Influence of surface melting on the deuterium retention in pure and lanthanum oxide doped tungsten

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Abstract

The issue of material melting under transient heat fluxes is one of the key concerns associated with the use of tungsten (W) divertor targets in ITER as the next step fusion device. This study aims to closely examine the impact of surface melting on fuel retention with respect to the performance in subsequent operations. Besides pure W, W - 1 wt. % La₂O₃ (WL10), which is considered as an advanced plasma facing material, is tested as a comparative study. The two tungsten grades are successively exposed to high heat flux (HHF) loads and low-energy deuterium (D) plasma. The thermal desorption spectroscopy (TDS) measurements indicate that D retention reduces by ~40% after shallow surface melting for both of the two W grades. On the other hand, D retention in the WL10 is almost double that in the pure W regardless of whether surface melting occurs or not. The TDS spectra exhibit two main desorption peaks at around 550 K and 700 K, which largely corresponds to grain boundaries and voids respectively. However, an additional peak appears unexpectedly around 1070 K for the melted WL10. We attribute this additional high-temperature peak to the formation of the hydrogen absorbing metal La in the resolidified layer, which is confirmed by transmission electron microscopy and energy dispersive X-ray spectrometry results.

Keywords: tungsten, lanthanum oxide, melting, microstructure, deuterium retention

1. Introduction

A poloidal divertor with a full-tungsten (W) armor will be used in ITER from the start of operations. It should tolerate high heat and particle fluxes as the main interface between the plasma and the material part [1,2].

A potential issue associated with the use of W divertor armor, however, is the material melting caused by off-normal events, as well as by leading edge exposures during steady-state and slow transients [3]. The melt layer motion, ejection, and evaporation result not only in the generation of substantial plasma contamination, but also in the reduced lifespan of plasma facing components [3,4]. The material microstructure and properties are significantly changed after resolidification, and can further influence fuel retention and recycling [5-7].

In most cases of macroscopic melting, coarse grains are observed in the resolidified layer [5,6,9,10]. Numerous spherical voids, in particular, are formed because of the cavitation in the W melt [5,6,8,9,10], which degrades the mechanical properties and the power-handling capabilities [5,9].

Our previous study has experimentally confirmed that the addition of lanthanum oxide (La_2O_3) can significantly suppress the cavitation in the melted W, which also leads to the dramatic reduction in evaporation and droplets ejection [6,10]. This indicates La_2O_3 -doping plays a dramatic role on alleviating the consequences of W melting in addition to its well-known advantages such as improving the mechanical properties and increasing the recrystallization temperature of W [11,12]. These features attracted our interest to investigate further by exposing the melted pure and La_2O_3 doped W to deuterium (D) plasma and compare their performance in terms of D retention.

In this study, we carry out a comprehensive test of the pure and La_2O_3 doped W as a plasma facing material under the synergy of HHF loads and D plasma. Low-energy D plasma irradiation is performed on these two W grades, which follows the melting pre-exposures by applying high heat flux (HHF) loads. The effect of surface melting on D retention is investigated in parallel with the impact of La_2O_3 -doping on the microstructure changes and the related influence on D retention is discussed.

2. Experiments

Rolled plates of commercial-purity tungsten (CPW) and W-1 wt. % La_2O_3 (WL10) respectively produced by Advanced Technology & Materials CO., Ltd., China and Beijing Tian-Long Tungsten & Molybdenum CO., Ltd., China are used in this study. They are fabricated from high-purity powder by sintering and rolled with a total reduction of ~80 % to a final thickness of 3 mm. The purity of CPW is > 99.96 wt. % by identifying the contents of more than 20 impurity elements, and the impurity content in WL10 is detected to be in the same level as CPW (< 0.04 wt.%). The features of W grains in rolled CPW and WL10 are quite similar, which are elongated

parallel to the plate surface with a typical length of 1-5 μm and an aspect ratio of 2-3. For WL10, La_2O_3 dopants with an average size of around 3 μm are dispersed in the W matrix. The detailed microstructure characterizations can be found in [6, 10].

Rolled CPW and WL10 plates with a surface area of 80 mm $\times$ 70 mm and a thickness of 3 mm are initially adiabatically exposed to dedicated HHF loads for surface melting in the HHF test facility GLADIS [13]. A Gaussian hydrogen neutral beam (~ 22 keV), with a center peak of 23 MW/m² and 80–100% of the central power covering the whole plate surface, is applied for 1.5–1.8 s. As a result, a circular melted area with a diameter of > 25 mm is obtained on the surface and thereafter small pieces with a square surface of 12 mm $\times$ 12 mm are cut from the melted area for D plasma exposure. The thickness of the small melted samples is not significantly changed in spite of the motion of the surface melt layer. Since the small melted samples consist of a resolidified surface layer and a fully recrystallized bulk [6,14], a recrystallized sample is chosen as the reference, which is cut from the unmelted region close to the melted area edge in the same HHF-loaded plate. That is to say the recrystallized sample experienced a peak temperature close to the melting point of W (3695 K) during HHF loading. Four kinds of samples, recrystallized CPW, melted CPW, recrystallized WL10, and melted WL10 are used in the present research.

Before the D plasma exposure, the recrystallized samples are mechanically polished to remove the damaged layer caused by energetic hydrogen particles bombardment [15], while the melted samples retain the newly formed resolidified surface. All samples are outgassed at 1273 K for 2 h at a background pressure of $< 1 \times 10^{-3}$ Pa in a vacuum furnace.

The D plasma exposure experiments are conducted in a low-pressure steady-state electron cyclotron resonance (ECR) plasma chamber ‘PlaQ’ [16]. The flux of D plasma is about 1×10^{20} D/(m²s) and the ion energy is 38 eV per deuteron. With an exposure duration of up to 17 hours the fluence reaches 6×10^{24} D/m². The exposure temperature is kept at 373 K during irradiation.

In order to characterize the microstructure resulted from HHF loading, fracture surfaces (2 mm $\times$ 3 mm) are prepared by **artificially** breaking thin rod-like pieces with a width of 2 mm and a length of 12 mm, which are cut next to the square samples from the 3 mm thick HHF-loaded plates. Then they are observed by a scanning electron microscope (SEM, TESCAN LYRA3 FEG-SEM/FIB) equipped with an Energy Dispersive X-ray (EDX) spectrometer. Detailed microstructure analysis is performed using a transmission electron microscope (TEM, FEI TECNAI G20), and the TEM samples are obtained by using a focus ion beam (FIB). Inductive coupled plasma atomic-emission spectrometry (ICP–AES) is used for the elemental analysis.

D desorption from the irradiated samples are measured with thermal desorption spectroscopy (TDS). The base pressure of the TDS chamber is better than 5×10^{-5} Pa. The sample is heated with a ramping rate of 1 K/s to 1273 K. The gases such as HD (mass 3), D₂ (mass 4), HDO (mass 19) and D₂O (mass 20) are monitored by a sensitive quadrupole mass spectrometer (MKS Microvision Plus). For calculating the total D retention, only the desorbed D in the form of HD and D₂ is included since the

partial pressure of deuterated water (HDO and D₂O) does not significantly change before and during the TDS heating process, and their signals could not be calibrated. The uncertainty of the TDS-measured D amount is about 7%, which is primarily due to the stability of the detector in the mass spectrometer and presence of deuterated water caused by the D release.

3. Results and discussion

Figure 1. Microstructures on the fracture surface of the recrystallized and melted CPW resulted from HHF loading. The orange line in (c) indicates the bottom of the resolidified layer. (b) and (d) illustrate the detailed feature on the fracture surface of the recrystallized and resolidified grain respectively.

The SEM images in figure 1 present the morphology on the fracture surface of the recrystallized and melted CPW due to HHF loading. Both exhibits largely intergranular fracturing of 3 mm in thickness, indicating the significant recrystallization embrittlement. For the recrystallized one (figure 1(a)), the recrystallized grains have an average grain size of around 90 μm that is clearly observable on the fracture surfaces. In addition, some small voids are found in the recrystallized CPW (figure 1(b)), which should be ascribed to the vaporization of low melting point impurities. The fracture of the melted CPW shows coarse resolidified grains over 100 μm in depth and fully recrystallized bulk below (figure 1(c)). In the resolidified layer, spherical voids varied in size (tens of nanometer to tens of micrometer) can be observed, suggesting intense bubble boiling in the W melt. Compared to the recrystallized microstructure, the main characteristic is that the columnar resolidified grains are perpendicularly orientated in relation to the surface.

Figure 2. (a) Fracture surface morphology of the recrystallized WL10 resulted from HHF loading. (b) illustrates the detailed feature on the fracture surface. (c, d) EDX results of the point 1 and 2 in (b), indicating they are La_2O_3 particle and void respectively.

For the recrystallized WL10, the average grain size is about $40\ \mu\text{m}$ (figure 2(a)), which is much smaller than that of CPW (figure 1(a)). A large number of micron-sized La_2O_3 particles (as the point 1 in figure 2(b) and (c)) can be found on the grain boundaries. They contribute to the smaller grains not only by impeding the grain boundary movement but would also by absorbing energy from the neighboring grains during its melting process, since the melting of La_2O_3 particles is supposed to occur due to its relatively low melting point $2586\ \text{K}$ [17]. At the same time, La_2O_3 -doping induces lots of interfaces and voids (as the point 2 in figure 2(b) and (d)).

A typical morphology of the melted WL10 is shown in figure 3(a). The recrystallized bulk is quite similar as the reference WL10 (figure 2(a)). The resolidified layer also includes columnar grains with $>100\ \mu\text{m}$ thick as observed in the melted CPW, but voids are rare, revealing the suppressed cavitation. After melting the micron-sized La_2O_3 particles are no longer present in the resolidified layer. The average concentration of lanthanum is only around $\sim 6.5\ \mu\text{g/g}$ according to ICP–AES measurements from three different positions in the resolidified layer, which implies intense vaporization of La_2O_3 in the liquid phase of W [10,18]. Instead, we find many nanoparticles as shown in the figure 3(b).

In order to identify the nanoparticle, several detailed analyses are done using a TEM sample obtained through FIB cutting on the fracture surface of a large resolidified grain (figure 3(c)). Point EDX analysis is performed inside the

nanoparticle as indicated by a white cross in figure 3(c). The result (figure 3(d)) shows that La is the main element, while O is negligible. A selected area electron diffraction (SAED) image of the nanoparticle is shown in figure 3(e). The bright diffraction spots come from the W grain with 0.2230 nm $\{110\}$ plane and $[111]$ crystal axis. In addition, we can see many hazy spots distributed on diffraction rings. We measure the three rings as indicated by dot lines in figure 3(e), and obtain the corresponding interplanar distances are 0.3025 nm, 0.2133 nm and 0.1737 nm, which fit γ -La (110) 0.3012 nm, (200) 0.2130 nm and (211) 0.1739 nm. The spots on the diffraction rings are checked using dark field images, and one of the dark field images corresponds to the spots indicated by a small orange circle in figure 3(e) is shown in figure 3(f). It is confirmed that these spots originated from the previously mentioned nanoparticle. These results demonstrate that the nanoparticles appear in the resolidified layer of WL10 is La. This means part of lanthanum oxide decomposed into La in the W melt. Since La cannot dissolve in W [19], it is reasonable to observe La particles in the W matrix.

Figure 3. (a) Microstructures on the fracture surface of the melted WL10 resulted

from HHF loading, and the orange line indicates the bottom of the resolidified layer. (b) Nanoparticles on the fracture surface of a resolidified grain. (c) A TEM sample prepared by FIB with coating of the surface shown in (b) with a layer of platinum (Pt). (d) EDX analysis of a point within the nanoparticle, where is indicated by the white cross in (c). (e) A SAED picture of the nanoparticle. (f) A dark field image corresponding to the hazy spots on a diffraction ring indicated by the small orange circle in (e).

Figure 4. TDS results of the four kinds of analyzed samples exposed to a fluence of 6×10^{24} D/m² with 38 eV/D at a temperature of 373 K. The heating rate is 1 K/s for all samples.

Figure 4 presents the D₂ and HD desorption rate in contrast to temperature during TDS heating process of the four analyzed samples. These results reveal that there are two main desorption peaks in common located at ~550 K and ~700 K respectively.

For the 550 K peak, the intensity is obviously reduced via surface melting, while increased through La₂O₃ doping. This kind of low temperature desorption peaks (up to 600 K) is supposed to be related to point defects, dislocations, and grain boundaries [20,21]. According to Manhard et al.'s study [21], a desorption peak around 600 K is significantly enhanced after recrystallization, where most of the dislocations are removed, and grain boundaries are the main D trapping site. Therefore, we ascribe this 550 K peak mainly to grain boundaries, based on the aforementioned

microstructure observations. Surface melting results in the coarse columnar grains becoming orientated perpendicular to the exposed surface, which not only reduces the grain boundary density but also enhances the outward D diffusion. Hence, the 550 K peak is reduced after surface melting (figure 4(c) and (d)). On the other hand, La_2O_3 dopant reduces the size of recrystallized grains, resulting in a relative strong intensity of the peak for WL10 (figure 4(b) and (d)).

With regard to the 700 K peak, the intensity of the recrystallized WL10 is much higher than other three samples. We infer that voids are the primary cause of this desorption peak [20,22]. Owing to La_2O_3 doping, a large number of W- La_2O_3 interfaces and voids were introduced (figure 2), which may have contributed to the noticeable emergence of the 700 K peak, and a similar phenomenon has been also reported in [23]. However, this peak is sharply reduced after the surface melting of WL10 (figure 4(d)), which should be attributed to the formation of the relatively dense resolidified layer (figure 3(a)).

It is worth to note that there is an additional desorption peak at around 1070 K (figure 4(d)) and it only occurs with the melted WL10. By comparing the microstructure features of the four samples, the unique feature of the melted WL10, namely the La nanoparticles present in its resolidified layer (figure 3) should be responsible for this additional peak.

Different from lanthanum oxide, La can react with hydrogen, and finally transforms into lanthanum hydride ($\text{LaH}_{1.9-3}$) [24,25]. This suggests the La nanoparticles can play an important role in absorbing D. During TDS measurement, D would release from lanthanum hydride and diffuse through W lattice to the surface [26]. Hence, the activation energy for its desorption reaches up to 2.47 eV/H, considering the enthalpy of formation for LaH_2 (around -1eV/H) [27], the solution energy of H in W (1.07 eV/H) [22] and the diffusion barrier of H in W (0.4 eV/H) [22]. This high activation energy indicates the appearance of the 1070 K desorption peak in the TDS spectrum [28].

To further confirm the impact of La in the D retention by hydrogenation, we do the estimation as follows. Taking into account the thickness of the resolidified layer $\sim 100 \mu\text{m}$ and the content of La is $\sim 9.0 \times 10^{-5} \text{ mol} / \text{m}^2$ according to the mass concentration $\sim 6.5 \mu\text{g/g}$, and assuming it completely transforms into lanthanum hydride during D implantation, it would consume $1.1 \times 10^{20} \text{ D} / \text{m}^2$ in form of LaD_2 , and $1.7 \times 10^{20} \text{ D} / \text{m}^2$ as LaD_3 , which match well with the measured deuterium release amount $1.4 \times 10^{20} \text{ D} / \text{m}^2$ by integrating the 1070 K peak (including D_2 and HD) in figure 4(d).

Figure 5. The total amount of D released from the four samples determined by TDS.

The total D release amount of the four samples found through TDS is illustrated in figure 5. They are all in the range of $(1 \times 10^{20} \sim 1 \times 10^{21})$ D/m², consistent with D retention in W-based materials reported in literatures [21,29], in which plasma exposures were carried out at the same conditions as this study. The total D release amount of the recrystallized CPW is 3.8×10^{20} D/m², while it drops to 2.2×10^{20} D/m² in the melted CPW. Similarly, in the case of WL10, it decreases from 7.1×10^{20} D/m² in the recrystallized sample to 4.5×10^{20} D/m² in the melted one. It seems that the surface melting associated with the formation of the ~ 100 μ m thick resolidified layer (less than 4% of the whole sample thickness) significantly reduces $\sim 40\%$ of the total D retention for the two tungsten grades.

For CPW, the reduction of the total D retention after surface melting is mostly caused by the drop after the 550 K desorption peak according to the TDS spectra (figure 4(c)), which is attributed to the formation of the coarse columnar resolidified grains. In the case of the WL10, the significant drop after the 700 K desorption peak (figure 4(d)), is the main reason of the reduced D retention. It should be related to the two main benefits obtained from surface melting. W-La₂O₃ interfaces are substantially removed, because the La₂O₃ dopants are eliminated due to evaporation [18]. Moreover, the cavitation in the W melt is suppressed by the “cleaning effect” related to the evaporation of the lanthanum additives [10], and consequently, few voids are formed in the resolidified layer.

The melting of metallic surface, especially of tungsten-based materials, is one of the most important topics for ITER and future fusion devices, and unfortunately it cannot be excluded due to possible loss of plasma control [30]. More attention has been focused on evaluating the performance of the resulting resolidified material [5,7]. Hamaji et al. [7] has investigated the D retention in resolidified W damaged by using an electron beam, the results show the effect of surface melting on the D retention is not significant. The present results in this study, however, clearly demonstrate that surface melting reduces the D retention in the damaged materials. Although the coarse

resolidified microstructure would undoubtedly reduce the mechanical property and the cracking threshold against thermal shocks [5,31], it does not denigrate the performance in terms of fuel retention.

On the other hand, the D retention is doubled because of the 1 wt. % La_2O_3 -doping, by comparing CPW and WL10 (figure 5). Though the D retention is dramatically reduced after surface melting, problems arise due to the transformation of lanthanum oxide into La, which can react with hydrogen. Our results indicate that only $\sim 6.5 \mu\text{g/g}$ La remaining in the $\sim 100 \mu\text{m}$ thick resolidified layer of the $\sim 3 \text{ mm}$ thick bulk could contribute to nearly 30% of the total D retention ($4.5 \times 10^{20} \text{ D/m}^2$) by forming lanthanum hydride. It is worth noting that surface melting of W armor would occur faster in ITER compared to this study, since more intense heat fluxes of $\sim \text{GW/m}^2$ would deposit on the W armor within milli-seconds during the transients, like ELMs (Edge Localized Modes) and disruptions [30]. Additionally, a quicker resolidification process is expected, because the W divertor is actively cooled in ITER [2]. In this context, less La would be vaporized from the W melt, since the duration for W to stay in liquid phase is shortened. Consequently, a higher content of La would remain in the resolidified layer, increasing the risk to fuel retention and recycling.

Dispersion strengthening is among the main strategies for ductilization, which addresses the brittleness of W at low temperatures [32]. However, the oxide and carbide dopants (e.g. La_2O_3 , Y_2O_3 and TiC) have been widely reported to significantly increase the D retention in advanced tungsten-based materials [23,29,33-35]. Moreover, similar with La, metal elements Y and Ti also have the ability to form high temperature hydrides [25]. Therefore, it is worth paying attention to the evolution of these oxide and carbide dopants under extreme irradiation conditions in tokamaks, and their potential for increasing D retention (grain boundary, interface, void and/or hydride) needs to be taken into consideration for the advanced plasma facing material development.

4. Conclusion

In this work, we prepare melted CPW and WL10 via dedicated surface melting exposure by applying HHF loading. Compared to the melted CPW, the main features of the melted WL10 consist in the smaller recrystallized grain size and the introduction of W- La_2O_3 interfaces and voids in the fully recrystallized bulk ($\sim 3 \text{ mm}$), and no voids/cavitation but the occurrence of La nano-particles in the resolidified layer ($\sim 100 \mu\text{m}$ thick), respectively

After low-energy D plasma exposure at 373 K with a fluence of $6 \times 10^{24} \text{ D/m}^2$, TDS results show that due to surface melting the D retention is significantly decreased by $\sim 40 \%$ for both CPW and ML10. The columnar resolidified grains orientated perpendicularly relative to the surface should be a key factor for this phenomenon, which not only reduces the density of grain boundaries but also can

enhance the D outward diffusion.

Though La_2O_3 is substantially vaporized in the melted WL10, a small amount of La ($\sim 6.5 \mu\text{g/g}$) remains in the resolidified layer. Since La can react with hydrogen and results in the formation of high-temperature hydride, it contributes to $\sim 30\%$ of the total D retention and the inducement of an additional desorption peak at $\sim 1070 \text{ K}$ in the TDS spectrum. In this context, despite the number of voids is significantly reduced in the resolidified layer of WL10 due to the suppression of cavitation compared to the melted CPW, the total D retention is still much higher in the melted WL10.

The presented results improve the current understanding of the fuel retention properties of damaged tungsten-based materials caused by melting exposures. In addition, important information is provided by showing the transformation of dopants due to extremely high heat loads, which should be taken into consideration from the point of view of future material selection and development.

Acknowledgements

The authors would like to thank Henri Greuner from IPP Garching for the support with the HHF tests in GLADIS. This work was supported by the National Nature Science Foundation of China under Grant 11775015 and a fund from the Science and Technology on Surface Physics and Chemistry Laboratory (No. 02020317). Yue Yuan acknowledges the support from the Young Elite Scientists Sponsorship Program from CAST under Grant 2017QNRC001. This work was supported by the Users with Excellence Project of Hefei Science Center of CAS under Grant 2018HSC-UE006.

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