of a Be flake dust before FIB treatments. A hatching region is a picked up area of the target in Fig.4.

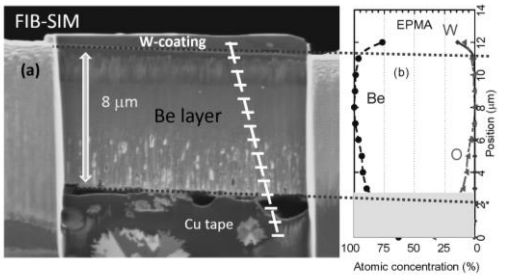

Figure.4 (a) Cross-section image of beryllium flake dust measured by SIM on FIB. A broken line shows measurement positions by EPMA. (b) An atomic concentration is measured by EPMA for the cross-section.

site in Fig. 4(a), white dots, which were damaged regions in the Be flake measured by TEM, were shown. For a thin film of this Be flake dust made by FIB, diffraction patterns of TEM were observed as shown in Fig. 5. From the comparison with the Be flake dust and reference data of metal beryllium, similar lattice pattern, which lattice constants using analytical results are a=2.4Å in the Be flake and 2.264Å [9] in the metal beryllium by reference data, is shown in Fig.5. Characterizations of the Be flake were shows, such as the dense crystal structures and chemical bindings of beryllium oxide with damages.

In Ref.10, retention deuterium concentration in C, Be and W deposits under co-deposition is reported. For beryllium, retention in beryllium oxide with carbide is one order of magnitude larger than metal beryllium. Hence, characterizations of beryllium in ILW materials important for discussions of hydrogen isotope retention and tritium inventories.

3.2. Hydrogen isotope retention in dust particles

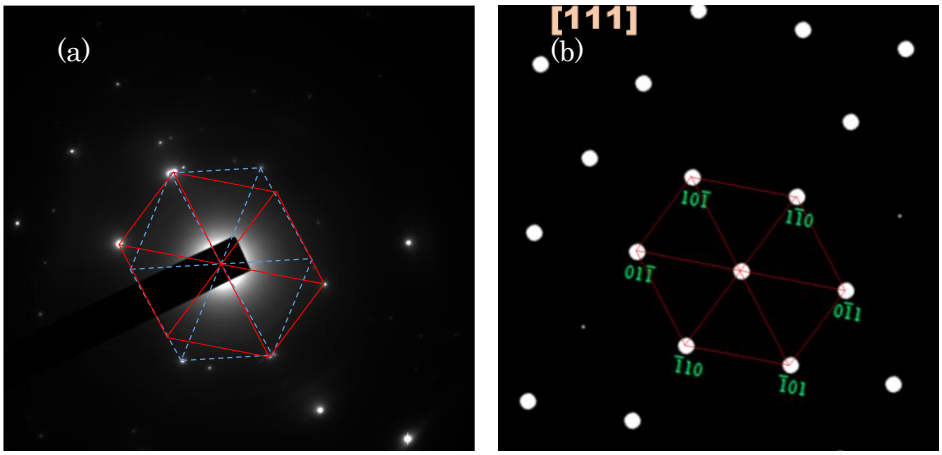

Figure.5 Diffraction pattern in the zone axis incident measured by TEM for (a) a Be flake dust as shown in Fig.3 and (b) reference data of metal beryllium.

Quantitative tritium measurement for ILW dust particles was done using LSC. Dust particles wiped-off by polycarbonate membrane filter in glass pod were caught and put in Hionic-Fluor cocktail with Soluene-350. Detected tritium activity from ILW dust particles at the inner divertor was 0.1 GBq/g, as estimated from the analysis of 2.3 mg of dust particles; the mass was determined by the microbalance. Some particles placed in the cocktails remained after the LSC analysis, and it was considered that not total tritium inventory from dust particles. Hence,

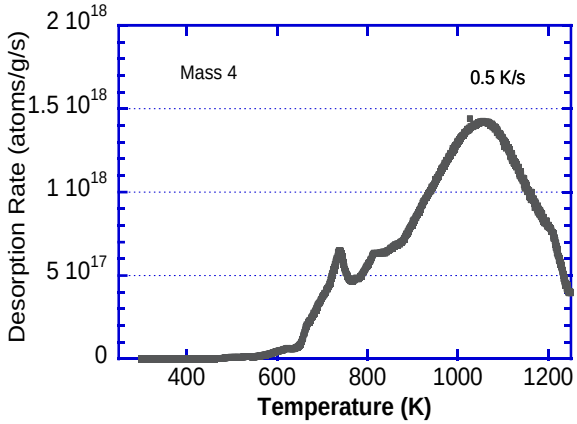

Figure.6 Thermal desorption spectrum for ILW dust particles.

it is thought that this result is underestimated.

Retention and trapping characteristics of hydrogen isotopes in a small amount of dust particles (4.4 mg) were evaluated by TDS measurements. It was a part of the JET-ILW dust (0.7 g) collected over a wide surface area in the inner divertor. The thermal desorption spectrum is shown in Fig.6. The total D retention evaluated mainly from the mass 4 signal of D_2 is 5.2 x 10^{18} D atoms, and an average retention of the dust particles in the inner divertor was estimated to be 1.2 x 10^{21} atoms/g. The first report of 2011-2012 campaign showed approximately 1.7 x10^{26} D atoms were puffed and 3.7 x 10^{23} D retained (0.2 %) mainly in the Be deposition layers at the inner divertor [11]. Total retained D in dust particles is estimated to be 8.2 x10^{20} D atoms in the inner divertor, less than 0.41% to the retention in the divertor tile surface, which shows that metal dust is not considered as a tritium retention source. It should be noted that D desorption is increased at the high temperature (> 800 K) and peaked at 1050 K, which does not appear in the standard beryllium deuteride powder [12]. It is necessary to investigate of relationship between hydrogen isotope retention, and surface morphologies and oxidations on JET-ILW dust particles.

4. Summary

A comprehensive analysis of collected dust and divertor tiles in the JET -ILW after the first campaign in 2011-2012has been carried out at IFERC.

Analysis of the cross-section structure as well as surface one started from a large-size dust particle (~100 μm). For the flake-type beryllium (Be)-base dust, it was found that the damaged Be crystal structure with containing larger oxygen component near the surface (~2 μm). In ILW dust particles, beryllium dust

Tritium retention of ILW dust particles was measured using LSC, and tritium activity of 0.1 GB/g in the inner divertor using 2.3 mg dust particles is shown.

D retention in small weight of dust particles (4.4 mg) was evaluated to be 1.2 x 10^{21} atoms/g by TDS, which corresponds to 8.2 x 10^{20} atoms in dust particles (0.7 g) collected in the inner divertor. This result firstly suggests that retention in the dust is small, i.e. less than 1% of the total retention in deposition layers of the inner divertor target.

Acknowledgments

This work was supported by ITER Broader approach activity, NIFS budget KEMF078, and International Fusion Energy Research Centre (IFERC) in Rokkasho. The authors would like to thank Mr. T. Suzuki and the tritium engineering laboratory in QST for helpful support about tritium handlings, and Drs. S. Masuzaki, M. Tokitani, Y. Torikai and T. Nakano for a useful discussion. The authors would also like to thank the framework of the EUROfusion Consortium.

References

[1] BREZINSEK S., JET-EFDA Contributors, “Plasma-Surface interaction in the Be/W environment: Conclusions drawn from the JET-ILW for ITER”, Journal of Nuclear Materials, 473 (2015) 11.

[2] YAMANISHI T., ASAKURA N., TOBITA K., OHIRA S., FEDERICI G., HEIDINGER

R., KNASTER J., CLEMENT S., NAKAJIMA N., other BA IFERC and IFMIF/EVEDA Contributors, “Recent technical progress on BA program：DEMO activities and IFMIF/EVEDA”, Fusion Engineering and Design 109-111 Part B (2016) 1272.

[3] ASHIKAWA N., ASAKURA N., FUKUMOTO M., HAYASHI T., UEDA Y., MUROGA T., “Characteristics of tungsten and carbon dusts in JT-60U and evaluation of hydrogen isotope retention”, Journal of Nuclear Materials, 438, (2013) S659.

[4] ASAKURA N., HAYASHI T., ASHIKAWA N., FUKUMOTO M., “Investigation of carbon dust accumulation in the JT-60U divertor plasmas”, Journal of Nuclear Materials, 438, (2013) S659.

[5] ENDSTRASSER N., ROHDE V., BALDEN M., HUMRICKHOUSE P.W., TOUSSAINT U.,BRAAMS B., CHUNG H.K-., NEU R., the ASDEX Upgrade team”, “ Comparative study of the dust particle population sampled during four consecutive campaigns in full-tungsten ASDEX Upgrade”, Physica Scripta, T145 (2011) 014021.

[6] BALDEN M., ENDSTRASSER N., HUMRICKHOUSE P.W., ROHDE V., RASINSKI M., TOUSSAINT U., ELGETI S., NEU R., the ASDEX Upgrade Team, “ Collection strategy, inner morphology, and size distribution of dust particles in ASDEX Upgrade”, Nuclear Fusion (2014) 073010.

[7] WIDDOWSON A., ALEVS E., AYRES C.F., BARON-WIECHEC A., BREZINSEK S., CATARINO N., COAD J.P., HEINOLA K., LIKONEN J., MATTHEWS G.F., “Material migration patterns and overview of first surface analysis of the JET ITER-like wall” Physica Scripta, T159 (2014) 014010.

[8] BARON-WIECHEC A., FORTUNA-ZALESNA E., GRZONKA J., RUBEL M., WIDDOWSON A., AYRES C., COAD P., HARDIE C., HEINOLA K., MATTHEWS G.F., “First dust study in JET with the ITER-like wall : sampling analysis and classification”, Nuclear Fusion, 55 (2015) 113033.

[9] PETRUNIN V.F., Inorganic Materials 20 (1984) 46-48.

[10] ROTH J., TSITRONE E., KOARER T., PHILIPPS V., BREZINSEK S., LOARTE A., COUNSELL G.F., DOERNER R., SEHMID K., OGORODNIKOVA O., CAUSEY R.A., “Tritium inventory in ITER plasma-facing materials and tritium removal procedures”, Plasma Physics and Controlled Fusion, 50 (2008) 103001.

[11] HEINOLA K., WIDDOWSON A., LIKONEN J., ALVES E., BARON-WIECHEC A., BARRADAS N., BREZINSEK S., CATARINO N., COAD P., KOVIYUANTA S., KRAT S., MATTHEWS G.F., MAYER M., PETERSSON P., and JET Contributors, “Long-term fuel retention in JET ITER-like wall”, Physica Scripta, T167 (2016) 014075.

[12] DOERNER R., BALDWIN M.J., BUCHENAUER D., TEMMERMAN G.De., NISHIJIMA D., “ The role of beryllium deuteride in plasma-beryllium interactions”, Journal of Nuclear Materials, 390-391 (2009) 681.