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Pantleon, Wolfgang

Published in:
Physica Scripta

Link to article, DOI:
[10.1088/1402-4896/ac2854](https://doi.org/10.1088/1402-4896/ac2854)

Publication date:
2021

Document Version
Peer reviewed version

[Link back to DTU Orbit](#)

Citation (APA):
Pantleon, W. (2021). Thermal stability of the microstructure in rolled tungsten for fusion reactors. *Physica Scripta*, 96(12), Article 124036 . <https://doi.org/10.1088/1402-4896/ac2854>

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Thermal stability of the microstructure in rolled tungsten for fusion reactors

Wolfgang Pantleon¹

¹ Department of Mechanical Engineering, Technical University of Denmark, Kgs. Lyngby, Denmark

E-mail: pawo@dtu.dk

Received xxxxxx

Accepted for publication xxxxxx

Published xxxxxx

Abstract

Plasma-facing components of future fusion reactors will have tungsten-based materials as armor. Annealed pure tungsten is brittle at room temperature restricting its use as plasma-facing material, whereas plastically deformed tungsten behaves in general more ductile at ambient temperatures. During operation as plasma-facing material at high temperatures, the deformation structure induced by plastic deformation becomes unstable. Restoration processes as recovery, recrystallization, and grain growth will alter the microstructure and impair the desired mechanical properties. In particular, recrystallization will reinstate the intrinsic brittleness of tungsten. Achieving a thorough understanding of the occurring restoration mechanisms (for long times at temperatures as close to the desired operation temperatures as possible) and quantifying the temperature-dependent recrystallization kinetics are essential for assessing the materials performance and an informed materials selection. The thermal stability of differently rolled pure tungsten plates is reviewed with the aim of predicting the materials lifetime; the impact of different activation energies on the selection of armor materials highlighted. The concept of a recrystallization temperature constituting a threshold temperature below which recrystallization does not occur is dismissed.

Keywords: tungsten, thermal stability, recovery, recrystallization, recrystallization temperature

1. Introduction

Tungsten is considered as armor material for plasma facing components as the first wall of the blanket or the divertor due to its excellent properties (as the highest melting point of 3695 K of all materials) [1]. While pure tungsten behaves ductile at high temperatures, its brittleness at low temperatures impedes application. Undeformed polycrystalline tungsten shows a transition from brittle to ductile behavior in the temperature range between 200 °C and 300 °C [1]. The brittle-to-ductile transition temperature depends on the materials microstructure and can be lowered by plastic deformation (even below room temperature, cf. [2,3]) rendering the use of plastically deformed tungsten as armor of plasma-facing components advantageous.

Unfortunately, deformation structures are thermally unstable and the materials microstructure will undergo different restoration processes during operation at high temperatures: recovery, recrystallization, and grain growth (grain coarsening). The energy stored in the defects introduced by plastic deformation (mainly dislocations) constitutes the driving force for their elimination thereby restoring the properties before plastic deformation [4]:

- During recovery, the stored energy decreases gradually by mutual annihilation of defects and rearrangement of dislocations without any long-range motion of high angle boundaries.
- During recrystallization, motion of high angle boundaries eliminates the defect content in the regions swept by the moving boundaries.

- During grain growth, grains coarsen by motion of their boundaries driven by the boundary energy itself.

The microstructure of tungsten-based plasma-facing components manufactured involving any plastic deformation will not be stable for an infinite lifetime; microstructural restoration processes will occur unavoidably reinstating the brittleness of undeformed tungsten through lowering the brittle-to-ductile transition temperature. The goal of materials qualification must hence be an assessment of the expected lifetime before alterations in the microstructure become crucial. In order to facilitate the search for a material resisting microstructural changes for a certain period, ideally the kinetics of microstructural changes must be determined at the desired operation temperature. When this is infeasible, in view of the expected long operation times of several years, the faster restoration kinetics at higher annealing temperatures must be quantified thoroughly in dependence on temperature in order to allow extrapolation to expectedly lower operation temperatures. This methodology is employed on several rolled plates of pure tungsten from different manufacturers after discussing the metallurgical foundations of thermally activated restoration kinetics.

2. Background

2.1 Thermal activation

The reaction rate of thermally activated processes follows in general an Arrhenius relation

$$\dot{\Gamma} = v \exp\left(-\frac{Q}{RT}\right) \quad (1)$$

with a jump frequency v , the universal gas constant R , the absolute temperature T and an activation energy Q . The reaction rate depends strongly on temperature, but no threshold temperature exists below which a thermodynamically permissible process entirely ceases to occur. If the process occurs unaltered, the reaction progress at different temperatures is additive and obtained for any time-dependent temperature history $T(t)$ by time integration

$$\Gamma = \int_{t_0}^t \dot{\Gamma}(t') dt' = \int_{t_0}^t v \exp\left(-\frac{Q}{RT(t')}\right) dt'. \quad (2)$$

2.2 Recovery

Recovery is a thermally activated process with a characteristic slowing down of the recovery rate from an initially high rate. For neither too long, nor too short times it follows an empirically established logarithmic time dependence, e.g. for the hardness

$$HV = HV_0^* - m \ln t. \quad (3)$$

Such a slow decrease of macroscopic properties or defect densities does not comply with an exponential decay with constant activation energy [5-7] (with occasional erroneous

claims of the opposite [8]); the required activation energy rather increases in the course of annealing depending on the amount of recovery (as confirmed abundantly [5-7,9]).

Kuhlmann [5] has rationalized the logarithmic time dependence at intermediate times as thermally activated decay of the flow stress of the recovering material

$$\frac{d\sigma}{dt} = -c \exp\left(-\frac{Q_0 - \sigma \Delta V}{k_B T}\right) \quad (4)$$

with a stress-dependent activation energy $Q_0 - \sigma \Delta V$ through an activation volume ΔV with Boltzmann constant k_B and a constant pre-factor c . Taking into account the proportionality $HV = C\sigma$ between hardness and flow stress with a coefficient $C \approx 3$ [10], a hardness evolution

$$HV = HV_0 - \frac{Ck_B T}{\Delta V} \ln\left(1 + \frac{t}{t_0}\right) \approx HV_0^* - \frac{Ck_B T}{\Delta V} \ln t \quad (5)$$

is obtained reflecting (for neither too short nor too long times) the empirically observed logarithmic dependence. Accordingly, the slope $m = \partial HV / \partial \ln t$ of the temporal evolution $HV(t)$ in equation (3) is proportional to the absolute temperature T and not affected by the activation energy Q_0 , i.e. the one in the absence of any flow stress, but allows determining the temperature-independent activation volume

$$\Delta V = \frac{Ck_B T}{m}. \quad (6)$$

2.3 Recrystallization

Atomistic jumps from on one side of a boundary to the other side facilitate motion of high angle boundaries and govern recrystallization. Consequently, recrystallization becomes a thermally activated process with an activation energy similar to the activation energy of self-diffusion, either through the bulk of grains or along short circuit paths.

For a concise description of the progress of recrystallization, two different microstructural processes have to be taken into account: formation of new defect-free nuclei with nucleation rate \dot{N} and their growth with rate \dot{G} . For uniform nucleation and growth, i.e. when nuclei arise randomly distributed in the volume and the growth rate in any direction is the same for all growing grains at any instant of time, Kolmogoroff [11] has derived the recrystallized volume fraction

$$X = 1 - \exp\left(-\int_0^t \dot{N}(t') f_d \left(\int_{t'}^t \dot{G}(t'') dt''\right)^d dt'\right) \quad (7)$$

analytically with a shape factor f_d depending on the dimensionality d of the growth. The analytical result simplifies for either constant nucleation rate or site-saturated nucleation and a constant, isotropic growth rate to the Johnson-Mehl-Avrami-Kolmogoroff (JMAK) equation

$$X = 1 - \exp(-b^n t^n) \quad (8)$$

where the Avrami exponent n characterizes both, the dimensionality of the growth and the time dependence of the nucleation rate, and b constitutes an effective combined nucleation and growth rate. Taking into account a potential incubation time t_{inc} before the onset of recrystallization, the more convenient form applied here is obtained

$$X = 1 - \exp\left(-b^n (t - t_{inc})^n\right). \quad (9)$$

Qualifying the recrystallization kinetics of a material in view of their use as armor of plasma-facing components relies on the time after which recrystallization has occurred in half of the volume, i.e. the time to half recrystallization

$$t_{X=0.5} = t_{inc} + b^{-1} (\ln 2)^{1/n}. \quad (10)$$

This time to half recrystallization roots on a sequence of three thermally activated processes: incubation, nucleation and growth. There is no *a priori* reason to presume that all three processes require the same activation energy. Nevertheless, all processes occur by atomic jumps and a similar activation energy it is not entirely inconceivable, an assumption, which would have to be justified *a posteriori*.

2.4 Recrystallization temperature

Occasionally, a *recrystallization temperature* is reported as the minimum temperature at which notable recrystallization occurs (when heating continuously or after a certain time). As a rule of thumb attributed to Tamman, this *recrystallization temperature* is about 0.4 of the absolute melting temperature, i.e. about 1200 °C for tungsten. The concept of such a *recrystallization temperature* is helpful for technical processes not lasting longer than a few hours; for instance, to distinguish hot working from cold working above and below this temperature for which (dynamic) recrystallization may or may not occur during the forming process. For lasting operation at high temperatures, however, it remains a matter of time before recrystallization will commence, if there is a deformation structure present. When waiting long enough, deformation-induced defects will disappear, as they are thermodynamically metastable.

The actual *recrystallization temperature* depends on the state of the material and the entire processing history, e.g. the starting grain size, the plastic strain achieved during deformation and not at least on the annealing time investigated. Cold deformation to a larger strain increases the defect density and the driving force for recrystallization, recrystallization may occur faster and its onset becomes notable at lower temperatures.

Despite the *law of recrystallization* that “increasing the annealing time decreases the temperature for recrystallization” [12], the misleading interpretation is commonly adapted, that recrystallization does not occur below a certain temperature – in conflict with experimental evidence of the contrary, e.g. [13-15]. A threshold

temperature below which recrystallization may not occur does not exist and “the stubborn idea that a metal has a fixed recrystallization temperature in the sense that it has a melting point” [16] must be abandoned. It is only a question of the time available, as thermally activated processes may occur with extremely low rates. An assessment of plasma-facing materials supposed to operate for several years based on a *recrystallization temperature* is not suitable.

2.5 Materials

Pure tungsten plates rolled to different thickness reductions from two different manufactures are investigated: (1) comparably thick tungsten plates from AT&M, Beijing, after warm rolling to a thickness reduction of 67 % (W67) [13], 80 % (W80) [14], and 90 % (W90) [15] and (2) thin tungsten plates from Plansee SE, Reutte, with final thicknesses of 2 mm, 1 mm, 0.5 mm and 0.2 mm [17,18]. The two former (TP2 and TP1) are solely warm-rolled (at an elevated temperature below the nominal recrystallization temperature), whereas additional cold rolling is applied for the thinnest two (TP0.5 and TP0.2).

Small pieces cut from the as-received plates and encapsulated in evacuated glass ampoules are put in furnaces pre-heated to selected annealing temperatures between 1100 °C and 1400 °C and removed after desired annealing times. After inspection for any sign of accidental oxidation (leading to discarding of the test), the chosen surface of the specimens is ground and polished to remove any surface roughness. Macro hardness measurements are performed with a Vickers indenter for a dwell time of 10 s and a load of 10 kgf (W67, W80, W90) and 0.5 kgf (TPs) in compliance with ASTM E29-17.

From the hardness evolution $HV(t)$ at each annealing temperature, the kinetics of the restoration processes is quantified. For the times before the onset of recrystallization, recovery kinetics is analyzed according to equation (3) allowing to extrapolate the recovery behavior $HV_{rec}(t)$ to larger times for which recrystallization occurs simultaneously. For partially recrystallized conditions, the measured macro hardness

$$HV = X HV_{rx} + (1 - X) HV_{rec} \quad (11)$$

is a combined effect of recrystallized regions with volume fraction X having a low hardness HV_{rx} and non-recrystallized regions with volume fraction $1-X$ having undergone recovery and reached a hardness HV_{rec} . Extrapolating the recovery behavior $HV_{rec}(t)$ obtained before onset of recrystallization by means of equation (3) allows to determine the recrystallized fraction present in the microstructure

$$X = \frac{HV_{rec} - HV}{HV_{rec} - HV_{rx}} \quad (12)$$

from the measured macro hardness HV .

3. Results

3.1 Annealing behavior

During annealing of deformed tungsten plates, the beneficial mechanical properties from plastic deformation (as increased strength and hardness) degrade gradually due to the occurring restoration phenomena. The hardness evolution during annealing of W67 and W90 at 1175 °C shown in figure 1 reveals two distinct stages for each of the plates (The proposed identification of individual restoration processes from the hardness evolution is supported by microstructural investigations presented elsewhere [15,19,20]).

Initially the hardness decreases only slightly with a continuously decreasing rate – a behavior typical for recovery. The significant decrease in the hardness after 400 h (W67) or 16 h (W90) indicates the occurrence of recrystallization in both plates – although annealed at a temperature below the commonly reported *recrystallization temperature* of 1200 °C. At 1175 °C, restoration in W90 is much faster than in W67 deformed to a lower thickness reduction despite their small difference in initial hardness.

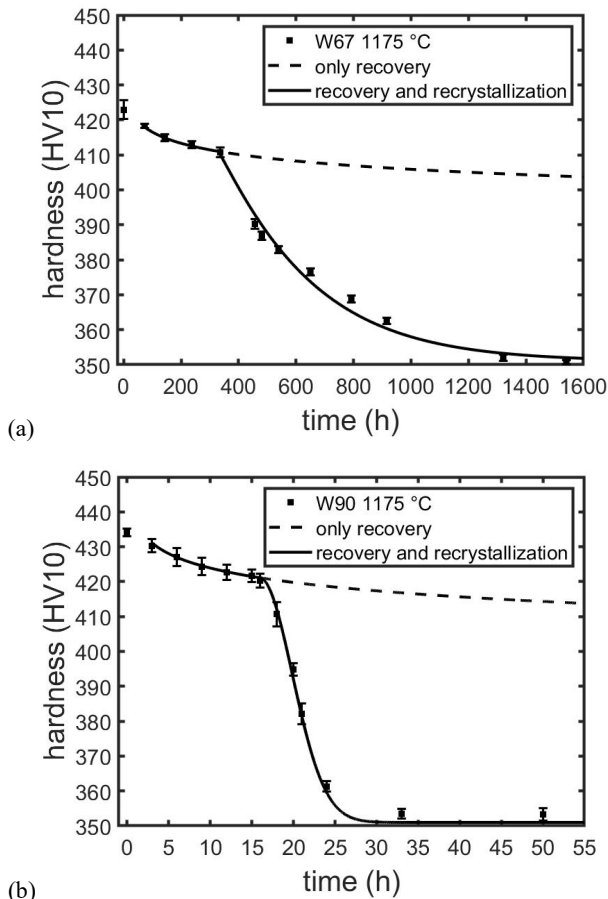


Figure 1. Hardness evolution during annealing at 1175 °C for two pure tungsten plates warm-rolled to different thickness reductions: a) 67 % (W67) and b) 90 % (W90). The lines present descriptions by appropriate models for recovery and recrystallization kinetics.

3.2 Recovery kinetics

For both thick tungsten plates, W67 and W90, recovery is revealed clearly at all investigated temperatures and values of m/T of $4.1 \cdot 10^3$ Pa/K and $3.1 \cdot 10^3$ Pa/K are obtained from fitting equation (3), leading by means of equation (6) to activation volumes of $66 b^3$ and $49 b^3$, respectively. These activation volumes originate from the activation area $\Delta A = \Delta V/b$ swept by a dislocation with Burgers vector b before annihilation. The shorter distance between dislocations and dislocation boundaries in the deformation structure of the material deformed to a larger thickness reduction (W90, cf. [15]) causes a smaller area swept and hence a smaller activation volume.

3.3. Recrystallization kinetics

Based on the extrapolated hardness evolution due to recovery $HV_{rec}(t)$, the recrystallized volume fraction is determined according to equation (12) and illustrated for W67 in figure 2. As obvious from the fits with and without an incubation time, equation (9) describes the experimentally determined recrystallized fractions only when properly taking into account such an incubation time. Comparing the measured hardness values to the modelled kinetics in figure 1 supports this assessment. While recovery kinetics gives a concise description of the early stage, the combined kinetics of recovery and recrystallization achieves the same for the entire hardness evolution.

3.3 Activation energies

Accurate model descriptions of the restoration kinetics are achieved in the outlined manner for all investigated plates at different annealing temperatures ranging from 1100 °C to 1400 °C. From the temperature dependence of the time to half recrystallization for each of the plates, an effective activation energy is obtained assuming an Arrhenius relation

$$t_{X=0.5} = t_0 \exp\left(\frac{Q}{RT}\right). \quad (13)$$

Analogously, other characteristic times as the time to half hardness loss, the incubation time or the inverse of the effective nucleation and growth rate are evaluated. Table 1 summarizes the activation energies obtained for all investigated plates. The activation energies of the different processes are in general comparable to activation energies of bulk self-diffusion (502-586 kJ/mol) or short-circuit self-diffusion (377-460 kJ/mol) [1]. For instance, all activation energies for W67 correspond to that of bulk diffusion, whereas the ones of W90 to that of short circuit diffusion; the difference rooting in a higher amount of defects (in particular low angle boundaries) present in W90 [15]. The similar activation energies obtained for all individual characteristic times for W67 (and analogously for W90) confirm the earlier speculation of a common activation energy for all three

involved processes (incubation, nucleation and growth) as they rely on atomistic jumps to allow dislocations to re-organize to cause nucleation, formation of viable nuclei and boundary migration. In the case of the thin plates, the lower activation energies for b^{-1} than for $t_{X=0.5}$ are attributed in part to issues in defining an effective combined nucleation and growth rate, cf. [18]. As long as a single process (incubation, nucleation or growth) is dominating, the activation energy should correspond to either the one of bulk or the one of short circuit diffusion. If the incubation time and the time for nucleation and growth become comparable in equation (10) and both processes possess different activation energies, effective activation energies between both values may result for $t_{X=0.5}$ as seen for the thinnest plates TP0.5 and TP0.2 [18].

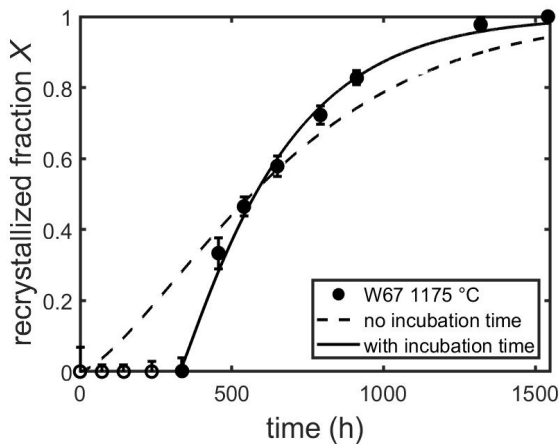


Figure 2. Evolution of the recrystallized volume fraction during annealing at 1175 °C for a pure tungsten plate warm-rolled to 67 % thickness reduction (W67) fitted to recrystallization kinetics with and without any incubation time.

4. Discussion

As recrystallization limits the use of deformed tungsten as plasma-facing material (by reinstating its brittleness), the time to half recrystallization allows estimating the lifetime under operation conditions. Figure 3 summarizes the times to half recrystallization for all investigated plates. The different slopes of their corresponding Arrhenius relations according to equation (13) originate from the different activation energies between 579 kJ/mol and 352 kJ/mol in table 1. The dashed lines extrapolating the behavior to temperatures out of the investigated range have a twofold purpose: they allow predicting the behavior at potential operation temperatures; additionally, they highlight a potential danger when ranking material at a particular annealing temperature without taking into account their different temperature dependence.

4.1 Quasi-stationary condition

For application as armor of the first wall of the blanket, none of the investigated rolled tungsten plates will reach half recrystallization at an expected temperature of maximal

800 °C before the scheduled replacement after five years. This is different for the armor of the divertor: the dotted line in figure 3 indicates the expected operation period of two (full power) years. For an operation temperature of 1175 °C, none of the materials is suitable as half recrystallization occurs too soon. If the operation temperature can be limited to 1000 °C, on the other hand, all plates except the highly deformed W90 will have sufficient thermal stability. For a surface temperature of 1050 °C for a heat flux of 10 MW/m² under normal operation conditions [21], only the moderately deformed W67 and three of the thin plates are suitable. For the best plates, half the volume will not recrystallize before 6.1 years (W67), 7.5 years (TP2), or 12.8 years (TP1).

For performing such extrapolations, accounting for the different activation energies becomes essential. When comparing the three thick plates at temperatures of 1250 °C or less, the most deformed plate W90 reaches half recrystallization always before the lesser deformed W80. As the lowest deformed plate W67 recrystallizes even latter, W67 should be preferred for the desired operation temperatures. An entirely different conclusion would be reached, if the recrystallization behavior would be studied at higher temperatures only and the different activation energies not taken into account properly. For instance, at 1500 °C, W90 would appear more stable against recrystallization than both less deformed counterparts W67 and W80.

An appropriate armor material must therefore be selected based on (i) investigations as close to the desired operation temperature as possible (not solely on high temperature behavior as in [22]) and (ii) determination of the temperature dependence of the recrystallization processes. A thorough quantification of the activation energy (as in case of the time to half recrystallization) for each material is indispensable.

Figure 3 emphasizes again the absence of a threshold temperature below which tungsten does not recrystallize and contradicts simultaneously frequently made opposing claims. The commonly reported *recrystallization temperature* of 1200 °C refers only to notable recrystallization after short periods (as realized earlier [13,15] and affirmed later [23]).

Table 1. Activation energies of different characteristic times for recrystallization of differently rolled pure tungsten plates. (Asterisks mark values not obtained due to a partially recrystallized as-received condition [14] or limited temperature data [18].)

Plate	Activation energy kJ/mol				Avrami exponent n	Ref.
	$t_{\Delta HV/2}$	$t_{X=0.5}$	t_{inc}	b^{-1}		
W67	568	579	568	600	1.1	[13]
W80	*	548	*	*	*	[14]
W90	357	352	369	328	2	[15]
TP2	*	529	*	235	2	[18]
TP1	*	569	*	236	2	
TP0.5	*	468	*	285	2	
TP0.2	*	363	*	285	2	

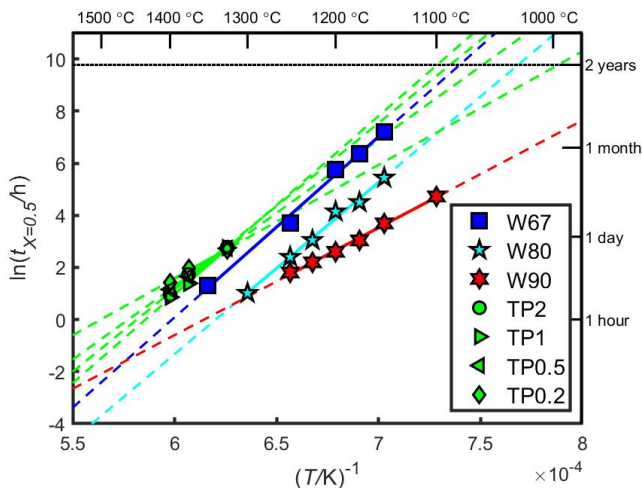


Figure 3. Arrhenius plot for time to half-recrystallization depending on annealing temperature for differently rolled tungsten plates.

4.2 Temporary overheating

When assessing the lifetime of plasma-facing materials based on the time to half recrystallization, the temperature under quasi-stationary conditions must be considered as well as occasionally increased temperatures during short time intervals t_{over} . Higher heat fluxes during slow transients rise the surface temperature e.g. to about 1550 °C for 15 MW/m² [21]. The progress of thermally activated processes at overheating temperature T_{over} is equivalent to an effective time

$$t_{eff} = t_{over} \exp\left(\frac{Q}{R}\left(\frac{1}{T_{stat}} - \frac{1}{T_{over}}\right)\right) \quad (14)$$

at the surface temperature T_{stat} under quasi-stationary conditions. Overheating during slow transients will impede the lifetime under normal conditions strongly depending on the activation energy. A temporary overheating by 500 K during a single transient lasting 1 s reduces the time to half recrystallization by 1.8 h in case of the lowest activation energy 352 kJ/mol, but by 521 h for the highest activation energy 579 kJ/mol. The materials (W67 or TP1) preferred for quasi-stationary conditions might not be an optimal choice depending on the heat flux, frequency, and duration of slow transients; the plate TP0.2 with low activation energy and not too short times to recrystallization might be suited best, if the quasi-stationary surface temperature is kept below 1000 °C.

5. Conclusions

No threshold temperature exists below which a thermally activated process will not occur. The lower the temperature, the slower rolled tungsten will undergo recrystallization. The commonly reported *recrystallization temperature* provides a convenient estimate for the occurrence of recrystallization within a timeframe of few hours or a day. During extended periods operating as plasma-facing material at high temperatures, it will be only a matter of patience to reach the

onset of recrystallization. For low temperatures as 800 °C, recrystallization in half the volume will not occur in the investigated rolled tungsten plates before five years, whereas the possibility of recrystallization becomes relevant during operation at higher temperatures. For a surface temperature of 1050 °C at the divertor, at least some of the investigated rolled tungsten plates will have sufficiently stable microstructures to withstand recrystallization in more than half the volume before the end of two full power years.

Acknowledgements

This work has been carried out partially within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 and 2019-2020 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission. The assistance of Angel Alfonso and Umberto Maria Ciucani is gratefully acknowledged.

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