

Three-Dimensional Morphology of Blister-Like Structures and Deuterium Retention in Tungsten Exposed to Low-Energy, High-Flux D Plasma

V.Kh. Alimov¹, S. Lindig², M. Balden², K. Isobe¹, W.M. Shu³, J. Roth², T. Yamanishi¹

¹ *Tritium Technology Group, Japan Atomic Energy Agency, Tokai, Ibaraki, 319-1195, Japan*

² *Max-Planck-Institut für Plasmaphysik, EURATOM Association, D-85748 Garching, Germany*

³ *ITER International Organization, CEA Cadarache, 13108 Saint Paul-lez-Durance, France*

Three-dimensional morphology of blister-like structures and deuterium retention in recrystallized tungsten have been examined after exposure to a low-energy (38 eV/D), high-flux (10^{22} D/m²s) deuterium plasma at ion fluences in the range from 10^{26} to 10^{27} D/m² and various temperatures. The methods used were scanning electron microscopy equipped with focused ion beam, thermal desorption spectroscopy, and the $D(^3\text{He},p)^4\text{He}$ nuclear reaction at ³He energies varied from 0.69 to 4.0 MeV. Blister-like structures with various shapes and sizes depending on the exposure fluence and temperature are formed on the W surface. In doing so, cracks with distorted areas (<600 K) and large cavities (100-1000 μm^3) at grain boundaries (≥ 500 K) were observed beneath the surface. The surface blister-like structures and the defects underneath are correlated along crystallographic orientation of the W grains in accordance to the low-indexed sliding systems. The defects are mobile and accumulate under D plasma exposure. Samples exposed near room temperature do not form such large cavities even by subsequent heating up to 1300 K. The D plasma exposure at temperatures above 700 K does not lead to formation of blister-like structures. At ion fluences of 10^{26} - 10^{27} D/m², the D retention increases with the exposure temperature, reaching its maximum value of about 10^{22} D/m² at 480-530 K, and then decreases to about 10^{19} D/m² at 800 K.

Keywords: blistering; deuterium; deuterium retention; high ion flux; tungsten

I. Introduction

Due to its favourable physical properties, like low erosion yield and high melting temperature, tungsten (W) is foreseen as a candidate material for plasma-facing high heat-flux structures in fusion reactors in operation and under construction. As plasma-facing material, tungsten (W) will be subjected to bombardment with low-energy, high flux deuterium (D) and tritium particles including helium (He) ash. This implantation process leads to concerns about

hydrogen isotope retention after long-term deuterium-tritium plasma exposure. Available data (reviews [1-3] and references therein) have shown that the hydrogen isotope retention in W materials exposed to high-flux hydrogen plasmas differs from that for ion implantation. In addition, there is evidence of blistering occurred on tungsten surface exposed to hydrogen plasmas with ion energies well below the displacement threshold [4-8]. However, not much is known about the hydrogen retention in different W

materials irradiated with low-energy hydrogen ions and exposed to low-energy, high-flux hydrogen plasmas. Especially, the temperature dependence of the hydrogen isotope retention after exposure to the plasmas at ion fluences $\geq 10^{26}$ ions/m² was not studied systematically.

Quite often the D retention is accompanied by formation of blister-like surface structures ([3] and references therein, [6, 8, 9-12]). As it was shown recently, an appearance of two-types of blister-like structures, small ones (around 1 μm) and large ones (10-30 μm), after exposure to low-energy D plasma at temperatures around 500 K, coexists at the maximum in the D retention [6, 8]. The three-dimensional (3D) morphology of the defects is characterized by cavities inside large blisters and cracks along grain boundaries [8, 11, 12]. Detailed analysis of the sub-surface structure belonging to the blister-like surface structure was fulfilled recently for clarification of the underlying mechanisms for morphology changed by D impact [13]. It has been found that there is strong variation on the surface topography and 3D morphology depending on fluence, temperature and grain orientation.

This work focuses on study of the temperature dependence of sub-surface morphology and D retention in recrystallized W exposed to low-energy, high flux D plasma at high ion fluences ($\geq 10^{26}$ D/m²).

II. Experimental

Polycrystalline tungsten (A.L.M.T. Corp., Japan) with a purity of 99.99 wt.% fully recrystallized after cutting and polishing was used. The recrystallization process was performed by annealing at 2070 K for 1 hour in pure hydrogen (protium) atmosphere. The W grain size was 20-200 μm . In the linear plasma generator used for

delivering a plasma beam comparable to the edge plasma at ITER divertor, the W samples were exposed to the D plasma highly enriched with species of D₂⁺ to over 80% [14]. The bias voltage of -80 V was applied to the W sample resulting in an incident energy of 38 eV/D, taking into account the plasma potential of about -4 V measured by a Langmuir probe. The incident deuterium ion flux was fixed at 10²² D/m²s, and the samples were exposed to ion fluences of 10²⁶ and 10²⁷ D/m². The sample was passively heated by the plasma itself and the exposure temperature was set by the thermal contact between the sample and the cooled holder.

The D concentration in the plasma-exposed W samples was measured by means of the D(³He, α)H reaction, where both, the α particles and protons were analyzed. To determine the D concentration at larger depths, an analyzing beam of ³He ions with energies varied from 0.69 to 4.0 MeV was used. The proton yields measured at different ³He ion energies allow measuring the D depth profile at depths of up to 7 μm [15].

Deuterium retention in the W samples was monitored ex-situ using thermal desorption spectrometry (TDS). An infrared heater was used to heat the samples at a ramp rate of 0.5 K/s and the sample temperature was raised to 1300 K. HD and D₂ molecules released during TDS run were monitored by a quadrupole mass spectrometer (QMS). To calculate the relative contribution of the recorded HD and D₂ masses to the total release of deuterium, the partial currents of the QMS were normalized as described in Ref. [16]. Standard D₂ leak with an inaccuracy lower than 10% was employed to calibrate the QMS after each TDS analysis.

The surface topography was examined by scanning electron microscopy (SEM), whereas the

three-dimensional sub-surface morphology of the plasma-exposed W samples was analyzed by a field emission SEM combined with a focused ion beam (HELIOS NanoLab 600, FIE) [13].

To reveal correlation of observed blister-like structures to the crystallographic orientation of individual grains, the grain orientation was determined by electron backscattering diffraction (EBSD). A 20 keV electron beam with an incident angle of 70° to the normal of the analyzed surface area was typically used for the EBSD analysis

with the HKL Nordlys II detector and “HKL Channel 5” analysis software [13].

III. Results

III.A. Surface topography and sub-surface morphology

After exposure to a fluence, Φ , of 10^{26} D/m² at temperatures, T_{exp} , in the range from 320 to 370 K, only sparse low-dome blisters with sizes of a few microns or less are observed (Fig. 1a).

At $T_{\text{exp}} = 400\text{-}500$ K, the blisters become

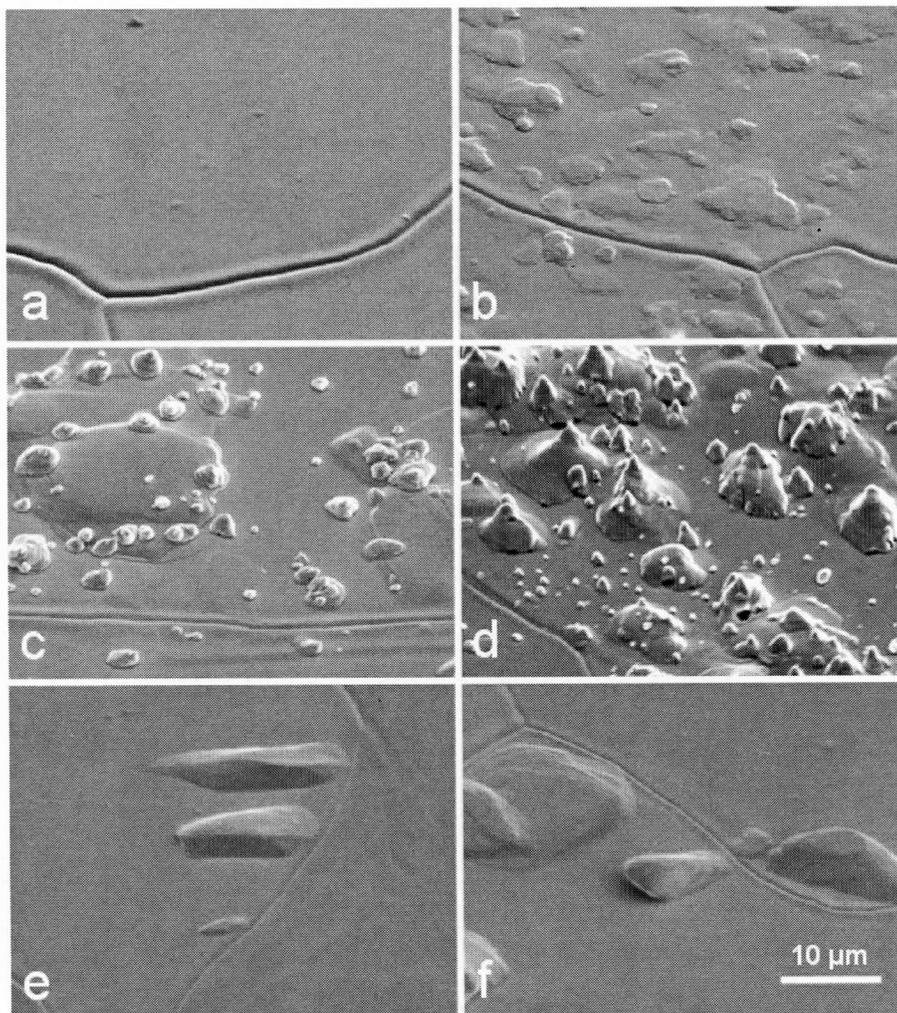


Fig. 1. SEM images of recrystallized W exposed to low-energy, high ion flux D plasma with ion fluences of 10^{26} D/m² (a, c, e – left part of the figure) and 10^{27} D/m² (b, d, f – right part of the figure) at temperatures of 360 K (a, b), 530 K (c), 460 K (d), 600 K (e), and 595 K (f). The magnification of all images is the same. The surfaces were tilted at an angle of 45° to the electron beam.

much denser and the domes of blisters become higher. Peculiar changes occur at $T_{\text{exp}} = 520\text{-}570\text{ K}$, where two kinds of blisters appear: large low-dome blisters with sizes of a few tens of microns and small cone-shaped blisters with diameters of several micrometers (Fig. 1c). At exposure temperatures in the range from 580 to 670 K, the small blisters disappear, whereas the large blisters become sparser with the increasing temperature (Fig. 1e).

The increase in fluence to 10^{27} D/m^2 leads to the appearance of dense and low-dome blisters with sizes up to $15\text{ }\mu\text{m}$ even at $T_{\text{exp}} = 320\text{-}370\text{ K}$ (Fig. 1b). Two kinds of blisters appear already at

exposure temperature of 460 K (Fig. 1d). At higher exposure temperatures, the temperature dependence of blistering at $\Phi = 10^{27}\text{ D/m}^2$ (Fig. 1f) is similar to that at 10^{26} D/m^2 .

For both fluences, no blisters appear at the exposure temperatures above 700 K.

According to EBSD analysis, shape and position of the blisters formed on each individual grain are correlated to the grain orientation (Fig. 2). The elongation directions of the blisters coincide with the projections of the (110) high symmetry planes of the W crystal onto the surface [13].

Three-dimensional sub-surface morphology

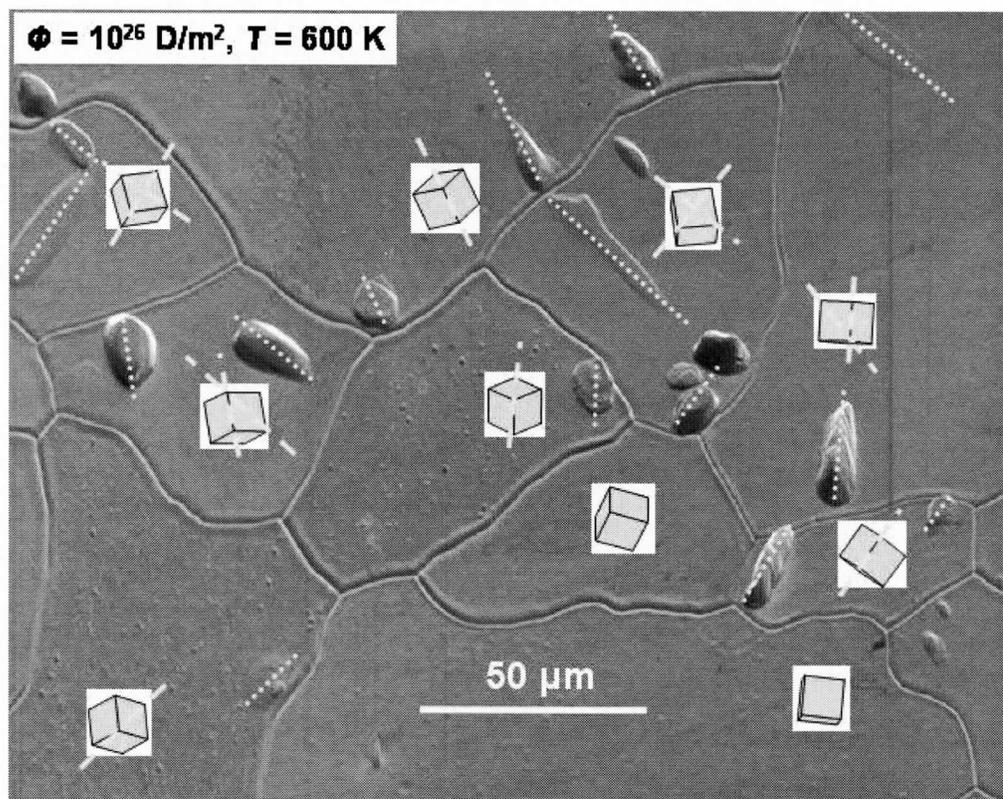


Fig. 2. Various elongated flat and high-dome blisters on the surface of recrystallized W exposed to low-energy, high ion flux D plasma with an ion fluence of 10^{26} D/m^2 at 600 K. The orientation of the grains was determined by EBSD and is represented by the inserted cubes. The projected (110) sliding planes are partly marked. The surface was tilted at an angle of 70° to the electron beam. The image was stretched in vertical direction in order to correct the tilt shortening (it was not done in Figs. 1 and 3).

of blister-like structures at $\Phi = 10^{27}$ D/m² ranges from fine intragranular crack-like structures for the W exposed at temperatures around room temperature (Fig. 3a) to large cavities formed after exposure at elevated temperatures (Fig. 3c). The cross-section of the small blisters is correlated with crack-like defects with width of ~ 0.1 μm and length of ~ 5 μm situated mainly inside the crystallites and some times at grain boundaries beneath the surface at depths of at least 10 μm (Figs. 3a and 3b).

The cross-section of the large blisters formed at elevated exposure temperatures exhibits always a cavity deep beneath the surface, which has contact with a grain boundary (Fig. 3c). The thickness of the material above the cavity easily reaches 10 μm , some of the cavities extent even to more than 50 μm beneath the surface. The volume of these cavities is roughly equal to the volume of the blister above the surface and is typically in the range of 100-1000 μm^3 [13]. The shape of the surface topography is a transformed image of the cavity shape. The cavities are in most cases not exactly below the blisters. The lateral shift is correlated to the grain orientation.

Figure 3b highlights another important feature of the D plasma-exposed W surface: large distorted regions around the cracks, visible as different greyscales inside a grain (due to the channelling contrast of the backscattered electrons, areas of equal crystallographic orientation, i.e. grains, appear with the same greyscale). In a few cases, such distorted subsurface regions are present without any significant indication on the surface [13].

For W samples exposed around room temperature, subsequent heating up to 1300 K (e.g., during TDS experiments) does not anneal the distorted regions and does not form large

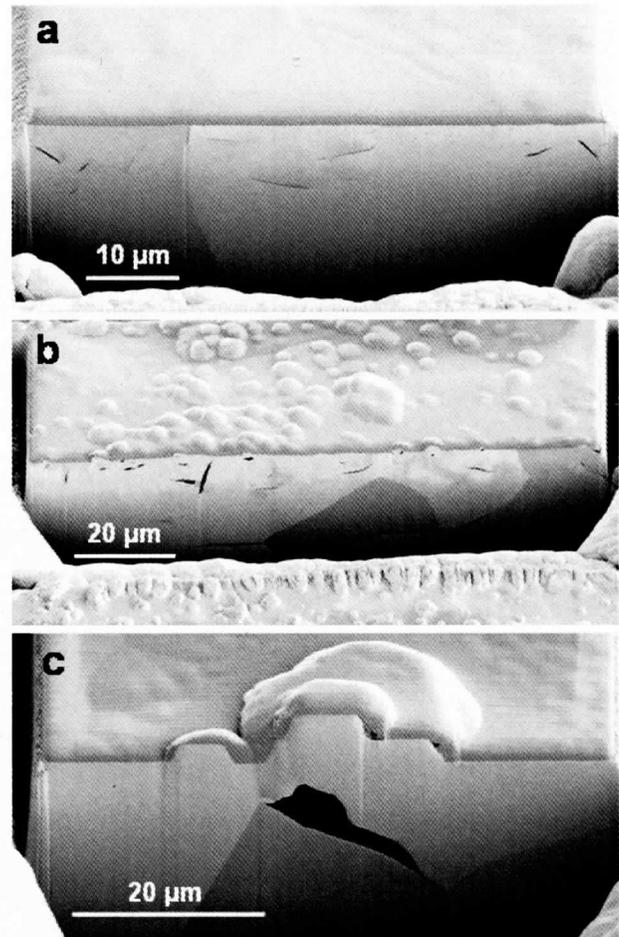


Fig. 3. Cross-sectional images of recrystallized W exposed to low-energy, high ion flux D plasma with an ion fluence of 10^{27} D/m² at 360 K (a), 480 K (b), and 595 K (c). Take note that prior the cross-sectioning the surfaces were coated with a Pt-C film. The surfaces were tilted at an angle of 52° to the electron beam and cross-section respective by -38° .

cavities, as occur in samples exposed at elevated temperatures [13].

III.B. Depth profiles

Deuterium depth profiles in the recrystallized W exposed to the D plasma at various temperatures are shown in Fig. 4 for ion fluences of 10^{26} and 10^{27} D/m².

In the recrystallized W exposed at $T_{\text{exp}} = 330$ K to $\Phi = 10^{26}$ D/m², the D depth profile is characterized by a sharp near-surface concentration maximum of 1-2 at.%, and, at depths above 1 μm , by a concentration of about 10^{-2} at.% slowly decreasing into the bulk (Fig. 4a). As the exposure temperature increases up to 530 K, the D concentration in the near-surface decreases, whereas the concentration at depths of 1-3 μm reaches the maximum value of about 1 at.%. Further increase of the exposure temperature leads to a uniform decrease of the D concentration (Fig. 4a).

As the ion fluence increases to 10^{27} D/m² at $T_{\text{exp}} = 320$ K, the D concentration at depths of 1-2 μm is significantly increased (Fig. 4b). At $T_{\text{exp}} = 480$ K, the D profile demonstrates, in addition to the near-surface peak, the concentration of about 1 at.% with no decrease into the bulk up to a depth of 7 μm .

For both ion fluences, the maximum D concentration is about 1 at.%, and this D concentration is achieved at the exposure temperatures of 480-535 K. This high D concentration could be due to accumulation of D₂ molecules in cavities created during the D plasma exposure.

After D plasma exposure at temperatures of 725-745 K, the D depth profiles demonstrate concentration minimum at depths of 0.5-2 μm (Fig. 4a,b). Unfortunately, we have no clear explanation of reasons for this local decrease in the D concentration.

III.C. Thermal desorption spectra

Thermal desorption spectrum obtained after exposure to a fluence $\Phi = 10^{26}$ D/m² at $T_{\text{exp}} = 320$ K demonstrates two strongly pronounced

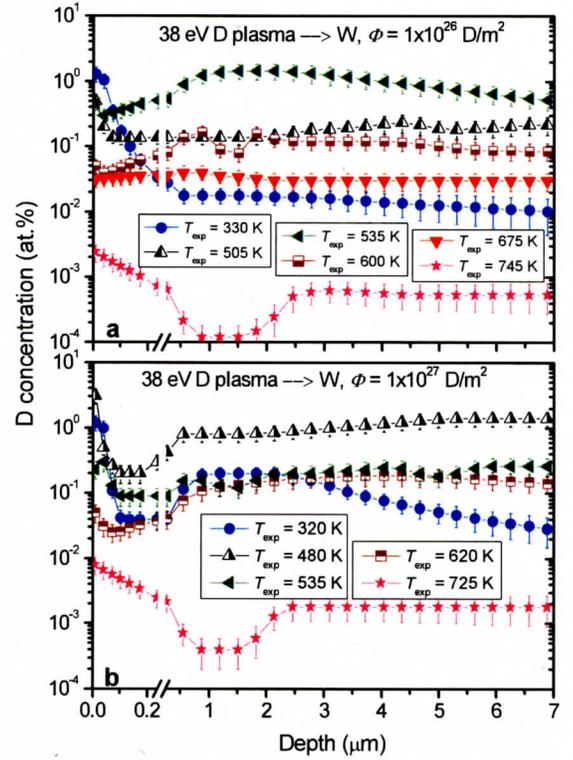


Fig. 4. Depth profiles of deuterium retained in recrystallized W exposed to low-energy, high ion flux D plasma with ion fluences of 10^{26} (a) and 10^{27} D/m² (b) at various temperatures.

peaks at temperatures $T_{\text{peak}} \cong 500$ and 650 K (Fig. 5).

At $\Phi = 10^{27}$ D/m² and $T_{\text{exp}} = 330$ K, the positions of these peaks are shifted towards higher temperatures and are located at $T_{\text{peak}} \cong 590$ and 780 K. The amount of deuterium released as the second TDS peak ($T_{\text{peak}} \cong 780$ K) increases by a factor of about 10 as compared to that for a fluence of 10^{26} D/m². This fact denotes strong W structure modification due to plasma exposure at near-room temperatures at $\Phi \geq 10^{26}$ D/m². At exposure temperatures above 500 K, as a fluence increases from 10^{26} to 10^{27} D/m², the amount of deuterium released during TDS run increases by a factor of about 3.

It should be noted that the tenfold increase in an ion fluence at temperatures in the range from 320 to 620 K results in shift of TDS peaks towards higher temperatures (Fig. 5). This fact points to a plasma-induced evolution of defects, which are responsible for trapping of deuterium.

After plasma exposure at temperatures and fluences when blisters are formed, the TDS spectra demonstrate bursting release (Fig. 5). Numerous bursting peaks indicate that deuterium accumulates in tungsten in the form of molecules inside cracks and cavities, corresponding to the formation of blisters. Thus, during TDS heating deuterium releases directly when blisters burst or firstly dissociates from molecules into atoms (thus

high temperature is required) and then atoms diffuse to the surface where they recombine and release as molecules.

It follows from the TDS spectra (Fig. 5a) that for D ion fluence of 10^{26} D/m², the main TDS peak is shifted toward higher temperature as the exposure temperature increases. This effect is also observed for ion fluence of 10^{27} D/m² after D plasma exposure at 330 and 480 K. However, at exposure temperatures from 515 to 620 K the main TDS peak is positioned practically at the same temperature $T_{\text{peak}} \cong 950$ K. It should be noted that this peak temperature is highest among all TDS spectra for both ion fluences and all exposure temperatures in the range from 320 to 620 K.

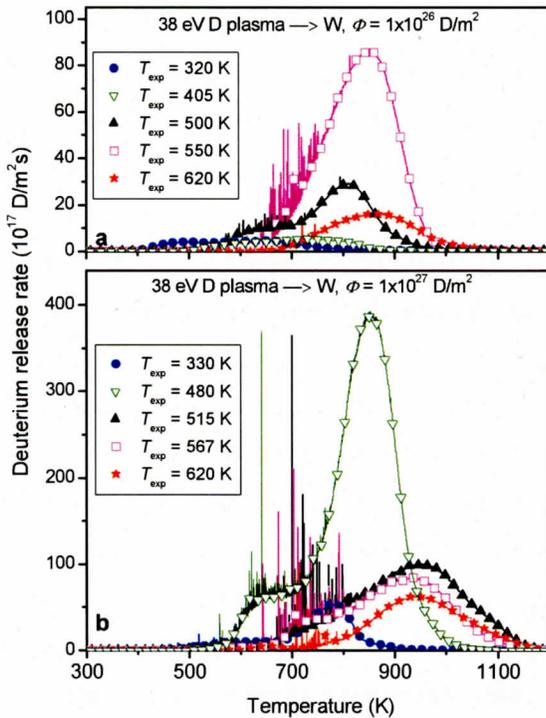


Fig. 5. Thermal desorption spectra of deuterium released from recrystallized W exposed to low-energy, high ion flux D plasma with ion fluences of 10^{26} D/m² (a) and 10^{27} D/m² (b) at various temperatures. TDS heating rate was 0.5 K/s.

III.D. Total retention

In the recrystallized W exposed to the D plasma at $\Phi = 10^{26}$ D/m², the deuterium retention is $(2-3) \times 10^{20}$ D/m² at $T_{\text{exp}} = 320$ K and, as exposure temperature increases, rises to its maximum of about 7×10^{21} D/m² at $T_{\text{exp}} = 530$ K and then decreases down to about 10^{19} D/m² at $T_{\text{exp}} = 775$ K (Fig. 6).

Increasing the fluence to 10^{27} D/m² at exposure temperatures in the range from 320 to 500 K results in significant increase of the D retention. At $\Phi = 10^{27}$ D/m² the maximum retention of about 10^{22} D/m² is observed at $T_{\text{exp}} = 480$ K. At exposure temperatures of 550-650 K, the D retention at $\Phi = 10^{27}$ D/m² decreases but remains higher by a factor of about 3 than that at $\Phi = 10^{26}$ D/m². At T_{exp} above 750 K, the D retention is about 10^{19} D/m² for both ion fluences.

From the comparison of the TDS and NRA data (Fig. 6) it is clear that at $\Phi = 10^{26}$ D/m² and irradiation temperatures up to 700 K about 50% of retained deuterium is localized in the sub-surface

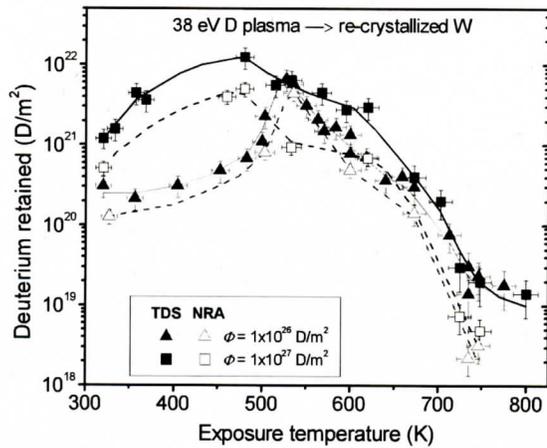


Fig. 6. Deuterium retention in recrystallized W exposed to low-energy, high ion flux D plasma with ion fluences of 10^{26} and 10^{27} D/m^2 , as a function of the exposure temperature. The total deuterium retention was determined by thermal desorption spectrometry (TDS) (solid points), whereas the D retention up to a depth of $7 \mu\text{m}$ was measured by nuclear reaction analysis (NRA) (open points).

layer up to $7 \mu\text{m}$. However, at $\Phi = 10^{27}$ D/m^2 the fraction of deuterium retained at depths above $7 \mu\text{m}$ significantly increases.

IV. Discussion

For tungsten irradiated with D ions at energies well below the displacement threshold, the mechanism of plastic deformation due to deuterium super-saturation [17] must be considered for modification of the sub-surface structure and formation of trapping sites for deuterium [9, 18]. During exposure to low-energy, high-flux D plasma, the D concentration in the implantation zone greatly exceeds the solubility limit and stresses the matrix lattice until plastic deformation occurs to alleviate these tensions [17]. This deformation is assumed to be responsible for

the generation of vacancies, vacancy complexes and microscopic cavities at depths of several tens of micrometers and the concurrent accumulation of diffusing deuterium. It may be suggested that the stress-induced plasticity of tungsten appears at high concentrations of solute hydrogen and increases as the temperature grows.

On the other hand, according to the theory of formation of superabundant vacancies in the presence of interstitial hydrogen atoms [19], the formation energy of a vacancy is decreased owing to vacancy-hydrogen cluster formation and the configurational entropy of the system at high hydrogen concentration. Recently Shu [11] considered that the mechanism of vacancy formation due to the lowering of the vacancy formation energy by trapping of deuterium can also be valid for tungsten exposed to low-energy, high-flux D plasma. The deuterium-vacancy clusters may diffuse deeply into the bulk and agglomerate resulting in microscopic cavities.

At long-term irradiation the diffusing D atoms recombine on the cavity surfaces, increasing thus the D_2 gas pressure inside these cavities. The cracks, distorted areas, and large and small cavities beneath the surface (Fig. 3) are caused by stress exceeding the toughness of the material. The stress may be increased by the gas overpressure inside the cavities. At elevated temperatures exceeding the brittle-to-ductile-transition temperature, the stress can be relaxed by dislocations moving along lattice planes through the whole crystallite leading to the cavities at the grain boundaries [13, 20]. This corresponds to the observed material migration above the surface, i.e., the blister-like surface topography. It may be suggested that the overpressured large and small cavities are responsible for appearance of large low-dome and small cone-shaped blisters,

correspondingly. It should be noted that for all small blisters, deep holes or pits were formed in the internal blisters [11]. Thus, formation of small cone-shaped blisters at $T_{\text{exp}} = 520\text{-}570\text{ K}$ may be explained by deuterium gas migration toward the surface at conditions of high plasticity of recrystallized W.

At near-room temperatures the stress relaxation results in plastic deformation and crack formation inside the grains. Thus, the temperature dependence of the structure modification correlates well with the temperature dependence of fracture toughness for W [20, 21].

The fact that annealing of samples with high amount of the retained D does not lead to formation the large cavities, as it does during exposure to the plasma, indicates that just the transient D concentration maintained during implantation is responsible for the stress state. It can be expected that the mobility of vacancies, voids, and dislocations in tungsten is affected by the transient D inventory during exposure to low-energy, high flux D plasma.

From the known low-indexed sliding systems [20], the lateral location of the cavity and surface structure as well as the direction of the elongation of the blisters could be explained (Figs. 2 and 3c) [13], but further detailed investigations are necessary.

The absence of blister-like structures as well as the low D retention at temperatures above 700 K indicates that the requirement for extended defect production is not fulfilled. Possibly, it could be explained by the strongly increased solubility and diffusivity [1] avoiding that the transient D concentration exceeds the solubility. Also it could be speculated that the defects in W anneal at these temperatures, the reemission is

increased, and therefore, the transient D concentration is strongly lowered.

The co-incidence of the maximum of the D retention in the temperature dependency (Fig. 6) with the appearance of highest density of structural changes (Fig. 3) is evident. Deuterium is retained mainly at vacancy-type defects and in gas-filled cavities and cracks, both in the form of D_2 molecules and D atoms. According to van Veen et al. [21], the D_2 gas inside the voids is expected to be released during annealing at 400-600 K (detrapping energy $\approx 1.0\text{ eV}$), whereas the D atoms bound on the inner surface of voids (binding energy $\approx 1.7\text{ eV}$) is expected to be released at 700-1000 K, depending on depths where the voids are generated. On the other hand, deuterium TDS peaks at 640 and 520 K are well explained by trap energies of 1.34 and 1.07 eV, which is related to trapping of a first and second D atom at a vacancy, respectively [22]. TDS spectra for the plasma-exposed W (Fig. 5) demonstrate a variety of trapping energies for deuterium and, therefore, a variety of vacancy defects and cavities/cracks formed in the sub-surface layer. Obviously that during D plasma exposure at temperatures below 600 K deuterium is accumulated in the form of D_2 molecules and D atoms, whereas at temperatures above 600 K deuterium is retained as D atoms solely.

In closing it is pertinent to note that the fluence and temperature dependence of the D retention is a consequence of the sub-surface structure modification and accumulation of deuterium in the cracks and cavities.

V. Summary

Significant temperature dependence of surface topography, sub-surface morphology and deuterium retention is found for recrystallized W

exposed to low-energy (38 eV/D), high-flux (10^{22} D/m²s) D plasma at ion fluences of 10^{26} and 10^{27} D/m². At temperatures of 320-400 K, only sparse blisters with diameters of 0.5-2 μm are formed on the W surface. In this case the D depth profiles are characterized by a near-surface concentration maximum of 1-2 at.% and, in the sub-surface layer (at depths from 1 to 7 μm), by a concentration of 0.02-0.2 at.% slowly decreasing into the bulk. As the exposure temperature increases, the blisters become much denser. Two kinds of blisters appear: large blisters with sizes of 10-30 μm and small cone-shaped blisters with diameters of less than a few μm . As this takes place, the D concentration in the sub-surface layer reaches 1 at.%. No blisters appear at temperatures above 700 K, and the D concentration at depths of several micrometers is about 10^{-3} at.%.

At exposure temperature around 360 K, fine crack-like defects and large distorted areas around the cracks are observed inside grains at depths of at least 10 μm . At higher temperatures (above 500 K), large cavities at the grain boundaries at depths of 10-50 μm are the dominating features, whereas the fine-crack structure with the distorted areas lose their significance. A correlation of shape and position of the defect structures underneath the surface blister-like structure with the crystallite orientation is obvious. A possible explanation follows from the low-index sliding systems in W.

Post-irradiation annealing up to 1300 K does not form the large cavities. This implies that the mobility of vacancies and voids in tungsten is affected by the transient D concentration maintained during exposure to the plasma.

Stress-induced plastic deformation caused by deuterium super-saturation within the near-surface layer and formation of superabundant

deuterium-vacancy clusters are suggested as mechanisms for nucleation and growth of microscopic cavities at depths of several tens of micrometers. At long-term plasma exposure, the diffusing D atoms recombine on the cavity surfaces, increasing thus the gas pressure inside these cavities, which may support the formation of blister-like structures.

Acknowledgements

The portion of this work performed at JAEA was supported by MEXT/Japan through JSPS (Kagaku-kenkyuhi-hojyokin, No.19055009) and ITER International Organization under Task Agreement ITER/TA/08/490.

References

- [1] R.A. Causey, T.J. Venhaus, "The use of tungsten in fusion reactors: a review of the hydrogen retention and migration properties", *Phys. Scripta*, **T94**, 9 (2001).
- [2] R.A. Causey, "Hydrogen isotope retention and recycling in fusion reactor plasma-facing components", *J. Nucl. Mater.*, **300**, 91 (2002).
- [3] C.H. Skinner, A.A. Haasz, V.Kh. Alimov, et al., "Recent advances of hydrogen retention in ITER's plasma-facing materials: beryllium, carbon, and tungsten", *Fusion Sci. Technol.*, **54**, 891 (2008).
- [4] W. Wang, J. Roth, S. Lindig, C.H. Wu, "Blister formation of tungsten due to ion bombardment", *J. Nucl. Mater.*, **299**, 124 (2001).
- [5] T. Venhaus, R. Causey, R. Doerner and T. Abeln, "Behavior of tungsten exposed to high fluences of low energy hydrogen isotopes", *J. Nucl. Mater.*, **290-293**, 505 (2001).
- [6] W.M. Shu, E. Wakai, T. Yamanishi, "Blister bursting and deuterium bursting release from

- tungsten exposed to high fluences of high flux and low energy deuterium plasma”, Nucl. Fusion, **47**, 201 (2007).
- [7] W.M. Shu, A. Kawasuso, Y. Miwa, et al., “Microstructure dependence of deuterium release and blistering in the near-surface region of tungsten exposed to high flux deuterium plasmas”, Phys. Scripta, **T128**, 96 (2007).
- [8] W.M. Shu, M. Nakamichi, V.Kh. Alimov, et al., “Deuterium retention, blistering and local melting at tungsten exposed to high-fluence deuterium plasma”, J. Nucl. Mater., **390-391**, 1017 (2009).
- [9] A.A. Haasz, M. Poon, J. Davis, “The effect of ion damage on deuterium trapping in tungsten”, J. Nucl. Mater., **266-269**, 520 (1999).
- [10] K. Tokunaga, M.J. Baldwin, R.P. Doerner, et al., “Blister formation and deuterium retention on tungsten exposed to low energy and high flux deuterium plasma”, J. Nucl. Mater., **337-339**, 887 (2005).
- [11] W.M. Shu, “High-dome blisters formed by deuterium-induced local superplasticity”, Appl. Phys. Lett., **92**, 211904 (2008).
- [12] W.M. Shu, A. Kawasuso, T. Yamanishi, “Recent findings on blistering and deuterium retention in tungsten exposed to high-fluence deuterium plasma”, J. Nucl. Mater., **386-388**, 356 (2009).
- [13] S. Lindig, M. Balden, V.Kh. Alimov, et al., “Subsurface morphology changes due to deuterium bombardment of tungsten”, Phys. Scripta, **T 138**, 014040 (2009).
- [14] G.-N. Luo, W.M. Shu, H. Nakamura, et al., “Ion species control in high flux deuterium plasma beams produced by a linear plasma generator”, Rev. Sci. Instrum., **75**, 4374 (2004).
- [15] V.Kh. Alimov, M. Mayer, J. Roth, “Differential cross-section of the $D(^3\text{He,p})^4\text{He}$ nuclear reaction and depth profiling of deuterium up to large depths”, Nucl. Instr. and Meth., **B 234**, 169 (2005).
- [16] P. Franzen, B.M.U. Scherzer, W. Möller, “Re-emission of deuterium from pure and boronized graphite”, Nucl. Instr. And Meth., **B67**, 536 (1992).
- [17] J.B. Condon and T. Schober, “Hydrogen bubbles in metals”, J. Nucl. Mater., **207**, 1 (1993).
- [18] V.Kh. Alimov, J. Roth, M. Mayer, “Depth distribution of deuterium in single- and polycrystalline tungsten up to depths of several micrometers”, J. Nucl. Mater., **337-339**, 619 (2005).
- [19] Y. Fukai, Y. Ishii, Y. Goto, K. Watanabe, “Formation of superabundant vacancies in Pg-H alloys”, J. Alloys. Comp., **313**, 121 (2000).
- [20] P. Gumbsch, J. Riedle, A. Hartmaier, H.F. Fischmeister, “Controlling factors for the brittle-to-ductile transition in tungsten single crystals”, Science, **282**, 1293 (1998).
- [21] P. Gumbsch, “Brittle fracture and the brittle-to-ductile transition of tungsten”, J. Nucl. Mater., **323**, 304 (2003).
- [21] A. van Veen, J. de Vries, D. Segers, G.J. Rozing, “Hydrogen defect interaction in tungsten observed with PA”, in: P.C. Jain, R.M. Singru and K.P. Gopinathan (Eds.), *Positron Annihilation*, p. 543-545, 1985, World Scientific Publ., Singapore.
- [22] M. Poon, A.A. Haasz, J. Davis, “Modelling deuterium release during thermal desorption of D^+ -irradiated tungsten”, J. Nucl. Mater., **374**, 390 (2008).