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Thermal shock response of deformed and recrystallised tungsten



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HIGHLIGHTS

- Microstructure has a strong influence on the mechanical properties of tungsten.
- Threshold values vary with microstructure by a factor of 2.
- Thermal shock damage patterns depend on the microstructure.
- Cracks parallel to the surface evolve the risk of enhanced erosion and overheating.
- Recrystallisation may lead to enhanced erosion for high pulse numbers.

ARTICLE INFO

Article history: Received 14 September 2012 Received in revised form 8 May 2013 Accepted 21 May 2013 Available online 18 June 2013

PACS: 28.52.-s 28.52.Fa 62.20.-x

65.40.De

Keywords: Tungsten High heat flux tests

Thermal shock Microstructure Recrystallisation

ABSTRACT

The thermal shock response of tungsten as a plasma facing material (PFM) strongly depends on its mechanical properties and consequently on its microstructure. In order to characterise this influence, deformed tungsten, both in its stress relieved and recrystallised condition, was exposed to 100 ELM like thermal shock events in the electron beam facility JUDITH 1. The induced thermal shock damages were analysed by scanning electron microscopy, optical microscopy and laser profilometry. Tensile tests at different temperatures show that the mechanical properties such as fracture strength and strain depend on the grain orientation and microstructure. Transmission electron microscope images of the as received and the recrystallised material show that the defect density of the recrystallised samples is decreased. Threshold values such as damage and cracking threshold vary with microstructure by a factor of 2. Also the induced thermal shock damages and surface modifications are strongly depend on the microstructure. Surface roughening due to plastic deformation is more pronounced in the recrystallised state and crack parameters as well as crack propagation is influenced by grain orientation due to preferential crack formation along grain boundaries.

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1. Introduction

The response of plasma facing materials (PFMs) to thermal shock loads is a major issue for future fusion devices like ITER and DEMO. Especially in the divertor region, the chosen materials have to withstand severe environmental conditions in terms of steady state (up to $10\,\text{MW}\,\text{m}^{-2}$), slow transient (up to $20\,\text{MW}\,\text{m}^{-2}$) and transient heat loads (up to $1\,\text{GW}\,\text{m}^{-2}$ and above). Beside these thermal loads PFMs are also exposed to high particle fluxes of hydrogen, helium and neutron, which will deteriorate the material properties and therefore have an impact on the thermal shock response. Under

these conditions, tungsten is one of the most promising materials for application as PFM especially in the divertor region. Its main advantages are a high melting point, high thermal conductivity, low sputtering yield and low tritium retention. But tungsten has also some drawbacks such as the high atomic number and the brittleness at low temperatures [1–4].

Thermal shock damages induced by ELM like events simulated in the electron beam facility JUDITH 1 (Juelich Divertor Test Facility in Hot Cells) comprise surface modifications and crack formation. How pronounced these damages are, depends not only on the test conditions such as absorbed power density and base temperature but also on the material's thermal and mechanical properties as well as on its microstructure. Both are strongly influenced by the manufacturing process of the material in terms of deformation and heat treatment.

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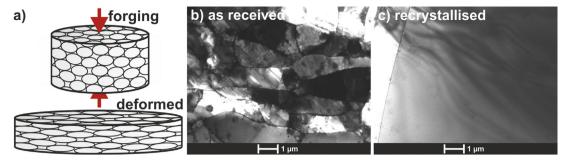


Fig. 1. Microstructure of single forged W-UHP: (a) schematic view of forging process and the resulting grain structure; (b) TEM image of the as received material; (c) TEM image of the recrystallised material.

In order to characterise the influence of a strongly elongated grain structure on the thermal shock response as well as the mechanical properties, deformed, so-called single forged, tungsten was exposed to 100 ELM like cyclic thermal shocks at various base temperatures to address the influence of the brittleness of the material. Additionally, the material was recrystallised and also exposed to the same cyclic thermal shocks to study the influence of recrystallisation, which will take place during the operation of ITER or DEMO.

2. Material properties and experimental settings

The investigated material is W-UHP (ultra high purity tungsten) with a purity of 99.9999 wt% provided by the PLANSEE AG, Austria [5]. The forging of the sintered blank (diameter ca. 80 mm and height ca. 116 mm) was applied in axial direction to increase the density and the mechanical strength of the material and to form round blankets with a diameter of ca. 160 mm and height of 29 mm. Finally, the material was stress relieved for 2 h at 1000 °C. Due to this manufacturing process, the grains are heavily elongated to disc like shape (Fig. 1a). Selected parts of the material were recrystallised at a temperature of 1600 °C for 1 h according to the information given by the manufacturer.

Closer investigation of the material by transmission electron microscopy (TEM) and metallographic means showed that there are significant differences between the as received and recrystallised material. Optical microscope images showed that the grains of the as received tungsten are strongly elongated perpendicular to the forging/deformation direction. After recrystallisation the average grain size is larger (as received: top view (forging direction) $53~\mu m \times 67~\mu m$, cross section (perpendicular to forging direction) $48~\mu m \times 170~\mu m$; recrystallised: top view $76~\mu m \times 126~\mu m$, cross section $74~\mu m \times 147~\mu m$) and the grain structure is more homogeneous. Furthermore, the TEM images in Fig. 1 show that the defects such as dislocations and subgrain boundaries in the recrystallised

material (Fig. 1c) are strongly reduced in comparison to the as received (Fig. 1b) one.

The mechanical properties of W-UHP were characterised by tensile tests with a deformation speed of $0.2 \, \mathrm{mm/min}$ (strain rate ca. $10^{-4} \, \mathrm{s^{-1}}$) at elevated temperatures of $300 \, ^{\circ}\mathrm{C}$, $500 \, ^{\circ}\mathrm{C}$ and $1000 \, ^{\circ}\mathrm{C}$. In order to quantify the influence of the grain orientation on the mechanical strength of the material, tensile test specimens were manufactured with grains orientated parallel (longitudinal) and perpendicular (transversal) to the loading direction. Additionally, tensile tests of recrystallised longitudinal specimens were performed. One sample for each temperature with the respective microstructure was manufactured (9 in total) with the dimensions: length = $26 \, \mathrm{mm}$, width = $8 \, \mathrm{mm}$, gauge thickness = $3 \, \mathrm{mm}$, gauge length = $15 \, \mathrm{mm}$ and a curvature radius of $1.5 \, \mathrm{mm}$. The results of these tests are plotted as engineering stress–strain curves in Fig. 2.

The focus was on the qualitative analysis of the curves and the comparison of different grain structures rather than the exact values of parameters such as the ultimate tensile strength or the fracture strain. A general expected result is that for higher temperatures the strength of the material decreases while the ductility increases. The comparison of all three grain orientations and structures shows that the longitudinal specimens have the highest strength, the recrystallised specimens have the highest fracture strain and the transversal specimens show brittle behaviour even at $1000\,^{\circ}$ C. This pronounced anisotropy of the mechanical properties due to the deformation during the production process (longitudinal and transversal) is known as "texture strengthening". The lower strength but improved ductility of the recrystallised material results from the reduced defect density [6,7].

For the thermal shock tests in JUDITH 1 [8] samples with the dimension $12 \text{ mm} \times 12 \text{ mm} \times 5 \text{ mm}$ were cut for all three grain orientations. All samples were polished to a mirror finish to create an undamaged well-defined starting state. The samples were loaded with ELM relevant power densities between 0.16 and 1.27 GW m⁻². These values were calculated by taking an electron absorption coefficient of 0.46 into account. The exposed area $(4 \text{ mm} \times 4 \text{ mm})$ was

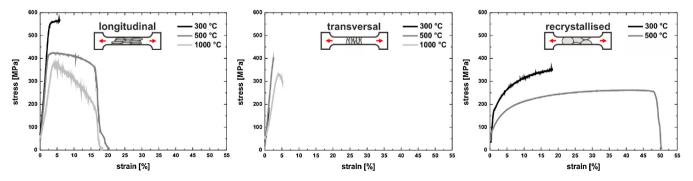


Fig. 2. Stress-strain diagrams for W-UHP at 300 °C, 500 °C and 1000 °C for longitudinal (left), transversal (middle) and recrystallised (right) specimens.

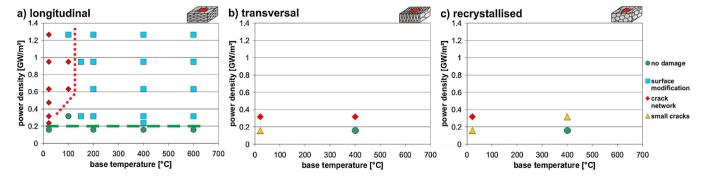


Fig. 3. Thermal shock response of W-UHP with longitudinal (a), transversal (b) and recrystallised grain structure (c) after 100 thermal shock events at different power densities and base temperatures. Damage (dashed line) and cracking (dotted line) threshold are defined for the longitudinal specimens.

scanned with a focused electron beam (diameter of ca. 1 mm) at very high scanning frequencies (47 kHz in x-direction and 43 kHz in y-direction), a single pulse duration of 1 ms and a total number of 100 pulses. The loading frequency was ca. 0.5 Hz to allow a complete cool down after each individual pulse. In addition to tests performed at room temperature (RT), a graphite holder with a tubular heating cartridge was used to achieve base temperatures up to $600\,^{\circ}$ C.

After the exposure in JUDITH 1 the induced damages were investigated by SEM and laser profilometry. Subsequently, the cross sections of the samples were investigated by metallographic means to analyse the crack propagation into the bulk material.

3. Results and discussion

The results of the thermal shock tests in JUDITH 1 and an overview of the induced thermal shock damages for all three grain structures are depicted in Fig. 3. Longitudinal specimens (grains elongated parallel to the loaded surface) are used as reference grain structure due to its highest mechanical strength in comparison to the other two grain structures (Fig. 2). The results in Fig. 3a show that the induced thermal shock damages allow the determination of damage (dashed line in Fig. 3a) and cracking (dotted line in Fig. 3a) threshold values valid for 100 pulses. These threshold values, their location and meaning for this material/grain orientation were discussed in detail in [9].

Based on the obtained result of the damage mapping of the longitudinal grain orientation, the thermal shock test of the transversal (grains elongated perpendicular to the loaded surface) and the recrystallised specimens (heat treated longitudinal specimens) were focused on more specific loading conditions around the damage threshold at 0.16 and 0.32 GW m $^{-2}$ as well as below and above the cracking threshold at RT and 400 °C. These threshold values are also in good agreement with results obtained in previous disruption like single pulse experiments [10]. For both grain structures, transversal (Fig. 3b) and recrystallised (Fig. 3c), the damage threshold decreases below 0.16 GW m $^{-2}$ at least at RT. The cracking threshold also increases for both grain structures above 400 °C. However the transversal specimens show thermal shock crack networks at 400 °C while the recrystallised material only shows small discontinuous cracks.

Fig. 4 gives an overview of the loaded surfaces and the corresponding cross sections for all three grain structures at RT and 0.32 GW m⁻². The thermal shock crack pattern of the longitudinal (Fig. 4a) and recrystallised (Fig. 4c) samples appear to be very similar. Both show an arbitrary crack network within the loaded area. In contrast to that, the thermal shock cracks of the transversal (Fig. 4b) sample clearly follow the grain orientation. The cracks are arranged parallel to each other and are sometimes connected via nearly perpendicular cracks. A comparison of the corresponding

Table 1Maximum and average values of the crack parameters for W-UHP with longitudinal (L), transversal (T) and recrystallised (R) grain orientation/microstructure loaded at RT and $0.32 \, \text{GW m}^{-2}$.

	L	T	R
Max. crack distance (µm)	1740	2989	1612
Avg. crack distance (µm)	481	246	392
Max. crack width (µm)	6	4	35
Avg. crack width (µm)	4	2	23
Max. crack depth (µm)	227	354	447
Avg. crack depth (μm)	156	230	183

cross sections (Fig. 4d-f) shows that longitudinal and recrystallised samples fail similar. The cracks grow perpendicular to the loaded surface into the material and at a certain depth they start to propagate parallel to the surface. This reduces the thermal conductivity significantly and evolves the risk of overheating/melting and enhanced erosion of surface parts. In contrast, there is no parallel crack propagation for the transversal samples. Furthermore, for the transversal and recrystallised samples cracks propagate preferentially intergranular through/into the material, while the cracks for the longitudinal samples show also transgranular crack propagation.

In order to investigate the induced thermal shock damages and their dependence on the microstructure, crack parameters such as crack distance (distance between adjacent cracks), width and depth (Table 1) as well as the arithmetic mean roughness R_a (Table 2) were measured. Major differences between the three grain orientations are a higher crack density (reciprocal crack distance) for the transversal material and the significantly lager crack width (ca. seven times larger) for the recrystallised material as well as its higher roughness at 0.32 GW m⁻² at RT and 400 °C.

The reason for the observed differences in the thermal shock response of W-UHP are the varying mechanical properties depending on the microstructure (Fig. 2). Transversal samples show brittle behaviour even at very high temperatures and low tensile strength in comparison to the longitudinal samples. This weak and brittle mechanical behaviour deteriorates the thermal shock response (in terms of threshold values) in comparison to the longitudinal grain orientation. However, transversal samples show no crack formation

Table 2 Arithmetic mean roughness R_a in μ m for W-UHP with longitudinal (L), transversal (T) and recrystallised (R) grain orientation/microstructure at the corresponding loading conditions.

Grain structure	RT			400°C		
	L	T	R	L	T	R
$\begin{array}{c} 0.16\text{GW}\text{m}^{-2} \\ 0.32\text{GW}\text{m}^{-2} \end{array}$	0.18 0.46	0.24 0.38	0.22 2.22	0.07 0.58	0.19 0.68	0.24 1.14

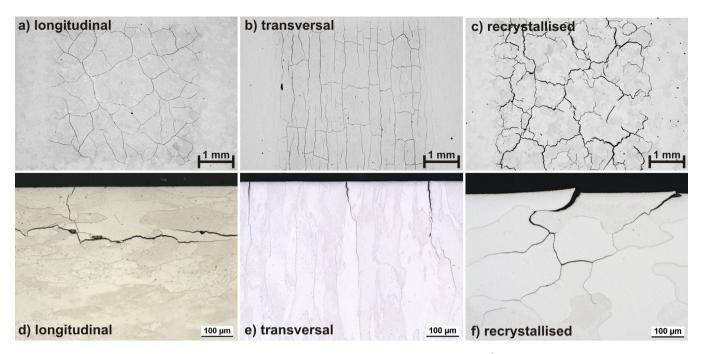


Fig. 4. SEM images of the thermal shock crack networks for all three grain structures (a-c) loaded at RT and 0.32 GW m^{-2} ; light microscopy images of sample cross sections for all three grain structures (d-f) loaded at RT and 0.32 GW m^{-2} .

parallel to the loaded surface, which reduces the risk of overheating and enhanced erosion of whole parts of the surface. Additionally, the high crack density acts a natural castellation of the surface and reduces the thermal stresses after the initial crack formation significantly. This results in comparable arithmetic mean roughness values for the longitudinal and transversal samples although the transversal samples are brittle.

Recrystallised samples have also a lower yield/tensile strength than the corresponding longitudinal, but a much higher elongation. This results in a lower damage threshold and crack formation even at 400 °C. Due to the higher ductility the recrystallised material shows only small discontinuous and arbitrarily distributed cracks at $0.32\,\text{GW}\,\text{m}^{-2}$ and $400\,^{\circ}\text{C}$, in contrast to the transversal samples. The closer investigation of the induced thermal shock damages showed that the crack distances and the surface roughness is significantly higher than for the other two grain orientations. These results are explained by the smaller yield strength and a reduced cohesion between grains. Another effect of the reduced cohesion of the recrystallised material is that cracks propagate only intergranular through the material and not transgranular like for the longitudinal samples. This indicates that it is much easier for the cracks to follow the grain boundaries even if they have to change their propagation direction several times. The reason for the low cohesion is the agglomeration of vacancies at grain boundaries during the recrystallisation process [11].

4. Conclusion

The material parameters of tungsten do not only depend on the material composition but also on the production process and hence on the microstructure. Especially the mechanical properties are strongly influenced by the grain orientation and structure. In order to investigate the impact of these differences on the thermal shock response of tungsten as a PFM, heavily deformed and recrystallised W-UHP samples were exposed to 100 ELM like thermal shock events.

The results show that both, the strongly elongated grain structure and the recrystallisation, influence the thermal shock response of tungsten. Not only threshold values such as damage and cracking

thresholds vary with microstructure but also the induced thermal shock damages and surface modifications depend on it. For the recrystallised state surface roughening due to plastic deformation is more pronounced. Crack parameters as well as crack propagation is strongly influenced by grain orientation due to preferential crack formation along grain boundaries. These changes can be traced back to microstructural and grain structure effects such as the "texture strengthening" in case of the longitudinal/transversal grain orientation or the thermally activated agglomeration of lattice defects at the grain boundaries of the recrystallised material.

Based on these results it can be stated that for none of the tested grain structures severe damage formation, namely cracking and roughening, can be avoided under these very high power densities. Longitudinal samples have the highest damage threshold (valid for 100 pulses) but show cracking parallel to the surface, which may lead to overheating and enhanced erosion. In contrast to that, transversal samples show no parallel cracks but have worse threshold values and very high crack densities, which could also lead to enhanced erosion especially for very high pulse numbers [12]. Recrystallisation of the material, which will take place during the steady state loading, will increase the erosion of complete grains and could therefore lead to an intolerable contamination of the plasma.

Acknowledgements

The authors would like to thank Dr. E. Wessel and V. Gutzeit for their kind assistance with the SEM/LM pictures. This work, supported by the European Communities under the contract of Association between EURATOM/Forschungszentrum Jülich, was carried out within the framework of the European Fusion Development Agreement. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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