Transient grating spectroscopy of thermal diffusivity degradation in deuterium implanted tungsten

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Abstract

Using transient grating spectroscopy, we measure thermal diffusivity and surface acoustic wave speed in tungsten exposed to different fluences of deuterium plasma. Scanning electron microscopy (SEM) shows the formation of surface blisters that have similar morphology for all fluences. A significant reduction in thermal diffusivity and surface acoustic wave speed occurs as a result of plasma exposure. A saturation of the thermal diffusivity reduction with fluence is seen. Deuterium ion flux density appears play a more important role in the thermal diffusivity reduction than the fluence. These results have important implications for plasma facing components in future fusion reactors.

Keywords

Deuterium plasma exposure, thermal conductivity, fusion armour materials, transient grating spectroscopy, surface acoustic wave

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Main Text

Mitigating climate change will require new solutions for sustainable power generation. Nuclear fusion could provide a carbon-neutral and long-term power source. However, there are major engineering challenges to be addressed on the path to its commercialisation. One such challenge is the performance of plasma-facing materials, especially those used in the divertor, which will face the harshest conditions. Tungsten is the front runner for divertor armour due to its high melting point, low sputtering yield, strength at high temperature and high thermal conductivity [1]. As such, a detailed exploration of its performance under fusion reactor relevant conditions is of high importance [1–3].

Understanding material property evolution due to harsh environments is key for the success of ITER and DEMO fusion programs [3]. It is anticipated that tungsten divertor armour in a tokamak reactor will be exposed to high heat fluxes (~10 MW/m² during steady state operation and ~20 MW/m² during slow transients [4,5]), intense irradiation with 14.1 MeV fusion neutrons and bombardment with ions (predominantly hydrogen and helium isotopes). A particular challenge is that hydrogen isotopes readily diffuse into the metal matrix, where they can substantially modify material properties and structure [6–9]. Trapping of deuterium at defects, as well as self-trapping have been observed to lead to the formation of voids in the material and the emergence of surface blisters [7–9]. Since tungsten armour components will be exposed to high heat loads, this raises concerns, as blisters are expected to degrade the heat removal capability of plasma facing components by reducing near-surface thermal diffusivity. This in turn would lead to higher than predicted surface temperatures, potentially causing localised melting. In this study we quantify the effect of deuterium plasma exposure on the thermal diffusivity of tungsten over a range of operation-relevant temperatures, flux densities and exposure times.

Deuterium injected into the material from the plasma affects the near surface region up to depths of several microns [10]. Bulk thermal transport measurement techniques are not suitable for measuring the properties of this thin layer. Instead a different approach, called transient grating spectroscopy (TGS) [11–14] is used in this study. It allows selective probing of the properties of few-micron-thick surface layers. A detailed description of the technique is provided elsewhere [12,14]. Briefly, in TGS two short, coherent laser pulses (0.5 ns, 532 nm) are overlapped on the sample surface with a well-defined angle. Interferences of the beams creates an intensity grating with wavelength \( \lambda \) on the sample surface. Some of the light is absorbed, leading to the formation of a temperature grating in the sample. This temperature grating decays with time as thermal energy diffuses from maxima to minima and into the bulk. In addition, rapid thermal expansion also launches two counter-propagating surface acoustic waves (SAWs).

Both the SAWs and the temperature grating lead to a modification of the sample surface height. This “transient” grating acts as a reflective diffraction grating in the sample and can be probed by diffraction of a second beam (continuous wave, 561 nm). The thermal diffusivity of a surface layer with thickness \( \lambda/\pi \) determines the rate at which the diffracted signal decays. Hence, by analysing the amplitude decay of the diffracted probe beam, the thermal diffusivity can be determined [13].

The TGS setup used in this study incorporates the simultaneous dual heterodyne measurement method proposed in [14]. A transient grating wavelength on the sample of \( \lambda = 2.758 \mu m \) is used. The average pump laser power at the sample was 1.9 mW (1 kHz repetition rate with 1.9 \( \mu J \) per excitation) and the total probe beam power was ~100 mW. The size of probe and excitation spots on the sample was 90 \( \mu m \) and 140 \( \mu m \) respectively (1/e² level), and measurements were performed in a vacuum of -1 \( \times 10^{-3} \) mbar.
Polycrystalline tungsten disc samples with 20 mm diameter and 1 mm thickness were prepared from 99.95% purity material (main impurities: Mo, Pb, Si, all below 160 appm) procured from Goodfellow. The sample surface was mechanically ground with SiC abrasive paper down to 2500 grit, followed by mechanical polishing with 3 and 1 μm diamond suspension on NLH polishing cloths. The samples were electrochemically polished in 0.5 wt % NaOH water solution at 12 V and then heat treated at 1300 K in vacuum (\(10^{-5}\) mbar) for 1 hour. Supplementary figure S1 shows a representative electron back scatter diffraction (EBSD) map of the microstructure. Deuterium plasma exposures were performed at the linear plasma generator Pilot-PSI [15]. Ion flux profiles were obtained from Thomson scattering and the surface temperature was measured using an IR camera. Details of the exposures can be found in [11]. TGS measurements were performed for three lines across each sample with a step of 200 μm, and with a spacing of 200 μm between the lines. For each position, ten measurements were recorded, each averaging over 2000 pump pulses. The resulting traces were fitted to extract thermal diffusivity and SAW speed [16]. Variation in the measured thermal diffusivity was consistently less than 5 %, and less than 0.07 % for the SAW speed. Varying the starting point of the fit changed the thermal diffusivity values by less 2 %.

Supplementary figure S2 shows an example TGS trace and the fit used to extract thermal diffusivity and SAW speed. Scanning electron microscopy of the samples was performed on an Auriga Zeiss dual-beam FEG SEM-FIB.

To investigate how thermal diffusivity varies with deuterium flux, fluence and exposure temperature, an experimental matrix with the following four exposure conditions was used: high temperature high dose (HT HD), high temperature low dose (HT LD), low temperature low dose (LT LD) and low temperature high dose (LT HD). Temperature and dose are determined at the centre of the plasma beam and are hence maximum values. “Low temperature” refers to ~450 K, “high temperature” to ~650 K, “low dose” to ~5 \(10^{25}\) m\(^{-2}\) (70 seconds exposure), “high dose” to ~\(10^{27}\) m\(^{-2}\) (1400 seconds exposure). The Gaussian nature of the ion beam allowed a spatial variation of flux and fluence across the sample surface. A 2 mm wide region around the sample edge was shielded from the plasma. Fig. 1 show the deuterium ion fluence profiles and temperature profiles for all samples. During the exposure the central region of the two samples exposed at high temperature (HT samples) was contaminated with molybdenum originating from the plasma source. Points from these regions are excluded from this study.
Figure 1. (a) Profile of incident deuterium ion fluence for the four samples. The reference fluence at the shielded sample edges is zero. (b) Surface temperature profiles for the four different samples. The temperature of the shielded edges is assumed to be consistent with the near-Gaussian nature of the temperature profile. The black-dashed box in the centre depicts the molybdenum-contaminated regions. The shielded region is depicted by the grey shaded box.

SEM micrographs of the deuterium exposed regions show prominent blistering in all samples (Fig. 2), while unexposed regions are blister free (see supplementary Fig. S3). For the low temperature (LT) samples there is no obvious variation of blister morphology with spatial position. The blister diameter is ~0.5 μm, consistent with previous studies on tungsten exposed to deuterium plasma at similar flux densities and temperatures [17]. Micrographs from the LT-LD sample (Fig. 2 (a) and (b)) and the LT-HD sample (Fig. 2 (c) and (d)) shows similar blistering, despite the fact that the LT-HD sample received a 20 times higher deuterium dose than the LT-LD sample. This suggests that, at least for the conditions considered here, the blistering effects is largely independent of deuterium fluence. The micrographs of samples exposed to deuterium at the higher temperature (HT) (Fig. 2 (e) and (f)) show blisters that are somewhat smaller. However again the blister morphology seems to vary little with deuterium dose.
Thermal diffusivity measurements were performed as line scans across the samples and include the unexposed edges as a built-in reference. Fig. 3 shows thermal diffusivity and SAW speed plotted as a function of position in the LT-HD sample. Profiles for the other samples are shown in supplementary figures S4-S6. The unexposed sample edges are easily identified by their higher thermal diffusivity and SAW speed (Fig. 3). The changes between unexposed and exposed regions are also very evident from TGS traces representative of both areas (supplementary figure S7). The unexposed regions (Fig. 3) have thermal diffusivity of \((6.6 \pm 0.2) \times 10^{-5} \text{m}^2\text{s}^{-1}\), which agrees very well with the literature value for tungsten at room temperature of \((6.4 - 7.1) \times 10^{-5} \text{m}^2\text{s}^{-1}\) [18–21]. Thermal diffusivity in the exposed region is reduced to values between 5.5 and \(6.3 \times 10^{-5} \text{m}^2\text{s}^{-1}\). At the very centre of the LT-HD sample there is a further drop to \(5.0 \times 10^{-5} \text{m}^2\text{s}^{-1}\). The SAW speed of the unexposed edges is \(2668 \pm 2 \text{ms}^{-1}\), in good agreement with previous measurements [22]. The SAW speed drops steeply from the unexposed to exposed region and then continues to gradually decrease towards the centre of the sample. The oscillations of SAW speed in the exposed region are reliably found in repeated measurements. This feature is not evident in the flux or temperature profiles (Fig. 1). Instead it can be ascribed to slight elastic anisotropy of tungsten, which results in a grain-orientation dependence of SAW speed. The variation we observe is \(-10 \text{ms}^{-1}\), which agrees with previous orientation-dependant measurements of SAW speed in a tungsten single crystal [22]. EBSD (supplementary figure S1) showed grain sizes of 200 - 400 \(\mu\text{m}\) in the present samples, which is consistent with the period of the SAW speed variations in the implanted region.
Figure 3. Thermal diffusivity (a) and SAW speed (b) profiles for the low temperature high dose (LT HD) sample. Error bars are the standard deviation for each point, obtained over 10 measurements. The x-axis is corrected for the centre of the incident ion beam. The shielded region is depicted by the grey box.

Figure 4. Measured thermal diffusivity plotted as a function of fluence and temperature for the four samples considered. Data from the high temperature high dose sample (HT HD) is depicted by circles, high temperature low dose (HT LD) by triangles, low temperature low dose (LT LD) by diamonds and low temperature high dose (LT HD) by squares. Reference fluence is zero, which is the value for the unexposed region.

Fig. 4 summarises the thermal diffusivity measurements, plotted as a function of exposure temperature and deuterium fluence. The unimplanted regions in all four samples have thermal diffusivity greater than $6.5 \times 10^{-5} \, \text{m}^2\text{s}^{-1}$ in agreement with previous studies [18–21]. There is a significant drop in thermal diffusivity between virgin and deuterium-exposed areas, irrespective of
fluence and exposure temperature. Samples exposed to the higher deuterium fluence show a larger reduction in thermal diffusivity compared to their low dose counterparts. The drop in diffusivity between the LD and HD samples (from $6.0 \times 10^{5} \text{ m}^2\text{s}^{-1}$ to $5.5 \times 10^{5} \text{ m}^2\text{s}^{-1}$), is similar in magnitude to the drop between the LD samples and unexposed regions (from $6.6 \times 10^{5} \text{ m}^2\text{s}^{-1}$ to $6.0 \times 10^{5} \text{ m}^2\text{s}^{-1}$). However, HD samples received a 20 times higher dose than LD samples, meaning that the drop in thermal diffusivity is not proportional to dose/fluence.

A similar trend has been observed by nuclear reaction analysis (NRA), where the sub-surface deuterium concentration in deuterium plasma-exposed tungsten samples was measured for different fluences [10]. NRA profiles showed a large difference in deuterium concentration between unimplanted regions and regions exposed to a fluence of $-5 \times 10^{25} \text{ m}^{-2}$, similar to the exposures considered here. The deuterium concentration in samples exposed to $-10^{27} \text{ m}^{-2}$ was only slightly higher. This suggests that with increasing fluence the deuterium concentration, blistering and thermal diffusivity reduction saturate. The fluence at which this saturation occurs may vary and depends on a number of factors such as the crystallography, heat treatment and surface polishing [7,8]. Since we observe little change in thermal diffusivity when fluence is increased by more than one order of magnitude, it is likely that our observations already fall within this saturation regime. It is also interesting to note that thermal diffusivity seems to correlate quite well with blister morphology, which remains largely unchanged between low and high dose exposed samples.

Previous studies suggested a number of different mechanisms for thermal diffusivity reduction due to plasma exposure/ion implantation [16,20,21,23,24]. Defects created by high energy ions or neutrons can function as effective electron scattering sites, thereby reducing thermal diffusivity, which for tungsten at room temperature is dominated by electron-mediated transport. For example significant reductions in thermal diffusivity have been reported for self-ion implanted tungsten [23,24]. However, the deuterium ion energy used in this study (~50 eV) was much lower than usual self-ion implantation energies (150 keV - 30 MeV)[24,25], and below the threshold required to produce displacement damage [26]. Hence displacement damage is not expected play a prominent role in our observations. Studies have shown that gases such as deuterium and helium can self-trap when injected into the tungsten matrix, i.e. an interstitial deuterium atom is attracted to another [16,27–30]. These gas interstitials then cluster, forming bubbles with increasing pressure inside, resulting in blisters. From the SEM micrographs in figure 2, it is clear that there is extensive surface blistering in all samples used in this study. These blisters are surface manifestations of such sub-surface gas-filled cavities [31]. These act as additional scattering centres and also suppress thermal diffusivity by reducing thermal contact between the surface and the bulk.

An interesting question concerns the substantial drop in thermal diffusivity seen at the centre of the LT-HD sample (Fig. 2 (a)). A similar effect is observed in the LT-LD sample (supplementary Fig. S4). Both samples were exposed to deuterium beams with a near-Gaussian profile and the same flux density (Fig. 1). However, the LT-HD sample received a 20 times larger fluence. The rather similar profile of thermal diffusivity as a function of spatial position in both samples, suggests that fluence has relatively little influence on thermal diffusivity degradation. The substantial reduction in thermal diffusivity at the centre of both samples, where flux density is highest, points to flux density playing a much more prominent role in controlling thermal diffusivity degradation.

These results have important implications for the design of armour components in future fusion reactors. In test components pristine tungsten was observed to undergo extensive melting at heat fluxes of $27-30 \text{ MW/m}^2$ [4], while future fusion reactors are expected to have peak heat fluxes of 10-20 $\text{ MW/m}^2$ in the divertor [5]. A reduction of thermal diffusivity will decrease the maximum heat flux that can be accommodated without melting. As such it is key that degradation of thermal transport
properties is accounted for in the design of armour components. A positive conclusion from this study is that the deuterium-exposure-induced reduction in thermal diffusivity appears to saturate quickly, rather than continuing to progress with extended exposure.

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Supplementary Information: Transient grating spectroscopy of thermal diffusivity degradation in deuterium implanted tungsten

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Supplementary Figure S1: Microstructure of the investigated tungsten following heat treatment determined by EBSD: (a) Orientation map (IPFY); (b) misorientation map; (c) identified grains.
**Supplementary Figure S2:** Transient grating signal and fitted trace for the unimplanted region in the low temperature high dose (LT HD) sample.

**Supplementary Figure S3:** SEM micrographs for the unimplanted regions in the LT LD (a), LT HD (b), HT LD (c) and HT HD (d) samples.
Supplementary Figure S4: Thermal diffusivity profile for the low temperature low dose (LT LD) sample. Error bars are the standard deviation for each point, obtained over 10 measurements. The red shaded region depicts data excluded from the analysis due to surface contamination. The unexposed region of the sample is shaded in grey.

Supplementary Figure S5: Thermal diffusivity profile for the high temperature low dose (HT LD) sample. Error bars are the standard deviation for each point, obtained over 10 measurements. The red shaded region depicts data excluded from the analysis due to surface and molybdenum contamination. The unexposed region of the sample is shaded in grey.
Supplementary Figure S6: Thermal diffusivity profile for the high temperature high dose (HT HD) sample. Error bars are the standard deviation for each point, obtained over 10 measurements. The red shaded region depicts data excluded from the analysis due to molybdenum contamination. The unexposed region of the sample is shaded in grey.

Supplementary Figure S7: Transient grating signal for a single measurement at the unimplanted edge (blue) and implanted centre (red) of the LT HD sample.