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Deuterium release and microstructure of tantalum-tungsten twin limiter exposed in TEXTOR-94

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Abstract

Ta–W twin limiter experiments had been carried out in TEXTOR-94. D_{β} and D_{γ} intensities were observed during discharges. As a result, the intensities from the Ta surface were less than those from the W surface. This is attributed to the release ratio of molecules and atoms from the surface, i.e. less atomic release and more molecular release from Ta. Concerning long term D retention in Ta, D distributed uniformly over the limiter and the content was about 4×10^{-5} D per Ta atom. The total amount in the Ta limiter was estimated 3×10^{20} D in the half limiter. After exposure, the Ta bulk showed a significantly modified surface: grain growth, wrinkle like deformations and local recrystallization. However, open cracks were not observed in Ta exposed in the same condition in which W created big open crack. © 2002 Elsevier Science B.V. All rights reserved.

1. Introduction

High-Z materials will be used as plasma facing materials (PFM) in future fusion devices [1]. Therefore, the studies on their performances under plasma load in existing fusion devices are great interest. In the tokamak TEXTOR, a high-Z research program has been carried out and high-Z test limiters such as tungsten and molybdenum have been exposed to the plasma [2–8]. To expose two different materials in a single discharge, twin limiter experiments have been performed. These twin limiter experiments allow one to compare directly two different materials. First, a tungsten (W) and graphite twin limiter has been investigated and compared to W

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and C from various points [4–8]. Recently, tantalum (Ta) and W twin limiter experiments were carried out. In the case of Ta and W, both materials have similar masses, therefore, the materials have similar reflection coefficients for D. However, Ta is an exothermic hydrogen absorber and W is an endothermic one. Hence, the behaviours of D around the surfaces will be different between Ta and W, especially, in release process of D.

Another interesting point is the performance of Ta as a PFM. Ta has a great advantage in its high ductility. On the other hand, W has difficulty in machining due to its brittleness and it must be operated above its ductile brittle transition temperature [9]. From these points, it is reasonable to apply Ta instead of W. However, Ta has disadvantages of its poor thermal conductivity [10], its hydrogen storage property [11] and hydride formation [12]. Therefore, it is necessary to investigate the performance of Ta under plasma load. In the present paper, we discuss the difference of the deuterium released from Ta and W surfaces, deuterium retention in bulks and microstructures of the exposed test limiters.

2. Experimental procedure

TEXTOR-94 is a limiter tokamak with circular plasma of a minor radius of 0.46 m and a major radius of 1.75 m. The plasma is limited by ALT-II limiter. In these experiments, it had been operated with 350 kA plasma current, 2.23 T toroidal magnetic field and 6 s discharge duration. The electron density was being varied from 2×10^{19} to 6×10^{19} m⁻³ in density ramp discharges and the density was kept around 3×10^{19} m⁻³ in flattop discharges. Additional heating was provided by a neutral beam injector injecting tangentially with a power of 1.4 or 1.8 MW.

The experimental setup is schematically shown in Fig. 1. The twin limiter was inserted from the top into TEXTOR-94 scrape off layer through a limiter-lock manifold. The twin limiter was 12 and 8 cm long in toroidal and poloidal directions, respectively and the spherical face of the limiter in contact with plasma was the radius of 7 cm. One half of the limiter was made of Ta (Plansee, 99.95%) and the other half was made of W (Plansee, 99.95%). The twin limiter can be rotated in such a way that Ta or W face to either the ion-drift-side or the electron-drift-side. The twin limiter was positioned at either 46.5 or 47.5 cm from the plasma centre.

The interaction between plasma and twin limiter was observed by spectroscopy, i.e. by monitoring line spectra of D neutral species from surfaces. Radial distributions



Fig. 1. Schematic view of the experimental setup. The Ta–W twin limiter was inserted from the top to the scrape off layer. The interaction between plasma and limiters was observed by spectrometric cameras and a pyrometer.

of spectra line intensities emitted from ions and neutrals around the twin limiter were measured by an image intensified CCD-camera coupled to a monochromator. The spectra were recorded in the wavelength range around 409–435 nm where D_{δ} (410 nm), CII (426.7 nm), WI (429.5 nm) and D_{γ} (434.0 nm) were able to be observed. The two-dimensional intensity distribution of D_{B} was observed from the direction tangential to the limiter surface with another CCD-camera through interference filters at 486 nm. The third CCD-camera viewed the limiter surface from the bottom through an infrared transmission (850-1100 nm) filter to construct the temperature distribution on the twin limiter surface. The surface temperature was monitored with an optical pyrometer that pointed to the area of maximum power loading. Edge electron temperature and density were measured at the equatorial plane by He-beam diagnostics.

After the plasma exposure, the limiter surfaces and cross-section of Ta were observed by optical microscopy. Parts of the limiters were cut into pieces $(10 \times 10 \times 5 \text{ mm})$ and heated at 1273 K for 3 h in a thermal desorption chamber to estimate long term D retention.

3. Results

3.1. Deuterium release from the surface

Fig. 2 shows the D_{β} intensities in front of the Ta and the W test limiters as a function of the surface temperatures measured by the pyrometer in the flat top discharge phases. The surfaces were heated up to 1600 K by power load under the comparable D flux ($\Gamma_D = 5 \times 10^{22}$ m⁻² s⁻¹). As shown in Fig. 2, the intensity from the Ta surface was less than that from the W surface. The intensity from the W surface was independent of the surface temperature, however, the intensity from the Ta surface started to increase gradually above 1300 K and was approaching to the value of W.



Fig. 2. D_{β} intensity in front of Ta and W as a function of surface temperature during flat top discharges in TEXTOR-94.





Fig. 3. Radial profile of D_{γ} intensity decay in front of Ta (# 86789) and W (# 86768) at 1200 K. The D_{γ} intensity was normalized by incoming D flux onto the surface. Two exponential decays can be seen.

Fig. 3 shows the radial profiles of D_{γ} intensity in front of Ta and W at 1200 K, below the increase point of D_{β} intensity from Ta. The vertical axis indicates D_{γ} intensities normalized to the D incident flux that is calculated by edge plasma parameters. The horizontal axis indicates the distance from the plasma centre. In this axis, the surface of twin limiter is located at 46.5 cm. As can be expected from Fig. 2, the intensity from the Ta surface was less than that from the W surface, in particular, near the surface (x > 46.5 cm). And both radial profiles seem to have at least two exponential components.

3.2. Retained deuterium in the twin limiter

Thermal desorption measurements were carried out to estimate the amount of the retained D in the exothermic Ta and the endothermic W. Fig. 4 shows the D contents as a function of the distance from the exposed surface. The long term retention of D in Ta was about 4×10^{-5} D per Ta atom. The distribution in Ta was uniform over the whole limiter, which was probably caused by large diffusion coefficient even at room temperature (5.2×10^{-6} cm² s⁻¹ [12]). The total D retention in Ta can be calculated to 3×10^{20} D atoms. The W bulk contained in 2 orders of magnitude less D than the Ta bulk and contents in the W bulk were very close to the detection limit.

3.3. Microstructure of tantalum twin limiter

The Ta surface was heavily damaged by the plasma exposure. On the other hand, the W surface showed neither significant grain growth nor surface modifica-



Fig. 4. D contents in Ta and W as a function of the distance from the exposed surface. Ta was exposed for 136 s and W was exposed for 231 s.

tion, which had already been presented in previous papers [3–5,9]. Fig. 5(a) and (b) shows the typical microstructures of Ta surface around the maximum power load, and Fig. 5(c) and (d) shows the cross-section. In the Ta surface, recrystallization localized not only at the highest heat load point but also at the surrounding area. The surface was mostly covered with wrinkle-like plastic deformation and closed cracks. The cross-section showed recrystallization, plastic deformation and the grown grains which size was in the order of centimetres.

When the twin limiter experiments started from the limiter temperature of 420 K, open cracks running over whole limiter were created in the W limiter (Fig. 6). This



Fig. 5. Microstructures of Ta, (a,b) the top surface and (c,d) the cross-section. The limiter was exposed to the plasma for 136 s.



Fig. 6. Pictures of Ta and W limiters exposed to the plasma.

was reported in a previous paper [9]. The Ta test limiter, however, was free from open cracks, even though Ta showed the significant surface modification. The ductility of Ta could avoid creating large open cracks.

4. Discussion

4.1. Deuterium release and retention

As shown in Fig. 2, the intensity of D_{β} in front of Ta was less than that of W and started to increase at a certain temperature. One should note that D_{β} intensities represent only the atomically released component. Concerning the tendency observed in the Ta surface, a similar phenomenon has been founded in graphite limiter experiments in TEXTOR [13] and in laboratory experiments [14]. According to these studies, the hydrogen was released as a mixture of atoms and molecules at low temperature and the ratio of atomic release increased at high temperature. In other words, the temperature dependence of D_{α} and D_{β} intensities is attributed to the ratio of molecular and atomic particle release. Ta which is an exothermic hydrogen absorber has similar properties as graphite from a viewpoint of hydrogen recycling. The temperature dependence shown in Fig. 2 can be explained by the same logic. Namely, molecular and atomic releases had been mixed at the lower temperature and then atomic release began to become the dominant above 1300 K. This interpretation is also supported by recent laboratory experiments of Ta and W [15]. In their work, it was concluded that the molecular component caused the difference between the D_{α} intensities from Ta and W surfaces. It is necessary to evaluate directly the D_2 band intensity as a function of temperature in the next experiments.

The radial profile could be distinguished into two exponential components as shown in Fig. 3. A simple TRIM calculation combined with CR-model [16] indicated that the shorter decay component (λ_1) could be fitted by the atomically released components from the surface. And the longer decay length (λ_2) seemed to be fitted by not only the component of reflected particles that have higher energy (longer penetration length) but also the component of molecular released particles. Namely, the radial profile can be explained that D_{γ} intensities by the released atoms from the surfaces are dominant component in the region of decay length λ_1 and D_{γ} intensities by the dissociated atoms from molecules released from the surfaces and reflected atoms are dominant component in the region of decay length λ_2 . Further calculations are necessary to understand clearly the species and energy of release particles.

The total retention was found about 3×10^{20} D atoms. One can estimate the net implantation rate as 4×10^{16} D cm⁻² s⁻¹ (limiter surface area 60 cm² and duration 136 s). This number is quite small compared to the incoming D flux ($10^{22}-10^{23}$ D m⁻² s⁻¹). The fraction incoming D to retained D is less than 10^{-5} . Possibly a part of implanted D desorbed after the shots.

The difference of the retained D between in the Ta bulk and in the W bulk was attributed to the essential difference between exothermic and endothermic hydrogen absorbers. In this point, W is obviously superior to Ta.

4.2. Microstructure of Ta twin limiter

The manufacture process of Ta limiter was reported as followings: first, 60% of forging was done on Ta ingot, secondary, the forged Ta was mechanically processed to the shape of the limiter and finally no stress relieving process was done. This process could leave considerable stress in the bulk that would be the strong driving force of grain growth especially above the recrystallization temperature (1500–1700 K) and, in fact, it is well known that grains in Ta could easily grow in a short time. Ta which has a poor thermal conductivity was easily heated up over the recrystallization temperature and thus huge grains (Fig. 5) were created.

Another remarkable point is the plastic deformation in the shrinkage process after thermal expansion. This rapid cooling-down phase over more than 1000 K will cause tensile stress in the bulk in a very short time. As it is often the case of fast deformation process, deformation twins, slip lines and cracks were created. The wrinkle-like plastic deformations seems to be deformation twins and slip lines caused stress relief during rapid cooling phase.

Due to the additional effect of hydride formation in exothermic hydrogen absorber such as Ta, the additional energy of hydride formation could cause very localized recrystallization which scattered not only in the area at the maximum heat load but also in the surrounding area.

5. Summary

Ta and W twin limiter experiments had been carried out in a tokamak TEXTOR-94. As a result of the observation of D_{β} and D_{γ} intensities during discharges, the intensities from the Ta surface were less than that from the W. This difference is attributed to the release ratio between molecules and atoms from the surfaces. Concerning long term D retention in Ta, D distributed uniformly because of diffusion over the limiter and the content was about 4×10^{-5} D per Ta atom, which is more than in 2 orders of magnitude higher than the content in W bulk. The difference of the retained D between in the Ta bulk and in the W bulk was attributed to the essential difference between exothermic and endothermic hydrogen absorbers. In this point, W is obviously superior to Ta.

Even though exposed Ta showed significantly modified surface: remarkable grain growth, deformation twins, slip lines and localized recrystallization, no formation of open crack formation in Ta was observed in the same condition in which W created big crack. This is a possible advantage of Ta as a high-Z component.

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