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Inverse identification of tungsten static recrystallization kinetics under high thermal flux

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Abstract

Understanding of recrystallization phenomenon is essential to apprehend damage process of tungsten armored plasma facing components and to optimize their use in tokamak environment. In ITER, plasma facing components will reach extreme surface temperature value up to 2000°C. Up to now, recrystallization kinetics of ITER tungsten grade were investigated from 1150°C to 1350°C. In order to understand tungsten recrystallization process on the wider relevant temperature range, kinetics have to be investigated at higher temperature and on several tungsten grades. Usually, kinetics are investigated by performing successive isothermal annealings on tungsten samples. Due to number of ITER tungsten grades and large temperature range (from 500°C to 2000°C), an important amount of tungsten samples have to be prepared to investigate recrystallization kinetics on ITER representative conditions. In this paper, an innovative way is proposed to obtain recrystallization kinetics of ITER tungsten grade based on the use of small-scale plasma facing component tested under high heat flux. Thanks to the use of this inverse method, tungsten recrystallization kinetics are identified at 1347°C$^{\pm 31}$, 1478°C$^{\pm 31.5}$, 1584°C$^{\pm 31.5}$and 1690°C$^{\pm 33.5}$by using experimental measurements and Johnson-Mehl-Avrami-Kolmogorov model. Then, obtained kinetics are used as input data in numerical post-treatments to obtain tungsten recrystallization gradients after 500 thermal cycles at 20MW/m^2.

Keywords: tungsten, inverse method, recrystallization, kinetics, simulation, ITER

1. Introduction

For the ITER divertor, plasma facing components are made with tungsten as armor material, bonded on a copper alloy tube as heat sink structural material and cooled by water. Such components withstand high heat flux up to 20 MW/m^2 and consequently satisfy ITER requirements [1, 2, 3, 4, 5, 6]. However, due to high heat flux, the loaded surface reaches extreme temperature values up to 2000°C and strong temperature gradient is generated on a thickness in the range of 6 mm [7, 8]. 2000°C is large enough to alter tungsten microstructure by recrystallization. Understanding of recrystallization phenomenon is essential to apprehend damage process of such components and to optimize their use in tokamak environment. Hereafter, this means tungsten recrystallization kinetics have to be investigated. Up to now, kinetics were obtained by isothermal annealings on one tungsten grade relevant with ITER specifications (called here abusively ITER tungsten grade) on a restricted temperature range from 1150°C to 1350°C [9]. To fulfill the gap and then understand tungsten recrystallization phenomenon on the entire temperature range, an innovative way is proposed in this paper. Based on the use of small-scale plasma facing component tested under high heat flux, this inverse approach aims to benefit from thermal temperature gradient generated experimentally to obtain tungsten recrystallization kinetics.

In the first part of this paper, inverse methodology is described. Second, based on hardness measurements, tungsten recrystallization kinetics are identified thanks to Johnson-Mehl-Avrami-Kolmogorov model (JMAK) [10]. Then, numerical integration of obtained kinetics highlights that consistent tungsten recrystallization gradients are obtained.

2. Assumptions

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

JMAK model (equation 1) can be use to describe the recrystallized fraction (X) evolution of material over the annealing time [10]. tinc is representing the incubation time, to take into account recovery phase before recrystallization [10], n is the Avrami exponent set at 1.098 [9] and b is thermo-dependent parameter. Recrystallization is a thermally activated process. Relationships between JMAK parameters and annealing temperature over temperature range are usually decribed by Arrhenius laws [10, 9]. In

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this way, inverse approach aims to propose a method to estimate b and tinc parameters at specific annealing temperatures and aim to identify Arrhenius relationships for these both parameters on a large temperature range. For that, X has to be estimated over different annealing times per annealing temperature.

$$X = \frac{HV_{in} - HV}{HV_{in} - HV_{rx}} \tag{2}$$

According to equation 2 [9, 11], X can be estimated from hardness measurements, with HV the measured hardness after any annealing time at a specific annealing temperature, HV_{rx} the measured hardness on fully recrystallized material and HV_{in} the measured hardness on initial material. Indents are performed at CEA Cadarache (IRFM) using a load of 10Kg and a dwell time of 10s [9].

In this way, based on the use of small-scale plasma facing component tested under high heat flux, inverse approach aims to benefit from thermal temperature gradient obtained experimentally. Indents (hardening tests) at specific positions, corresponding to a loaded annealing temperature are performed. Annealing temperatures are identified thanks to use of the thermal temperature gradient obtained numerically. Measured hardness related to same annealing temperature per block are averaged and then converted in X (equation 2).

To obtain parameters from equation 1, each X obtained is assigned to an equivalent annealing time ($t_{annealing}$). $t_{annealing}$ per investigated block is defined as a function of number of loaded cycles (N_{cycle}) and steady state loaded time (Δt) (equation 3). Parameters b and tinc are then identified per investigated annealing temperature thanks to the use of least squares method regarding equation 1 and Arrhenius laws fitting. Finally, Arrhenius relationships are used as input data in numerical anisothermal JMAK post treatment to model tungsten recrystallization gradient after 500 thermal cycles at $20MW/m^2$. Anisothermal JMAK model is commonly used in literature for anisothermal paths [12].

$$t_{annealing} = N_{cycle} * \triangle t \tag{3}$$

3. Material and method

3.1. Materials

In preparation of the actively cooled divertor of WEST tokamak (PHASE 2), mock-ups were manufactured. Composed of 7 tungsten blocks supplied from Advance Technology and Materials, these were bonded on Cu-OFHC interlayer (1mm) finally bonded on a copper alloy tube (CuCrZr) used as heat sink structural material [13]. Tungsten block dimension is 27.4mm (width) * 26mm (height) * 12mm (depth). The minimum distance from tungsten upper surface to the copper interlayer is 6 mm. Tube inner diameter is 12mm, while the outer diameter of the tube is 15mm. These mock-ups were exposed to electron beam

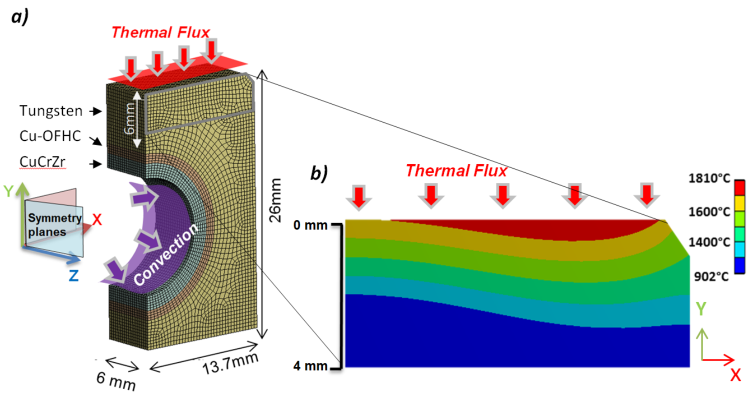

Figure 1: a) Numerical modeling, b) Thermal temperature gradient obtained under 20 MW/m^2

JUDITH 1 at Forschungszentrum Julich [13]. In this paper, two tungsten blocks (M4 and M5) from one mock-up named M2-2 in [13] are investigated. These were exposed to cyclic high heat flux (10s ON / 10s OFF):

-1000 cycles at $10MW/m^2$ followed by **300** cycles at $20MW/m^2$ for M4

-1000 cycles at $10MW/m^2$ followed by **500** cycles at $20MW/m^2$ for M5.

For thermal loading at 10 MW/m^2 (in total 10 000s), tungsten reaches maximum temperature close to 1000°C being not enough to have an impact on tungsten microstructure [9].

3.2. Finite elements modeling

Inverse approach aims to benefit from thermal temperature gradient observed under $20MW/m^2$ to obtain tungsten recrystallization kinetics. For that, temperature gradient and equivalent annealing time over cycles is estimated using numerical simulation performed on finite elements code (ANSYS 17.2).

Numerical model presented in figure 1 (a), is representative to studied tungsten blocks M4 and M5. Thanks to the use of symmetry planes only quarter part of block is modeled taking into account the presence of $1*1mm^2$ chamfer in the modeling. 38376 quadratic elements are used to mesh the 3D geometry. Heat conductivity and coefficient of thermal expansion used are those presented for tungsten, Cu-OFHC and CuCrZr in [7]. In this study, 20 MW/m^2 heat flux is applied on the upper surface and CEA routine [14] is used to calculate heat transfer coefficients (T°=22°C, v=10m/s and p=2.25 MPa) due to water cooling during high heat flux tests at the inner wall pipe. Figure 1 (b), highlights that thermal loadings at 20 MW/m^2 involve strong temperature gradient leading to extreme temperature values from 1810°C at the loaded surface to 902°C 4mm in the depth of the block.

Maximum surface temperature obtained is compared to that measured by infrared camera during the experimental campaign [13] and revealed that numerical model estimates consistent thermal results. This map is used for

	M4 (300 cycles)	M5 (500 cycles)
Δt=5s	1500s	2500s
Δt=10s	3000s	5000s

Table 1: Equivalent annealing time assumed per block

the identification of positions related to specific annealing temperature.

3.3. Annealing time

$t_{annealing}$ (equation 3) has to be estimated for each studied block. Figure 2 (a) shows the temperature evolution (over one thermal cycle) of two nodes placed on both sides of annealing temperature range explored. Steady state is obtained after 5s heating. Consequently, conservative study should be achieved assuming Δt=5s. However, considering Δt=5s in this study, involves that heating phase (from 0s to 5s heating) is neglected. To evaluate the impact of this assumption, kinetics will be investigated assuming two different $t_{annealing}$ per block (tableau 1) with Δt=5s which involved shorter time to obtain fully recrystallized material (conservative study) and Δt=10s which assumed that entire heating phase play a role in the microstructure change.

4. Results and discussion

4.1. Hardness measurements

Figure 2 (b) shows indents performed on the external surface of block M4 and zoom figure 2 (c) displays hardness values for each indents. Zoom highlights that hardness increases by the depth. Similar measurements are performed on block M5. Average hardness values obtained for blocks M4 and M5 are presented in table 2. Due to a deteriorated surface of block M5, no indent is performed for positions related to annealing temperature equal to 1584°C and 1690°C (see figure 1 for global thermal gradient). Moreover, 3 indents are used to obtain average hardness for position related to annealing temperature equal to 1478°C. Indents measure approximately 200μm diagonal which involve vertical temperature gradient in the order of 50°C. Temperature assigned for each indent is consequently given with an incertitude of ±25°C. Hardness values are then averaged over minimum of 3 indents obtained for 3 measurements related to a same loaded temperature (figure 2, c). The maximum horizontal temperature variation over indents varying from ±6°C at 2.6mm depth to ±9°C at 0.6mm depth which involves a maximum final incertitude of ±33.5°C on the average temperature estimated for each investigated zones (figure 2, c).

To estimate HV_{in}, 20 indents are performed on tungsten plate (28*28*12mm^3) supplied by Advance Technology and Materials (AT&M). Average hardness (HV_{in}) obtained (458 HV_{10}±2) is compared with average hardness obtained from indents performed far from the M4 loaded surface (451 HV_{10}±5) revealing that material supplied can be used to estimate HV_{rx}. Isothermal annealing at 1250°C (96 hours) is achieved on tungsten sample (9*12*3mm^3) obtained from surface tungsten plate to estimate HV_{rx}. 20 indents are performed on the sample after annealing. Average hardness (HV_{rx}) obtained is equal to 378 HV_{10}±2. Based on HV_{in} (458 HV_{10}±2) and HV_{rx} (378 HV_{10}±2) each hardness values obtained for blocks M4 and M5 are converted in X thanks to equation 2 (table 2).

Indents are also performed at 8.1mm from the edge of block M5 in order to valid the inverse method on another part of the block. Due to thermal gradient, 5 hardness measurements are performed over the depth. Consequently, hardness is not averaged and error bars presented Figure 4 (b) are representative to statistic obtained for HV_{in} (±2).

4.2. Recrystallization kinetics by inverse method

At annealing temperatures equal to 1584°C and 1690°C only one X is provided which corresponds to measurements performed on M4. To provide another point for the determination of these kinetics, some assumptions are made for the determination of X related to the block loaded at 500 cycles (M5). In any case, the X related to M5 for a given annealing temperature is higher than the one obtained for same annealing temperature on M4. At 1584°C, X is assumed equal to 98.185% which is the average between the value obtained after 300 cycles (95.75%) and fully recrystallized fraction. Then at 1690°C, X is fixed at 100%. Parameters b and tinc are obtained per annealing temperature by fitting equation 1 using least squares method (table 3) for both given annealing times. Related recrystallization kinetics obtained for each annealing temperature (assuming Δt=5s) and Arrhenius laws are given in figure 3. Arrhenius straight as the method imposes it (R^2=0.998).

4.3. Discussion

Figure 4 (a) shows tungsten recrystallization gradient obtained numerically after 500 cycles at 20MW/m^2 using determinated kinetics at 3.5mm from the edge assuming Δt=5s and Δt=10s. Average measurements obtained at 3.5mm from the edge of blocks are used to fit kinetics parameters. Consequently, figure 4 (a) shows consistent evolution of tungsten recrystallized fraction with regard to experimental data displayed table 2. It shows also that the realistic annealing time is between Δt=5s and Δt=10s.

Figure 4 (b) highlights recrystallized fraction evolution over the block depth at 8.1mm from the edge. It shows consistent predictions of X tendency.

In this paper, two tungsten blocks are used to fit tungsten recrystallization kinetics. In further studies, number of investigated blocks could be increased in order to optimize the fitting procedure and provide more consistent data. Also, to increase statistics and minimize temperature incertitude per investigated kinetic, micro-indents could be considered.

	T(°C)	$1347^{\pm31}$	$1478^{\pm31.5}$	$1584^{\pm31.5}$	$1690^{\pm33.5}$
500 cycles	Hv_{10}	$448^{\pm2}$	$403^{\pm4.5}$	$385^{\pm3.5}$	$377^{\pm4}$
(20 MW/m²)	X (%)	$13^{\pm2}$	$68.25^{\pm6}$	$91.75^{\pm4}$	$100^{\pm5}$
300 cycles	Hv_{10}	$427^{\pm3.5}$	$400^{\pm1}$	-	-
(20 MW/m²)	X (%)	$38.37^{\pm4}$	$72.91^{\pm1.5}$	$98.125^{\pm1.875}$	100

Table 2: Average hardness values and related recrystallized fraction obtained

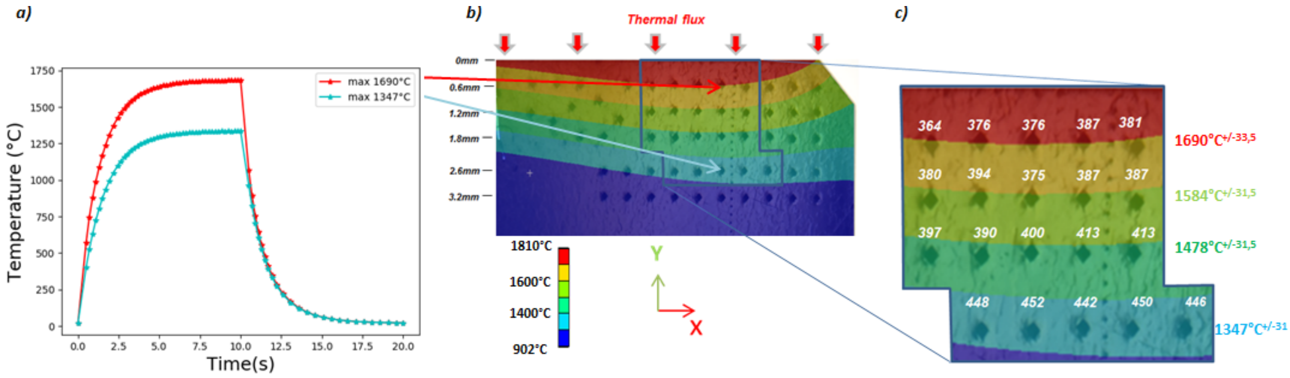

Figure 2: a) Temperature evolution of two nodes, b) indents performed on M4 are assigned to temperature, c) hardness values obtained for M4 and related annealing temperature

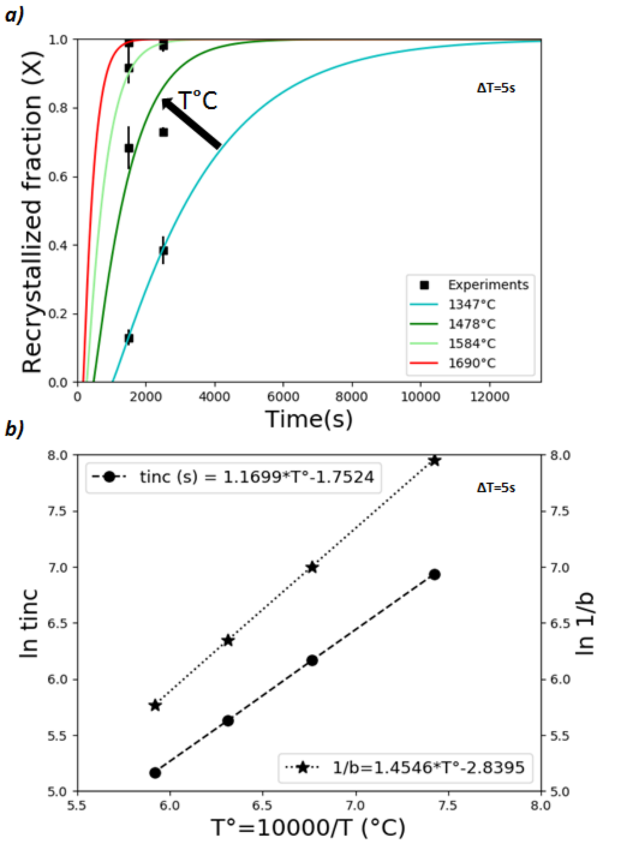

Figure 3: a) Recrystallization kinetics obtained by inverse method and b) related Arrhenius relationships assuming Δt=5s

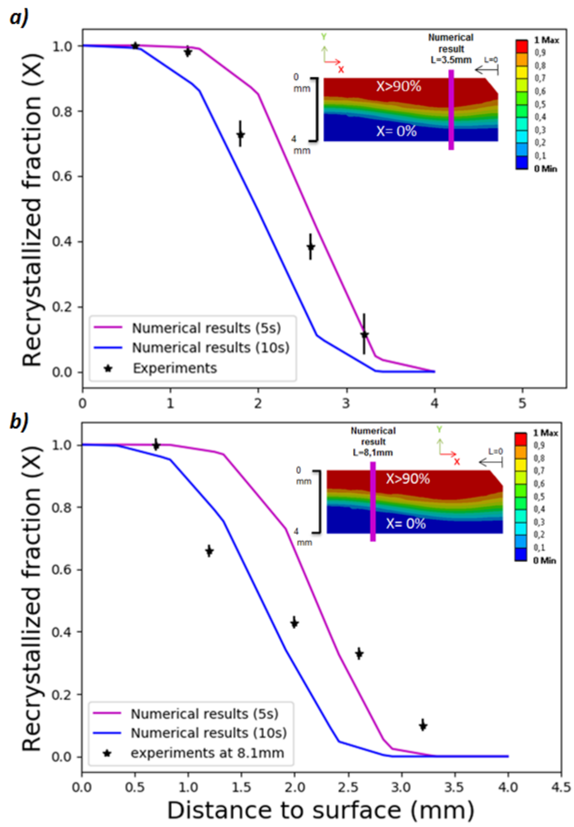

Figure 4: Evolution of the recrystallized fraction obtained numerically for both annealing times assumed (5s and 10s) and comparison with experiments performed at: a) 3.5mm from the edge of the block, b) 8.1mm from the edge of the block.

5. Conclusion

This paper proposes an innovative way to investigate tungsten recrystallization kinetics used as armored mate-

	b				tinc (s)			
	$1347°C^{\pm 31}$	$1478°C^{\pm 31.5}$	$1584°C^{\pm 31.5}$	$1690°C^{\pm 33.5}$	$1347°C^{\pm 31}$	$1478°C^{\pm 31.5}$	$1584°C^{\pm 31.5}$	$1690°C^{\pm 33.5}$
Δt=5s	0.000355	0.000909	0.00175	0.00312	1026s	476	279	176
Δt=10s	0.000175	0.000461	0.00089	0.00160	2052s	1004	611	397

Table 3: JMAK parameters identified for two annealing times using least squares method

rial in plasma facing components. Here above, kinetics were obtained at $1347°C^{\pm 31}$, $1478°C^{\pm 31.5}$, $1584°C^{\pm 31.5}$and $1690°C^{\pm 33.5}$. JMAK parameters (b and tinc) obtained are extrapolated over the explored temperature range using Arrhenius relationships. To finish, these are used as input data in numerical post-treatments and consistent tungsten recrystallization gradients are obtained.

6. Acknowledgments

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