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Deuterium retention and morphological modifications of the surface in five grades of tungsten after deuterium plasma exposure

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HIGHLIGHTS

- D retention for used W grades varies by more than one order of magnitude.
- Hydrogen loading-induced damaging manifests in surface modifications and retention.
- Effects of plasma flux on retention and surface modifications are discussed.
- Higher fluxes produce more severe hydrogen loading-induced damaging.

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ABSTRACT

Five tungsten (W) grades were simultaneously exposed to deuterium (D) plasma with 10^{20} D/(m^2 s) of 38 eV/D up to 10^{26} D/m^2 at 500 K specimen temperature. The D inventories and their depth profiles within the topmost 12 μm were determined by nuclear reaction analysis (D(^{3}He, p)α). Morphological modifications at and below the surface were analysed by confocal laser scanning microscopy and scanning electron microscopy assisted by focused ion beam cross-sectioning. The observed variation of the D inventory by more than one order of magnitude (0.5–15 × 10^{20} D/m^2) is attributed only to the different properties of each W grade. Spherical blisters and stepped flat-topped extrusions are observed depending on the W grade. These modifications are interpreted as an indication for hydrogen loading-induced damaging. The exposure conditions and W grades were chosen to allow a comparison between published data sets.

1. Introduction

Tungsten (W) is envisaged as plasma-facing material for future fusion devices, e.g. [1,2]. In recent years many studies regarding the hydrogen retention and surface morphology were performed on various W grades, e.g. [3–20]. The hydrogen retention varies by orders of magnitude depending on the ion beam and plasma exposure conditions (e.g., hydrogen flux, fluence, impact energy, temperature). Even if some dependences on exposure parameters were investigated in detail (e.g., on fluence and temperature), often the material and its final preparation vary from study to study. This variation can be expected to influence the retention [19,21,22] and the surface morphology evolving during plasma exposure. Therefore, this variation hinders the comparability between different studies. It is still under discussion how the used W grades contribute to the observed variation in retention and to the observed morphological modifications of the surface, which range from about spherical blisters to stepped, flat-topped as well as volcano-like extrusions, e.g. [5,6,15,16]. For assessing the retention and the erosion behaviour of tungsten grade used in future fusion devices, this question must be answered and a more basic understanding is necessary, also of the recrystallized material.

In this article, "**W grade**" is defined as the combination of the tungsten base material, its processing and the specimen preparation, which includes in particular polishing and heat treatments.

Two extensive studies of two groups [7–10] and [11–15] were performed with detailed parameter scans. The two groups investigated the deuterium retention with D depth profiling and the surface morphology evolution, each on well-prepared surfaces, but for different tungsten grades and different exposure conditions, in particular deuteron flux and fluence.

The first group [7–10] used as the typical exposure condition 38 eV/D, 6 × 10^{24} D/m^2, and 370 K at a flux of ~10^{20} D/(m^2 s). They used a quantified plasma source [23] ("PlaQ") to expose rolled W material after four different heat treatments up to recrystallization. They performed fluence, energy and temperature scans around the condition given above, resulting in D inventories varying by more

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than three orders of magnitude up to nearly 10^{21} D/m^2. They also studied the blister size and areal density of the observed spherical blisters.

The second group [11–15] used W material from another manufacturer after polishing and after recrystallization. They determined the temperature dependence of the D inventory and the surface modifications at two higher fluences (10^{26} D/m^2 and 10^{27} D/m^2) also for 38 eV/D but with a hundred times higher flux ($\sim 10^{22}$ D/(m^2 s)) at the Linear Plasma Generator ("LPG") at Japan Atomic Energy Agency, Tokai, Japan [24]. They detect D inventories up to slightly more than 10^{22} D/m^2 and a variation of D inventory with temperature between 300 and 800 K of more than three orders of magnitude. A variety of surface modifications – from large flat-topped (tens of µm) to volcano-like extrusions of different size (hundreds of nm up to several µm) – were observed and their sub-surface morphology was analysed.

The first aim of the present study is to allow a comparison between the two data sets briefly described above by using the same W grades as well as by choosing suitable exposure conditions at PlaQ, i.e., 10^{26} D/m^2 and 500 K. Therefore, only one main parameter varies to each published data set: fluence to [7–10] or flux to [11–15]. The second aim is to provide data belonging to a series of experiments dedicated to study the influence of the ion flux during plasma exposure on surface morphology and deuterium retention. This joint action covers a flux range of about 4 orders of magnitude by using different exposure devices, from PlaQ with moderate flux of 10^{20} D/(m^2 s) [23] via LPG with high flux of 10^{22} D/(m^2 s) [24] to Pilot-PSI with very high flux of 10^{24} D/(m^2 s) [25].

Therefore, in the study presented here, five different tungsten grades – including the ones predominantly studied in [7–15] – were exposed simultaneously to a D plasma in PlaQ, i.e., receiving the same exposure (specimen temperature: 500 K; D impact energy: 38 eV/D; flux: 10^{20} D/(m^2 s); fluence: 10^{26} D/m^2). The fluence of 10^{26} D/m^2, which needed 12 days of continuous exposure at PlaQ, was chosen in order to allow the comparison with the higher flux experiments, e.g., with $\sim 10^{24}$ D/(m^2 s) at Pilot-PSI, DIFFER, Netherlands [25], where accumulating the same fluence takes only $\sim$100 s.

2. Experimental details

2.1. Specimens and preparation

Two tungsten base materials produced by two manufacturers (Plansee SE, Austria and A.L.M.T. Corp., Japan) were used for the five investigated specimens. The base material from Plansee has a guaranteed purity of 99.97 wt.%, while the Japanese material has a higher nominal purity of 99.99 wt.%. The Plansee material was delivered after two different deformation processes: rolling to a $\sim$0.8 mm thick plate, or swaging to a rod of 40 mm diameter. The Japanese material has an unknown deformation history. Furthermore, two additional grades were produced by recrystallization of the material of both manufacturers. To distinguish these five specimens, they will be termed **WP-R, rc-WP-R, WP-S, WJ,** and **rc-WJ** throughout this paper (**P** = Plansee, **J** = Japan, **R** = rolled, **S** = swaged, **rc** = recrystallized).

The deformation process, e.g., hot- and cold-rolling, forging or swaging, leads to variations in the grain morphology (size and shape) and in preferred grain orientation, depending on the degree of deformation and specimen shape. In general, the grains are elongated along the rolling direction or the rod axis.

For the rolled tungsten from Plansee (**WP-R**) the (sub-)grain dimension perpendicular to the plate surface is the smallest with 0.5–1 µm, while parallel to the surface the typical size is 1–5 µm [26,27] (see Section 2.4). For this specimen type, the plate surface is the surface exposed to the plasma. The specimen size is 12×15 mm^2. In the swaged material from Plansee, the grains are elongated parallel to the rod axis with a length of 5–10 µm. Perpendicular to the rod axis the grains have typical sizes of 2–5 µm. As specimens, 15×12 mm^2 plates ($\sim$0.8 mm thickness) were cut with the 15×12 mm^2 surface perpendicular to the rod axis (**WP-S**). Therefore, the grains are elongated perpendicular to the specimen surface, which was later exposed to the plasma. Please note that this criterion – grains elongated perpendicular to the surface – is one requirement for W used in ITER [28] and is, therefore, frequently used in literature to quote such material as "ITER-grade", e.g. [11,13,19]. The specimens of the Plansee materials were polished as described in [26] to ensure removing all mechanical damage by residual grain deformation.

Specimens of $10 \times 10 \times 2$ mm^3 were cut from the Japanese material with their 10×10 mm^2 planes perpendicular to the main deformation direction (**WJ**). This leads also to elongated grains perpendicular to the specimen surface ("ITER-grade"). These specimens were polished by the Japanese manufacturers to a mirror finish (for quality of the polishing see [11,13]). The typical grain size parallel to the specimen surface is 1–3 µm, while perpendicular to the surface the size is 2–5 µm [11].

Two different treatments were applied to produce the two recrystallized specimens. The polished specimen of the rolled Plansee material was heated to 2000 K for 0.5 h in helium atmosphere in house (**rc-WP-R**). The polished specimen of the Japanese material was heated by the manufacturer to 2070 K for 1 h in hydrogen atmosphere (**rc-WJ**). The deformation processing of that specimen was different from that of the specimen **WJ.** The grain size parallel to the surface for the recrystallized specimens **rc-WP-R** and **rc-WJ** are 10–30 and 20–100 µm, respectively. The different size could be due to the differences in the degree of deformation, the impurity content and the recrystallization conditions.

The preferred orientation of the grains parallel to the exposed surface of the five specimens was determined by X-ray diffraction (XRD 3003 PTS diffractometer, Seifert/GE). The two faces of the specimens of the rolled Plansee material have different rolling texture, one dominated by (1 0 0) and (1 1 1) planes parallel to the surface and the other by (1 1 0) planes, i.e., still easily recognisable after recrystallization [27]. These textures are slightly altered by the recrystallization procedure [27]. For the specimen **rc-WP-R** the side dominated by (1 1 0) planes was exposed to the plasma, while for the specimen **WP-R** the side dominated by (1 0 0) planes. The swaged Plansee material has a ⟨1 1 0⟩ fibre texture. Therefore, the exposed surface of **WP-S** is dominated by (1 1 0) planes. The Japanese material shows also a type of rolling texture and the exposed specimen surface of **WJ** is also dominated by (1 1 0) planes. For the recrystallized specimen **rc-WJ**, no conclusive texture analysis was possible due to the large grain size and the statistical small number of analysed grains. An orientation analysis in the frame of another study on such recrystallized specimens [29] points to a soft texture with a higher amount of grains close to (1 1 0) and a lower amount of grains close to (1 1 1).

Before exposure, all specimens were heated at 1200 K for 1 h for degasing, stress relieving and removing of oxide layers.

Equivalent specimens to **WP-R, rc-WP-R, WJ,** and **rc-WJ** – same material and treatment – were studied in [7–10,25,26], [7,8], [11,15], and [12–17], respectively. All equivalent specimens of rolled Plansee material originate from a single sintering batch to ensure the same initial microstructure and chemical composition. Specimens cut from the same rod of swaged Plansee material as the specimen **WP-S** were investigated in [18–20]. Unfortunately, these specimens [18–20] were polished with a different procedure than the recipe described in [26].