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Reciprocal and recrystallization kinetics of differently rolled, thin tungsten plates in the temperature range from 1325 °C to 1400 °C

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ABSTRACT

The thermal stability of thin tungsten plates of four different thicknesses achieved by warm- and (in two cases) cold-rolling is investigated in the temperature range between 1325 °C to 1400 °C. Hardness testing of annealed specimens allows tracking the degradation of the mechanical properties and, indirectly, the involved restoration processes during the microstructural evolution. For all four tungsten plates, a concise description of both, isothermal and isochronal annealing treatments is achieved using well-established models for the kinetics of recovery and recrystallization. A systematic dependence of the recovery kinetics at different temperatures on the hardness loss during recovery at a particular temperature has been identified. For the recrystallization kinetics, an Avrami exponent of 2 is observed in general. On the other hand, the activation energies revealed for different characteristic times of the recrystallization process depend on the plate thickness (or more precisely, the defect density stored). These findings, in particular, an activation energy far below that of short circuit diffusion, indicate the possibility of persisting nucleation throughout annealing.

1. Introduction

On the path to utilize fusion energy in future power plants, one of the toughest hurdles yet to be overcome is the development of armor materials for the plasma facing components of the first wall and the divertor. For the latter, operational requirements call for improvements in thermal stability [1] such that a desired lifetime of at least two years can be achieved [2]. The armor materials face persistent high heat and particles fluxes, demanding extraordinary material properties to resist heat and mechanical stresses. This resistance has to combine with exceptional thermal conductivity, particularly at the designed high operation temperatures, in order to fulfil the purpose of fast heat transport. Accounting for the requirement for low-activation materials [3], tungsten is considered the most promising choice for plasma facing components in terms of high thermal conductivity, strength, creep resistance and low sputtering yield [4–6]. On the contrary, tungsten is in general brittle at room temperature [5,7–10], although severe plastic deformation considerably reduces the ductile-to-brittle transition temperature [5,11].

Improving the mechanical behaviour of tungsten by plastic deformation though is not free of complications. When kept at high temperatures as in operating fusion reactors, plastically deformed tungsten will undergo restoration processes: recovery, recrystallization and grain growth will occur and alter the microstructure. These phenomena, in particular recrystallization, restore the intrinsic brittleness of annealed tungsten by replacing the beneficial deformation structure with an equiaxed, almost defect-free new grain structure. Different amounts of plastic work done to the material lead to a different stored energy in the material in the form of defects and correspondingly to a different tendency of the material to recrystallize. Considering that a certain ductility (introduced by plastic deformation) and a sufficient thermal stability (to maintain the deformation structure) both are required for tungsten to be applied as plasma facing material, a thorough understanding of their mutual interrelation and their dependence on the plastic deformation is of paramount importance. In extension of earlier investigations on plates [12–14] and foils [15,16], the thermal stability of rolled, thin tungsten plates (TPs) is characterised with focus on recovery and primary recrystallization. Following their microstructural assessment in the as-received state as well as after isothermal annealing [17], new data on isochronal annealing are presented. Based on the obtained isochronal data the temperature range between 1300 °C and 1400 °C is determined as a decent compromise between the desired operational temperatures and the feasibility of performing annealing experiments (with reasonably short annealing times to observe the above-mentioned phenomena). A quantitative description of all annealing treatments is provided in terms

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of sound and well-established models for the evolution of recovery and recrystallization.

2. Materials and Methods

Four plates (100 mm × 250 mm cut from larger pieces) of four different thicknesses of 99.97% technically pure tungsten [18] were acquired from Plansee SE (Reutte, Austria). The two thicker plates (2 mm (TP2) and 1 mm (TP1)) were manufactured solely by warm-rolling, whereas the two plates with smaller thicknesses (0.5 mm (TP0.5) and 0.2 mm (TP0.2)) experienced final cold-rolling steps.

The as-received condition of the thin plates had been investigated previously [17] as summarized in Table 1. The microhardness of the as-received condition reveals an increasing hardness with decreasing thickness of the plates with exception of TP1 showing the lowest hardness of the batch. Microstructurally, with exception of TP1, the TP s show the typical characteristics of rolled metals: increasing grain elongation along the rolling direction and decreasing chord length along the normal direction (from 538 nm (TP2) to 327 nm (TP0.5) and 229 nm (TP0.2) with exceptional 564 nm for TP1), but also an increasing volume fraction of the rotated cube texture component {100} [011] with increasing thickness reduction. The details of the more elaborate microstructural analysis including an assessment of the heterogeneity of the plates is reported in [17]. Smaller specimens were cut from the plates, encapsulated in quartz glass ampoules in an argon atmosphere and annealed in a general-purpose tube furnace NaberTherm RHTC 80-230/15. Details on the production route, sample sizes and cutting, protective atmosphere encapsulation, direction identification and impurity content are reported in [17].

Different series of annealing experiments were performed on the TP s. The new isochronal series with a constant annealing time of 2 h at temperatures ranging from 800 °C to 1450 °C allowed to assess their recovery behaviour and to confirm the interesting temperature range for isothermal annealing. Isothermal experiments were formerly performed at five different temperatures between 1300 °C and 1400 °C and reported in [17]. Out of these five isothermal experiments, three series performed with annealing times up to 67 h at the temperatures of 1325 °C, 1375 °C and 1400 °C are taken into account for evaluation of the recrystallization kinetics.

Vickers hardness measurements with load 0.5 kg and dwell time 10 s were performed on the surface parallel to the rolling plane, i.e. containing the rolling and the transversal direction. Due to the small thickness of the thin plates, only this surface allowed performing a sufficient number of hardness indents (at least 10) for performing a statistically sound analysis.

### 3. Results

#### 3.1. Isochronal annealing and model description of recovery kinetics

To assess the relevant temperature range for isothermal experiments, isochronal annealing experiments for 2 h were performed at temperatures from 800 °C to 1450 °C. With increasing annealing temperature more pronounced hardness reductions are observed (as seen in Fig. 1). In general, two different regimes can be distinguished: the (almost) linear decrease of the hardness with temperature observed at lower temperatures is attributed to recovery, whereas the more pronounced hardness decrease at higher temperatures indicates onset of recrystallization.

In modelling the recovery behaviour, Kuhlmann's recovery model [19,20] based on thermal-activated dislocation motion was employed. According to this interpretation, recovery kinetics is governed by an Arrhenius-like relationship for the evolution of the resistance $\sigma$ against dislocation motion (for not too low $\sigma$)

<table>
<thead>
<tr>
<th>Plate</th>
<th>As-received condition</th>
<th>Hardness at first stagnation (late recovery)</th>
<th>Hardness at second stagnation (full recrystallization)</th>
<th>Average loss during recovery</th>
<th>Average loss during recrystallization</th>
<th>Average total loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>TP2</td>
<td>541 ± 2</td>
<td>507 ± 2</td>
<td>496 ± 4</td>
<td>504*</td>
<td>449 ± 2</td>
<td>479 ± 1</td>
</tr>
<tr>
<td>TP0.5</td>
<td>595 ± 3</td>
<td>500 ± 9</td>
<td>479 ± 2</td>
<td>501*</td>
<td>414 ± 4</td>
<td>445 ± 2</td>
</tr>
<tr>
<td>TP0.2</td>
<td>642 ± 2</td>
<td>479 ± 2</td>
<td>477 ± 8</td>
<td>468*</td>
<td>415 ± 4</td>
<td>432 ± 2</td>
</tr>
</tbody>
</table>

$\Delta H/V_{\text{rec}}$ is an average between all three temperatures. Therefore, the individual losses during recovery and recrystallization ($\Delta H/V_{\text{rec}}$ and $\Delta H/V_{\text{rex}}$, respectively) do not add exactly to the total loss ($\Delta H/V_{\text{total}}$).
\[
\frac{dQ}{dt} = -c \exp\left(-Q_0 - \sigma \Delta V \right)
\]

with an effective activation energy \( Q_0 - \sigma \Delta V \) lowered from the intrinsic activation energy \( Q_0 \) of recovery by the resistance \( \sigma \) through an activation volume \( \Delta V \). The activation volume \( \Delta V = \frac{b_{\text{Burgers}} \Delta A_{\text{act}}}{b_{\text{Burgers}}} \) quantifies the area \( \Delta A_{\text{act}} \) swept by a dislocation with Burgers vector \( b_{\text{Burgers}} \) if released from an obstacle by thermal activation. Due to the proportionality \( HV = Ce \) between Vickers hardness and flow stress (with \( C \approx 3 \) [21]), an equivalent behaviour of the hardness is expected.

Kuhlmann’s evolution Eq. (1) can be integrated in time,

\[
HV = HV_0 - \frac{CRT}{\Delta V} \ln \left(1 + \frac{t}{t_0}\right)
\]

leading for not too short times \( t \) to a logarithmic dependence of the hardness

\[
HV = HV_0 - \frac{CRT}{\Delta V} \ln(t)
\]

on the annealing time \( t \). For a constant annealing time \( t = t_{\text{isochr}} \) as in the isochronal experiments, Eq. (3) describes an almost linear decrease in hardness with annealing temperature \( T \)

\[
HV = HV_0 - AT
\]

with a slope

\[
A = \frac{CR}{\Delta V} \ln(t_{\text{isochr}}).
\]

Note, that the integration constant

\( t_0 = (RT/c\Delta V)\exp(-(Q_0 - \sigma \Delta V)/RT) \) in the exact Eq. (2) causes a weak additional temperature dependence of the effective initial hardness value \( HV_0^* \) and slight deviations from linearity for large temperature variations.

The lowest temperature for which a significant loss of hardness (with respect to the as-received condition) due to recovery can be noticed differs strongly between the thin plates, ranging from 1100 °C for the TP2 to 800 °C for TP0.2. For fitting of the linear temperature dependence according to Eq. (4) to the obtained hardness values after annealing for 2h, hardness values from lower annealing temperatures which were within a standard deviation of the as-received material hardness were excluded. Also, measurements belonging to a condition where recrystallization already set in are not considered in the analysis, such as that of TP2 for 2 h at 1325 °C in Fig. 1a, clearly belonging to the regime of more pronounced hardness decrease. For all four thin plates a good fit of the linear temperature dependence of Eq. (4) is obtained in specific temperature intervals as seen from Fig. 1. For example, for TP2 the model description fits the data in the temperature interval from 1100 °C to 1350 °C very well \( (R^2 = 0.95) \). The obtained values for the back-extrapolated initial hardness \( HV_0^* \) and the recovery coefficient \( A \) are summarized in Table 2. Both, the back-extrapolated initial hardness and the recovery coefficient, increase with decreasing thickness but for TP1, showing the lowest values for both the parameters. From the results of these isochronal experiments, it appeared most appropriate to perform isothermal annealing treatments in the temperature range between 1300 °C and 1400 °C for longer periods of time.

3.2. Isothermal annealing and model description of recrystallization kinetics

Three isothermal annealing experiments performed at 1325 °C,
\[ VM = X V n b n t t \ln(\ln(1)) \ln(\ln(1)) \]

and recrystallization

\[ HV = X V n b n t t \]

\[ HV_{\text{total}} = HV_{\text{recovery}} + HV_{\text{rec}} \]

\[ \Delta HV_{\text{rec}} = HV_{\text{rec}} - HV_{\text{as-received}} \]

\[ \Delta HV_{\text{rec}} = HV_{\text{recovery}} - HV_{\text{as-received}} \]

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\[ \Delta HV_{\text{rec}} = HV_{\text{recovery}} - HV_{\text{as-received}} \]
Evolution of the recrystallized fraction in thin tungsten plates during isothermal annealing at different temperatures: (a) TP2 at 1325 °C, (b) TP1 at 1325 °C, (c) TP0.5 at 1325 °C, (d) TP0.2 at 1325 °C, (e) TP2 at 1400 °C, and (f) TP0.2 at 1400 °C. The dashed line represents a fit of the modified JMAK model according to Eq. (6) with fixed Avrami exponent $n$ of 2. The obtained fitting parameters are listed in Table 3.
In the cases where there is a recognizable incubation period in the hardness evolution, the incubation times follow the same trend as the rate coefficients $b$ (and show a deviating behaviour for TP1 as well). For annealing of TP0.5 for 1375 °C and the entire isothermal annealing at 1400 °C, no evidence for an incubation time necessary to describe the kinetics was found (as recrystallization onsets immediately, cf. Figs. 3e and f, for TP2 and TP0.2, respectively) and a vanishing incubation period was assumed.

As seen in Fig. 3 for quite different starting and annealing conditions, application of the listed parameters in Table 3 resulted in a rather good description of the datasets. From the governing evolution Eq. (6), the times to achieve half-recrystallization (i.e. $X_V = 0.5$) are inferred finally:

$$t_{X_V = 0.5} = \frac{1}{b} \ln\left(1 - X_V\right) + t_{inc} = \frac{1}{b} \ln\left(2\right) + t_{inc}. \quad (9)$$

In this manner, three characteristic times describing the duration of thermally activated processes are obtained: the time to half recrystallization $t_{X_V = 0.5}$, the incubation time $t_{inc}$ and the inverse of the rate coefficient $b^{-1}$, describing the combined effect of nucleation and growth of recrystallizing grains. For all three characteristic times, a temperature dependence according to an Arrhenius law

$$t = t^\text{exp}\left(\frac{Q}{RT}\right) \quad (10)$$

is assumed allowing determination of their corresponding activation energies $Q$.

As shown in Fig. 5 and summarized in Table 3, the Arrhenius plots (semi-logarithmic plots of the time to half recrystallization or $b^{-1}$ vs. the inverse of the absolute annealing temperature) resulted in two different ranges for the activation energies: in the case of the times to half recrystallization ranging from $363 \pm 47 \text{ kJ/mol}$ for TP0.2 to $569 \pm 27 \text{ kJ/mol}$ for TP1, while in the case of the coefficients $b$ from $235 \pm 75 \text{ kJ/mol}$ for TP2 to $285 \pm 142 \text{ kJ/mol}$ for TP0.5.

4. Discussion

4.1. Recovery behaviour

Probing the as-received state by hardness testing should reflect the increasing plastic deformation of the material with decreasing thickness through an increased dislocation density stored in the material, causing an increased Vickers hardness of the thinner plates. This expectation is satisfied in general, disrupted though by the too low hardness of TP1 only. The deviating behaviour of TP1 has been further substantiated by inspecting the microstructure of the as-received state using EBSD[17] and attributed to eventual recrystallization during or between the warm-rolling passes rationalizing the similar grain size of TP1 and TP2 along ND (564 nm and 538 nm, respectively [17]). Peculiar differences in the behaviour of TP1 are also revealed in the recovery kinetics analysed here: the recovery coefficient $A$ of TP1 is the smallest between all the investigated thin plates (see Table 2).

According to Kuhlmann’s model, changes in hardness during recovery are not simply proportional to the hardness after plastic deformation. The present hardness value rather determines the further hardness loss by a reduction in activation energy for recovery from the materials intrinsic value $Q_0$ through an activation volume $\Delta V$ (cf. Eq. (1)). The recovery coefficient $A$ (defined in Eq. (5)) is related to the microstructure present in the material in the as-received state through the activation volume $\Delta V$ due to the presence of a dislocation structure as a result of plastic deformation. An increased dislocation content in the material implies a smaller spacing between dislocations. Dislocations with Burgers vector $b_{\text{Burgers}}$ released in a thermal-activated event will travel only a shorter distance, pass through a smaller area $\Delta A_{\text{inc}}$, and hence have a smaller activation volume $\Delta V = b_{\text{Burgers}} \Delta A_{\text{inc}}$. The smaller activation volume will in turn lead to a faster recovery kinetics and a higher recovery coefficient $A$. Consequently, larger plastic deformation should indirectly lead to a larger recovery coefficient $A$. As discussed earlier, the final plate thickness cannot be used as a measure for the plastic deformation as the resulting hardness does not depend systematically on the plate thickness (due to the observed deviations for TP1). Instead the recoverable increase in hardness due to deformation, corresponding to the hardness loss $\Delta H_{\text{inc}}$ during recovery up to the first stagnation value, is used as a measure for the induced plastic deformation and, indirectly, the stored dislocation density. Relating the recovery coefficient $A$ and the hardness loss during recovery for all four plates, as in Fig. 6, for an annealing temperature of 1325 °C, reveals a systematic linear dependence. (A similar linear dependence is observed when relating the coefficient $A$ to the average hardness loss during recovery between all three investigated temperatures as the values differ only slightly as seen in Table 1.) This non-trivial relationship

<table>
<thead>
<tr>
<th>Plate</th>
<th>Incubation time $t_{inc}$</th>
<th>Rate coefficient $b$</th>
<th>$Q_{0.5}$</th>
<th>$Q_0$</th>
<th>Maximum possible temperature for 2 fpy</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1325 °C</td>
<td>1375 °C</td>
<td>1400 °C</td>
<td>1325 °C</td>
<td>1375 °C</td>
</tr>
<tr>
<td>TP2</td>
<td>10.7 ± 0.1</td>
<td>3.0 ± 0.7</td>
<td>0</td>
<td>0.153 ± 0.005</td>
<td>0.319 ± 0.097</td>
</tr>
<tr>
<td>TP1</td>
<td>10.8 ± 0.4</td>
<td>1.6 ± 1.5</td>
<td>0</td>
<td>0.168 ± 0.037</td>
<td>0.347 ± 0.173</td>
</tr>
<tr>
<td>TP0.5</td>
<td>7.8 ± 0.3</td>
<td>2.5 ± 0.2</td>
<td>0</td>
<td>0.111 ± 0.013</td>
<td>0.306 ± 0.024</td>
</tr>
<tr>
<td>TP0.2</td>
<td>3.2 ± 0.7</td>
<td>0.2 ± 4.9</td>
<td>0</td>
<td>0.074 ± 0.050</td>
<td>0.120 ± 0.011</td>
</tr>
</tbody>
</table>

As shown in Fig. 3 for quite different starting and annealing conditions, application of the listed parameters in Table 3 resulted in a rather good description of the datasets. From the governing evolution Eq. (6), the times to achieve half-recrystallization (i.e. $X_V = 0.5$) are inferred finally:

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4.1. Recovery behaviour

Probing the as-received state by hardness testing should reflect the increasing plastic deformation of the material with decreasing thickness through an increased dislocation density stored in the material, causing an increased Vickers hardness of the thinner plates. This expectation is satisfied in general, disrupted though by the too low hardness of TP1 only. The deviating behaviour of TP1 has been further substantiated by inspecting the microstructure of the as-received state using EBSD[17] and attributed to eventual recrystallization during or between the warm-rolling passes rationalizing the similar grain size of TP1 and TP2 along ND (564 nm and 538 nm, respectively [17]). Peculiar differences in the behaviour of TP1 are also revealed in the recovery kinetics analysed here: the recovery coefficient $A$ of TP1 is the smallest between all the investigated thin plates (see Table 2).

According to Kuhlmann’s model, changes in hardness during recovery are not simply proportional to the hardness after plastic deformation. The present hardness value rather determines the further hardness loss by a reduction in activation energy for recovery from the materials intrinsic value $Q_0$ through an activation volume $\Delta V$ (cf. Eq. (1)). The recovery coefficient $A$ (defined in Eq. (5)) is related to the microstructure present in the material in the as-received state through the activation volume $\Delta V$ due to the presence of a dislocation structure as a result of plastic deformation. An increased dislocation content in the material implies a smaller spacing between dislocations. Dislocations with Burgers vector $b_{\text{Burgers}}$ released in a thermal-activated event will travel only a shorter distance, pass through a smaller area $\Delta A_{\text{inc}}$, and hence have a smaller activation volume $\Delta V = b_{\text{Burgers}} \Delta A_{\text{inc}}$. The smaller activation volume will in turn lead to a faster recovery kinetics and a higher recovery coefficient $A$. Consequently, larger plastic deformation should indirectly lead to a larger recovery coefficient $A$. As discussed earlier, the final plate thickness cannot be used as a measure for the plastic deformation as the resulting hardness does not depend systematically on the plate thickness (due to the observed deviations for TP1). Instead the recoverable increase in hardness due to deformation, corresponding to the hardness loss $\Delta H_{\text{inc}}$ during recovery up to the first stagnation value, is used as a measure for the induced plastic deformation and, indirectly, the stored dislocation density. Relating the recovery coefficient $A$ and the hardness loss during recovery for all four plates, as in Fig. 6, for an annealing temperature of 1325 °C, reveals a systematic linear dependence. (A similar linear dependence is observed when relating the coefficient $A$ to the average hardness loss during recovery between all three investigated temperatures as the values differ only slightly as seen in Table 1.)
roots in the reduction of the activation volume due to an increased defect content (dislocation density). Remarkably, even the data from TP1 follow the same linear relationship, indicating that it is indeed the microstructure of the as-received state, rather than the thickness reduction, which is governing the annealing behaviour.

In view of this correspondence between the hardness loss during recovery and the recovery kinetics, all other findings concerning the hardness in the as-received condition and even all microstructural information appear consistent: the higher the induced defect density by rolling, the higher the initial hardness, the higher the hardness loss achievable during recovery, the lower the activation volume for recovery and the faster the recovery kinetics. This general trend follows the reduction in plate thickness except for the deviating behaviour of TP1, manifested in a less deformed microstructure, a lower initial hardness after rolling and a relatively higher activation volume for recovery compared to TP2. This is very likely caused by a lower work-hardening experienced by TP1 which could be possibly a consequence of dynamic recrystallization of TP1 occurring during one of the warm-rolling passes or static recrystallization at the elevated temperatures between the passes.

4.2. Recrystallization behaviour

The observed recrystallization kinetics is concisely described by the JMAK model with an Avrami exponent of 2 and different activation energies for the different characteristic times governing the recrystallization process. It should be noted that the two solely warm-rolled plates, TP2 and TP1, as well as the two plates with final cold-rolling, TP0.5 and TP0.2, show pairwise quite similar activation energies. In the case of the time to half recrystallization, TP2 and TP1 showed activation energies \( Q_{\text{N}} \approx 0.5 \) comparable to the activation energy of self-diffusion in tungsten (502-586 kJ/mol) [5], while the activation energies \( Q_{\text{G}} \approx 0.5 \) of TP0.5 and TP0.2 are similar to that of short-circuit diffusion (377-460 kJ/mol) [5].

For the growth/nucleation coefficient \( b^{-1} \), on the other hand, all activation energies were found to be even below the reported range for self-diffusion. This rather surprising observation of a too low activation energy might originate from the fact that the coefficient \( b \) is actually describing the combined effect of nucleation and growth of recrystallizing grains and not a single thermal-activated process. If both processes follow individual thermal-activated events with different activation energies for nucleation and growth, \( Q_N \) and \( Q_G \), respectively, and correspondingly different Avrami exponents \( n_N \) and \( n_G \), their combined effect can be assessed (based on rate theory and [22]) in the following manner.

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**Fig. 5.** Arrhenius plots for a) the time to half recrystallization b) the inverse of the rate coefficient \( b \) allowing the evaluation of activation energies for different characteristic times of the recrystallization process.

**Fig. 6.** Recovery of thin plates during isochronal annealing: recovery coefficient \( A \) vs. hardness loss during recovery \( \Delta H_{\text{V,0.5}} \) at 1325 °C (i.e. difference between hardness of as-received condition and hardness of severely recovered condition seen as the first stagnation in hardness evolution).
The effective activation energy for the combined rate coefficient $b$

$$Q_b = \frac{n_b Q_D + n_n Q_N}{n_D + n_N}$$  \hspace{1cm} (12)

is, hence, a weighted sum of the activation energies of both, nucleation and growth. An effective Avrami coefficient $n = n_b + n_N$ of 2 can be rationalized in two different idealized manners [22]: either two-dimensional growth ($n = 2$) with site-saturated nucleation ($n_b = 0$) or quasi-one-dimensional growth ($n = 1$) with continuous nucleation with constant nucleation rate ($n_N = 1$), the corresponding activation energies being $Q_b = Q_D$ and $Q_b = (Q_D + Q_N)/2$, respectively. In the latter case, a nucleation process with comparably low and almost vanishing activation energy ($Q_N = 0$, a fair assumption at least for the heavily cold-rolled plates TP0.5 and TP0.2), would lead to $Q_b = Q_D/2$ and, hence, allow to explain values for the activation energy lower than that of self-diffusion governing the growth process. Values of the activation energy for the rate coefficient $b$ close to half of the activation energy of self-diffusion suggest such an interpretation in terms of sustained nucleation (with negligible activation energy) and quasi-one-dimensional growth.

The value for activation energy for the time to half recrystallization obtained for TP0.5 is slightly above the reported values [5] for short circuit diffusion, the one of TP0.2 slightly below. This might be a consequence of the time to half recrystallization being actually the sum of two different times describing different thermal-activated processes, the time to incubation of recrystallization and the time for the progress of recrystallization (cf. Eq. (9)). A physical interpretation of the activation energy to half recrystallization has rather to be based on the individual processes.

Analogous to the activation energy, pairwise similar hardness values for the warm- (TP2 and TP1) and cold-rolled (TP0.5 and TP0.2) thin plates were obtained in the fully recrystallized state (second stagnation in Table 1) for all three annealing temperatures. This indicates the presence of pairwise similar deformation structures before annealing with larger differences between the deformation structures after warm- and cold-working (leading also to pairwise similar hardness values in the later stages of recovery).

In general, the recrystallization behaviour of the thin plates follows the same trend as seen for the recovery behaviour: the onset of recrystallization occurs earlier (as seen from a shorter incubation time $t_{inc}$) and the kinetics (as seen from the higher rate coefficient $b$) becomes faster with higher thickness reduction and hence larger plastic deformation. The deviating behaviour of TP1 is attributed again to its initial structure with lowest hardness and largest chord length.

An interesting systematic dependence between the hardness loss during recovery and the rate coefficient $b$ for recrystallization of the different plates is revealed from Fig. 7: the higher the rate coefficient $b$ of a plate, the lower its hardness loss during recovery. (The same trend is observed for the total hardness loss, whereas the hardness loss during recrystallization is almost the same for all plates.) Considering that the nucleation/growth coefficient $b$ reflects the rate at which recrystallization takes place due to the energy stored in the deformation structure and that the hardness loss during recovery quantifies the loss of driving force for the recrystallization process due to recovery, the linear dependence $\Delta HV = \Delta HV^* - kb$ between both parameters is rationalized. The relation reflects the competition of both processes about the same driving force, i.e. the stored energy in the as-received state: A large hardness loss during recovery eliminates part of the driving force for recrystallization and causes slower recrystallization kinetics. On the other hand, a large hardness loss during recovery will cause a fast recovery kinetics as discussed above in connection with Fig. 6. Remarkable, in both cases the extraordinary behaviour of TP1 concerning its plastic deformation and related defect density as expected from the achieved thickness reduction becomes nicely rationalized when considering its lower hardness loss during recovery instead of the thickness reduction.

Finally, the concise description of the recrystallization kinetics achieved in the investigated temperature range (cf. Fig. 3) and the obtained activation energies can be used to extrapolate the recrystallization kinetics to the service temperature at which the materials are expected to operate in a fusion reactor (tacitly assuming that the same process will hold at these temperatures as well). For instance, the maximal service temperatures for which the recrystallized volume fraction in the materials will not exceed 50% during two years of service can be assessed. These temperatures (cf. Table 3) range between 996 °C for TP0.2 and 1099 °C for TP1. These extrapolated temperatures will enable the use of these thin plates as plasma-facing armor for the first wall (at an expected service temperature of 800 °C) for 2 years, but are far below the temperature expected for the plasma-facing divertor components experiencing high heat fluxes [26].

5. Conclusions

The thermal stability of four plates of pure tungsten with different thicknesses was investigated to assess the effect of progressive thickness reduction through rolling. Isothermal annealing at three different temperatures was performed up to complete primary recrystallization. The microstructural evolution during annealing was tracked by Vickers hardness testing. Well-established kinetic models for recovery and recrystallization allowed a concise description of the observed hardness evolution. The obtained kinetic parameters for the different phenomena are consistent with the expectations of an increased amount of plastic deformation and hence an increased dislocation density with increasing rolling reduction for all plates but TP1: the latter is attributed to the lower as-received hardness of TP1 caused probably by dynamic recrystallization through one of the warm-rolling passes or static recrystallization between them.

Describing the recovery kinetics by Kuhlmann’s model revealed a linear dependence of the recovery coefficient $A$ on the hardness loss during recovery for all plates (instead of the plate thickness or thickness
reduction). Description of primary recrystallization through the JMAK model yielded Avrami exponents equal to 2, incubation times and nucleation/growth coefficients $b$, all reflecting the work-hardening due to the rolling experienced by the plates and featuring a lower activation energy for recrystallization for plates which underwent a larger work-hardening. The small values for the activation energy of the nucleation and growth coefficient $b$, less than the activation energy for short-circuit diffusion, indicate a sustained nucleation with rather low activation energy. Systematic correlations between the hardness loss due to recovery, the recovery coefficient $A$ or the nucleation/growth coefficient $b$ for recrystallization were found, indicating the paramount role of the microstructure and the therein stored energy in ruling the annealing behaviour.

In general, an increased work-hardening in the tungsten plates results in a reduced thermal stability as characterized by the time to achieve recrystallization in half of the material. For an envisaged time in service of 2 years for plasma-facing components before half of the volume of the materials recrystallizes, the investigated plates are expected, from extrapolation based on the obtained kinetic parameters and activation energies, to endure service temperatures between 1000 °C and 1100 °C. These temperatures are well above those foreseen for the armor material of the first wall (800 °C), but too low compared to those simulated for the divertor implying possible application in the former case only.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declaration of Competing Interest

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

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Supplementary materials


References