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Thermal shock resistance of tungsten with various deformation degrees under transient high heat flux

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Abstract

Tungsten (W) is considered as the most promising plasma facing material in fusion device, which will be exposed to steady and transient heat loads. Usually, the deformation degree of W influences its thermal shock properties. So we evaluated the thermal shock resistance of rolled pure tungsten (PW) with 60%, 90% deformation degrees and W-1.0wt%La2O3 (WL10) with 52%, 88% deformation degrees using electron beam at an absorbed power density of 0.22 GW m⁻² for 5 ms and further discussed the relationship between the thermal shock resistance and microstructures, thermal-mechanical properties. The absorbed beam current (30 mA), electron beam acceleration voltage (120 kV) and loaded area (4 × 4 mm²) is used to estimate the absorbed power density. The results indicated that PW-90%, LW-88% exhibited smaller grain size, higher relative density, microhardness but lower strength, thermal conductivity compared to PW-60%, LW-52%. The cracking threshold was < 0.22 GW m⁻² for PW-60%, LW-52% and > 0.22 GW m⁻² for PW-90%, LW-88%. Elliptic left pores in PW-60% and LW-52% aggravated the effect of stress concentration cracking during the transient high heat flux test and decreased the cracking threshold although they exhibited high strength and thermal conductivity.

1. Introduction

Due to the favorable properties including high melting point, high thermal conductivity, excellent erosion resistance, low tritium inventory and good high temperature mechanical property, tungsten (W) is considered as the most promising plasma facing material to be used in future fusion devices [1–6]. During plasma operation, W have to withstand severe environmental conditions in terms of steady and transient heat loads as well as high particle fluxes [7–11]. The transient high heat flux will cause severe damage of the W such as roughing, cracking, melting, particle release, droplet ejection and recrystallization, etc [12, 13]. Thermal shock resistance of W was related to the microstructure (grain size and grain orientation, etc), basic properties (yield strength (σ), bulk density (ρ), Young’s Modulus (E), Poisson ratio (μ), thermal conductivity (λ), coefficient of thermal expansion (α), specific heat (c_p) etc) and thermal diffusion time scale, thermal diffusion depth, pulse duration [13, 14]. Usually, thermal shock resistance can be expressed by the thermal stress intensity factor (R_a), which can be calculated by the following equation (1):

\[ R_a = \frac{\sigma \lambda (1 - \mu)}{\alpha E \beta C_p} \]  

(1)

It has been proved that the composition and deformation degree determined the microstructure and thermal-mechanical properties of W [15, 16]. Therefore, it is necessary to evaluate the thermal shock resistance of deformed W and W alloys with various deformation degrees. Pintsuk et al [17] investigated the thermal shock property of two deformed W grades with various deformation degrees and focused on the relationship between the cracking characteristics and grain size, ductile to brittle transition temperature (DBTT). The slight deformed
W exhibited a lower cracking threshold of \( 23 \text{ MW/m}^2 \) than \( 39 \text{ MW/m}^2 \) for heavy deformed W, which was explained by the rise of DBTT. Besides, Zhou et al. [12] also evaluated the thermal shock performance of the swaged W with various deformation degrees. Higher deformation degree resulted in smaller major crack density and the reason was not analyzed. Summarily, the thermal shock properties of deformed W with various deformation degrees were few reported and the relationship between the thermal shock resistance and microstructures, thermal-mechanical properties was also rarely investigated.

In this paper, rolled pure tungsten (PW) with 60\%, 90\% deformation degrees and rolled W-1.0wt%La2O3 (WL10) with 52\%, 88\% deformation degrees were prepared. Then microstructure, thermal-mechanical properties and thermal shock resistance were characterized and the relationship between the thermal shock resistance and strength, thermal conductivity, especially relative density, i.e., left pores was discussed in detail.

### 2. Experiments

#### 2.1. W preparation

Commercial PW and WL10 powders with average particle size of about 3 ± 0.2 μm were densified by cold isostatic pressing and intermediate frequency sintering at 2373 K for 2 h. Subsequently, for PW, the sintered billets were rolled from 30mm-thick to 12 mm and 3 mm with deformation degrees of 60\% and 90\% and the rolled PW samples were labeled as PW-60\%, PW-90\%, respectively. For WL10, the sintered billets were rolled from 25 mm-thick to 12 mm and 3 mm with deformation degrees of 52\% and 88\% and the rolled WL10 samples were labeled as LW-52\%, LW-88\%, respectively. Intermediate annealing was necessary to reduce induced stresses and to maintain sufficient workability. Finally, these deformed W sheets were annealed at 1373 K in hydrogen atmosphere for 2 h to relax residual stresses. Impurities of the PW and WL10 are listed in table 1.

#### 2.2. Thermal shock test

The thermal shock facility was described in [18]. Thermal shock tests were performed at an absorbed power density of 0.22 GW m\(^{-2}\) with pulse duration of 5 ms at room temperature for 1 cycle. The sketch of W sample installation during thermal shock test is shown in figure 1. Sample size was 12 × 12 × 3 mm\(^3\) and the scanning area was 4 × 4 mm\(^2\). The elongated W grains were aligned perpendicular to the heat transfer direction. The absorbed power density (\( P_{\text{abs}} \)) was calculated by equation (2):

\[
P_{\text{abs}} = \frac{U I_{\text{abs}}}{S}
\]

\( U \) is the acceleration voltage of electron beam, 120 kV. \( I_{\text{abs}} \) is the absorbed current, which is recorded during testing. \( S \) is the scanning area, 16 mm\(^2\).

#### 2.3. Characterization

Microstructures were characterized via optical microscopy and scanning electron microscopy (SEM) as well as field emission scanning electron microscopy equipped with an electron backscattered diffractometer (EBSD) detector. Density was measured using Archimedes’ method. Vickers microhardness was measured under a load of 1.96 N for 10 s. Three points bending (3PB) specimens of 37 × 4 × 3 mm\(^3\) were produced to measure the fracture strength. Thermal conductivity was measured by the laser flash method. Surface roughness were measured by a laser profilometry and a confocal laser scanning microscopy.

### 3. Results and discussion

#### 3.1. Microstructure

Figure 2 shows the microstructures on the cross-section surfaces of rolled PW and WL10 with various deformation degrees. Grains were elongated along the rolling direction. More noises left on the PW-90\% and LW-88\% should be resulted from their larger residual stress. The grain size characteristics of these W materials

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**Table 1. Chemical impurities of the PW and WL10, <ppm.**

| Sample | Impurities |
|--------|------------|
| PW     | C < 20, Fe < 15, Ni < 10, Al < 10, Cr < 10, Ca < 10, As < 10, Si < 10, K < 10, S < 5, Mg < 5, Mn < 5, Co < 10, O < 25, N < 10, Mo < 18, Ti < 5 |
| WL10   | C. < 20, Fe < 15, Ni < 10, Al < 15, Cr < 10, Ca < 10, As < 10, Si < 10, K < 10, S < 20, Mg < 10, Bi < 1, Sn < 5, Pb < 1 |
in terms of feret min, feret max, aspect ratio and area are summarized in figure 3. Obviously, PW-90% and LW-88% exhibited smaller feret min, grain area and larger feret max, aspect ratio compared with PW-60% and LW-52% due to the larger deformation degrees (90% and 88% versus 60% and 52%). Besides, LW-52% and LW-88% exhibited larger aspect ratio compared with PW-60% and PW-90%, which can be attributed to the pinning effect on grain boundary movement and dislocation movement induced by La$_2$O$_3$ particles. The distribution and size of La$_2$O$_3$ particles are shown in figure 4. La$_2$O$_3$ particles in LW-88% exhibited smaller feret min and larger aspect ratio than that in LW-52% due to the larger deformation degree. It should be noted that the aspect ratio of La$_2$O$_3$ was 21.3 ± 12.3, much larger than 12.8 ± 4.4 of tungsten matrix in LW-88%. This phenomenon can be caused by the slight coarsening of tungsten grain induced by stress relief annealing at 1373 K, which was close to the recrystallization starting temperature [19]. Summarily, grain size decreased with the increase of deformation degree for both PW and WL10 basing on the aspect ratio and feret min simultaneously.

3.2. Basic properties

Basic properties in terms of relative density, microhardness, 3PB strength and thermal conductivity for the rolled PW and WL10 with various deformation degrees are summarized in figure 5. Obviously, for the former two properties, relative density and microhardness increased with the deformation degree for both W materials. Severer plastic deformation would facilitate the close of left pores from sintering thereby lead to higher relative density. Figure 6 shows the fracture surface of rolled PW and WL10 with various deformation degrees. A certain number of pores were detected on the fracture surface of PW-60%, LW-52% and no pore was observed on the fracture surface of PW-90%, LW-88%. Besides, microhardness order from the minimum to maximum was PW-60%, LW-52%, PW-90% and LW-88%, which was opposite with the arrangement of grain size due to the Hall-Patch relationship.

Generally, strength of metal is increased with deformation degree due to the work hardening mechanism. However, 3PB strength decreased with the increase of deformation degree for both W materials. 3PB strength was 1069, 995 MPa for PW-60%, PW-90% and 1312, 1068 MPa for LW-52%, LW-88%, respectively. Lower strength in severer rolled PW and WL10 should be resulted from microcracks. On the one hand, crack-tips can be formed in the following manners [20]: (a) merging of edge dislocations on the same glide plane; (b) stress concentration induced by dislocation pile-up when the dislocation motion was inhibited by grain boundaries; (c) dislocation interaction between two slip bands; (d) sideway of incomplete dislocation walls formed at the location of slip bands intersecting. Furthermore, dislocation merging, dislocation pile-up, dislocation interaction, slip bands intersecting and sideway of incomplete dislocation walls would be facilitated by severe plastic deformation. Thus the formation risk of crack-tips would be increased with deformation degree. On the other hand, the constitutive equation of W during plastic deformation can be expressed using the equation (3):

$$\sigma_d = 181.213e^{\frac{1338.452}{T_e}} \epsilon^{0.31} e^{-0.023\epsilon}$$  (3)
where \( \sigma_d \) is the deformation resistance, \( T_d \) is the deformation temperature, \( \varepsilon \) is deformation degree and \( \dot{\varepsilon} \) is deformation rate. Generally, \( T_d \) is decreased with the increase of rolling pass. Thus for the PW-90% and LW-88%, both larger deformation degree and lower deformation temperature would result in the higher deformation resistance, which would further facilitate the formation of microcracks.

Thermal conductivity of metal is approximately equal to electronic thermal conductivity (\( \lambda_e \)). The fundamental expression for \( \lambda_e \) can be calculated on the basis of the theory of thermal conductivity for classical gas [21]:

\[
\lambda_e = \frac{1}{3(\alpha c_e \nu_e l_e)}
\]  

(4)

where \( c_e \) is the electronic heat capacity (per electron), \( n \) is the number of conduction electrons per volume, \( \nu_e \) is the electron speed and \( l_e \) is the electron mean free path. In this paper, pores, La\(_2\)O\(_3\), grain size and dislocation density were the main factors influencing the thermal conductivity of W. Firstly, the interfacial area of W increased because of the existence of pores and La\(_2\)O\(_3\) particles, which facilitated the scattering of electron and decreased the mean free path of electron. Pores (or cracks) blocking heat transfer has been also reported in [22, 23]. Secondly, small grain size, i.e., more grain boundaries and more dislocations also facilitated the scattering of electron and decreased the mean free path of electron. Grain boundary and dislocation inhibiting heat transfer has been reported in [24, 25]. Thirdly, thermal conductivity of La\(_2\)O\(_3\) was much lower than that of W matrix. Therefore, rolled PW and WL10 with larger deformation degrees exhibited lower thermal conductivity compared with ones with smaller deformation degrees, which was resulted from the smaller grain size and more dislocations (larger deformation degree usually resulted in more dislocations). Besides, rolled PW exhibited higher thermal conductivity compared with WL10 with close deformation degree, which can be
Figure 3. Grain size of PW-60% (a), PW-90% (b) and LW-52% (c), LW-88% (d).

Figure 4. Size and distribution of $\text{La}_2\text{O}_3$ particles in LW-52% (a), (c) and LW-88% (b), (d).
Figure 5. Relative density, microhardness, bending strength and thermal conductivity of rolled PW and WL10 with various deformation degrees.

Figure 6. Fracture surface of PW-60% (a), PW-90% (b) and LW-52% (c), LW-88% (d).
mainly attributed to the electron scattering effect and low thermal conductivity of La₂O₃. Briefly, severer rolled PW and WL10 exhibited higher relative density, microhardness but lower strength, thermal conductivity.

3.3. Thermal shock resistance and mechanism

Figure 7 shows the surface modifications of PW-60%, PW-90% and LW-52%, LW-88% samples after thermal shock tests. Obviously, cracks were only formed on the PW-60% and LW-52%. So it can be concluded that the cracking threshold was below 0.22 GW m⁻² for PW-60%, LW-52% and above 0.22 GW m⁻² for PW-90%, LW-88%. Furthermore, PW-60% and LW-52% also exhibited higher surface roughness compared with PW-90% and LW-88% as shown in figure 8. Higher surface roughness in PW-60% and LW-52% can be attributed to the cracks. Figure 9 shows the three-dimensional surface modifications of PW-60% and LW-52% after the thermal shock test. The areas around the cracks showed obviously swelling.

Usually thermal shock property of W was determined by its microstructure, thermal properties and mechanical properties simultaneously. The relationship between the thermal shock property and strength, thermal conductivity, left pores was investigated in the present paper. In the thermal shock test, compressive stresses and tensile stresses were induced on the loaded surface during the heating and cooling stages, respectively. Assuming the heat flux direction is y axis, rolling direction is x axis and transverse direction is z axis. The surrounding cold bulk prevented the volume expanding along x and z directions during the thermal shock test. In other words, \( \varepsilon_x = \varepsilon_z = 0 \).

\[
\varepsilon_x = \frac{\sigma_x}{E} - \mu \left( \frac{\sigma_y}{E} + \frac{\sigma_z}{E} \right) - \alpha T = 0
\]

\[
\varepsilon_z = \frac{\sigma_z}{E} - \mu \left( \frac{\sigma_x}{E} + \frac{\sigma_y}{E} \right) - \alpha T = 0
\]

\[
\varepsilon_y = \frac{\sigma_y}{E} - \mu \left( \frac{\sigma_x}{E} + \frac{\sigma_z}{E} \right) - \alpha T
\]

\( \varepsilon_x, \varepsilon_y \) and \( \varepsilon_z \) is the strain along x, y and z axis, respectively. \( \sigma_x, \sigma_y \) and \( \sigma_z \) is the stress along x, y and z axis, respectively. \( T \) is the temperature rise. It can be calculated that:
At the beginning of the cooling process, \( \sigma_x = \sigma_z = \frac{T \alpha E}{1 - \mu} \) (8)

Furthermore, during the thermal shock test, the surface temperature rise can be approximately calculated by the equation (9) [26] and \( \sigma_{\text{max}} \) can be expressed using equation (10):

\[
T = 2P_{\text{abs}} \sqrt{\frac{t}{\pi \lambda \rho c}}
\]

\[
\sigma_{\text{max}} = \frac{2P_{\text{abs}} \alpha E}{1 - \mu} \sqrt{\frac{t}{\pi \lambda \rho c}}
\]

Thus lower thermal conductivity would result in higher \( T \), i.e., higher thermal stress. If the \( \sigma_{\text{max}} \) exceeded \( \sigma_f \), cracking would be occurred.

Basing on the basic properties as mentioned in 3.2 section, PW-60% and LW-52% exhibited higher thermal conductivity compared to PW-90% and LW-88%. Theoretically, PW-60% and LW-52% would suffer higher \( \sigma_{\text{max}} \) and would show better thermal shock resistance considering their higher strength. But in fact, PW-60% and
LW-52% exhibited worse thermal shock properties as shown in figure 7, which should be resulted from their low relative density, i.e., more left pores.

Spherical sintering pores were elongated along rolling direction during plastic deformation and elliptic pores were survived in PW-60% and LW-52% as the deformation degree was not large enough. Subsequently, the elliptic pores will cause the local stress concentration during the thermal shock test, which would increase the cracking risk. The stress induced by stress concentration can be calculated by the equation (11) [27] assuming that the major axis of elliptic pore was along the rolling direction:

$$
\sigma_{sc} = 2\sigma_s \frac{c}{\sqrt{c_r}}
$$

where $\sigma_{sc}$ is the stress induced by stress concentration. $\sigma_s$ is the stress from surrounding. $c_r$ ($c_r = \frac{b^2}{c}$, b and c is the major axis and minor axis) is the curvature radius of the pore. Therefore, $\sigma_{sc}$ is much higher than $\sigma_s$ and when $\sigma_{sc}$ exceeds $\sigma_c$, cracks will be formed. Thus the elliptic left pores in PW-60% and LW-52% would aggravate the effect of stress concentration cracking during the transient high heat flux test and decrease the cracking threshold although they exhibited high strength.

Table 2 shows the thermal shock damage of W materials with various relative densities or deformation degrees. In [17], the cracking threshold increased with deformation degree, which was similar to our results. In [28], relative density influenced the melting or erosion threshold rather than the cracking threshold. It can be seen that relative density and deformation degree influenced the thermal shock resistance of W under transient high heat flux significantly, which should be further investigated systematically in the future. Besides, the cracking threshold of W during transient high heat flux was also influenced by the fabrication history and chemical composition. For example, the cracking threshold at RT and 5 ms pulse duration was <0.15 GW m$^{-2}$ for the sintered PW and rolled PW [29], 0.25 GW m$^{-2}$ for the forged PW [30], 0.33–0.35 GW m$^{-2}$ for the CVD-W, W-TiC [30] and 0.44–0.66 GW m$^{-2}$ for the W-K [31]. Obviously, the cracking threshold of W was increased by introducing suitable second phase. Thus dense W strengthened by the second phase can be the most promising candidate material for the PFM especially for the divertor in fusion reactor.

### 4. Conclusions

In this work, rolled PW with 60%, 90% deformation degrees and WL10 with 52%, 88% deformation degrees were prepared. Then microstructure, thermal-mechanical properties and thermal shock resistance were characterized and the relationship between the thermal shock properties and strength, thermal conductivity, especially relative density, i.e., left pores was discussed.

1. Grain size decreased with the increase of deformation degree for both PW and WL10 basing on the aspect ratio and feret min simultaneously.

2. PW-90%, LW-88% exhibited higher relative density, microhardness but lower strength, thermal conductivity compared to PW-60%, LW-52%.

3. Cracking threshold was <0.22 GW m$^{-2}$ for PW-60%, LW-52% and >0.22 GW m$^{-2}$ for PW-90%, LW-88%.

4. Elliptic left pores in PW-60% and LW-52% aggravated the effect of stress concentration cracking during the transient high heat flux test and decrease the cracking threshold although they exhibited high strength and thermal conductivity.

### Table 2. Thermal shock damages for W materials with various relative densities or deformation degree at RT.

| Sample | Deformation degree, % | Relative density, % | Cracking threshold, GW m$^{-2}$ | Melting or erosion threshold, GW m$^{-2}$ | References |
|--------|-----------------------|---------------------|---------------------------------|------------------------------------------|------------|
| PW-60% | 60%                   | 98.4                | <0.22                           | /                                        | Our        |
| PW-90% | 90%                   | 99.2                | >0.22                           | /                                        | Our        |
| LW-52% | 52%                   | 98.6                | <0.22                           | /                                        | Our        |
| LW-88% | 88%                   | 99.6                | >0.22                           | /                                        | Our        |
| M1     | small                 | /                   | 0.22–0.33                       | 0.55–0.88                                | [17]       |
| M2     | large                 | /                   | 0.44–0.55                       | 0.55–0.88                                | [17]       |
| W02    | /                     | 94.68               | <0.22                           | 0.22–0.27                                | [28]       |
| W10    | /                     | 97.49               | <0.22                           | 0.27–0.44                                | [28]       |
| W30    | /                     | 98.22               | <0.22                           | 0.11–0.16                                | [28]       |

References:
1. et al [27], 2. [28], 3. [29], 4. [30], 5. [31].
Certainly, W with low density, i.e., more survived sintering pores are not suitable for the PFM in fusion reactor. However, the cracking threshold of W with high density in the present work was still lower than those of dispersion strengthened W materials such W-TiC, W-K et al in literatures. Thus dense W strengthened by the suitable second phase can be the most promising candidate material for the PFM especially in the divertor components.

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