Nuclear Materials and Energy 17 (2018) 279–283

Contents lists available at ScienceDirect

Nuclear Materials and Energy

journal homepage: www.elsevier.com/locate/nme

Tritium retention characteristics in dust particles in JET with ITER-like wall

T. Otsuka[a,*], S. Masuzaki[b], N. Ashikawa[b], Y. Hatano[c], Y. Asakura[d], Tatsuya Suzuki[d], Takumi Suzuki[d], K. Isobe[d], T. Hayashi[d], M. Tokitani[b], Y. Oya[e], D. Hamaguchi[d], H. Kurotaki[d], R. Sakamoto[b], Hiroyasu Tanigawa[d], M. Nakamichi[d], A. Widdowson[f], M. Rubel[g], JET Contributors[1]

[a] Kindai University, 3-4-1 Kowakae, Higashi-Osaka 577-8502, Japan
[b] National Institute for Fusion Science, 322-6 Oroshi-cho, Toki 509-5292, Japan
[c] University of Toyama, Gofuku 3190, Toyama 930-8555, Japan
[d] National Institutes for Quantum and Radiological Science and Technology, Rokkasho Aomori 039-3212, Japan
[e] Shizuoka University, Shizuoka, 422-8529, Japan
[f] Culham Centre for Fusion Energy, Culham Science Centre, Abingdon, OX 14 3DB, UK
[g] Royal Institute of Technology (KTH), 100 44 Stockholm, Sweden

ARTICLE INFO

Keywords:
JET-ILW
Dust
Tritium
Imaging plate
Electron prove microscopic analysis

ABSTRACT

A tritium imaging plate technique (TIPT) in combination with an electron-probe microscopic analysis (EPMA) were applied to examine tritium (T) retention characteristics in individual dust particles collected in the Joint European Torus with the ITER-like Wall (JET-ILW) after the first campaign in 2011–2012.

A lot of carbon (C)-dominated dust particles were found, which would be pre-existing carbon deposits in the JET-C or released carbon particles from the remaining carbon-fiber components in the JET-ILW. Most of T was retained at the surface of and/or in the C-dominated dust particles. The retention in tungsten, beryllium and other metal-dominated dust particles is relatively lower by a factor of 10–100 in comparison with that in the C-dominated particles.

1. Introduction

Tritium (T) retention in dust generated in fusion devices with carbon (C) walls is influencing safety of reactor operation, due to the high content of radioactive hydrogen isotopes in carbon particles [1]. To mitigate this issue a full metal wall will be installed in ITER. After the successful experiments of the Joint European Torus with the ITER-like Wall (JET-ILW) [2], the generation of dust and debris was reduced by a factor of ~100, and the morphology and composition of metallic dust particles have been characterized by many methods [3–6]. Under the framework of Broader Approach (BA) activity in Japan [6], amounts of T on/in dust generated in JET-ILW have quantified T by liquid scintillation counting and thermal desorption methods [7]. The results showed that the amount of T per unit mass of the JET-ILW dust was not as low as expected [8] and rather comparable with that per unit mass of dust generated in JET with carbon wall (JET-C). Since various species of metallic and metal oxides dust would be generated in JET-ILW and then ITER, it is important to understand and determine tritium (T) retention and its behavior in individual dust particles with repect to their microstrutures and compositions. In the present study, we have applied a tritium imaging plate technique (TIPT) in combination with an electron-probe microscopic analysis (EPMA) to examine T retention characteristics in individual dust particles collected at the inner diverter region in JET-ILW after the first campaign in 2011–2012 [2].

2. Experimental

Dust particles were collected by a vacuum cleaning method at the surfaces of the tiles at the inner divertor region in JET with ILW after the first campaign 2011–2012 [9]. The total amount collected from the inner divertor only was on the level of 0.77 g and a part of this (0.14 g) was sent as a sample to International Fusion Engineering Research Center (IFERC) at Rokkasho in Japan [6,7]. A tiny amount of dust was taken from that sample and gently mounted on the surface of a disk made of indium (In). The intensity of T retained at the surface of and/or in the dust particles was evaluated by TIPT [10,11]: emission of β-rays

* Corresponding author.
E-mail address: teppei.otsuka@ele.kindai.ac.jp (T. Otsuka).
[1] See author list in the paper, X. Litaudon et al., Nucl. Fusion, 57 (2017) 102001.

https://doi.org/10.1016/j.nme.2018.11.001
Received 30 July 2018; Received in revised form 1 November 2018; Accepted 1 November 2018
Available online 27 November 2018

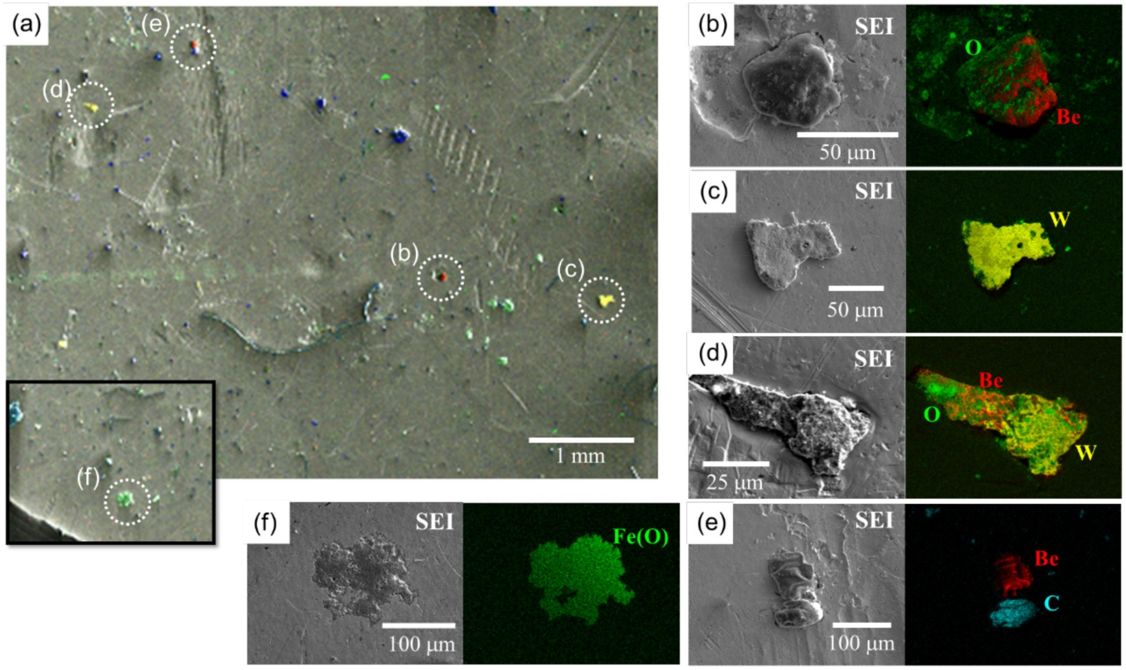

Fig. 1. Secondary electron image of (a) the whole surface area of In disk with EPMA image of C, Be, O and W, and magnified EPMA image of (b) a Be-dominated dust particle, (c) a W-dominated particle, (b) a W-dominated particle co-deposited with Be, (e) a Be-dominated dust particle neighboring a C-dominated particle and (f) an Fe oxide dust particle.

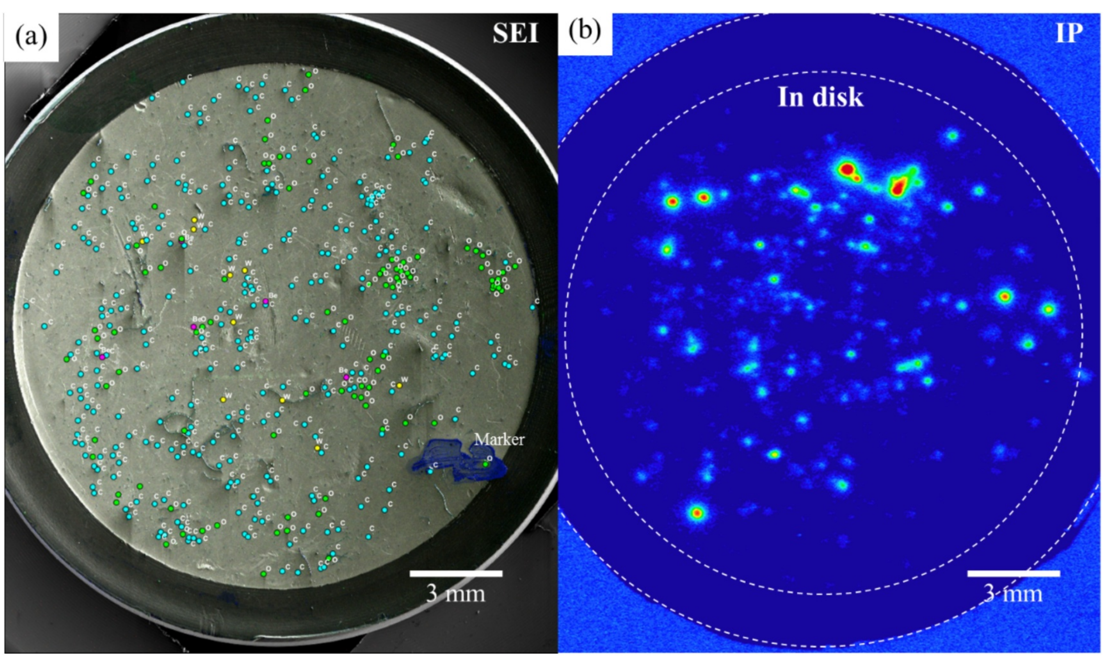

Fig. 2. (a) EPMA image showing composition of dust particles. Small circles with superscript labels indicate the location and composition (C, O, Be and W) of dust particles. (b) IP image showing T distribution on the In disk.

Table 1
Composition and number of dust particles with/without tritium.

Main elements included in dust	Number of dust particle	Number of dust particle with tritium	Number density of dust particles with tritium (%)
C	322	124	85
Metals (Fe, Ni) and Si oxides	93	11	<7
Be	5	4	<3
W	11	8	<6

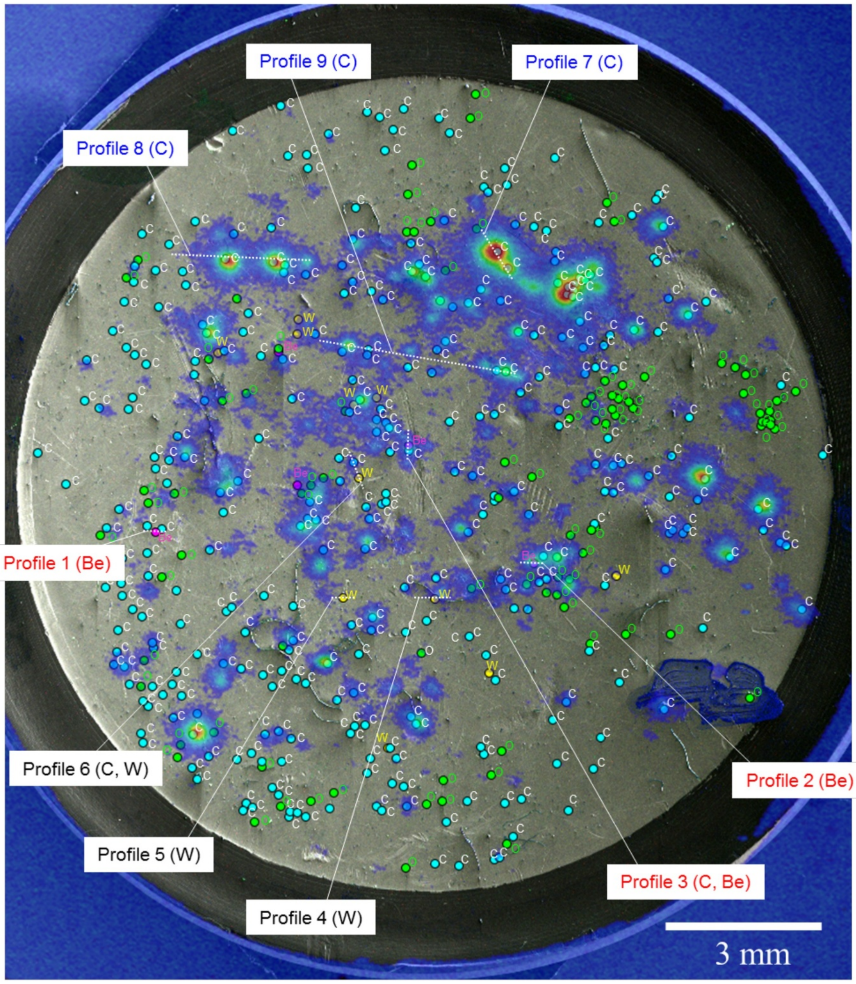

Fig. 3. Super-imposed image of Fig. 2(a) EPMA image and (b) IP image. White dashed lines indicate the position of line profiles shown in Figs. 4.

from T to the imaging plate (IP) was measured over a whole surface of the In disk for 1 h at room temperature. The spatial resolution of TIPT was 25 µm. Afterward, Electron Probe Microscopic Analysis (EPMA) was conducted to determine morphology (composition and structure) of the dust particles on the same surface area where TIPT was conducted. EPMA (or wavelength dispersive X-ray spectroscopy, WDS) using 10 keV electron beam gives information on the elemental composition with around 1 µm spatial resolution. The compositional analysis of the whole In disk was performed with a resolution of 20 µm and the detailed analysis of individual dust particles was separately analyzed with a resolution of 1 µm. The obtained maps (images) of the T distribution and elemental compositions were super-imposed to reveal characteristic features of T retention in the individual dust particle with the aid of image analysis software FIZI [12].

3. Results and discussion

3.1. Compositions of dust particles

Fig. 1(a) is an image showing a secondary electron image (SEI) with EPMA data for each element at the surface of the In disk. In the image, small dots of blue, green, yellow, and red indicate the distribution of the elements, carbon (C), oxygen (O), tungsten (W) and beryllium (Be), respectively, on the background of black & white grey colors of SEI. Although one dot in the image represents an individual dust particle greater than 20 µm × 20 µm in size, the elements in the individual particles and their locations on the disk were clearly identified. All the detailed features or characteristics of the individual dust particles were well summarized in [3–5,9] and are briefly introduced here to categorize which element dominated in each dust particle. Fig. 1(b) shows a dust particle mainly composed of Be with O, approximately 50 µm × 50 µm in size and rather flat surface. This Be-dominated dust particle is likely to be a flake of deposit plausibly oxidized after removal from the JET vessel. Fig. 1(c) shows a W-dominated dust particle, which would be a flake of deposit or agglomerates originating from the W coating. One flake appears in combination with Be and O indicating that it originates from co-deposits as shown in Fig. 1(d). Two distinct dust particles were identified in Fig. 1(e) as a Be-dominated dust particle and a C-dominated one, which were close to each other by coincidence during the sample preparation. There were many oxides with various elements, such as iron (Fe), nickel (Ni) and silicon (Si) which might originate from structural components like steels and Inconel alloys, and diagnostic tools.

Fig. 2(a) clearly indicates the location of C-, Be-, W-dominated dust particles and metals oxide dust particles as small spots with superscript labels (C, Be, W, and O) across the whole surface of In disk. The number of each type of dust particles were counted by hand and summarized in Table 1. Very few Be- and W-dominated dust particles were found in comparison with C-dominated and metals oxide dust particles, indicating that loose metallic dust particles were not generated in significant numbers from the ILW. Note that, though the ILW was a full metal plasma-facing wall, carbon-fiber composites exist in the vessel as (a) divertor carrier structures and, (b) the substrate material of inner and outer divertor tiles where the plasma-facing surfaces and sides are covered with W coatings but the back of tiles are not. A small number of C-particles could be released from these components during the

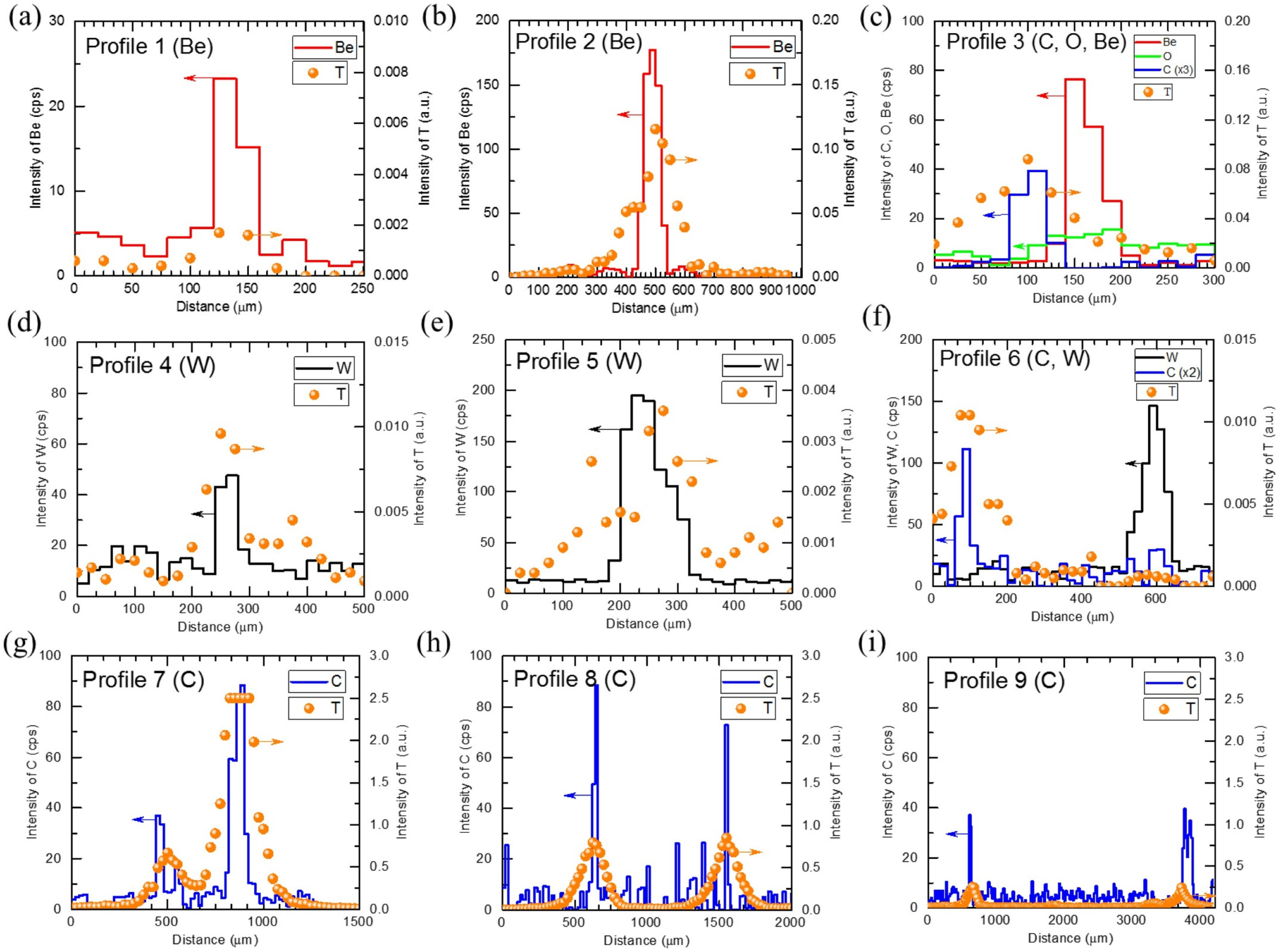

Fig. 4. Line profiles of intensity of T and elements, (a)–(c) for Be-dominated dust particles, (d)–(f) for W-dominated ones and (g)–(i) for C-dominated ones.

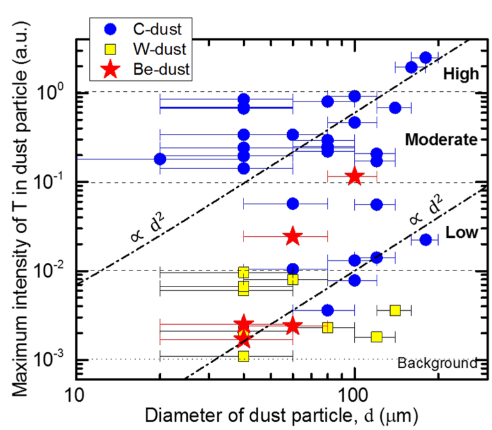

Fig. 5. T retention characteristics of the dust particles summarized for each element with respect to the size of the particle.

installation of plasma facing components for ILW. Additionally, a small amount might originate from C-rich deposits which could remain from the JET-C phase in places difficult to access by cleaning performed before the ILW installation, for example on the surface of the water-cooled louvers in the remote divertor corners where C-based deposits may still be present.

3.2. Tritium retention of the dust particles

Fig. 2(b) is an IP image showing the distribution of T intensity on the whole surface area of the In disk. The scale indicates increasing T amount as the colour changes from green to yellow to red on the background colour of blue. A variety of different colours and sizes of green and red spots indicate different amounts of T retained on surfaces or in the bulk of individual dust particles and their locations on the In disk. In order to examine quantitatively which type of dust particles retain T or not, the images of the elemental compositions (Fig. 2(a)) and T distribution (Fig. 2(b)) are super-imposed together as shown in Fig. 3. Two facts were immediately drawn from Fig. 3, (i) not all dust particles retain T and (ii) T was found at locations where the dust particles were not recognized in the EPMA image with the resolution of 20 μm. The former was summarized in Table 1 showing that 61% and 88% of the C-dominated and metals oxide dust particles did not retain T. Non-T containing particles could be attributed to their generation plausibly by inert gas discharges and hydrogen plasma in JET-C or at the remote area in JET-ILW, or possible contamination of ubiquitous dust from surroundings during the sample collection and the transport. The latter will be discussed later with respect to relationships of T retention characteristics and sizes of dust particles.

Fig. 3 and Table 1 clearly show T retention characteristics of individual dust particles. At first, it is important to note again that the number of Be-dominated, W-dominated, and metal oxide dust particles retaining T were very small. Consequently, more than 85% of the total dust particles retaining T were the C-dominated ones.

Line profiling of T content and elements for several dust particles were conducted along white dashed lines depicted in Fig. 3 and summarized in Fig. 4(a)–(c) for Be-dominated, (d)–(f) for W-dominated, and (g)–(i) for C-dominated dust particles. Note that the intensity of T and elements were separately taken from the TIPT and EPMA results, respectively. In each figure, the distribution of T corresponds with the distribution of the element in the dust particle. Assuming a circular cross-section of the dust particle, for EPMA (shown on the left axis), a full width of a peak intensity of the element would correlate to a particular size of dust particle represented as a diameter of the particle, *d*. For TIPT (shown on the right axis), the broader width of the peak intensity of T than that of the element is due to T β-ray emission from the surface of the dust particle into the In bed or air in gaps surrounding the dust particle. For T retained in the bulk of a dust particle, such effects would be much smaller for heavier elements like W, than C and Be due to attenuation of T β-ray in the bulk of the dust particle. Except for one particle shown in Fig. 4(b), T retention on/in the Be-dominated dust particles was lower than for C-dominated dust particles. As could be expected from earlier researches [13,14], neighboring Be-dominated and C-dominated dust particles, shown in Fig. 1(e), indicate in Fig. 4(c) that T was highly retained in the C-dominated dust particles. In Fig. 4(d)–(f) for the W-dominated dust particles, the maximum peak intensity of T was lower by a factor of 100 compared with that for the C-dominated dust particles. Although the relatively higher T content of the C-dominated dust particles may influence the measurement of the T retention behavior, it is worth noting that intensities of T in Fig. 4(g)–(i) are widely distributed.

Assuming that T retention on/in the dust particle is proportional to the peak intensity of T, T retention characteristics of the dust particles are summarized in Fig. 5 for each element with respect to the size of the particle, i.e. the diameter of the particle, *d*. 4 Be-dominated, 8 W-dominated, and 30 C-dominated dust particles were randomly selected and measured. In the figure, T retention in the C-dominated dust particles is distributed over three orders of magnitude, which are categorized into three levels; high, moderate and low in T content. T retention of the Be-dominated and W-dominated dust particles were lower by a factor of 10–100 than that of the C-dominated ones in the medium T retention category. The dashed and chained lines are guides to the eye indicating the square of the diameter, d^2. T retention at the surface of a dust particle is expected to increase with surface area, however the deviation and wide distribution of T retention from the d^2 line shown in Fig. 5 indicates that T trapping in not only in the near surface region but also in the bulk of the dust particles. Bulk trapping could occur on the inner walls of either porous structures (shell or layer) or grain boundaries in the dust particles [5]. The trace of T retention which is not correlated with dust particles in Fig. 3 is likely attributed to T retention on/in dust particles smaller than several μm or nm size since such sized dust particles could not be measured due to a lack of EPMA resolution in the present study. The impact of such smaller dust particles on T retention could be studied by measuring T distribution with TIPT in combination with both EPMA and transmission electron microscopy (TEM), with the aid of focused ion beam (FIB).

4. Conclusions

The major contribution of this work to studies of dust in tokamaks is the first-ever combined determination of tritium and elemental composition of individual dust particles using TIPT and EPMA as complementary techniques. The combination of these two techniques allows the characteristics of T retention for the individual particles in dust samples from the inner diverter region after the JET-ILW first campaign (2011–2012) to be investigated.

Although it should be stressed that the amount of dust retrieved by vacuum cleaning was small, below 1 g in total [9], T retention in the JET-ILW dust was larger than expected [7]. The majority of particles in the dust sample were carbon which would be attributed to pre-existing C-deposits from JET-C operations or released C-particles from carbon fiber components present in the JET-ILW. T was mostly retained at the surface of and/or in the C-dominated dust particles. The most important outcome of the study is that the retention in W, Be and other metal-dominated dust particles is lower by a factor of 10 –100 in comparison with that found for the C-dominated particles.

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. This work was supported in part by JSPS KAKENHI Grant no. JP26289353, the ITER Broader Approach Activities and NIFS Bilateral Collaboration Grant no. NIFS13KUHR023.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.nme.2018.11.001.

References

[1] J. Winter, Dust: a new challenge in nuclear fusion research? Phys. Plasmas 7 (2000) 3862–3866.
[2] S. Brezinsek, et al., Beryllium migration in JET ITER-like wall plasmas, Nucl. Fusion 55 (2015) 10.
[3] A. Baron-Wiechec, et al., First dust study in JET with the ITER-like wall: sampling, analysis and classification, Nucl. Fusion 55 (2015).
[4] J.C. Flanagan, et al., Characterising dust in JET with the new ITER-like wall, Plasma Phys. Control. Fusion 57 (2015) 014037.
[5] M. Rubel, et al., Dust generation in tokamaks: overview of beryllium and tungsten dust characterisation in JET with the ITER-like wall, Fusion Eng. Des. 136A (2018) 579–586.
[6] S. Masuzaki, et al., Analyses of microstructure, composition and retention of hydrogen isotopes in divertor tiles of JET with the ITER-like wall, Phys. Scri. 170 (2017) 014031.
[7] N. Ashikawa, et al., Determination of retained tritium from ILW dust particles in JET, this issue (2018).
[8] J. Roth, et al., Tritium inventory in ITER plasma-facing materials and tritium removal procedures, Plasma Phys. Control. Fusion 50 (2008) 20.
[9] A. Widdowson, et al., Material migration patterns and overview of first surface analysis of the JET ITER-like wall, Phys. Scri. 159 (2014).
[10] T. Otsuka, T. Tanabe, Application of a tritium imaging plate technique to depth profiling of hydrogen in metals and determination of hydrogen diffusion coefficients, Mater. Trans. 58 (2017) 1364–1372.
[11] T. Otsuka, Y. Hatano, Tritium retention in individual metallic dust particles examined by a tritium imaging plate technique, Phys. Scri. 167 (2016) 014010.
[12] J. Schindelin, et al., FIZI: an open-source platform for biological-image analysis, Nat. Methods 9 (2012) 676–682.
[13] J.P. Coad, et al., Erosion/deposition issues at JET, J. Nucl. Mater. 290 (2001) 224–230.
[14] M. Rubel, et al., Beryllium and carbon films in JET following D-T operation, J. Nucl. Mater. 313 (2003) 321–326.