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Sequential and Simultaneous Thermal and Particle Exposure of Tungsten

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Abstract

The broad array of expected loading conditions in a fusion reactor like ITER necessitates high requirements on the materials that qualify as plasma facing materials (PFMs). Tungsten that has been selected as PFM for the divertor region, the most affected part of the in-vessel components, must thus sustain severe, distinct exposure parameters. Accordingly, comprehensive experiments investigating sequential and simultaneous thermal and particle loads were performed on double forged pure tungsten, not only to investigate whether the thermal and particle loads cause damage but also if the sequence of exposure maintains an influence. The exposed specimens showed various kinds of damage like roughening, blistering, and cracking at a base temperature where tungsten could be ductile enough to compensate the induced stresses exclusively by plastic deformation [1]. The adverse effect of hydrogen on the material performance was pointed out in common with the influence of the loading sequence.

Introduction

The in ITER expected environmental conditions for the in-vessel components and thus for the armour materials are severe and complex. The armour materials, or so called PFMs, must withstand not only steady state loads up to 20 MW/m² but also transient events (up to 1 GW/m² and above) like edge localised modes, disruptions, etc. and high particle fluxes such as hydrogen, helium and, neutron irradiation. Especially, the divertor will be the most affected part of the in-vessel components and therefore of special relevance in terms of performance, durability, and economy [2]. However, experiments that cover all anticipated exposure parameters are inherently difficult to realise. Nevertheless, comprehensive experiments investigating at least several exposure parameters at once were executed in the last years to predict the performance and the lifetime of the components [3-5]. In the experiments described in this article, various loading parameters were combined and samples with different microstructures (grain orientation, recrystallized) were investigated. In detail, the linear plasma generator PSI-2 [3] was used to simulate thermal and particle exposures with varying sequences. At this, the particle loads were represented by deuterium plasma while a Nd:YAG laser was used to simulate the fusion relevant transient events with power densities in the range of 0.19-0.64 GW/m². All experiments were executed at a base temperature of 400° C with a maximum number of transient events of 1000. This work focuses on the influence of sequential loading on the damage behaviour of tungsten, possible changes of the morphology, and consequential risks regarding the proposed area of application.

Experimental

The experiments investigating the impact of sequential and simultaneous thermal and particle loads were realized in the linear plasma generator PSI-2, located at Forschungszentrum Jülich. PSI-2 offers the opportunity to study synergistic effects of plasma-wall interactions among others. The experiments were performed on industrially available, double forged pure tungsten (Plansee SE, Austria; with a purity

of 99.97 wt.%) [1]. Due to the forging process, the grains have an elongated structure. Taken this elongated structure into account, two types of samples were produced, one with a perpendicular (transversal, T) grain orientation to the loaded surface and one with a parallel (longitudinal, L) orientation. Moreover, the same material in the recrystallized state (R) was investigated. Samples with the dimensions $12 \times 10 \times 5 \text{ mm}^3$ were cut and afterwards polished to a mirror finish to reduce the surface roughness and to obtain a well-defined reference surface structure. The targets were mechanically clamped to an actively cooled sample holder equipped with an ohmic heater. This heater guaranteed that the specimens were heated to a base temperature of 400°C which was measured by a thermocouple. Furthermore, a Nd:YAG laser with a wave length $\lambda = 1064 \text{ nm}$ and an energy of 32 J was used to simulate the transient events with a repetition frequency of 0.5 Hz and a pulse duration of 1 ms . Thereby, the absorbed power densities covered a range of $0.19\text{--}0.64 \text{ GW/m}^2$. For the particle exposure, deuterium plasma with a flux $\sim 6 \cdot 10^{21} \text{ m}^{-2} \text{ s}^{-1}$ and a fluence up to $\sim 1.2 \cdot 10^{25} \text{ m}^{-2}$ (1000 pulses) was used. The particles were accelerated with a bias voltage of -60 V towards the sample. After the termination of the tests, each sample held four exposure spots, where the power density and plasma parameters were retained but the order of the loading sequence and the pulse number were changed. In detail, the first spot was primarily exposed to 100 transient thermal events and subsequently to plasma. On the second spot the loading was simultaneous, while the third and fourth exposure point saw initially the plasma followed by 100 and 1000 thermal pulses, respectively. This method offers the opportunity to simultaneously study the thermal shock behaviour and the influence of hydrogen as well as the order of exposure on the material performance. After the experiments in PSI-2, the induced damages and surface modifications were analysed by light microscopy, scanning electron microscopy (SEM), cross sections and focused ion beam (FIB).

Results and Discussion

Figure 1 illustrates exemplarily SEM images of the loaded surfaces of longitudinal (L) and transversal (T) samples. The analysis of the exposed tungsten targets revealed that there are significant differences in the surface morphology and damage structure in dependence of the loading sequence and the sample type.

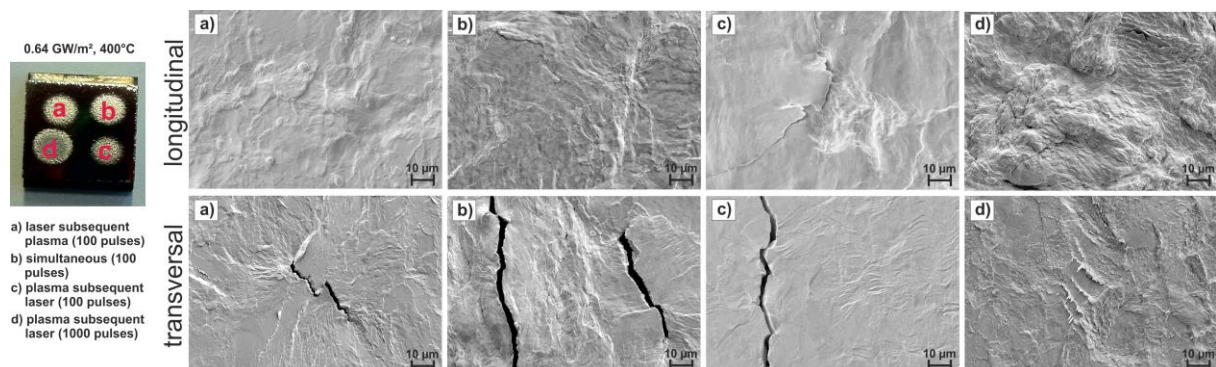


Figure 1: SEM images of the loaded surfaces after sequential exposure (explained on the left side). Samples with a longitudinal grain orientation (top row) showed no crack formation, while transversal (bottom row) and recrystallized samples exhibited cracks for almost all tested sequences and power densities.

The longitudinal samples compensated the induced stresses solely by plastic deformation. This plastic deformation (surface roughness) became more distinct with the increase of the absorbed power density and pulse number. After 1000 pulses, the plastic deformation was high enough to cause a decrease of the thermal contact to

the bulk material which resulted in sporadically molten parts. The molten tungsten solidified on single samples in scale like structures which can be seen on figure 2. These scale like structures were also found on transversal samples. FIB images brought no clear and authoritative evidence that the source of these structures was pressed out material or grains. Moreover, recrystallization was detected after 1000 pulses at 0.64 GW/m² with a depth of 1.5 mm in the entire laser spot area (~ 3 mm).

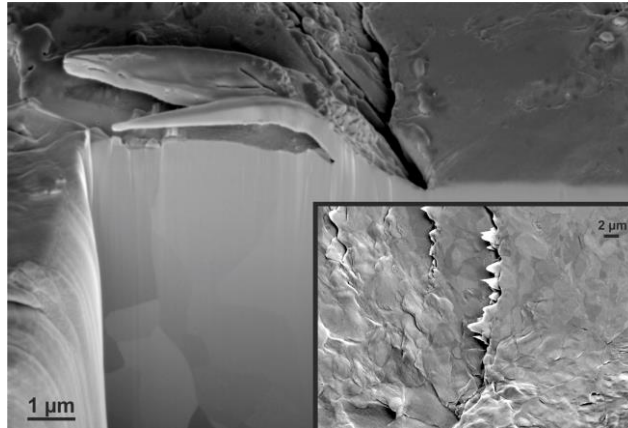


Figure 2 Focused ion beam image of the scale like structures in detail after 1000 pulses at 0.64 GW/m² and a SEM image in the right, bottom corner which shows a larger part of the loaded surface and solidified tungsten.

Furthermore, the particle exposure led to the diffusion of hydrogen into surface near regions where it was detected as blisters. The amount of blisters fluctuated massively according to the loading sequence. This sequence related fluctuation can be explained by the fact that diffusion, retrapping and, rerelease of hydrogen are exponentially dependent on the temperature. Therefore, the highest quantity of blisters was detected when the samples initially were exposed to laser beam and subsequently to plasma. Simultaneous loads and sequences that started with plasma exposure and terminated with transient events exhibited a small number of blisters or no blisters which can be reducible to the temperature rise at any laser pulse (e.g. 0.38 GW/m² → $\Delta T \sim 700^\circ\text{C}$) that increases the mobility of the trapped hydrogen atoms and causes deeper diffusion into the material or degassing.

The analysis of samples with a transversal grain orientation to the loading direction showed besides the increasing surface roughness with rising applied power densities also crack formation. The crack formation was detected for power densities higher 0.19 GW/m² and each sequence. For this reason, the crack width was measured for each, single sequence and compared among them to search out if there were significant differences in dependence of the exposure conditions. Such dependence could not be determined. The revealed average crack widths after tests at 0.38 GW/m² were around 2 μm and after experiments at 0.64 GW/m² around 11 μm . Moreover, the crack depth was investigated via cross sections (Figure 3, right picture) and it became apparent that the cracks propagated up to 495 μm deep into the material depending on the extent of the power density. Regarding this cracking, which was not investigated in [1], it has to be initiated by the trapped hydrogen which causes a distortion in the tungsten lattice structure and therefore, generates stresses. These stresses in addition to the thermal shock stresses declined the cracking threshold and led to enhanced crack formation even at 400° C base temperature.

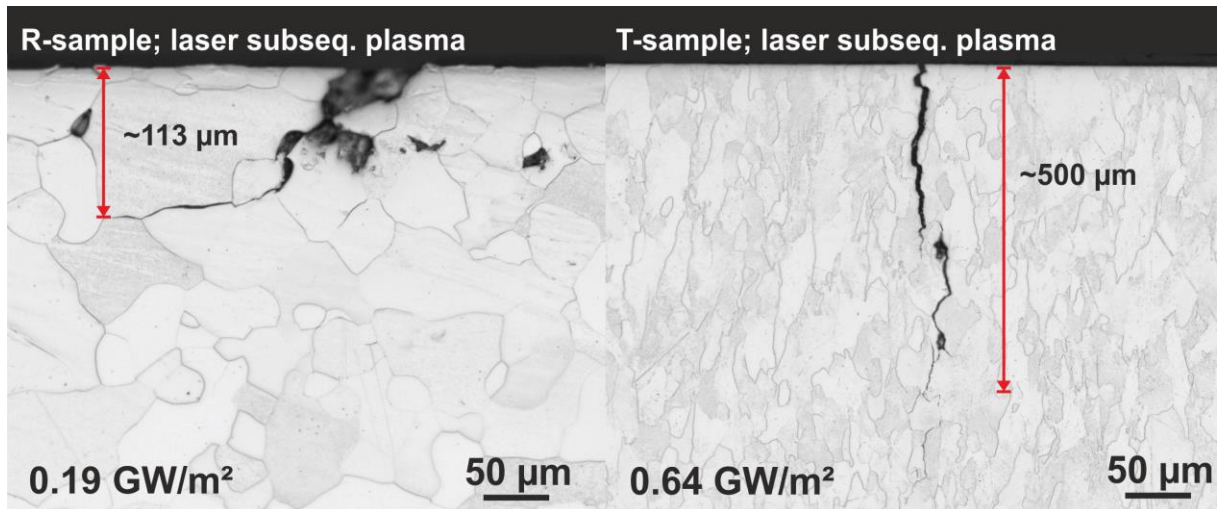


Figure 3 Cross sections of a recrystallised sample (left) and a transversal (right) after exposure. R-samples already cracked at the lowest, tested power density while T-samples started to crack at power densities above 0.19 GW/m².

However, recrystallized samples showed the weakest performance independent of the power density and loading sequence. In detail, besides roughening and swelling, the material cracked at all testing parameters due to the mechanical properties which are influenced by the microstructure. As a result of the recrystallization process, vacancies agglomerate at the grain boundaries which in turn increase the open volume between adjacent grains. In addition, atomic hydrogen is trapped among others by these vacancies and when the accumulated hydrogen forms molecules this leads to additional stresses in the material. For that reason the crack width was measured and resulted in an average value of 2 µm. Accordingly, the analysis of the cross sections showed that the cracks penetrated up to 500 µm deep into the material, both perpendicular and horizontal. Associated with this the cross section preparation was accompanied by a substantial loss of grains which reflects also the weakness of recrystallized tungsten.

Consequently, the thermal shock behaviour is influenced by the hydrogen embrittlement primarily for the T and R samples. R samples showed due to the degradation of mechanical properties even crack formation outside the loaded area and deep inside the bulk material. However, the crack density, width and depth were smaller compared to [1]. L samples were vastly resistant against crack formation which could be attributed to their microstructure.

Conclusion

The presented results exhibit the significant influence of simultaneous and sequential thermal and particle loadings on the thermal shock performance of tungsten. It was clearly recognized that the order of exposure has a substantial impact on the surface modification and damage formation. To be more precise, sequences with deuterium exposure at the end led to the distinctly marked deuterium accumulations in the surface near lattice which could be detected based on a considerable amount of blisters on the sample surface. In addition, simultaneous exposure and sequences that ended with transient events i.e. laser exposure showed less marked or no blisters. This formidably decrease of blisters can be explained with the degassing of trapped deuterium and/or a deeper diffusion into the bulk material.

Furthermore, it was found out that deuterium has an adverse impact on the damage behaviour and exacerbates the performance substantially. This exacerbation was reflected in crack emergence on recrystallized samples and samples with transversal grain orientation, which was additionally accompanied by melting and the formation of scale like structures. This shows conspicuously the embrittlement due to hydrogen accompanied by a shift of the cracking threshold [1]. Especially, the crack formation on tungsten with transversal grain orientation could be critical due to the fact that this is the preferred orientation for applications in ITER. In addition, horizontal crack propagation would massively influences the thermal contact to the bulk material and thus causes melting and in the worst case the loss of whole sample parts. To figure out if the preferential tungsten type may lead to complications and/or premature failure, experiments with higher base temperatures but apart from that with the same test conditions are indispensable. Of course, the test conditions are different to ITER, concerning the base temperature, the missing particle load during the transients and the two orders of magnitude lower particle load for the steady state condition but as mentioned in the introduction experiments that cover all anticipated exposure parameters are inherently difficult to realise

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