Recrystallization behaviour of high-flux hydrogen plasma exposed tungsten

Citation for published version (APA):

Document license:
CC BY

DOI:
10.1016/j.jnucmat.2020.152748

Document status and date:
Published: 01/03/2021

Document Version:
Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

• A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
• The final author version and the galley proof are versions of the publication after peer review.
• The final published version features the final layout of the paper including the volume, issue and page numbers.

Link to publication

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

• Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
• You may not further distribute the material or use it for any profit-making activity or commercial gain
• You may freely distribute the URL identifying the publication in the public portal.

If the publication is distributed under the terms of Article 25fa of the Dutch Copyright Act, indicated by the “Taverne” license above, please follow below link for the End User Agreement:
www.tue.nl/taverne

Take down policy
If you believe that this document breaches copyright please contact us at:
openaccess@tue.nl
providing details and we will investigate your claim.
Recrystallization behaviour of high-flux hydrogen plasma exposed tungsten

V. Shah a, J.T.S. Beune a, Y. Li a,b, Th. Loewenhoff c, M. Wirtz c, T.W. Morgan b, J.A.W. van Dommelen a,#

a Department of Mechanical Engineering, Eindhoven University of Technology, PO Box 513, Eindhoven, 5600 MB, the Netherlands
b DIFFER - Dutch Institute for Fundamental Energy Research, De Zaale 20, Eindhoven, 5612 AJ, the Netherlands
c Forschungszentrum Jülich, Institut für Energie- und Klimaforschung, Jülich 52425, Germany

A R T I C L E   I N F O

Article history:
Received 22 October 2020
Revised 26 November 2020
Accepted 12 December 2020
Available online 17 December 2020

Keywords:
Tungsten
Hydrogen
Plasma exposure
Hardness
Recrystallization kinetics

A B S T R A C T

Knowledge of a material’s thermal stability under extreme synergistic particle and heat loads is crucial for developing high performance reactor materials. In this work, the recrystallization behaviour of tungsten under the influence of hydrogen is investigated by low energy high flux hydrogen plasma exposure for various lengths of time. The microstructural changes following exposure are probed by micro-indentation, electron back-scatter diffraction measurements and the characteristic time for recrystallization is assessed using the Johnson–Mehl–Avrami–Kolmogorov (JMAK) model. A recrystallization activation energy in the range of 425 to 440 kJ mol⁻¹ is determined, identical to that of oven annealed samples, thereby indicating an insignificant influence of hydrogen plasma on the recrystallization kinetics of tungsten.

© 2020 The Author(s). Published by Elsevier B.V. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/)

1. Introduction

Over the past decade, the refractory metal Tungsten (W) has been identified as the leading candidate material for the plasma facing components (PFCs) for the divertor of future fusion reactors due to its favourable high temperature properties. The lifetime of these W PFCs is crucial for the efficiency, economics and good performance of the reactor as the divertor performs the vital function of extracting heat, helium and other impurities from the plasma [1,2]. The PFCs are subjected to intense particle fluxes of fast neutrons, low energy hydrogen (H) and helium (He) ions and radiation, leading to high heat loads (~10 to 20 MWm⁻² along with high energy transients) [3,4]. Fundamentally speaking, the prolonged exposure of metals at high temperatures assists in microstructural restoration by annihilation of defects, nucleation and the growth of new defect free grains to restore the equilibrium state; a process commonly termed as recrystallization and grain growth [5]. Under the expected loading conditions in the ITER tokamak, the recrystallization of at least top few mm of the ITER bulk-W PFCs is anticipated [6]. Recrystallization of W during high heat flux loading has been observed experimentally by several studies following laser, electron, as well as plasma exposure [7–10]. The recrystallization of bulk W has been surmised as detrimental for the lifetime of the PFCs due to a loss of strength and hardness, and an increase in the brittle-to-ductile transition temperature (BDTT), consequently promoting crack formation and adversely affecting the thermal fatigue resistance of the PFCs [11–13]. Since, the “ITER grade” W is defined within a narrow range of parameters produced by heavy deformation, the occurrence of recrystallization also implies the loss of the designed benefits of the purposely engineered microstructure of W. However, it should be noted that the picture is not fully complete, as there have been studies depicting an increase in the total elongation following recrystallization as compared to deformed state (similar testing temperature), implying a decrease in BDTT [11,14,15].

Besides the heat loads, the influence of particles loads, i.e. neutrons and plasma ions, on the kinetics of the recrystallization process at the microstructural scale are not well defined. Primarily, the expected consequence of neutron irradiation in W PFCs is an overall increase in the internal stored energy because of the high density of defects created (vacancies/ self interstitial clusters, dislocation loops as well as network dislocations), thereby leading to an increase in the recrystallization rate as compared to the thermal only case [16–18]. Also, the synergistic interaction of the neutron induced defects with plasma species, specifically the He atoms, will assist in the formation of gas filled voids, frequently termed

---

# Corresponding author.
E-mail address: J.A.W.vanDommelen@tue.nl (J.A.W. van Dommelen).

https://doi.org/10.1016/j.jnucmat.2020.152748
0022-3115/© 2020 The Author(s). Published by Elsevier B.V. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/)
He bubbles [19]. In general, the effect of bubbles on the kinetics of the recrystallization process have been well known [20]. For example, doping of W with potassium (K) has been practised for decades to retard recrystallization by producing K bubbles, eventually resulting in microstructural strengthening [21,22]. A similar effect of retarding recrystallization by He bubbles has been reported under high energy He ion irradiation of W [23–25]. Mechanistically, the impediment of recrystallization in the presence of bubbles is similar to the effect of second phase particles (precipitates), with the bubbles/particles exerting a drag force (Zener drag) on the moving grain boundaries during recrystallization [5]. The magnitude of the drag force, in the case of bubbles, is governed by the size of the bubble, showing a further dependency on the irradiation/annealing temperature [25]. In the case of H atoms, the identical conclusion on the role of gas atoms has been drawn by Loewenhoff et al. [26], where recrystallization of W was suppressed during H plasma exposure regardless of the high temperatures. Contrarily, the presence of dissolved H atoms in Palladium (Pd) and Vanadium (V) has been reported to accelerate the kinetics of the recrystallization and grain growth process by lowering the formation energy of vacancies along with a change in mobility of dislocations and other defects [27,28]. Thus, given the inconsistent conclusions from these studies, the fundamental question on the role of H atoms as expander or inhibitor of the recrystallization process ensues and specifically for the fusion reactor case, understanding its influence is critical for obtaining accurate lifetime estimates of the W PFCs. The present work addresses this question by using the unique high-flux linear plasma facility Magnum-PSI to perform synergistic high heat and H particle exposure of W for varying time intervals at temperature ranges foreseen in PFCs. This is followed by a systematic post-mortem analysis to determine the recrystallization kinetics and compared with literature studies.

2. Experimental

2.1. Test samples

A tungsten bar, manufactured by Plansee using sintering and bi-directional forging was used as the source for the samples. These top hat shaped samples had an upper surface of 30 × 30 mm and a thickness of 10 mm (Fig. 1), and were produced at Forschungszentrum Jülich using electro-discharge machining. The machined samples had elongated grains with an average aspect ratio of 4.5, oriented perpendicular to the plasma exposure plane, while the average grain size (equivalent circular diameter) was approximately 21 μm. The bi-directional forging based manufacturing of the material induced a strong (110) type fibre texture (pre-dominantly α fibre, see Appendix A), alike to a wire drawing process [29].

2.2. Magnum PSI: hydrogen plasma exposure

The H plasma exposure was carried out in a continuous mode using the linear plasma device Magnum-PSI [30]. This is equipped with superconducting magnets and capable of generating continuous ion fluxes of the order 10^{24} to 10^{25} m^{-2} s^{-1} as anticipated in future fusion reactors [31,32]. For the investigation, a total of 5 samples were employed, with exposure times varying from 10 min to 6 hr. The plasma conditions were monitored using Thomson scattering (TS) and gave electron temperatures (T_e) 2 to 3 eV and electron densities (n_e) of 10^{20} to 10^{21} m^{-3}. During exposure, the machine settings were constantly monitored to maintain a peak surface temperature of 1500 °C. The targets were exposed at floating potential and following sheath acceleration, this led to a maximum ion energy of 14 eV at the target surface, corresponding to a projected implantation range of approximately 1 nm. This energy is well below the displacement threshold energy (85 to 90 eV [33]), thereby assuring no generation of vacancies. The plasma beam had a Gaussian particle and heat flux distribution with a full width half maximum (FWHM) of approximately 12 ± 1 mm, resulting in a non-uniform temperature distribution radially over the exposed surface as shown in Fig. 1b. The sample surface temperature at the sample centre was continuously tracked using a multi-wavelength pyrometer (FAR Associates® FMPI) with a spot size of ≈ 3 mm. Additionally, the entire surface was monitored every 8 min via a fast infrared (IR) camera (FLIR SC7500MB). Here, due to the extreme length of some of the exposures, continuous recording was not possible. The sample temperature was also continuously tracked via an N-type thermocouple mounted in a hole machined at the bottom of the sample (Fig. 1). This gave values between 600 °C and 800 °C for all samples, with variations due to differences in clamping of the target samples to the water cooling system.

2.3. Post exposure characterization

Post plasma exposure, insights into the microstructural changes due to recrystallization were probed by performing micro-indentations, as recrystallization is characterized by a distinct reduction in hardness. The micro-indentation measurements were performed using a CSM micro-indenter with an indentation load of 4.9 N (HV0.5). For better statistics and to scrutinize the effect of the non-uniform temperature distribution on the microstructure evolution of the exposed surface, the indentations were performed in a mapping mode with a minimum step size of 200 μm. For selected samples, the indentation measurements were complemented with electron back-scatter diffraction (EBSD) mapping, thereby allowing to correlate the indentations with the microstructural state. The EBSD based mapping along the plasma exposed surface was performed using a TESCAN-MIRA SEM with an acceler-

![Fig. 1](image-url) Schematic showing the geometry of the samples employed for the H plasma exposure (a) front view (b) isometric view (not to scale). All the given dimensions are in millimetres. The particle flux in the beam has a Gaussian like distribution (indicated in (a)) with a FWHM of 12 ± 1 mm, leading to a non-uniform temperature distribution along the exposed surface as shown in (b).
ation voltage of 20 kV and a step size of 0.25 to 3.5 μm, depending on the microstructural state.

3. Results and discussion

The temperature distribution along the exposure plane obtained from the IR recordings for varying exposure times are shown as 2D maps in Fig. 2a. An exception is the 6 hr exposure, where the temperature map was estimated based on the plasma beam parameters (FWHM = 13 mm determined using Thompson scattering) and constrained by the pyrometer data due to erroneous IR recording arising from copper deposition from the source and related contamination of the IR measurement. The influence of copper deposition on the hardness measurements is expected to be negligible, as the deposited copper layer has a thickness of not more than few nanometres. As seen in the temperature maps (Fig. 2a), small variations in the alignment of the plasma during exposure between samples manifests as a shift in the position of the hot zone from the centre of the sample. Additionally, variations in temperature gradient along the exposure plane, i.e. away from the beam spot and towards the sample edge can be observed between different exposures (for example: 10 min vs. 3 hr). This can be attributed to variation in the heat transfer capacity of the clamping between the exposures.

Fig. 2b shows the exposure time dependent hardness (HV) maps acquired from the micro-indentation measurements. At a first glance, independent of the exposure time, significantly lower HV values can be observed in the areas close to the centre of the sample, while the area with low HV values increases with increasing time (for example: between 1 hr and 3 hr). It is worthwhile to note that for lower exposure time (10 min to 1 hr), the above statement does not hold entirely. On further close comparison of the temperature and the HV maps (Fig. 2a and Fig. 2b), a distinctive relationship between the high temperature and low HV value (vice versa) regions can be realized. Thereby, clearly depicting the recrystallization-induced softening due to microstructural changes during exposure. Characteristically, all the hardness maps dominantly display sharp transitions between the low and high HV value zones. These transition regions correspond to the relatively lower recovered/partially recrystallized fractions as higher temperatures favour recrystallization. Fig. 2c shows a large scale EBSD based mapping of the recrystallized area following 1 hr exposure in terms of a inverse pole figure (Z-IPF) map. The recrystallized microstructure (dominantly with preferred orientation [101], green coloured) clearly follows the radial temperature profile, with the recrystallization extent radially extending up to ≈ 4.4 mm along the exposure plane from the hot-spot centre, conforming to the radii of low HV zone in Fig. 2b (black box in 1 hr HV map). To fur-
ther ascertain the recrystallized state in Fig. 2c, Fig. 2d presents the grain scale misorientation analysis in terms of a grain orientation spread (GOS) map. The majority of grains (99.93% area fraction) tend to have an orientation spread below 3°, which is typically the threshold value for discriminating recrystallized grains especially in W [9,15].

The HV values extracted from the varying time dependent HV maps (Fig. 2b) and correlated with the corresponding temperature distribution (Fig. 2a) along the exposure plane are shown in Fig. 3a. As highlighted before, the temperature dependent hardness profile depicts a two stage behaviour, where small drops in HV values at low temperatures occur pre-dominantly due to recovery, while large drops in HV occur at high temperatures pre-dominantly due to recrystallization. It is important to point out that for some hardness measurements, the deviations tend to be large (for example, high HV values for 1 hr exposure). Nonetheless, the temperature dependent HV trend for all exposure times displays an identical and consistent behaviour. The temperature dependent parametric functions determined from the varying exposure time data sets (Fig. 3a) are further shown as black lines in Fig. 3a. Here, the parametric function represents an error type function having the form HV = 0.5 (1 − erf (T − a)/b)c + d, with HV denoting the hardness and T denoting the temperature; while a, b, c and d are constants determined by non-linear least square regression procedure. The scattering of hardness data at lower temperatures prevents a definite analysis of the kinetics in terms of the combined recovery and recrystallization processes. Thus, the recovery kinetics are not treated explicitly in the following analysis.

For assessing the recrystallization kinetics during exposure, the parameter $t_{\text{half}}$, representing the time scale for half hardness-loss (or half recrystallization), is introduced. This parameter defines the thermal stability of the deformed microstructure, and is obtained from the parametric functions (black line) shown in Fig. 3a by determining the temperature at which half hardness-loss occurs for a given exposure time. Table 1 underlines the temperature evolution of $t_{\text{half}}$, and an apparent drop of nearly 150°C can be seen between 10 min (1400 °C) and 6 hr (1250 °C) exposure, implying a reduced thermal stability at longer exposure times.

To assess the recrystallization kinetics over a wide temperature range, the temperature dependent recrystallized fraction $(X)$ for a given exposure time are determined from the mean HV values (Fig. 3a). Next, the modified Johnson-Mehl-Avrami-Kolmogorov (JMAK) model having the form $X = 1 - \exp (-b^n(t - t_{\text{inc}})^p)$ is used to determine the kinetics [5]. Here, the coefficient $b$ represents the thermal activation, the Avrami exponent $n$ describes the nucleation mechanism including the growth dimensionality, $t_{\text{inc}}$ represents the incubation time before start of recrystallization, and they are determined by a non-linear least square fitting procedure. The temporal evolution of the recrystallized fraction determined from the above model for different temperatures is shown in Fig. 3b (solid lines). For illustration purpose, the data set of recrystallized fraction at 1350 °C, evaluated from hardness for different expo-

![Fig. 3. (a) The evolution of hardness with respect to temperature for different exposure time and their corresponding parametric functions (black line) used for determining the time dependent temperature at which half-hardness loss occurs ($t_{\text{half}}$). (b) The temperature dependent temporal evolution of the recrystallized fraction. The solid lines represent the evolution at different temperatures based on the JMAK model, while the circular markers illustrate the recrystallized fraction data determined from hardness following different exposure times at 1350 °C.](image-url)

**Table 1**

<table>
<thead>
<tr>
<th>$T$ [°C]</th>
<th>$t_{\text{half}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1399 ± 9.4</td>
<td>10 min</td>
</tr>
<tr>
<td>1371 ± 3.8</td>
<td>30 min</td>
</tr>
<tr>
<td>1366 ± 4.8</td>
<td>1 hr</td>
</tr>
<tr>
<td>1255 ± 3.3</td>
<td>3 hr</td>
</tr>
<tr>
<td>1247 ± 8.6</td>
<td>6 hr</td>
</tr>
</tbody>
</table>
sure times is also plotted in Fig. 3b (circular markers), while the time for half recrystallization $t_{1/2}$ determined from these curves (Fig. 3b) are provided in Table 2. For the accounted temperatures in Fig. 3b, the coefficients $b$ and $t_{1/2}$ tend to be strongly temperature-dependent. At the same time, the Avrami exponent $n$ displays a weak temperature dependence, with an average value of 1.4 (for more details, refer to Appendix C). This value is within the range 0.78 to 2, reported by Alfonso et al. [34,35] and Wang et al. [36] for warm-rolled tungsten with varying deformation levels. Fundamentally, the characteristic time for the recrystallization process and the temperature are known to follow an Arrhenius-type relation [5], thereby allowing a measure of activation energy to be obtained. This is demonstrated in Fig. 4, where the aforementioned parameter $t_{1/2}$ is plotted. For obtaining precise insights on the role of H atoms on the kinetics, the data set from the work of Alfonso et al. [35] corresponding to oven annealed samples (1100 °C to 1250 °C) is used for comparison, extrapolated linearly to higher temperatures and is shown in Fig. 4. The $t_{1/2}$ (blue square and circular markers) data points in Fig. 4 show similar behaviour as the reference linear fit for the temperature range 1150 °C to 1350 °C with some scatter, while for higher temperatures, i.e. > 1400°C, the deviation from the reference tends to be higher. The error bars in the $t_{1/2}$ data points account for the maximum temperature error of the IR camera. The scatter of the $t_{1/2}$ could be due to the heterogeneous annealing approach used in the present work as compared to oven annealing. Additionally, using the slope of a linear fit of $t_{1/2}$ (not shown in Fig. 4), an activation energy of 433 kJ mol$^{-1}$ (± 2%) is obtained. This activation energy is comparatively higher than 352 kJ mol$^{-1}$ (± 4%) for a highly deformed warm-rolled W plate (90%), as reported by Alfonso et al. [35]. Moreover, Alfonso et al. [34] have also reported an activation energy of approximately 579 kJ mol$^{-1}$ for moderately deformed warm-rolled W plate (67%), much higher than the 90% rolled plate. The difference in the activation energies between the two plates has been further reasoned based on the microstructural features, specifically the low-angle grain boundary spacing (LAGB), i.e. with increasing degree of deformation (67% to 90%) the LAGB spacing reduces (2 μm to 0.67 μm). However, in the present case the relation between the lowering of activation energy with decreasing LAGB spacing does not hold true. The LAGB spacing in the present work is approximately 2.75 μm (see Appendix A), marginally higher than the moderately deformed W plate. This discrepancy can possibly understood as due to difference between the forming process, i.e. bi-directional forging versus rolling, and the consequent variance in the defect density. Nonetheless, the determined activation energy tends to be within the 377 to 460 kJ mol$^{-1}$ range for the grain boundary diffusion, and is much lower than that of 502 to 586 kJ mol$^{-1}$ for bulk diffusion [37]. This therefore implies that the recrystallization kinetics tend to proceed pre-dominantly by grain boundary diffusion.

Elaborating on the role of H for recrystallization kinetics, one could expect that retardation or acceleration of recrystallization due to H would dramatically influence the activation energy. For example, the diffusion of Ar atoms and presence of dissolved Ar atoms in W has been shown to delay the onset of recrystallization, ultimately resulting in a higher activation energy as compared to vacuum annealing [38]. Also, considering the case where the H atoms form complexes and retard recrystallization by pinning the grain boundaries (similar to second phase precipitates), the activation energy would increase, as demonstrated by Zan et al. [39] for oxygen reinforced W. On the other hand, for the case where H atoms tend to favour excess vacancy concentration by lowering their formation energy [27,28], the activation energy would decrease in comparison to the pristine state, as excess vacancies accelerate recrystallization [18,40]. However, following the current kinetic analysis and the observation of activation energy representative of grain boundary diffusion in the present work, identical to the work of Alfonso et al. [35], it can be concluded that low energy H plasma exposure of W has an inconsequential effect in accelerating or retarding the recrystallization process. One of the plausible explanations for this behaviour can be the combination of low implantation depth of H atoms and relatively high temperatures during exposure, eventually resulting in high desorption rates as well as limited long range bulk diffusion, and thus an overall lower H bulk concentration. Additionally, since high temperatures prevent trapping events between the H atoms and lattice defects (high energy traps such as dislocations have a de-trapping energy of 1.25 eV [41]), pre-dominantly all the H atoms would exist as freely diffusing interstitial atoms, consequently contributing marginally to elastic stresses in the lattice. In a nutshell, the observation of unaltered recrystallization kinetics despite long term heat and H plasma exposure could be due to the lower bulk retention of H atoms and the resulting weak interaction with the lattice defects. This conclusion is further supported by observations of negligible deuterium (D) concentration in bulk W following nuclear reaction analysis (NRA) of high flux, high fluence D exposure of W monoblocks performed by Morgan et al. [42]. Nevertheless, further investigations specifically addressing the oven annealing behaviour of samples (like in the present work) will allow to decouple and provide additional insights into the effects of microstructure and H plasma. Moreover, distinguishing between the role of different impurities, i.e. H, He, and beryllium (Be), the major impact on the rate of recrystallization is expected due to the He as compared to H, as it has a stronger binding tendency with lattice defects (predominantly promoting the formation of He bubbles). On the other hand, the role of Be is unclear, hence difficult to ascertain and demands further investigation.

### Table 2

<table>
<thead>
<tr>
<th>T [°C]</th>
<th>$t_{1/2}$ [hr]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1450</td>
<td>5.3</td>
</tr>
<tr>
<td>1350</td>
<td>1.18</td>
</tr>
<tr>
<td>1300</td>
<td>1.87</td>
</tr>
<tr>
<td>1250</td>
<td>6.05</td>
</tr>
<tr>
<td>1200</td>
<td>22.61</td>
</tr>
</tbody>
</table>

Fig. 4. Arrhenius plot depicting the time for half-hardness loss/half recrystallization ($t_{1/2}$) determined from indentation measurements following H plasma exposure, in addition to data points from oven annealed (no H exposure) W samples [35]. The blue coloured square markers represent the time for half-hardness loss from Table 1, while the circular markers represent the time for half-recrystallization obtained from Table 2. The activation energy determined for H exposed samples (blue markers) is approximately 433 kJ mol$^{-1}$, which is higher than for the oven annealed non-exposed samples (approximately 352 kJ mol$^{-1}$, red markers), but within the range 377 to 460 kJ mol$^{-1}$ for grain boundary diffusion. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
4. Conclusion

Summarising, the present work dealt with investigating the recrystallization kinetics of W samples exposed to H plasma for varying time. The characteristic time of recrystallization process, i.e. for half-hardness loss or half-recrystallized fraction (t_{half}) revealed activation energy similar to that of the oven annealed samples in the literature, with the recrystallization operating mechanism being grain boundary diffusion. This agreement on activation energy between the present work and literature indicates that under low energy H plasma exposure, the influence of H atoms on the recrystallization kinetics of W is not significant. Future developments specifically addressing the synergistic influence of bulk displacement damage and H plasma exposure would allow a better understanding of the interplay between H atom and lattice defects on the microstructure evolution, eventually allowing more accurate and realistic predictions for future fusion reactors.

Data availability

The raw or analysed data reported in this work are available upon a reasonable request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

V. Shah: Conceptualization, Methodology, Visualization, Writing - original draft. J.T.S. Beune: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Visualization, Writing - review & editing. Y. Li: Conceptualization, Methodology, Visualization, Writing - review & editing. Th. Loewenhoff: Investigation, Methodology, Writing - review & editing. M. Wirtz: Investigation, Methodology, Writing - review & editing. T.W. Morgan: Conceptualization, Funding acquisition, Methodology, Project administration, Supervision, Writing - review & editing. J.A.W. van Dommelelen: Conceptualization, Funding acquisition, Methodology, Project administration, Supervision, Writing - review & editing.

Acknowledgements

DIFFER is part of the institutes organization of NWO. We acknowledge the support of the Magnum-PSI Facility Team at DIFFER. The Magnum-PSI facility at DIFFER has been funded by the Netherlands Organisation for Scientific Research (NWO.) and EURATOM. This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014–2018 and 2019–2020 under grant agreement no. 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission. Additionally, this research was carried out under project number T16010c in the framework of the Research Program of the Materials innovation institute (M2i) (http://www.m2i.nl) supported by the Dutch government.

Appendix A. Microstructure characterization: pre-exposure (deformed) state

Fig. 5 schematically shows the EBSD characterization of the microstructure before H plasma exposure (deformed) state. The Y-direction inverse pole figure (Y-IPF) map (Fig. 5a) clearly depicts

---

**Fig. 5.** EBSD based characterization of microstructure in pre-exposed (deformed) state (a)Y-direction inverse pole figure (IPF) map (b) grain boundary map (c) Inverse pole figure (d) Orientation distribution function (in Euler space), with $\phi_2$ sectioned at 45°. The low angle grain boundary spacing was determined from the intercepts between a chord and the boundaries with misorientation less than 15° as shown in (b). For statistics, 50 chords were laid over the grain boundary map, with the average low angle grain boundary spacing being approximately equal to 2.75 μm. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
the bi-directional forging induced elongation of grains with an average aspect ratio of 4.5. The microstructure clearly shows a high density of low angle grain boundaries (LAGB) due to deformation (Fig. 3b), with an average LAGB spacing of 2.75 μm. The LAGB spacing was calculated by counting the intercepts with LAGBs along a given line (shown in the magnified image Fig. 5b). Pre-dominantly α fibre texture was observed (Fig. 5c) with relatively high intensities for {001}(110) texture component, followed by {112}(110) and {111}(110) texture components (Fig. 5d).

Appendix B. Microstructure characterization: H plasma 3 hr exposure

The recrystallized region near the centre of the sample following 3 hr H plasma exposure is shown in terms of Z-direction (depth) inverse pole figure (Z-IPF) map in Fig. 6a. This region corresponds to the black box indicated in the 3 hr HV map in Fig. 2b. To further ascertain the recrystallized state, the grain orientation spread (GOS) map of the corresponding microstructure is shown in Fig. 6b, and as seen in the map, most of the grains tend to be below the recrystallization threshold orientation spread value of 3°. Thereby, confirming the low HV values observed in the corresponding region of the 3 hr exposure HV map (Fig. 2b).

Appendix C. Parametrization: JMAK model

Table 3 summarizes the temperature dependent coefficients of the JMAK model determined using non-linear least square regression analysis. Note that only temperatures ≥ 1200°C are considered here. For lower temperatures, accurate parametrization of the JMAK model could not be obtained, as the kinetics of recrystallization are slow, and hence experiments with longer exposure time than considered in the present work are required.

Table 3

<table>
<thead>
<tr>
<th>T [°C]</th>
<th>b</th>
<th>n</th>
<th>τec (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1200</td>
<td>1.02 × 10⁻⁵</td>
<td>1.3</td>
<td>500</td>
</tr>
<tr>
<td>1250</td>
<td>3.85 × 10⁻⁵</td>
<td>1.2</td>
<td>138</td>
</tr>
<tr>
<td>1300</td>
<td>1.24 × 10⁻⁴</td>
<td>1.5</td>
<td>0.76</td>
</tr>
<tr>
<td>1350</td>
<td>1.95 × 10⁻⁴</td>
<td>1.4</td>
<td>1.2 × 10⁻⁶</td>
</tr>
<tr>
<td>1450</td>
<td>2.54 × 10⁻³</td>
<td>1.61</td>
<td>0.95</td>
</tr>
</tbody>
</table>

References


