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Evolution of microstructure and texture of moderately warm-rolled pure tungsten during annealing at 1300 °C

Kang Wang¹, Haitao Sun¹, Xiang Zan¹,²,*, Umberto Maria Ciucani³, Wolfgang Pantleon¹,¹, Laima Luo¹,², Yucheng Wu¹,²,⁴

¹ School of Materials Science and Engineering, Hefei University of Technology, Hefei 230009, China;  
₂ National-Local Joint Engineering Research Centre of Nonferrous Metals and Processing Technology, Hefei 230009, China.  
³ Section of Materials and Surface Engineering, Department of Mechanical Engineering, Technical University of Denmark, Kongens Lyngby 2800, Denmark  
⁴ Research Centre for Powder Metallurgy Engineering and Technology of Anhui Province, Hefei 230009, China

Abstract: The mechanical behavior, microstructure and texture evolution were investigated during isothermal annealing at 1300 °C of pure tungsten moderately warm-rolled to 67% thickness reduction. The degradation of the mechanical properties is characterized by hardness testing. The microstructure and texture evolution during heat treatment were characterized by Electron Backscatter Diffraction. During annealing of the moderately warm-rolled tungsten, recrystallization occurred first, quickly followed by relatively slow grain growth. The recrystallized volume fractions determined from hardness measurements and microstructural characterization were essentially the same. The evolution of the grain sizes during recrystallization was analyzed independently for deformed and recrystallized grains. Quantitative texture analysis showed that the overall texture strength is enhanced after recrystallization. As recrystallization strongly affects the mechanical properties of tungsten, such insights in the annealing behavior of warm-rolled tungsten plates are valuable for an understanding of their performance as potential plasma-facing materials in future fusion reactors.
Key words: Warm-rolled tungsten, Recrystallization, Microstructure, Grain size, Texture

1. Introduction

Tungsten is a refractory metal with excellent properties: the highest melting point of all metals (3695 K), high strength at high temperatures, good thermal conductivity (approximately 1.75 W cm\(^{-1}\) K\(^{-1}\) at 293 K), low thermal expansion coefficient (4.32-4.68·10\(^{-6}\) K\(^{-1}\) at 293 K), high sputtering threshold energy and limited activation under neutron irradiation [1-3]. Due to its unique properties, pure tungsten is the most promising candidate material for the plasma-facing first wall and divertor components in fusion reactors [4, 5]. During service, these components experience extreme operating conditions from thermal loading, plasma exposure and neutron irradiation [6], especially huge heat fluxes (steady heat power of 10-15 MW/m\(^2\) with transients up to 20 MW/m\(^2\)) and temperature gradients (During transient processes, the surface temperature may exceeds 2000 °C which results together with an internal coolant tube temperature of 100 °C (for ITER) in a thermal gradient of about 200 K mm\(^{-1}\)) [6-8]. The combination of these demands puts strict requirements on the choice of plasma-facing materials.

Under the expected service condition with high temperatures (constantly up to 1200 °C at the divertor of ITER [8], but even higher during transients), recovery, recrystallization and grain growth phenomenon will occur in the chosen material, which in case of deformed pure tungsten will lead to microstructural and textural changes. During recrystallization, the originally deformed grains are consumed by dislocation-
free recrystallized grains, resulting in a decrease in the strength of the material and grain boundary embrittlement [9-11]. Recent investigations indicate that the resistances to blistering (an undesired surface modification) and irradiation damage were influenced significantly by the orientation of the surface grains [12-18]. In general, tungsten grains having low-indexed crystallographic planes at the exposed surface displayed better irradiation resistance under particle irradiation. For instance, grains with surfaces parallel to {001} have good erosion resistance and showed much less mass removal after the 30 keV Ga\(^+\) ion bombardment [16]. The resistance towards blistering of grains with surfaces near {100} is better than that of grains near {111} [18]. It is expected to control or alleviate both blistering and retention by selecting tungsten material with suitable preferred orientations for fusion reactors. The texture evolution of tungsten during long-term high temperature service will in turn affect the performance regarding irradiation and other properties. It is considered to be necessary and valuable to investigate the texture evolution of tungsten during annealing to establish the link between bulk texture and properties.

Generally, the microstructural evolution during annealing at high temperatures can be categorized into three main stages: recovery, recrystallization and grain growth. Recovery usually involves thermally activated motion, agglomeration, and annihilation of point defects and moreover annihilation and rearrangement of dislocations [19, 20]. Primary static recrystallization which follows recovery and also competes with it involves the replacement of recovered deformed structure by newly formed defect-free and more equiaxed grains. After completion of recrystallization, the recrystallized
Grains begin to grow as the annealing continues [19, 20]. The annealing behavior of tungsten (bulk plate, sheet and foil) has been reported from different investigations, which mainly focused on the recovery and recrystallization kinetics [21-25] and determined the recrystallization temperature through the combination of the changes of hardness and microstructure during isochronal annealing at different temperatures [26-29]. However, metals manufactured by different deformation processes, develop different deformation structures leading to different recrystallization behaviors [19]. Therefore, it is necessary to study the annealing behavior of tungsten plates with different rolling history and quantify the microstructure evolution during the whole annealing process.

The present research investigated the static recrystallization and grain growth behavior of a commercial pure tungsten plate warm-rolled to an intermediate rolling reduction of 67% (abbreviated as W67) during annealing at 1300 °C. Due to the short time during which solely recovery processes occur at the high temperature chosen, the recovery stage will be ignored. The present material differs from the tungsten plate warm-rolled to the same thickness reduction and discussed in [21] as it has been received from a different supplier (Advanced Technology & Manufacturing Co. Ltd., Beijing) following a different rolling scheme. The degradation of mechanical properties is followed by hardness testing; the microstructural and textural evolution is characterized using Electron Backscatter Diffraction (EBSD). In this manner, the work aims to further the understanding of the behavior of tungsten as plasma-facing material through service at the high operation temperatures in fusion reactors.
2. Materials and Experiments

The material used in this investigation was a warm-rolled commercially pure tungsten plate received from Beijing Tianlong Tungsten Technology Co. Ltd. The plate had been manufactured from tungsten powder by cold isostatic pressing and sintering under a hydrogen atmosphere at 2300 °C for 6 h. A sintered billet (with initial temperature of 1500 °C) was warm-rolled in several passes by unidirectional rolling to a thickness reduction of 67% to obtain the rolled plate. Small specimens of about 6×4×5 mm³ along RD×TD×ND (Rolling direction × Transverse direction × Normal direction) were cut from homogeneous parts central (with respect to ND) in the plate by wire cutting using electro discharge machining.

Different small specimens cut from the warm-rolled pure tungsten plate were individually isothermally annealed at 1300 °C for different times to investigate the evolution of microstructure and texture during annealing. All specimens were individually sealed in evacuated quartz glass tubes to prevent oxidation during the heat treatment. The tubes with the samples were put in a pre-heated furnace for annealing, removed individually after the desired annealing time and cooled to room temperature by air-cooling. For each desired annealing time a single small sample was used, for which both, hardness tests and microstructural investigations by EBSD, were performed.

All hardness measurements and EBSD results were obtained on transversal sections (i.e. RD-ND sections) close to the center region (with respect to ND) of the original plate. The surface was prepared metallographically by subsequent mechanical
grinding and polishing with silicon carbide paper. For EBSD investigations, the samples were additionally electropolished at room temperature with an aqueous solution containing 3 wt. % NaOH at a constant voltage of 10 V. Vickers hardness measurements were performed on the polished surfaces using a load of 3 kg and a dwell time of 10 s. The reported hardness values HV3±ΔHV3 are averages over 10 indents with the standard deviation ΔHV3 of the average; error bars in graphs represent ±ΔHV3 as well. An EBSD HKL Inca attachment with an EBSD system from Oxford Instruments in an S-3400N Hitachi SEM was used to determine the local orientations on large grids and to analyze the local texture. A step size of 1 μm was chosen in general for studying details in the microstructure and a larger step size (2 μm) was used for texture determination to allow increasing the mapped area and improving the statistics. Low angle boundaries (LABs) with misorientation angles between neighboring pixels from 2° to 15° and high angle boundaries (HABs) with misorientation angles above 15° are highlighted in boundary maps. In the present study, grains were identified and reconstructed based on a threshold misorientation angle of 15°. Grains were classified as recrystallized or deformed according to their internal average misorientation angle using the Tango software tool of the Channel 5 software package from Oxford Instruments. If the average misorientation angle in a grain exceeds 2°, the grain is considered as containing a deformed structure. Grains with an internal average misorientation angle less than 2° are identified as recrystallized. Additionally, the recrystallized volume fraction is determined as area fraction of the map covered by recrystallized grains. The crystallographic texture is represented using the orientation
distribution function (ODF) in Euler space. This function was calculated by the MTEX software package via series expansion to an expansion coefficient of 22 using a Gaussian half-width of 5° on the measured individual orientations.

3 Results and discussion
3.1 Vickers Hardness Analysis

The evolution of the Vickers hardness during isothermal annealing at 1300 °C is illustrated in Fig.1a. It can be seen that the hardness decreases dramatically during the first hours from the value 427 HV3 of the deformed state to a value of 370 HV3, where a short plateau appears in the curve. Subsequently, the hardness decreases slowly as the annealing time increases. The drastic decrease in hardness following a characteristic curve (e.g. [21]) is attributed to recrystallization; the temporary stationary state indicates completion of recrystallization from which the time for complete recrystallization is estimated to about 5 h; the subsequent slow decrease in hardness is induced by grain growth. The recrystallized volume fraction $X$ is calculated from the variation in hardness HV with annealing time (analogous to [21]) as:

$$X = \frac{HV_{def} - HV}{HV_{def} - HV_{rex}}$$

where $HV_{def}$ is the hardness value of the as-received state (427 HV3) and $HV_{rex}$ is that of the fully recrystallized state (370 HV3). The hardness of the as-received state is quite similar to that of a comparable tungsten plate warm-rolled to 67% thickness reduction [21]; the hardness of their recrystallized states differ slightly due to a different grain size after complete recrystallization.
The evolution of recrystallized volume fraction \( X \) during annealing as obtained from hardness testing using eq. (1) is shown in Fig. 1b. According to the recrystallized volume fractions obtained by hardness testing, specific samples with four different annealing times (0.5 h, 1 h, 5 h and 24 h) representing different stages of recrystallization (and grain growth) were selected for investigating the evolution of microstructure and texture by EBSD.

Fig. 1 Evolution of (a) Vickers hardness and (b) recrystallized volume fraction of warm-rolled tungsten W67 during annealing at 1300 °C.

3.2 Microstructure analysis

It is well known that the static recrystallization behavior of metals strongly depends on the initial microstructure of the deformation [19, 20]. Therefore, the microstructure of the tungsten plate after warm rolling was characterized by EBSD. Fig. 2a presents an orientation map of the as-received state obtained with a step size of 1 μm covering an area of 425×319 μm² colored according to the crystallographic directions along the normal direction of the plate. High angle boundaries with a misorientation angle above 15° are shown as black lines. The map reveals that there are
many grains with a <111> direction (in blue) or a <100> direction (in red) parallel to
the normal direction (<111>//ND or <100>//ND). Furthermore, many grains with high
local misorientation angles inside can be found, some even showing deformation bands.
In order to investigate the deformation structure, an even smaller step size is required
to reveal the details of the substructure within the grains. Fig. 2b shows a boundary map
for a smaller area of $256 \times 189 \, \mu m^2$ obtained with a finer step size of 0.5 \( \mu m \) close to the
region of Fig. 2a. An abundance of low angle boundaries (LABs) with misorientation
angles between $2^\circ$ and $15^\circ$ within the grains is characteristic for deformation structures
with many deformation-induced dislocation boundaries. Fig. 2b also reveals differences
in the density of LABs between the individual grains. Some grains exhibit large areas
with a rather low content of LABs appearing white, resembling recrystallized regions
and indicating a possible occurrence of recrystallization after the last rolling pass. In
contrast, other grains show fine and equiaxed recovered subgrains, which result from
dislocation annihilation and rearrangement during dynamic recovery.

![Fig. 2 Microstructures obtained by EBSD on the RD-ND section in the center of the warm-rolled tungsten W67 in the as-received state. (a) Orientation map coloring the crystallographic directions along the normal direction of the plate according to the inset. (b) Grain boundary map with high angle boundaries (with misorientation angles larger 15°) shown as black lines and low angle boundaries (with misorientation angles between 2° and 15°) shown as green lines.](image)
The microstructural evolution of W67 during isothermal annealing at 1300 °C is shown in Fig. 3 based on four selected annealing times (0.5 h, 1 h, 5 h and 24 h). Fig. 3a shows the microstructure of the sample annealed at 1300 °C for 0.5 h. The major part of this orientation map exhibits a large amount of deformed structure. Many non-recrystallized grains are still present. However, some recrystallized grains with different sizes are found, some of them exhibit approximately isometric shapes. Fig. 3b shows the microstructure of a sample annealed at 1300 °C for 1 h. The figure contains a large number of recrystallized grains, with only a small amount of deformed structure remaining. Fig. 3c shows the microstructure of a specimen which was annealed at 1300 °C for 5 h. The figure is mainly composed of recrystallized grains without LABs inside and no evidence for non-recrystallized grains is seen. The absence of any deformation structure indicates that complete recrystallization has occurred. The recrystallized grains are almost equiaxed with only a slight elongation along RD. Fig. 3d shows the microstructure of the sample annealed at 1300 °C for 24 h. Compared with the grains in Fig. 3c, the grains are significantly larger (cf. Table 1). Therefore, grain growth must have occurred between the annealing times of 5 h to 24 h.

The recrystallized volume fractions of the specimens annealed at 1300 °C for 0.5 h, 1 h and 5 h are evaluated from the areas covered by recrystallized grains in the representative orientation maps (see section 2). The corresponding recrystallized volume fractions of the three annealed samples are 0.30, 0.83 and 1.00, respectively. The recrystallized volume fractions obtained by hardness measurement according to eq.
are 0.35, 0.91 and 1.00 (cf. Fig. 1b). The recrystallized fractions obtained by the different measuring methods are quite similar indicating that the loss in hardness is indeed a direct consequence of the microstructural evolution during recrystallization.

Fig. 3 Orientation maps coloring the crystallographic directions along the normal direction of the specimen according to the inset for warm-rolled tungsten W67 annealed at 1300 °C for different times: (a) 0.5 h, (b) 1 h (c) 5h and (d) 24 h. High angle boundaries (with misorientation angles above 15°) shown as black lines and low angle boundaries (with misorientation angles between 2° and 15°) shown as white lines.

Fig. 4 shows the boundary misorientation angle distribution evolution of W67 during annealing at 1300 °C. A higher fraction (82%) of LABs is present in the as-received state. As recrystallization proceeds, the contribution of HABs increases while that of LABs decreases. At the later stages of annealing, the boundary misorientation
angle distribution approaches a Mackenzie distribution [30] describing the misorientation angle distribution between completely random orientations, i.e. in the absence of any texture, considering cubic symmetry. Certain deviations from the Mackenzie distribution caused by the texture of the material can still be observed. Even after annealing for 24 h, a small fraction of LABs not accounted for by the Mackenzie distribution is apparent from Fig. 4e. This is mainly caused by a small amount of deformation structure remaining in the microstructure as obvious from color variations in some of the grains in Fig. 3d.

Fig. 5 quantifies the evolution of the fraction of HABs and LABs. With an increase in annealing time, the fraction of LABs decreases, while the fraction of HABs increases correspondingly. Deformed and recovered grains contain many LABs inside, while there are only a few LABs inside almost defect-free recrystallized grains. Primary static recrystallization is defined by nucleation and growth of nuclei into the deformed or recovered substructure. More specifically, recrystallization occurs mainly through the formation and motion of HABs. Consequently, a reduction in the fraction of LABs is expected because the deformed or recovered regions become replaced by almost defect-free recrystallized grains.
Fig. 4 Boundary misorientation angle distribution of warm-rolled tungsten W67 (a) in as-received state and after annealing at 1300 °C for different times: (b) 0.5 h, (c) 1 h, (d) 5 h and (e) 24 h. The red line represents a Mackenzie distribution describing the misorientation angle distribution between completely random orientations considering cubic symmetry.

Fig. 5 Fraction of high angle boundaries on the RD/ND section of warm-rolled tungsten W67 after annealing for different times at 1300 °C.
Fig. 6 shows the grain size reported as equivalent circular diameter (ECD) as well as the aspect ratio (AR) of the individual grains identified in the orientation maps of the as-received and annealed specimens. Deformed and recrystallized grains are distinguished by their average internal misorientation angle being above or below 2°, respectively. Deformed grains in the as-received state (Fig. 6a) are mainly small having an ECD below 10 µm, with a few exceptions showing an ECD up to 75 µm. Most grains are far from equiaxed and quite large aspect ratios up to 8.1 are observed as expected from the thickness reduction by 67% corresponding to a theoretical aspect ratio of 9.

Fig. 6b and 6c show the size and aspect ratio of deformed and recrystallized grains in the partially recrystallized samples. The number of deformed grains with grain sizes below 30 µm and aspect ratios between 5 and 9 increases in the sample annealed for 0.5 h (Fig. 6b), but decreases in the sample annealed for 1 h (Fig. 6c). As the annealing time increases, the size of the deformed grains gradually decreases and very large deformed grains disappear. Recrystallized grains in the early stage of recrystallization (for the sample annealed for 0.5 h) (Fig. 6b) are mainly small and have an ECD below 10 µm, with a few exceptions showing an ECD up to 55 µm. Most recrystallized grains have aspect ratios below 3.5. Even in the later stage of recrystallization (Fig. 6c) many small recrystallized grains with sizes less than 10 µm are observed. The proportion of grains with an ECD above 30 µm is higher in Fig. 6c than in Fig. 6b. Figs. 6d and 6e show the size and aspect ratio of deformed and recrystallized grains in the fully recrystallized samples. The number of recrystallized grains appears to be decreasing while their size increases gradually. The ECD of recrystallized grains in Fig. 6d is
mainly below 65 µm except for two larger grains. Due to the growth of the grains, the number of grains with an ECD larger than 65 µm is significantly increased in Fig. 6e. As the annealing time increases, the aspect ratios of the recrystallized grains in Figs. 6c to 6e remain essentially below 3.5, except for a few individual grains with a larger aspect ratio.

*Fig. 6 Equivalent circle diameter and aspect ratio of all individual grains identified in the orientation maps of (a) the as-received warm-rolled tungsten W67 and specimens after annealing at 1300 °C for (b) 0.5 h, (c) 1 h, (d) 5 h and (e) for 24 h. Deformed*
and recrystallized grains are distinguished by their average internal misorientation angle being above or below 2°, respectively.

The statistical measures for the distributions of ECD and AR of deformed and recrystallized grains in different states are summarized in Table 1 for convenience. As expected the average AR of recrystallized grains is in general lower than that of deformed grains. As the annealing time increases, the average ECD of the deformed grains decreases, but that of the recrystallized grains increases. The average AR of the deformed grains increases slightly first and then decreases as the annealing time increases. The average AR of the recrystallized grains remained essentially unchanged during the recrystallization stage. According to Table 1, the variation coefficient of the ECD distribution decreases for both types of grains, deformed and recrystallized grains, as the annealing time increases, which indicates that the grain size distribution of deformed grains in the as-received state is relatively wide and that the size distributions of both, the deformed and the recrystallized grains, becomes more and more narrow during recrystallization. Between the two largest annealing times, the variation coefficient for recrystallized grains remains almost unaltered, indicating self-similarity and scaling during grain growth (c.f. [19]).
Table 1 Statistical measures (average value, standard deviation of the distributions, and variation coefficients characterizing the dispersion of the distribution) for grain size (ECD) and aspect ratio (AR) distribution of all deformed and recrystallized grains identified in different states of W67.

<table>
<thead>
<tr>
<th>Grain type</th>
<th>State</th>
<th>Number of detected grains</th>
<th>Average ECD ($\mu_{ECD}$) [μm]</th>
<th>Standard Deviation ($\sigma_{ECD}$) [μm]</th>
<th>Variation coeffic. ($\sigma_{ECD}/\mu_{ECD}$) ($c_{ECD}$)</th>
<th>Average aspect ratio ($\mu_{AR}$)</th>
<th>Standard Deviation ($\sigma_{AR}$)</th>
<th>Variation coeffic. ($\sigma_{AR}/\mu_{AR}$) ($c_{AR}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deformed grains</td>
<td>As received</td>
<td>364</td>
<td>9.6</td>
<td>12.3</td>
<td>1.28</td>
<td>2.6</td>
<td>1.5</td>
<td>0.56</td>
</tr>
<tr>
<td></td>
<td>1300 °C-0.5 h</td>
<td>328</td>
<td>7.6</td>
<td>7.9</td>
<td>1.04</td>
<td>2.9</td>
<td>1.6</td>
<td>0.55</td>
</tr>
<tr>
<td></td>
<td>1300 °C-1 h</td>
<td>127</td>
<td>4.8</td>
<td>4.6</td>
<td>0.94</td>
<td>2.4</td>
<td>1.1</td>
<td>0.47</td>
</tr>
<tr>
<td>Recrystallized grains</td>
<td>1300 °C-0.5 h</td>
<td>304</td>
<td>7.3</td>
<td>7.2</td>
<td>1.00</td>
<td>1.9</td>
<td>0.8</td>
<td>0.42</td>
</tr>
<tr>
<td></td>
<td>1300 °C-1 h</td>
<td>221</td>
<td>14.2</td>
<td>13.8</td>
<td>0.98</td>
<td>1.8</td>
<td>0.6</td>
<td>0.33</td>
</tr>
<tr>
<td></td>
<td>1300 °C-5 h</td>
<td>92</td>
<td>28.4</td>
<td>19.0</td>
<td>0.67</td>
<td>1.9</td>
<td>0.7</td>
<td>0.38</td>
</tr>
<tr>
<td></td>
<td>1300 °C-24 h</td>
<td>47</td>
<td>38.2</td>
<td>26.3</td>
<td>0.69</td>
<td>2.0</td>
<td>0.7</td>
<td>0.36</td>
</tr>
</tbody>
</table>

Fig. 7 shows the fraction of the deformed and recrystallized area occupied by grains of different sizes for respectively deformed or recrystallized grains in W67 after different annealing times at 1300 °C, i.e. Fig. 7a shows the distribution function of deformed grains in the regions still identified as deformed, whereas Fig. 7b presents the same for the recrystallized grains in the recrystallized regions. With the increase of annealing time, the area fraction occupied by small deformed grains of all the regions still identified as being deformed increases (Fig. 7a), while the area fraction of small recrystallized grains in all recrystallized regions decreases (Fig. 7b).
Fig. 7 Distributions of equivalent circle diameter of (a) deformed grains in the deformed part and (b) recrystallized grains in the recrystallized part for the as-received W67 and specimens after annealing at 1300 °C for different amounts of times.

As annealing proceeds, smaller recrystallized grains gradually grow (causing the increase in area fraction of larger recrystallized grains in Fig. 7b) and elongated deformed grains are gradually consumed. Individual parts (fragments) of larger grains become separated from each other by relatively equiaxed recrystallized grains formed at former grain boundaries between deformed grains (leading to an increase in area fraction of small deformed grains with annealing time in Fig. 7a). This is most likely the reason for the apparent decrease in the ECD of the deformed grains and the observed transient increase in the aspect ratio of the deformed grains (Fig. 6b and Table 1).

Fig. 8 shows the spacing of high angle boundaries along ND on the RD-ND section of W67 in as-received state and after annealing for different times at 1300 °C. As the annealing time increases, the HAB spacing decreases slightly and then increases gradually. Fragmented deformed grains and newly formed recrystallized grains cause
the HAB spacing to become smaller at the initial stage of annealing. In the later stages of annealing, the behavior of the recrystallized grains dominates the evolution of the HAB spacings: small recrystallized grains begin to grow during recrystallization and coarsen during grain growth, whereas only a few (small) deformed grains survive in the small region not consumed by recrystallization yet.

![Graph showing spacing between high angle boundaries](image)

**Fig. 8** Spacing between high angle boundaries (i.e. with misorientation above 15°) along ND on the RD-ND section of W67 in the as-received state and after annealing for different times at 1300 °C (obtained from orientation maps in Fig. 2a and Fig. 3).

The bars represent the accuracy of the averages as determined by the standard deviation of the averages.

The warm-rolled tungsten plate investigated here recrystallized at 1300 °C faster than expected from another pure tungsten warm-rolled to the same thickness reduction showing even a significant incubation time before onset of recrystallization [21]. Such a difference in the behavior illustrates the relevance of the rolling process and potential post-dynamic restauration after the last rolling pass. Recrystallization nuclei created by recovery during or after warm-rolling may initiate recrystallization without incubation.
at high temperatures and impede the material properties of a plasma-facing component
during operation.

3.3 Texture Evolution

Fig. 9 presents the $\varphi_2 = 45^\circ$ section of the orientation distribution function (ODF)
from as-received W67 and samples annealed at 1300 °C for different annealing times.
It can be observed that the texture of the material resembles typical rolling textures for
body-centered cubic materials consisting of $\theta$ ($<001>$/ND), $\gamma$ ($<111>$/ND), and $\alpha$
($<110>$/RD) fibers [19, 31, 32]. In the as-received condition, a dominating $\gamma$-fiber, a
less pronounced $\theta$-fiber and a weak $\alpha$-fiber texture component are observed. Relatively
high orientation densities are observed for the texture components along the $\gamma$-fiber for
the annealed conditions as well. The overall orientation density is not very strong,
which may be related to the relatively high (warm) rolling temperature. In addition to
the $\theta$-fiber, $\gamma$-fiber and $\alpha$-fiber texture components, many other diffuse texture
components are found in these ODF sections. This indicates that both the original and
recrystallized textures are weak. The highest orientation density in the recrystallized
samples is larger than that of the original material and the overall texture strength is
enhanced after recrystallization.

The texture index [33] and entropy [34] is the most frequently used measures to
quantify the overall texture strength in quantitative texture analysis. The texture index
is mainly used to compare the overall texture of samples in a series, the texture entropy
quantifies the deviation from a random orientation distribution. Both quantities, texture
index and entropy, are calculated from the orientation distribution function (ODF). The volume fractions of different texture components were calculated based on the individual orientations in the map. Table 2 presents the evolution of the volume fraction of the main fiber texture components (α-fiber, γ-fiber and θ-fiber) as a function of the annealing time. Overall, as the annealing time increases, the θ-fiber texture increases significantly, the α-fiber texture increases slightly and the γ-fiber texture does not seem to change much. In general, the texture index and the maximum orientation density increase and the entropy decreases (i.e. becomes more negative) as annealing time increases. This indicates that the texture of the as-received tungsten plate is more random than after recrystallization and that the texture becomes slightly stronger during recrystallization. The observed slight loss of texture strength as seen by a decrease in texture index and maximum orientation density and regain in texture entropy during annealing at 1300 °C between 5 h and 24 h, i.e. during grain growth, might be caused by orientation-independent growth, but annealing for a longer periods would be required to substantiate this observation.

The higher volume fraction of the θ-fiber texture component after recrystallization results in an increased fraction of grains having a {100} plane parallel to the rolling plane for the tungsten plate considered here. The higher frequency of such low-indexed crystallographic planes (compared to {111} planes resulting from a γ-fiber texture) should increase the radiation resistance of the plate (cf. [18]), if the rolling plane becomes the plasma-facing surface normal to the particle flux. It must be noted, however, that radiation resistance is not solely determined by grain orientation and
further investigations will be required to substantiate the claim.

Fig. 9 ODF section ($\phi_2=45^\circ$) of (a) the as-received warm-rolled tungsten W67 and specimens after annealing at 1300 °C for (b) 0.5 h, (c) 1 h, (d) 5 h and (e) for 24 h (determined from large orientation maps). (f) Schematic illustration of important texture components in body-centered cubic materials (from [19]).

Table 2 Quantitative texture parameters for the as-received condition of warm-rolled tungsten W67 and specimens after annealing at 1300 °C for different times. The corresponding numbers for a completely random orientation distribution are given for comparison purposes.

<table>
<thead>
<tr>
<th>Sample state</th>
<th>Volume fraction (%)</th>
<th>Texture index</th>
<th>Entropy</th>
<th>Max orientation density</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0-fiber</td>
<td>$\alpha$-fiber</td>
<td>$\gamma$-fiber</td>
<td></td>
</tr>
<tr>
<td>As-received</td>
<td>13.5</td>
<td>21.6</td>
<td>20.9</td>
<td>1.19</td>
</tr>
<tr>
<td>1300 °C-0.5 h</td>
<td>16.0</td>
<td>27.6</td>
<td>20.9</td>
<td>1.29</td>
</tr>
<tr>
<td>1300 °C-1 h</td>
<td>21.1</td>
<td>28.7</td>
<td>16.1</td>
<td>1.35</td>
</tr>
<tr>
<td>1300 °C-5 h</td>
<td>20.0</td>
<td>22.2</td>
<td>23.0</td>
<td>1.79</td>
</tr>
<tr>
<td>1300 °C-24 h</td>
<td>19.3</td>
<td>24.5</td>
<td>21.0</td>
<td>1.64</td>
</tr>
<tr>
<td>Random texture</td>
<td>10.0</td>
<td>19.7</td>
<td>13.2</td>
<td>1.00</td>
</tr>
</tbody>
</table>

4. Conclusions
The annealing behavior of pure tungsten warm-rolled to 67% thickness reduction was studied by Vickers hardness testing and EBSD analysis. Analysis of the evolution of microstructure and mechanical properties shows that W67 first undergoes fast discontinuous recrystallization during static annealing at 1300 °C. With progressing recrystallization, the elongated deformed microstructure is replaced by recrystallized grains with a lower aspect ratio. After completing recrystallization, the recrystallized grains begin to grow as the annealing continues. The grain size statistics shows that the size distribution of both types of grains, the deformed grains and the recrystallized grains, becomes sharper during recrystallization and stays self-similar during grain growth as seen from a constant variation coefficient. The average aspect ratio of recrystallized grains remained almost constant, whereas the deformed grains went through a transient increase. The results of the quantitative texture analysis showed that the overall texture of the warm rolled tungsten is in general weak. Nevertheless, during recrystallization the fraction of the θ-fiber texture component increases compared to the as-received condition, resulting in an increase in the overall texture strength after recrystallization and an alignment of a low-indexed crystallographic plane with the rolling plane considered beneficial for resistance against blistering and radiation damage. For the tungsten plate investigated here, recrystallization occurs particularly fast due to an absence of any incubation time compared to other tungsten material warm-rolled to the same thickness reduction. Manufacturing of warm-rolled tungsten for plasma-facing components should therefore aim to avoid formation of recrystallization nuclei during both, rolling at high temperatures and the subsequent
cooling process.

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References


