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Experimental investigation of the microstructural changes of tungsten monoblocks exposed to pulsed high heat loads


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ABSTRACT

Extending the lifetime of tungsten based plasma facing components for future fusion reactors remains an everlasting challenge. In this work, the microstructure of tungsten monoblocks exposed to a few thousand cycles of combined pulsed heat loads of 10 and 20 MWm⁻² (achieved via an electron beam) is thoroughly investigated. The heat exposure assisted surface roughening is observed to be significant. Build-up of thermal stresses in the monoblock results in severe geometrical distortions. The evolution of the microstructure of the tungsten monoblocks due to recrystallization is found to be substantial in the top 5.5 mm from the surface, and the relation between the recrystallization threshold and temperatures throughout the monoblock is investigated. Additionally, no traces of recrystallization-induced crack formation within the monoblock is observed. The recrystallization-induced microstructural evolution is investigated in terms of grain size, grain boundary distribution, and the recrystallization induced softening as determined from micro-hardness measurements. An adequate quantitative match between the changes in the microstructural features such as grain size, grain boundary character, and the related hardness is obtained. Moreover, the depth dependent microstructural recrystallized fraction in the monoblock is examined via hardness and EBSD measurements, and a comparison between the different methods is presented. The presence of a preferred crystal orientation of the recrystallized grains is observed and discussed in terms of the initial texture.

1. Introduction

The divertor component in magnetic confinement based tokamak reactors is often termed as the most critical component dictating the lifetime of the reactor [1]. Primarily, the divertor consists of plasma facing components (PFCs), which perform the vital functions of extracting heat from the burning plasma, and prevent the dilution of the burning plasma by capturing helium ions [2]. The refractory metal tungsten (W) is the main candidate material for PFCs due to its excellent high temperature properties, i.e. high melting temperature, high thermal conductivity and low swelling coefficient [3,4].

Considering the ITER application, the design of the tungsten based PFCs is based on a monoblock geometry brazed to an actively water cooled CuCrZr alloy based heat sink [5]. During normal operation, the tungsten monoblocks will be exposed to a combination of extreme particle loads, i.e. fast neutrons (14 MeV, φ = 10¹⁸ neutrons m⁻² s⁻¹) and gas atoms (H and He, φ = 10²⁴ atoms m⁻² s⁻¹) [6,7]. The fast neutrons will predominantly lead to lattice damage by generating defects as well as compositional changes by transmutation. Furthermore, the temperature driven diffusion and trapping of gas atoms at neutron induced defects will ultimately result into a myriad of microstructural and property changes, extending from the surface (e.g. micro-crack and blister formation [8–15], pinhole and bubble formation [16–20], nanotendril formation [21–24] to the monoblock bulk (e.g. irradiation hardening [25–28], thermal conductivity changes [28–30], gas atom retention [31–35]). The performance of tungsten under neutron (including ions as surrogate), plasma and synergistic neutron-plasma loads has been extensively studied in the past two decades, and a comprehensive review of the modelling and experimental efforts at different material length and time scales can be availed from the works of Hasegawa et al. [36], Marian et al. [37], Harrison [38] and Reith et al. [39].

Besides the particle loads, the thermal shock and thermal fatigue based performance of the monoblocks under severe repetitive heat loads is a major concern and termed as one of the limiting factors for the lifetime of PFCs. For steady state operations, the monoblocks will endure a pulsed heat load of 10 MWm⁻², resulting in high temperatures (≤ 1300°C) at the surface and a steep temperature gradient through the monoblock [4,5]. The base operating temperatures in the monoblock are determined to prevent premature failure of tungsten due to its
brittle-ductile transition at lower temperatures and microstructural changes by recrystallization at higher temperatures [40]. In addition to the steady state heat loads, the monoblocks will also have to withstand slow transients of about 20 MWm$^{-2}$ ($\leq 10$ s) and fast transients (also known as off-normal events) such as edge localized modes (ELMs), vertical displacement events (VDEs) and run-away electrons depositing high energy density ($1-60$ MJm$^{-2}$) within a very short time (0.1–300 ms), thereby resulting in an additional temperature upsurge on top of the operating surface base temperature [41,42].

The behaviour of tungsten (a monoblock geometry and other geometries) under such thermal loads has been studied predominantly with electron beams [43–45], laser devices [44,46] and pulsed plasmas [44,47] by several research groups, revealing surface damage phenomena such as macro-cracking, erosion, roughening due to plastic deformation, melting, and droplet formation [48–54]. Moreover, the observed cracking phenomenon has been attributed to the presence of residual stresses following the repetitive heating and cooling cycles. Depending on the microstructural state or temperature, these residual stresses can induce plastic deformation assisted roughening of the surface, thereby assisting in the crack initiation process and crack growth during the repeated loadings [55,56]. The strong dependence of the damage threshold on the grain size, grain orientation, processing/manufacturing route as well as the base surface temperature has been represented in terms of damage maps [49,51,56]. Along with the high heat load assisted surface modifications, microstructural changes due to recrystallization and grain growth in the sub-surface regimes have also been observed experimentally. The occurrence of recrystallization has been further associated with brittle failure due to a reduction of the grain boundary cohesion, thereby predominantly resulting in intergranular cracking (recrystallization embrittlement) along with faster damage evolution, ultimately adversely influencing the thermal fatigue resistance [57–60]. On the other hand, there have been studies depicting ductile behaviour of tungsten in recrystallized state with an increase in the maximum elongation strain (nearly double) as compared to the deformed microstructural state [61–64]. Thus, a direct connection between recrystallization and brittle failure is not trivial.

Taking into account the design specification of high heat flux (HHF) fatigue of monoblocks from the ITER full W divertor qualification program, the performance of small to medium scale W mock-ups under combined sequential heat loads of 10 MWm$^{-2}$ (5000 cycles, 10 s) and 20 MWm$^{-2}$ (300–1000 cycles, 10 s) has been explored extensively. For instance, Pintsuk et al. [65] investigated the damage evolution in several W mock-ups (armour thickness: 5–6.5 mm), manufactured by different suppliers (Ansaldo, Italy and Plansee, Austria) under combined heat loads of 10–20 MWm$^{-2}$ ($\leq 1000$ cycles, different loading schemes) via electron beam exposure (FE2000-France) as a function of W-CuCrZr joining technology and microstructural orientation (rod vs plate). The recrystallization depth was in the range 2–4 mm depending on the loading scheme, and the formation of longitudinal deep cracks (macro-cracks) in the rod material was observed along with roughening (for all orientations). To further scrutinize the deep cracking phenomenon reported by Pintsuk et al. [65], Li and You [66] developed a finite element based numerical model and attributed the low cycle fatigue (LCF) crack formation to the brittleness of tungsten in its recrystallized state, with the initiation of the crack near the armour surface, followed by growth along the depth due to the development of tensile stresses during the cooling stage. Similar macro-crack formation in W mock-ups (manufactured by Ansaldo, Italy; Plansee, Austria; or AT&M, China) exposed to a higher number of cycles (10 MWm$^{-2}$, 5000 cycles and 20 MWm$^{-2}$, 300–1000 cycles) via electron beam (FE2000-France, JEBIS-Japan, IDTF-Russia) was observed later on by Pintsuk et al. [67], Gavila et al. [68], Sun et al. [69] and Nogami et al. [70]. Apart from the macro-crack formation, the higher number of thermal cycles resulted in a recrystallization extent varying between 1.5–8 mm (depending on mock-up geometry) in addition to damage phenomena such as single grain ejection, severe plastic deformation at the surface and micro-cracking near the W-CuCrZr interface.

Contrary to the above studies, no macro-crack formation was noticed by Riccardi et al. [71] in W mock-ups produced from the ALMT, Japan and AT&M, China grade (manufactured by Atmostat, France; CNIM, France; or Research Instruments GmbH, Germany) and exposed to ITER specified HHF thermal fatigue test at IDTF-Russia, thereby highlighting the role of the tungsten grade on the HHF thermal fatigue performance, specifically the macro-crack formation. Apart from the inhibition of crack formation, severe geometric distortion and surface roughening were still reported with no details provided on the recrystallization behaviour and the structure-property relation. Hence, to accurately determine the damage evolution mechanisms and precisely predict the thermal fatigue lifetime of the monoblock under extended steady-state high heat loads, a thorough microstructural characterization of these monoblocks is essential.

In this work, the influence of pulsed HHF load exposure (few thousand exposure cycles, relevant to ITER conditions) on the performance of the tungsten monoblock is investigated. The novelty of the present work consists in a full macro- to micro-scale based characterization and analysis of the HHF exposed W monoblocks. At the macro-scale, the damage evolution of the surface is probed and quantified in terms of the mean roughness, while at the micro-scale, the microstructural characteristics are determined and analyzed by performing optical microscopy and EBSD measurements. The relation between the microstructure and its influence on mechanical properties is established by performing micro-indentation measurements. Moreover, for the first time within the context of the divertor monoblock application, the extent of temperature dependent microstructural softening (along the monoblock depth) is quantified and discussed using different characterization approaches. The paper is structured as follows. In Section 2, details regarding the HHF exposed monoblock samples and the experimental characterization techniques are provided. The results from the microstructural characterization investigations are presented and further discussed in Section 3. The conclusions from the present work are summarized in Section 4.

2. Experimental methodology

2.1. Mock-up fabrication and HHF exposure

The tungsten monoblocks investigated in the present work were manufactured by AT&M, China and were obtained in heat exposed state from Research Instruments GmbH, Germany. The monoblocks were produced by sintering followed by hot rolling, thereby resulting in elongated grains, which were oriented parallel to the heat flux direction in the monoblock consistent with the ITER specifications [72]. The corresponding monoblock geometry also follows the ITER specifications, with a width of 28 mm, axial length of 12 mm and armour thickness of 8 mm [5]. Figs. 1a,1b schematically show the monoblock geometry along with the sample reference system. Additionally, the copper interlayer within the monoblock was applied through diffusion bonding, whereas the CuCrZr tube was brazed on to the monoblock (RI GmbH). The detailed brazing procedure can be found in [71].

For HHF thermal fatigue testing, the tungsten monoblock assembly mock-ups, each consisting of 7 monoblocks, were exposed to an electron beam at the IDTF (ITER divertor testing facility) in St Petersburg, Russia. The HHF exposure scheme of the ITER qualification program was adopted, consisting of 5000 thermal cycles at 10 MWm$^{-2}$ and 1000 thermal cycles at 20 MWm$^{-2}$ [68]. The exposure time of each thermal cycle was 10 seconds with a dwell time of 10 seconds. The absorbed heat flux was probed via global calorimetry measurements from the thermocouple readings located at the inlet and the outlet of the mock-ups, whereas the surface temperatures were recorded via an infrared camera and two colour pyrometers [71]. Note that owing to the surface degradation mechanisms during HHF testing, the surface temperatures
HHF campaign (specifically, the heat flux of 20 MWm$^{-2}$) is used as the indicative surface temperature throughout the current work and the resulting estimated temperature profile along the monoblock depth is shown in fig. 1c.

2.2. Surface roughness

The heat exposure induced surface roughness was assessed using a Bruker NPFLEX$^\text{TM}$ optical interferometer. An area of $9 \times 9$ mm$^2$ was probed by stitching 20 individual scans. The scans were acquired using an objective lens of 5x (numerical aperture of 0.12) with a narrow green light source (wavelength of 537 nm). The mean roughness parameter ($R_a$) was determined from the dataset following in-plane tilt correction and interpolation based correction for missing data points. Similarly, for comparison, the mean roughness of the monoblock bottom surface was determined and is considered as the reference, i.e. the roughness of the unexposed state.

2.3. Microstructural characterisation

To investigate the microstructure of the monoblocks following heat exposure, characterization studies were performed along two different monoblock planes, i.e. the T-H plane and the W-H plane. The samples from the monoblock were obtained through electrical discharge machining (EDM), thereby nullifying the probability of machining induced microstructural changes. The sample machined along the T-H plane (monoblock cross-section) had dimensions of 15 (height) $\times$ 12 (width) $\times$ 5 (thickness) mm$^3$. For the W-H plane, only half of the monoblock section with dimensions of 8 (height) $\times$ 14 (width) $\times$ 5.5 (thickness) mm$^3$ was machined. The machined sections along the two planes and the investigated areas are depicted in fig. 2 through the highlighted regions (cyan and purple). Furthermore, for comparison, the reference microstructure, i.e. the initial (deformed) microstructure was characterized from the bottom part of the monoblock along the T-H plane and the W-H plane (not shown here).

The sample preparation for optical microscopy and EBSD based characterization consisted of standard metallography steps, i.e. mechanical fine grinding of samples with SiC papers (up to 4000 grit) followed by electropolishing with 1.5 wt.% NaOH (sodium hydroxide) solution at 15 V for approximately 40 seconds. Moreover, for optical microscopy based characterization, the grain structure was visualized by etching the samples with 30 wt.% H$_2$O$_2$ (hydrogen peroxide) at 70°C for 30 seconds. The optical images were acquired using a ZEISS$^\text{TM}$ Axioplan 2, equipped with a 5x objective lens (numerical aperture of 0.15) under bright field mode. The macro-scale optical images of the T-H and W-H planes were obtained by stitching manually acquired optical images with at least 20% overlap.

The EBSD based characterization was performed using a FEI Sirion XL-30 ultra high resolution scanning electron microscope (UHR-SEM), equipped with a field emission electron source (FEG) and an EDAX Hikari EBSD camera at 25 kV acceleration voltage (spot size of 5). Only the T-H plane was analyzed. The step size for the EBSD scan was based on the microstructural state, i.e. for the initial (deformed) microstructure, a step size of 0.5 μm was used with a scan area of $1.25 \times 0.75$ mm$^2$, whereas boundaries with misorientation $> 15^\circ$ are classified as high-angle (HAGBs), whereas boundaries with misorientation $> 15^\circ$ are termed as high-angle (HAGBs). The kernel average...
misorientation (KAM) maps from the scan data were estimated considering the first nearest neighbours with a threshold misorientation of 5°, whereas for the grain orientation spread (GOS) based analysis, a threshold spread value of 3° was used to define a recrystallized grain [76,77]. The average grain size was estimated from the EBSD scan data using the area weighted average approach [78].

2.4. Hardness measurements

The hardness maps along the T-H plane and the W-H plane were obtained by performing micro-indentation measurements using a CSM instruments Micro-Indenter, equipped with a diamond square-based pyramid indenter (Vickers indenter). All the indentation measurements were performed with a force of 1.96 N and a dwell time of 15 seconds between consecutive indents. The mapped area along the T-H plane was 8.0 × 11 mm², with the indentations spaced equidistantly 500 μm apart along the T direction and equidistantly 250 μm apart along the H direction. For the W-H plane, the mapped area was 7 × 14 mm² with identical indentation spacings as in the case of the T-H plane.

2.5. Recrystallized fraction determination

The temperature dependent microstructural recrystallized fraction along the T-H plane of the monoblock was determined by using the hardness and EBSD measurements. The recrystallized fraction from the hardness data was determined using the temperature dependent hardness reduction from the initial state relative to the maximum hardness reduction for a fully recrystallized microstructural state, following HHF exposure:

\[ X_T = \frac{H_{init} - H_T}{H_{init} - H_{rex}} \]  

(1)

where, \( X_T \) is the recrystallized fraction at temperature \( T \), \( H_{rex} \) is the hardness in the fully recrystallized state, \( H_T \) is the hardness at a given temperature \( T \). \( H_{init} \) denotes the hardness in the initial state (deformed state). Similarly, the recrystallized fraction \( X_T \) from the EBSD data was calculated using the temperature dependent fraction of the high-angle grain boundaries following the HHF exposure, expressed as:

\[ X_T = \frac{HAGB_{init} - HAGB_T}{HAGB_{init} - HAGB_{rex}} \]  

(2)

with HAGB_{ rex} representing the HAGB fraction for the fully recrystallized state, HAGB_T representing the HAGB fraction at temperature \( T \), and HAGB_{ init} representing the HAGB fraction for the initial (deformed) state.

Another approach based on the areal (volume) fraction of the grain orientation spread measured from the EBSD data set was also used to verify the temperature dependent microstructural recrystallized fraction.

3. Results and discussion

3.1. Macrostructural characterization

The effect of HHF exposure on the macrostructural changes of the monoblocks are displayed in fig. 3. Fig. 3a shows the top view of the heat exposed monoblock for repetitive HHF loads, indicating considerable roughening of the surface aided by plastic deformation along with some local melting and material erosion events. However, no roughening assisted formation of a macroscopic crack network along the heat exposed surface is observed. The damage extent of surface roughening is localized in the centre of the monoblocks, with relatively lower roughening induced damage at the edge of the monoblocks, conforming the presence of lower thermal stresses at the edge of the monoblock, in accordance with the thermo-mechanical finite element analysis based observations of Li and You [66] and Nogami et al. [70,79]. In addition to the roughening and the melting phenomena, severe distortions in the monoblock geometry occur, leading to a barrel like shape of the monoblocks.

This barreling phenomenon can be explained based on the thermo-mechanical analysis of the HHF exposed monoblocks [66,70,73], where the cyclic heating and cooling cycles during the HHF exposure have been reported to result in the development of alternating compressive (heating) and tensile (cooling) thermal stresses in the monoblock, which over time (with increasing number of exposure cycles) manifest as accumulated thermal stresses and hence plastic strain. The barrelling phenomenon is influenced by the location of the monoblock in the mock-up, with substantial barrelling of the monoblocks located near the centre. Monoblocks located at the either ends of the mock-up show the least amount of barrelling.

Fig. 3b presents an isometric view of the heat exposed monoblocks. From the image, the influence of the temperature profile along the monoblock is visible on the surface, with a change in reflectivity of the monoblock regions with relatively high temperatures, corresponding to recrystallized areas (indicated by the red lines in fig. 3b). The surface reflectivity based transition occurs at an approximate depth level of 5–7 mm from the top surface (depending on the plane of interest). Additionally, for the W-H plane, the reflectivity based transition profile follows the curved shape of the temperature profile, i.e. relatively high temperatures along the monoblock edges (regions away from the cooling tube) in comparison to the monoblock centre (regions near the cooling tube). Furthermore, fig. 3b shows that HHF exposure has a negligible effect on the structural integrity of the monoblocks, with no trace of macro-cracks running through the monoblock or exfoliation based damage. This superior HHF thermal fatigue performance of the monoblocks in comparison to previous studies [65,68–70] demonstrates the fact that recrystallized tungsten can indeed exhibit ductile...
behaviour. Moreover, the absence of cracking in the recrystallized state in the present case can be attributed to the plastic deformation assisted suppression of the macro-crack formation (during the low temperature cycle) as suggested by Nogami et al. [70].

The roughening magnitudes of the heat exposed monoblock surface obtained from the interferometry measurements are plotted in fig. 4. The investigated area in the reference state, i.e. the initial state (bottom surface of the monoblock) is shown in fig. 4a, whereas the corresponding topography map is displayed in fig. 4c. As observed in fig. 4c, the surface topography prominently consists of processing (machining) induced band like structures (horizontal lines), characterized by relatively low waviness and a mean roughness ($R_a$) equal to 0.72 μm. For the heat exposed state, the region of interest and the corresponding topography map are shown in fig. 4b and fig. 4d. Due to the HHF exposure, the localized roughening phenomena lead to a substantial change in the surface topography, ultimately resulting in an increased waviness of the surface. The mean roughness parameter ($R_a$) determined from the heat exposed surface topography is 41.86 μm, thereby exhibiting an increase of nearly 41 μm in $R_a$ with respect to the initial state.

For rectangularly shaped heat pulses, the effect of the pulsed heat loads on the surface damage is usually expressed in terms of the heat flux factor ($F_{HF}$) [80]. For the present case (absorbed power density = 20 MWm$^{-2}$; exposure time = 10 s), the maximum calculated $F_{HF}$ corresponds to a value of 63.24 MJm$^{-2}$s$^{-1/2}$. To further assess the surface roughening and to obtain a quantitative comparison with other studies (ELM alike loads simulated using an electron beam or laser beam, with higher power density with lower exposure time), fig. 5 shows the evolution of roughness as a function of the $F_{HF}$ along with the number of exposure cycles. For a lower number of exposure cycles irrespective of the $F_{HF}$ value, the magnitude of HHF exposure assisted roughening is relatively low (<3 μm). However, with increasing number of exposure cycles (few thousand), substantial roughening occurs, with $R_a$ exceeding 20 μm for $F_{HF}$ $\geq$ 50 MJm$^{-2}$s$^{-1/2}$. Furthermore, the $R_a$ value of 41.86 μm corresponding to 1000 exposure cycles determined in the present work (blue diamond in the roughness map) shows the extent of the surface damage produced by the long duration low power density pulsed heat loads (present case) in comparison to the shorter duration higher power density pulsed loads (literature studies [51,81,82]). This, clearly illustrates the severity of the pulsed low power density heat loads (20 MWm$^{-2}$) on surface degradation, which in combination with thermal shock events (predominantly ELMs) can aggravate the situation.

3.2. Reference initial (deformed) microstructural state

The microstructure of the monoblock along the T-H and the W-H plane in the initial state is shown in fig. 6. For the T-H plane (fig. 6a), the grain topology confirms the rolling processing of the monoblock with severe elongation of the grains. Also, the grains along the T-H plane tend to be oriented parallel to the heat flux direction, in
accordance with ITER specifications. In case of the W-H plane (fig. 6b), the grain topology demonstrates a heterogeneous type microstructure, with some grains being severely elongated. The microstructure along the W-H plane is characteristic for the rolling contact plane (plane in contact with rolls) and altogether, the grain topology exhibits a plate like structure with a lower degree of elongation as compared to the T-H plane. Irrespective of the plane in consideration, the images in fig. 6 also reveal a heterogeneity in the etching process with some grains being visible as more strongly etched in comparison to others. This inhomogeneous etching can be attributed to the inhomogeneous plastic deformation of grains during the rolling process, thereby resulting in substantial sub-grain formation within heavily deformed grains.

For detailed microstructural insight, the microstructure along the T-H plane was further examined in terms of crystal orientation, sub-grain formation and grain boundary characteristics by EBSD. Fig. 7 shows these EBSD mapping based results. The tube direction (T) inverse pole figure (IPF) map shown in fig. 7a reveals a preferred crystallographic orientation of the grains, with the majority of grains having their <111> direction parallel to the T direction (<111>||T, blue colour). Apart from the preferred crystallographic orientation, several areas inside the grains in the IPF map show variegation, thereby indicating the extensive formation of sub-grains as emphasised earlier. A qualitative characterization of the sub-grain formation due to plastic deformation and the corresponding measure of the stored strain energy can be obtained through the kernel average misorientation (KAM) parameter, which is obtained by averaging the measured misorientation between a given point (pixel) and its nearest neighbours [83]. As seen in the KAM map (fig. 7b), the accommodation of the plastic deformation via the formation of special sub-structures varies from grain to grain with a majority of grains exhibiting a kernel averaged misorientation in the range 0.3°–2.0° (for KAM based distribution see fig. 17c in Appendix B), suggesting a high stored strain energy due to dislocations inside the monoblock. Similarly, in terms of quantifying the overall character type of the grain boundaries in a typical rolled microstructure, fig. 7c reveals that the occurrence frequency of boundaries with a misorientation less than or equal to 15° (LAGBs) is the highest, whereas as the cumulative occurrence frequency of boundaries with a misorientation angle greater than 15° (HAGBs) is less
than 6%. To quantify the grain size, the areal based grain size distribution calculated from the EBSD data is shown in fig. 7d. The distribution tends to peak in the range 80–160 μm, with an average grain size of 96 μm, marginally larger than reported by Yin et al. [84] for AT &M ITER grade W (CEFTR).

3.3. Microstructural changes following high heat flux exposure (HHF)

The HHF induced microstructural changes along the T-H and the W-H plane of the monoblock were characterized using optical microscopy. Macro-scale based images reconstructed by stitching individual images are shown in fig. 8. The image shown in fig. 8a corresponds to the top 8 mm region of the monoblock cross-section (region highlighted in cyan in the monoblock sketch), thereby exposing the influence of the temperature profile along the monoblock depth. A substantial change in the grain structure by recrystallization and grain growth can be observed in fig. 8a, resulting in two different microstructures represented as zone A and zone B. Zone A has a recrystallized grain structure, whereas zone B is similar to the initial, deformed microstructural state. The transition between zone A and zone B is rather abrupt, and occurs at a depth of approximately 5.5 mm from the top surface. Taking into account the temperature profile along the monoblock depth, the transition between the recrystallized and initial grain structure occurs approximately around 1100°C, thereby providing an estimate of the estimated recrystallization threshold temperature. A recrystallization temperature of 1100°C is lower than the usually reported threshold temperature (1150°C - 1300°C depending on the deformation degree; based on 1 h static annealing studies [85–88]). The difference may be due to the higher cumulative annealing time (approximately 2.7 hours considering 1000 cycles of 10 s). Also, Yuan et al. [85] have shown that short pulsed HHF exposure (0.5-1.5 s; 23 MWm−2) leads to an increase in the recrystallization onset temperature (approximately 2480°C) due to higher heating rates, with faster recrystallization kinetics as compared to the ones observed in the steady state annealing experiments. However, in the present case, with comparatively long HHF pulses, the heating rates tend to be nearly identical as in the static annealing experiment, suggesting a negligible effect on the recrystallization onset temperature. Furthermore, the boundary between the recrystallized and original microstructure displays more or less a similar profile as the temperature profile along the monoblock cross-section (T direction). Additionally, the presence of a steep temperature gradient along the monoblock leads to a gradient in the size of the recrystallized grains (zone A in fig. 8a), with relatively large grains in the sub-surface region. The large grain growth in the near-surface region is attributed to the presence of high temperatures near the surface (≈ 1750°C-2200°C), which, in combination with repetitive small plastic strains (due to HHF loads), provide
the driving force for grain boundary movement, ultimately resulting in the abnormal grain growth phenomenon (AGG or secondary recrystallization). The AGG based reasoning for the development of large grains under HHF exposure is identical to the one provided by Ciulik and Taleff [89], for the development of large single crystals in Mo by plastic straining at higher temperatures and by Omori et al. [90] for a Cu-based alloy through cyclic heat treatments. Also, in case of W, similar AGG based phenomena have been observed by Farrell et al. [91], but the driving force for AGG in this study was attributed to differences in the impurity concentration. Ciulik and Taleff [89] have further subclassified the AGG phenomenon based on the concurrence with plastic straining, i.e. static AGG or dynamic AGG. However, distinguishing the observed AGG phenomenon as static or dynamic is not trivial and requires precise information on the magnitude of plastic strain that initiates the AGG, as well as the evolution of the microstructural state as a function of the number of exposure cycles.

The micrograph of the W-H plane shown in fig. 8b exhibits similar changes in the grain structure due to recrystallization and grain growth following repetitive heat exposure. However, contrary to the T-H plane, the transition boundary between the recrystallized (zone A) and the initial microstructure (zone B) varies as a function of the distance from the cooling tube. For regions close to the cooling tube, the transition occurs at a depth level of approximately 5 mm from the surface, whereas away from the cooling tube, the transition occurs at

Fig. 8. Low magnification optical images depicting the microstructural changes along the (a) T-H plane and (b) W-H plane of the monoblock due to HHF exposure. The optical images correspond to the regions highlighted in cyan (T-H) and purple (W-H). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
approximately 7 mm. This width dependent (W direction) microstructure transition is due to the temperature profile along the W-H plane as highlighted earlier, i.e. higher temperatures along the monoblock edges in comparison to the monoblock centre. Apart from the depth and width dependent microstructure transition, the recrystallized grain structure (zone A) along the W-H plane shows a similar topology as the T-H plane, with larger grains near the surface and relatively finer grains along the depth.

In addition, for both planes (T-H and W-H), the depths at which the transition between the recrystallized and the initial microstructure occurs are consistent with the values obtained from the macro-scale image depicting the change in surface reflectivity (fig. 3b). Besides the HHF induced microstructural changes in the monoblock, micro-cracks in the softer recrystallized microstructure or at the interface between the two zones A and B in figs. 8a and 8b are not visible.

To further scrutinize the observed temperature dependent recrystallization behaviour in the monoblock, EBSD mapping of the recrystallized region (zone A) in the T-H plane was performed and the results are shown in fig. 9 (the EBSD mapping of the microstructure transitional regime, i.e. recrystallized versus initial state is demonstrated in fig. 19, Appendix C). The height direction IPF map is shown in fig. 9a and the influence of the temperature on the grain size can be clearly observed. As pointed out earlier, the presence of relatively high temperatures (> 1750°C) in the near-surface region results in substantial AGG, ultimately resulting in grains with an average grain size of 450 μm. Also, variegation within the large surface grains is observed, thereby revealing the formation of sub-grains due to plastic deformation under the repetitive heat loads. For temperatures below 1750°C and above the recrystallization threshold temperature 1100°C, i.e. in the bulk monoblock, an nearly equiaxed grain structure with a bimodal grain size emerges. However, the variation in the temperature (depth) dependent average grain size is relatively small in the bulk monoblock (fig. 18, Appendix C). Also, the predominance of red coloured recrystallized grains in the IPF map indicates a preferred crystallographic orientation of the <001> direction parallel to the height direction (<001>||H).

In order to gain more insight into the distribution and the nature of the grain boundaries in the recrystallized state, the temperature (depth) dependent grain misorientation was calculated by partitioning the EBSD map. Fig. 9c schematically shows this temperature dependent grain boundary character distribution along the monoblock depth. The Mackenzie curve representing a random texture distribution is also plotted for comparison [92]. For regions with high temperatures (> 1750°C; near the monoblock surface), the distribution shows a peak for boundaries with a misorientation angle smaller than 10° (LAGBs), with a relatively low occurrence frequency of HAGBs. However, for regions with temperatures lower than 2000°C (in the bulk monoblock), the boundary distribution changes significantly, with the occurrence frequency of the
LAGBs decreasing as a function of temperature and a parallel increase in the frequency of the HAGBs. Also, for temperatures lower than 1750°C, the majority of the boundaries are of high-angle type. Thus, defining a transition from low-angle type at the surface to high-angle type in the bulk. Moreover, by comparing the temperature dependent grain boundary distribution with the random distribution (fig. 9c), it can be stated that none of the distributions matches the random distribution (fig. 9c), as recrystallization predominately results in development of HAGBs.

The plastic deformation of the near-surface grains during the repetitive heat loads (in the presence of high temperatures) is further assessed and evaluated in terms of the intragranular misorientation. For this purpose, the grains labelled 1 and 2 with an orientation spread of approximately 4° (recrystallized surface grain) and 1° (recrystallized bulk grain) from the GOS map (fig. 9b) are considered. Fig. 10 shows the intragranular misorientation within the grains. For the recrystallized surface grain 1 (fig. 10a), significantly higher intragranular misorientation occur with significant jumps in the cumulative misorientation profile (point to origin in fig. 10c corresponding to the red line in fig. 10a) reaching a magnitude of 8°. This qualitatively indicates the presence of geometrically necessary dislocations (GNDs), formed due to the plastic deformation of near-surface recrystallized grains. For the recrystallized bulk grain (fig. 10b), as expected, the intragranular misorientation is relatively low, with the cumulative misorientation profile being nearly flat around the magnitude of 2° (point to origin in fig. 10d corresponding to the red line in fig. 10b), i.e. the minimum angular resolution of the standard EBSD technique.

Recently, Ren et al. [88] attributed the improved ductility in cold rolled as well as annealed (below recrystallization temperature) tungsten to the presence of a higher fraction of LAGBs near the surface (fig. 9b). The amount of plastic strain in the recrystallized surface grains due to repetitive thermal loads is relatively low, as indicated by the lower GOS spread in comparison to the GOS spread for the initial (deformed) microstructural state (fig. 17 in Appendix B). Additionally, the temperature dependent areal based GOS distribution is shown in fig. 9d. For temperatures exceeding 200°C, i.e. near the surface region of the monoblock, the GOS based distribution reveals a significant area fraction of grains with a relatively higher orientation spread in recrystallized state (≈ 47%). While for temperatures between 200°C and the recrystallization threshold of 1100°C, i.e. in the monoblock bulk, the area fraction of grains with relatively low orientation spread increases (with decreasing temperature) and matches with the decrease in the LAGB fraction in fig. 9c, as recrystallization predominantly results in development of HAGBs.

Fig. 10. Intragranular misorientation for grains taken from different locations in the T-H plane of the heat exposed monoblock, where (a) and (c) represent the plastically deformed recrystallized surface grain and (b) and (d) represent the recrystallized grain located at a depth of 2.8 mm. Subfigures (c) and (d) also show the misorientation as a function of the distance (along the red line) from the origin (point O) within the grain. The point to point curve in (c) and (d) provides a measure of the misorientation between neighbouring points along the red line, whereas the point to origin curve provides a measure of the misorientation between all the points along the red line and the origin point O. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
3.4. Texture analysis

Fig. 11 presents pole figures for the initial and recrystallized microstructural states, corresponding to the EBSD maps shown previously in Fig. 7a and Fig. 9a. For the initial microstructural state (Fig. 11a), the orientation (111)||T exhibits the highest intensity of the order 3 times random, with relatively lower intensities for the orientation (110)||W (2.5 times random) and (110)||H (1.5 times random). Due to the HHF exposure, the recrystallized grains exhibit higher intensities (approximately 2.8) for the (001) orientation with a small rotation with respect to the width direction (W). Additionally, the orientations (111)||T and (110)||W are also observed for the recrystallized grains, however with relatively lower intensities of approximately 2 times random. These results illustrate the recrystallization promoted weakening of the initial texture, in accordance with the observations of Xia et al. [93] and Ren et al. [88]. Also, irrespective of the microstructural state, the maximum intensity of the texture does not exceed 3 times random, thereby suggesting a weak global crystallographic texture.

To gain more insight in the texture components present in the initial as well as the recrystallized state, the orientation distribution function (ODF) sectioned at an Euler angle \( \phi_2 \) equal to 0° and 45° are shown in Fig. 12. The common fibres for BCC metals are shown in Fig. 20 (Appendix D). In the initial state, the ODF sections (Fig. 12a) reveal predominantly the presence of the \( \varepsilon \) fibre \((\phi_1 = 90^\circ, \phi = 35^\circ, \phi_2 = 45^\circ)\) with the texture component \((112) < 111 >\), and \( \zeta \) fibre \((\phi_1 = 0^\circ-70^\circ, \phi = 45^\circ, \phi_2 = 0^\circ)\), characterized by the texture components \((011) < 100 >, (211) < 001 > \) and \((011) < 111 >\). Additionally, relatively higher intensities for the \( S \) type family components, i.e. \((214) < 121 >, (213) < 306 > \) at \( \phi_2 = 65^\circ \) (not shown here) are also observed, along with a weakly developed \( \theta \) fibre \((\phi_1 = 0^\circ, \phi = 0^\circ, \phi_2 = 0^\circ)\) family component \((001) < 100 >\) (as shown in 45° ODF in Fig. 12a). These observed fibre textures are distinctive for a shear type deformation in BCC metals [94].

For the recrystallized state, the ODF sections (Fig. 12b) exhibit identical fibres as in the initial state, i.e. \( \varepsilon, \zeta \) and \( \theta \); however the texture components tend to differ. The \( \zeta \) fibre \((\phi_1 = 55^\circ-90^\circ, \phi = 45^\circ, \phi_2 = 0^\circ)\) in the recrystallized state is mainly composed of the family components \((011) < 111 >\) and \((011) < 111 >\) with relatively high intensities as compared to the initial state. Additionally, a relatively stronger development of the \( \eta \) fibre \((\phi_1 = 0^\circ, \phi = 0^\circ-90^\circ, \phi_2 = 0^\circ)\) with texture component \((001) < 100 >\) identical to the \( \theta \) fibre also appears in the recrystallized state ODF sections. The preferred orientation close to the \( \eta \) fibre \((< 100 >||H)\) in the recrystallized state can be understood relative to the higher growth rate of grains oriented with...
their \(<100>\) parallel to the heat flow direction (height direction) [95]. Furthermore, the development of a recrystallization texture approaching the initial texture can occur by oriented nucleation and oriented growth mechanisms [96]. Based on the oriented nucleation theory, the texture is dictated by grains that nucleate with a preferred orientation from the heavily deformed grains (higher Taylor factor). On the other hand, within the oriented growth theory, the nucleated grains tend to be randomly oriented, however selective growth of some grains tends to dominate the global texture. Thus, for the present case, the recrystallization texture can be explained on the basis of the oriented growth theory due to the presence of a steep temperature gradient in the monoblock and the corresponding abnormal growth of the grains near surface and normal grain growth in the bulk. Similar reasoning with respect to development of recrystallization texture under ELM like transient heat loads in W has also been reported by Susolva et al. [59].

Additionally, the nature of the identified initial and recrystallized texture differs significantly from the ones usually reported in literature. For example, Xia et al. [93] investigated the rolling and annealing texture in rolled W sheet and reported the presence of \(\alpha\) and \(\gamma\) fibre texture in both states, whereas Suslova et al. [59] predominantly observed \(\alpha\) fibre as cold-rolling and annealing texture. Likewise, Ren et al. [88] reported a cold rolling and annealing texture primarily consisting of \(\alpha\), \(\gamma\) and \(\eta\) fibres, and recently the annealing assisted strengthening of \(\alpha\) fibre in hammered ITER grade W was reported by Tanure et al. [97]. The difference in the fibre textures between the present work and the literature are likely due to the processing route of the W sheets/rod, with hot rolling resulting in a distinct fibre texture as compared to cold rolled or hammered type processing. The hot rolling texture observed in the present study is in accordance with the one reported by Zhang et al. [98] in hot rolled W plate and Kim et al. [99] in hot rolled electrical steels.

The implications of the texture observed in the present work, i.e. predominantly grains with \(<111||T\) (\(\{(112)||W\) and \(011||W\)) as well as \(<001||T\) (relatively lower density) on the performance of the monoblock under synergistic particle and heat loads are significant. On the one hand, as reported by several literature studies [11–15], substantial blistering and nanostructure formation can occur for grains oriented with \(<111\) corresponding to the surface normal, resulting in a strong modification of the surface. On the other hand, grains with a \(<001\) surface normal, although being the least prone to blistering can promote surface modification by the development of wavy structures [100]. Simultaneously, surface grains with an orientation close to \(011\) can aid in faster diffusion of hydrogen species [101], whereas grains close to the \(112\) orientation will be susceptible to higher helium retention [102]. This could result in a performance loss of the monoblocks under combined particle and heat loads due to complex particle interactions. However, deeper investigations aiming to relate the material state as well as the crystallographic orientation to the

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**Fig. 12.** Orientation distribution function (Euler space) of the (a) Initial (deformed) state, (b) heat exposed (recrystallized) state determined using EBSD, with \(\varphi_2\) sectioned at 0° and 45°. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
Fig. 13. Hardness maps of the monoblock following high heat flux exposure along the (b) T-H plane and (c) W-H plane. The highlighted regions (cyan and purple) in (a) represent the mapped areas in (b) and (c). The mapped areas correspond to the optical images shown in fig. 8. A transition in hardness along the monoblock depth occurs due to a change in the microstructural state, i.e. recrystallized to initial (deformed) state. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 14. Temperature/depth dependent hardness along the T-H and the W-H plane of the heat exposed monoblock evaluated from the hardness map in fig. 13. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 15. Temperature (depth) dependent microstructural softening in the monoblock (T-H plane) following HHF exposure, determined and compared using different approaches. A typical temperature dependent sigmoidal function type softening behaviour is observed. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
thermal fatigue performance under combined HHF and plasma loads are required to substantiate such relations.

3.5. Structure-property relation: Hardness mapping

The effect of the microstructural changes following the HHF exposure on the mechanical properties was obtained through Vickers based micro-indentation measurements. Hardness maps for the T-H and the W-H plane of the heat exposed monoblock are shown in fig. 13, where fig. 13a schematically displays the areas where the hardness mapping was performed. For both planes, i.e. in fig. 13b (T-H) and fig. 13c (W-H), the recrystallization and grain growth assisted microstructural changes due to the high temperatures and high stored energy (dislocations) result in significant loss of hardness, with the softening regime extending approximately up to depth levels of 5.5 mm (even higher for the W-H plane). For the T-H plane, a sharp jump in the hardness is observed at depth values of 5.5 mm, whereas in case of the W-H plane, the increase in the hardness along the depth occurs sharply near the centre of the monoblock and more gradually along the edges. The increase in the hardness along the monoblock depth is due to the presence of the initial deformed microstructure characterized by a high dislocation density. Moreover, the depth values at which the transition in hardness is observed concurs with the microstructure transition depth values determined from the optical images (fig. 8). Additionally, the increase in the hardness near the surface regions due to the plastic deformation assisted sub-grain formation (GNDs for example in figs. 10a, 10c) is found to be insignificant. This can be due to the relative low density of the GNDs in the near-surface grains, ultimately having negligible contribution to hardness as compared to other defects.

Furthermore, the temperature (depth) dependent hardness profile along the depth in the T-H and the W-H plane is shown in fig. 14. A distinct two stage behaviour of the hardness profile as a function of temperature (depth) can be observed for both the planes. The initial reductions in hardness in the estimated temperature range 920°C-1100°C (in the bulk monoblock) may be due to the high stacking fault energy of W, promoting rapid recovery at lower temperatures by dislocation rearrangement, climb and cross-slip based mechanisms, along with partial recrystallization. The large reductions in hardness at higher temperatures (2250°C-1100°C) in the top 5–5.5 mm of the monoblock can be linked with the full recrystallization stage. A similar (annealing temperature dependent) two stage behaviour of hardness has also been reported by Tsuchida et al. [86], Guo et al. [87], Tanure et al. [97] for different ITER grades W. Although the transition in hardness along the W-H plane occurs more smoothly as compared to the T-H plane, the hardness profile along both planes exhibits nearly identical temperature/depth values with respect to the transition.

3.6. Recrystallized fraction

Fig. 15 compares the temperature dependent microstructural softening fraction determined using different approaches, i.e. hardness, percentage of HAGB fraction and GOS. The hardness based recrystallized fraction (open circles in blue) typically shows a temperature dependent sigmoidal behaviour (dashed black line), with negligible softening for temperatures below 900°C and complete softening for temperatures above 1100°C. A similar trend is observed for the recrystallized fraction determined using the HAGB fraction from the EBSD measurements, except for high temperatures (approximately > 1550°C), where the HAGB fraction based approach underestimates the recrystallized fraction. The discrepancy in the recrystallized fraction at higher temperatures (> 1550°C), i.e. in the top surface region of the monoblock is due to the pronounced plastic deformation assisted sub-grain formation with negligible influence on the hardness, hence, resulting in a relatively higher fraction of LAGBs as compared to HAGBs. Also, for temperatures higher than approximately 1950°C, an identical behaviour of the GOS based softening fraction can be observed, irrespective of the recrystallized threshold value. However, for temperatures below 1900°C, the GOS tends to show a nearly similar softening fraction as obtained by the hardness. Note that the GOS is quite sensitive to the recrystallized grain threshold value (illustrated by the difference between a threshold of 2° and 3° in fig. 15), specifically for regions where significant intragranular misorientations occur (near the surface of the monoblock), whereas for lower temperatures, the magnitude of the softening varies marginally with the recrystallized grain threshold value. In a nutshell, the different approaches confirm a consistent qualitative trend of the temperature dependent microstructural softening in the monoblock.

4. Summary and conclusions

The prime focus of the present work was on understanding the microstructural changes in the tungsten monoblocks under pulsed high heat flux (HHF) loads relevant to fusion reactors. For this, the actively cooled tungsten monoblocks exposed to several thousand cycles of pulsed HHF load were characterized by using a combination of interferometry, optical microscopy, electron back scatter diffraction and micro-indentation techniques. The main findings of the work are:

• Significant barreling of the monoblock geometry combined with plastic deformation assisted roughening of the monoblock surface was observed. These geometrical and surface modifications were attributed to the accumulation of thermal stresses during the pulsed loading of the monoblock. Additionally, the extent of such geometrical changes could worsen with high power density heat loads, thereby remarkably limiting the performance and lifetime of the monoblocks.
• Substantial microstructural change of the HHF exposed monoblocks due to recrystallization and grain growth was observed. The recrystallization depth in the monoblock was in the range 5-5.5 mm (from the exposed surface) with an abrupt transition between the recrystallized and initial microstructure. This abrupt transition was related to the estimated temperature profile in the monoblock, providing a measure of the recrystallization threshold temperature of approximately 1100°C. The relatively lower recrystallization threshold temperature observed in the present work clearly highlights the importance of long duration pulsed heat load studies for reactor like applications as compared to the standard annealing experiments.
• The presence of high temperatures and small plastic strains near the monoblock surface resulted in a secondary recrystallization phenomenon, with relatively large grains ($d_{avg} = 450 \mu m$) near the surface in comparison to the bulk of monoblock ($d_{avg} = 90-120 \mu m$).
• In contrast to the often reported brittleness of tungsten in the recrystallized state, no macro-crack or micro-crack formation was found in the recrystallized regime of the monoblock, thereby indicating that the recrystallization phenomenon by itself may not be detrimental for the lifetime of the monoblocks. Yet, second-order effects of recrystallization such as the segregation of impurities at grain boundaries may limit the lifetime by promoting brittleness.
• The absence of micro-cracking may be also related to the plastic deformation assisted formation of the LAGBs in the near surface region, increasing the strain compatibility between the surface grains, which promotes a ductile behaviour of the recrystallized W. However, quantitative predictions regarding the influence of recrystallization on cracking behaviour, specifically within the context of HHF loading conditions demand further investigations.
• The recrystallized microstructure displayed crystallographic texture identical to the initial state, however with an overall reduction in the intensity, i.e. weakening of the initial texture. The similarities between the initial and crystallographic texture may be due to the
presence of a strong temperature gradient in the monoblock, thereby suggesting an oriented growth type mechanism.

- Based on the observed crystallographic texture, the recrystallized grains tend to be susceptible to further surface modification as well as bulk retention of species during plasma exposure, ultimately compromising the service lifetime of the monoblocks.
- A two-stage temperature dependent softening behaviour was observed in the monoblock. The recovery and partial recrystallization stage, characterized by small reductions in hardness, was estimated to be in the temperature range 920°C-1100°C, whereas the full recrystallization stage, with a substantial reduction in hardness, was estimated to be above 1100°C.
- The hardness measurement based temperature dependent microstructural softening of the mono-block was compared with the one predicted by EBSD measurements, and a qualitative agreement of the microstructural softening trend between these methods was noticed. Hence, indicating the accuracies of both approaches for characterizing the recrystallization assisted microstructural softening.

For future work, the effect of the HHF exposure, specifically the long duration pulsed loading at 20 MWm⁻² in combination with the plasma ion exposure (synergistic as well as sequential) on the thermal fatigue behaviour need to be assessed for more accurate predictions of the monoblock lifetime. Furthermore, the influence of the particle loads, specifically the neutrons and the helium atoms, on the kinetics of the recrystallization process under such HHF conditions is still an open question. The neutrons may accelerate recrystallization in the monoblock as well as assist in dynamic recrystallization, while the helium bubbles (dominantly in the surface and sub-surface) may retard recrystallization. Thus, the extent of the effects of these particle loads on recrystallization need to be characterized experimentally by appropriate irradiation campaigns. Meanwhile, the presence of a heterogeneous microstructural state, i.e. recrystallized in the top few millimetres and initial (deformed) in the remainder of the monoblock may limit the long term performance of the monoblock. The influence of the grain size on the brittle-to-ductile transition temperature (BDTT) has been well documented, and the presence of a grain size gradient in the recrystallized regime of the monoblock (as observed in the present work) will extensively affect the mechanical behaviour, which is not thoroughly investigated yet. In this context, mechanical testing of such heterogeneous microstructures can help in developing more understanding of real-time failure scenarios.

Data availability

The raw or analyzed data reported in this study cannot be shared currently. However, in future the authors can be contacted for further information regarding the availability of data.

Declaration of Competing Interest

This study and the manuscript present no conflict of interest.

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Appendix A. Evolution of surface temperature

Fig. 16 shows the evolution of the monoblock surface temperature as a function of the number of HHF exposure pulses (20 MWm⁻²). The increase in the monoblock surface temperature is due to the HHF assisted roughening of the surface, ultimately increasing the emissivity of the surface. Also, in fig. 16, the surface temperature evolution is depicted for the monoblocks manufactured by ALMT (Japan), whereas in the present case, monoblocks manufactured by AT&M (China) have been investigated. However, irrespective of the manufacturer, the HHF exposure scheme as well as the HHF assisted roughening tend to be identical. Thus, the experimentally determined surface temperature (vs. number of pulses) for ALMT monoblocks can be extrapolated to AT&M monoblocks.

Fig. 16. Variation of surface temperature for W monoblocks (from 3 mock-ups) manufactured by ALMT (Japan) as a function of the number of pulses during HHF exposure at 20 MWm⁻², reproduced from [71], Copyright 2019, with permission from Elsevier. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
Appendix B. Initial microstructure: EBSD analysis

The complementary EBSD analysis of the initial (deformed) microstructural state is schematically depicted in fig. 17. Fig. 17(a) shows the grain orientation spread (GOS) map, whereas fig. 17(b) displays the areal based GOS distribution. As noticed in fig. 17(b), the areal based GOS distribution peaks within the range 6°-8°, typically characteristic of a deformed microstructure. Moreover, in fig. 17(c), the number fraction based kernel average misorientation (KAM) distribution is showed, corresponding to the KAM map, shown previously in fig. 7b.

Appendix C. Recrystallization and grain growth effects in monoblock

The HHF exposure assisted microstructural changes due to recrystallization and grain growth was extensively investigated in this study. Within this context, the supplementary information regarding the temperature dependent grain size determined along the monoblock depth from the EBSD technique is shown in fig. 18. Furthermore, fig. 19 displays the transition of the microstructure along the monoblock depth in terms of (a) IPF map and (b) GOS map. The IPF map in fig. 19a corresponds to the height direction and the recrystallized grains (no variegation) can be distinguished from the initial grains (deformed; with considerable variegation within the grains.). Also, the distinction between the recrystallized and the initial grains can be more easily perceived through the GOS map in fig. 19b, with the recrystallized grains revealing a relatively lower degree of orientation spread as compared to the initial grains (deformed).
Fig. 18. The evolution of the grain size as a function of the estimated temperature in the monoblock following the long duration HHF exposure. The x-axis based error bars indicate the degree of uncertainty in the temperature due to the non-steady state HHF exposure scheme and the resulting surface degradation. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 19. The temperature dependent transition of the microstructural state in the monoblock depth following the high heat flux exposure determined using EBSD, with (a) Height direction inverse pole figure (IPF) map and (b) Grain orientation spread (GOS) map. The top 400 μm (monoblock depth: 5.1–5.5 mm) region in (a) and (b) represents the recrystallized microstructure (specifically, note the relatively lower orientation spread of the grains), whereas the microstructure in the depth resembles the initial state (higher orientation spread due to plastic deformation assisted sub-grain formation). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
Appendix D. Crystallographic texture: BCC metals

Fig. 20 depicts the commonly observed rolling and annealing textures for BCC metals in Euler space, sectioned at Euler angle $\varphi_2$ equal to (a) 0° and (b) 45°. Additionally, the characteristic components pertaining to the different fiber textures are also shown.

![Image of Euler space sections for BCC metals](image_url)

Fig. 20. ODF sections in Euler space depicting the commonly observed texture fibres at (a) $\varphi_2 = 0°$ (b) $\varphi_2 = 45°$ in BCC metals, adapted from [103]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

References


