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Demonstrating tungsten fiber-reinforced porous-matrix tungsten composites for future fusion application

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Abstract

Tungsten fiber-reinforced tungsten (W_f/W) has been developed to improve the fracture toughness of W materials, as demonstrated in previous studies (2019 *Nucl. Fusion* **59** 086034; 2021 *Mater. Sci. Eng. A* **817** 141361). In the present study, we focus on the performance of the developed W_f/W materials under fusion-relevant test conditions and further demonstrate their use as plasma facing materials in future fusion reactors. Specifically, one set of W_f/W samples was exposed to Ne plasma to investigate the erosion resistance against plasma sputtering, in comparison to the reference ITER-grade W sample. In addition, deuterium (D) retention in the plasma-exposed W_f/W samples was studied via thermal desorption spectroscopy. Furthermore, laser thermal shock tests were performed on W_f/W to simulate the transient heat load condition and to investigate the material performance under extreme heat flux. With increasing porosity, W_f/W exhibits lower mass loss (net erosion) after Ne plasma exposure. Though porous, W_f/W composites unexpectedly show a comparable D retention to the reference bulk W, which is attributed to the openness of the pores in the matrix. Thermal shock testing results indicate a similar cracking threshold (0.38 GW m^{-2} , 1 ms) as compared with that of ITER-grade W materials. However, due to the lower thermal conductivity of porous matrix W_f/W , under extremely high loading conditions (1.6 GW m^{-2} , 2 ms) surface melting was observed. The present work demonstrates the great potential of the porous matrix W_f/W for future fusion application.

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Keywords: tungsten fiber-reinforced tungsten, short fibers, porous matrix, plasma erosion, deuterium retention, laser thermal shock

(Some figures may appear in colour only in the online journal)

1. Introduction

For use as plasma-facing materials (PFM) in future fusion reactors, candidates require advanced mechanical and thermal properties [3] to meet significant challenges in harsh environments. This is the case particularly in the divertor region, where surfaces are subjected to extensive particle and heat fluxes. Tungsten is one of the most suitable materials for use as PFM in the divertor region due to its favorable physical properties, such as low sputtering yield, high sputtering threshold, high thermal conductivity and high melting point. However, the intrinsic brittleness of W materials has strongly limited their applications in fusion reactors. This brittleness issue is foreseen to be further worsened when exposed to fusion plasma with neutron irradiation [4–7]. Intensive efforts have been devoted to overcoming the brittleness of tungsten or enhancing the fracture toughness. Toughening techniques can be divided into two groups, i.e., intrinsic and extrinsic toughening. The former principle includes adding nanoparticles [8, 9], grain refinement [10], and alloying [11]; while techniques applying composites, e.g., multilayer composites [12, 13] and tungsten fiber-reinforced composites [14–16], belong to extrinsic toughening mechanisms. Given the fact that cracking is almost inevitable in the harsh fusion environments with neutron embrittlement, the extrinsic toughening strategy seems to be more applicable for W materials. Relying on the extrinsic toughening mechanism [17], various types of tungsten fiber-reinforced tungsten composites (W_f/W) have been developed in the past few years [2, 16, 18–22], e.g., W_f/W contents 30% fiber mass fraction and an yttrium oxide interface between fibers and dense matrix to realize the toughening effect [16]. The improved fracture toughness relies on the extra energy consumption mechanisms (e.g., crack deflection, interface debonding, fiber ductile deformation, etc) enabled by the weak bonding between the fibers and matrix. In previous studies [16, 20, 23] on W_f/W , the weak bonding was mainly realized by a dedicated oxide interface. However, the introduction of the oxide interfaces turned out to be costly and technically challenging, and has therefore strongly limited the large-scale production of W_f/W . Recently, a new concept of porous matrix W_f/W (PoMa- W_f/W), with typically 80%–88% relative density tungsten matrix, has been proposed and fabricated [2, 22] applying a controlled matrix porosity instead of introducing oxide interface to realize the weak connection between fibers and the matrix [24]. It has been demonstrated [2] that such novel PoMa- W_f/W materials exhibit significantly improved fracture toughness and crack resistance compared with pure W. More importantly, it does not require the dedicated yttrium oxide coating to realize the weak bonding between fiber and

matrix, and thereby promising great potential in large-scale production for future application in fusion reactors.

In our previous work [2], it has been reported that PoMa- W_f/W exhibits an increased fracture toughness compared to pure W relying on the extrinsic toughening mechanism. In the present study, to further demonstrate the suitability of applying PoMa- W_f/W as PFMs in future fusion reactor, we investigate the materials performance under plasma exposure with focus on hydrogen isotopes retention and erosion [3, 25–27]. Specifically, PoMa- W_f/W with different relative density have been produced and exposed to deuterium (D) plasma at the linear plasma device PSI-2 in Forschungszentrum Juelich [28]. The D retention after plasma exposure was probed using thermal desorption spectroscopy (TDS). Neon (Ne) plasma was applied to study the erosion behavior of W_f/W under plasma exposure. In addition, the cracking behavior of the PoMa- W_f/W under transient heat load is studied. Pulsed laser was shot to sample surfaces to simulate the transient heat load during fusion operations.

2. Experimental

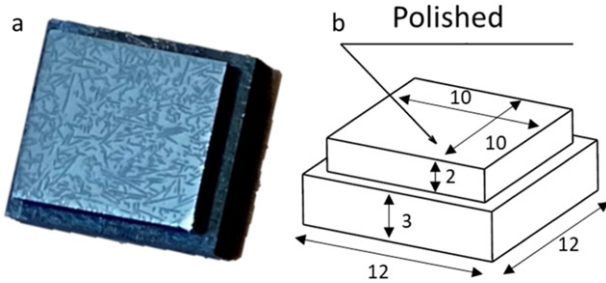
2.1. Sample preparation

Pure tungsten powders (OSRAM GmbH) with 5 μm average particle size and potassium doped short tungsten fibers (OSRAM GmbH) with 2.4 mm length and 0.15 mm diameter were employed as the raw materials to manufacture PoMa- W_f/W via field assisted sintering technology [22]. Fibers with a very fine and elongated grain structure, due to the drawing process upon production, show good elongation (up to $\sim 3\%$) and extremely high tensile strength (up to 3000 MPa) [29]. In addition, during the production, potassium (~ 75 ppm) doping is used to strengthen the microstructural stability. Potassium is insoluble in tungsten, forming dispersed nanobubble rows along the grain boundaries of the elongated grains, which can resist the growth of grains during recrystallization [30]. It has been reported that even after annealing at ~ 2170 K for 30 min, the potassium-doped W fibers can still show ductile behavior during tensile testing at room temperature [30].

For the consolidation process, short tungsten fibers and powders were mixed by manual shaking in a vessel. The mixture was then spread into a columnar graphite mold with 20 mm in diameter. Tungsten foils with a thickness of 0.025 mm were used between the to be sintered sample and the graphite mold to mitigate possible carbon contamination during sintering process [31]. To acquire W_f/W samples with different matrix porosity, various sintering temperatures were used, as listed in table 1. The sintering was performed in vacuum with a base pressure below 0.1 mbar. As a result,

Table 1. Sample production parameters.

	$W_f/W_{\#1}$	$W_f/W_{\#2}$	$W_f/W_{\#3}$	Reference W
Sintering temperature	~ 1820 K	~ 1670 K	~ 2170 K	—
Pressure	60 MPa	60 MPa	60 MPa	—
Fiber mass fraction	40%	40%	30%	—
Density	$\sim 85\%$	$\sim 78\%$	$\sim 93\%$	$\sim 99\%$

**Figure 1.** (a) Typical W_f/W irradiation sample, the dark particles are the short fibers; (b) sample geometry of plasma exposure and laser shock in PSI-2.

coin-shape samples (20 mm in diameter and ~ 6 mm in height) were produced. Here, the reference W in this work was made from sintering followed by a forging process manufactured by Plansee, according to ITER specifications with average (SEM measured) grain size between 1 and $10 \mu\text{m}$, slightly elongated perpendicularly to the exposed surface. A more detailed characterization can be found in [32]. This reference W here has been a widely investigated as the candidate for plasma facing material. The reason of choosing this material as the reference W is due to the fact that we would like to demonstrate the potential of using W_f/W as plasma facing material and compare the correlating behavior with other ‘standard’ candidates.

Afterwards, the samples were cut into standardized shaped samples for testing in PSI-2 device with a dimension shown in figure 1, using electrical discharge machining. The sample surfaces were afterwards mechanically ground and polished until $0.25 \mu\text{m}$ (polishing particle size) for a mirror finish. After polishing, the Ra of the reference W and $W_f/W_{\#1}$, $W_f/W_{\#2}$ and $W_f/W_{\#3}$ are 19 nm, 1355 nm, 393 nm and 239 nm, respectively; the Rz are 134 nm, 22 406 nm, 11 542 nm and 19 550 nm respectively. The reason for the high surface roughness of the PoMa- W_f/W is due to the relatively large height difference between the fiber and matrix after polishing and nature of high roughness of the porous surface.

The typical microstructure of the prepared W_f/W samples is shown in figure 2. Randomly oriented and distributed fibers can be seen in the tungsten matrix, which is in a porous state and shown in figure 2(b). For more details on the microstructure of the PoMa- W_f/W , the reader is referred to our previous study [2].

2.2. Plasma erosion

To investigate the plasma erosion resistance, PoMa- W_f/W and reference pure W were exposed to Ne plasma in PSI-2 device with active water cooling in the backside of the sample. The

sample temperature during Ne plasma exposure was monitored by a thermal couple attached to the backside of the sample. The average bias voltage between the neon plasma and the samples was 108 eV; the total exposure fluence was $3.65 \times 10^{24} \text{ Ne}^+ \text{ m}^{-2}$. During the exposure, the sample temperature was ~ 320 K.

The sample masses before and after Ne plasma exposure were measured by micro-balance to access the mass loss caused by the plasma erosion. The evolution of sample surface morphology due to erosion experiments was characterized using scanning electron microscope (SEM, Zeiss DSM 982).

2.3. Deuterium retention

An independent set of samples (including PoMa- W_f/W and reference W) were exposed to deuterium (D) plasma at PSI-2 devices to study the retention behavior of hydrogen isotopes in PoMa- W_f/W . Before the D plasma exposure, all the samples were annealed at 1270 K for 100 min in order to degas and release the residual stress. Experimental conditions were set similarly to the above-mentioned Ne plasma exposure, except for the incident ion species. The average bias voltage between the D plasma and the samples was 78 eV; the total fluence was $4.99 \times 10^{25} \text{ D}^+ \text{ m}^{-2}$. During the exposure, sample temperature was stabilized at around 470 K, which was monitored using a thermal couple attached to the back side of the sample.

After D plasma exposure, the samples were stored in a vacuum chamber (10^{-2} mbar) for 6 months. Afterwards, the total deuterium retention was measured by TDS up to 1000 K in ‘TESS’ [33] at IPP Garching Germany, where the sample was heated with a linear temperature ramp of 3 K min^{-1} and the released D-containing molecules are measured by quadrupole mass spectroscopy, including HD (mass 3), D_2 (mass 4), HDO (mass 19), D_2O (mass 20), and other mass channels, but only HD and D_2 were later taken into account for the quantification of D amount due to the difficulty in calibrations HDO and D_2O signal. The QMS signal for D_2 was calibrated to absolute desorption fluxes by an independent measurement using a calibrated leak (Laco Technologies). The calibration factor for HD is taken as a factor of 0.6 as that for D_2 signal.

2.4. Laser thermal shock

To investigate the materials behavior under transient heat load condition, laser beam exposure was applied in PSI-2 device. A Nd:YAG laser (LASAG FLS 352 N) was used ($\lambda = 1064 \text{ nm}$), with absorbed power densities between 0.19 GW m^{-2} and 1.6 GW m^{-2} [34]. The power profile of the laser beam is identical with the profile in [34]. A circular area with a diameter of 3.6 mm was exposed to 1–1000 pulses, with a pulse duration

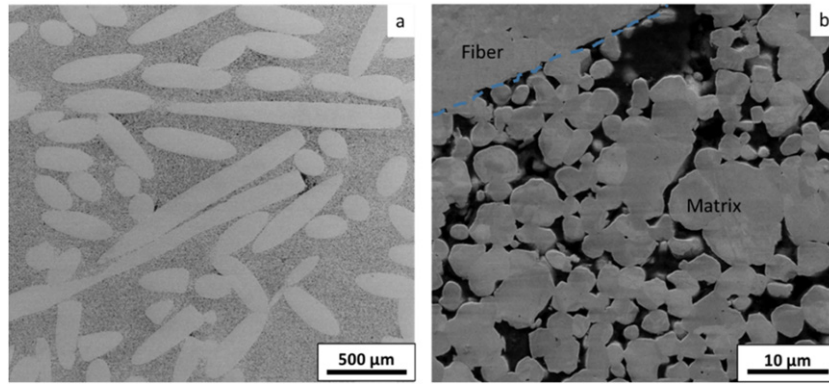


Figure 2. Typical microstructure of PoMa-W_f/W (85% relative density).

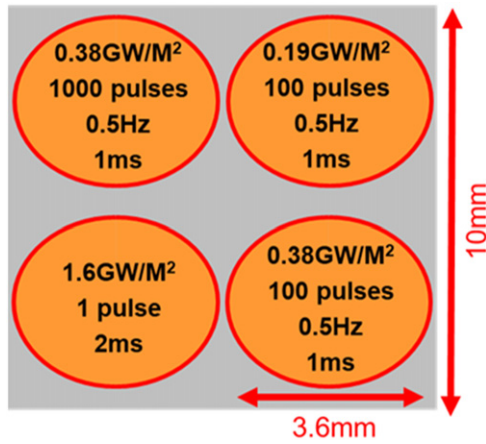


Figure 3. Schematic diagram of thermal shock spot on W and W_f/W samples.

of 1–2 ms. The pulse frequencies were 0.5 Hz in order to allow sample cooling down to room temperature after each pulse. For each sample, four spots were tested with different power densities, pulse numbers or durations. The laser shots parameters on each spot are shown in figure 3. The surface morphology after the laser beam shock was analyzed by SEM (Zeiss, Crossbeam 540) equipped with focus ion beam (FIB) for cross-sectioning.

3. Results and discussion

3.1. Plasma erosion

One major concern upon applying porous matrix for PFM is the possible particle flaking under bombardments of energetic ions. On the one hand, particle flaking leads to additional weight loss beyond that induced by plasma erosion. The exfoliated particles or pieces will impose a lot of impurities into the core plasma and may lead to plasma disruption. High net erosion and particle shedding will both limit the life time of plasma facing component and therefore should be avoided.

Assuming a normal incidence for all the impacting particles, the theoretical mass loss from a flat surface due to plasma

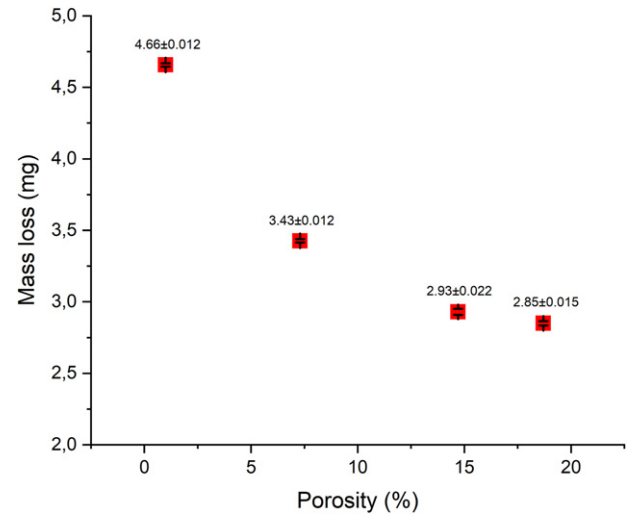


Figure 4. Weight loss vs porosity after Ne plasma (108 eV) irradiation for pure W and W_f/W with different matrix density.

sputtering can be calculated by [35]:

$$\Delta m_{\text{weight}} = \Phi \cdot Y \cdot S \cdot m_W. \quad (1)$$

Where Δm_{weight} is the theoretical mass loss, Φ the incident particle fluence, Y the sputtering yield at given ion energy, S the surface area, m_W the mass of the tungsten atom.

Applying the sputtering yield of W by 100 eV Ne ions in [35], the theoretical mass loss of pure W during the Ne plasma exposure is calculated as 4.0 mg. It should be pointed out that in equation (1) redeposition of sputtered particles is not considered. In other words, the experimentally measured weight loss after plasma erosion should be typically lower than that calculated by equation (1) due to redeposition of sputtered particles. In our experiments, the mass losses caused by plasma erosion for both the reference W sample and several PoMa-W_f/W samples with different porosity are measured, as plotted in figure 4. The reference W sample is plotted just assuming a 1% porosity, which shows slightly higher mass loss compared to the theoretical value (4 mg). This is attributed to the sputter erosion induced by other impurity ion species contained in the PSI-2 plasma beam during neon plasma operation [36], which is not considered in equation (1). However,

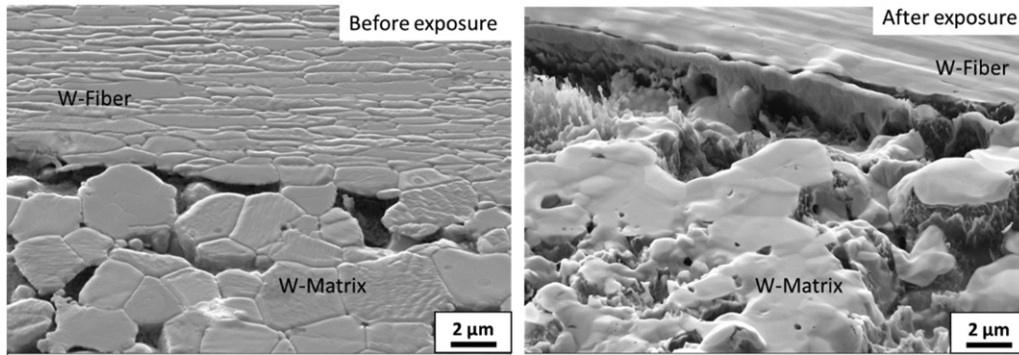


Figure 5. Surface morphology of W_f/W (85% relative density) before and after Ne plasma irradiation. In order to manifest the surface roughness more clearly, the samples are tilted for 54° here.

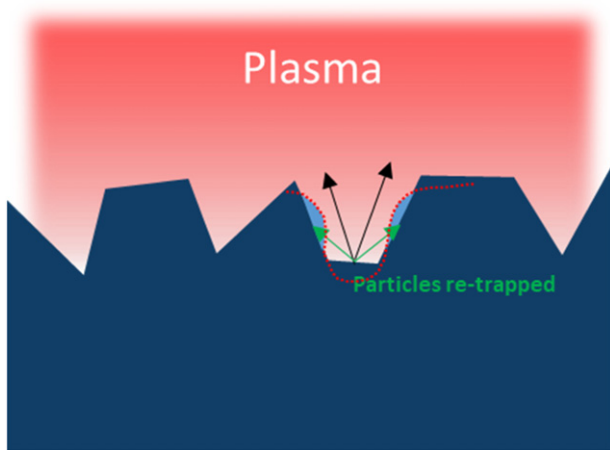


Figure 6. Illustration of surface roughness influence on the plasma erosion effect, dashed line marked the possible surface after erosion.

for the W_f/W with different porosities (figure 4), the mass losses are less than the theoretical value, despite the influence of the impurities. This indicates a relatively strong redeposition occurring on PoMa- W_f/W surfaces. Another interesting aspect in figure 4 is the decreasing trend of the mass loss as a function of the sample porosity, which will be discussed in next paragraph.

To understand the lower net erosion of PoMa- W_f/W , surface morphologies of samples with different porosity were characterized using SEM. Figure 5 represents the typical surface morphology of W_f/W sample (85% relative density) before and after Ne plasma exposure. Due to the high porosity, the surface before exposure looks already quite rough; pores/gaps between the tungsten grains are visible. Fine grain structures of tungsten fibers can also be seen on the surface as the smooth part. After Ne plasma exposure, great changes have taken place for the porous W matrix in terms of surface morphology. Grassy features have formed in the gaps and edges of single particle become roundish, similar to the results in [37]. These grassy features are typical morphology after redeposition. For tungsten fibers with flat surface areas present in the exposed surface, no significant change can be distinguished before and after Ne plasma exposure, demonstrating a less effective redeposition.

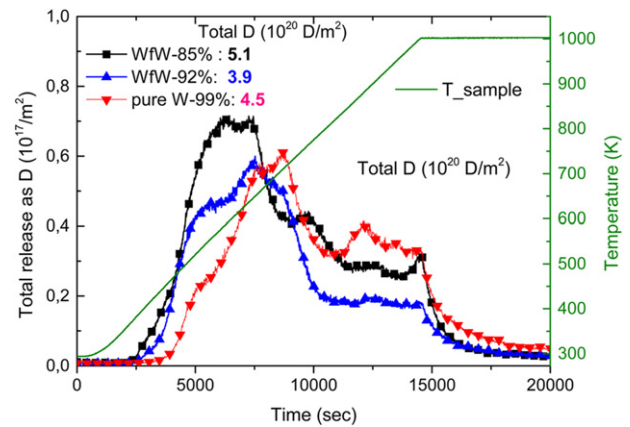


Figure 7. D retention measured by TDS test up to 1000 K (3 K s^{-1} , green) for the sample W_f/W with 85% density (black), 92% density (blue) and reference W (red).

Based on the above results, the lower mass loss of PoMa- W_f/W samples compared with the theoretical value can be referred to the re-deposition effect due to high surface roughness of the matrix. As sketched in figure 6, when the surface roughness is high, the particles being sputtered away can be re-trapped by the surface bumps with much higher probability, as it has been reported in [38, 39]. Additionally, according to the less mass loss of PoMa- W_f/W , together with the SEM observation, the loose matrix structure does not cause the shedding of W particles under plasma erosion.

3.2. Deuterium retention

Besides the concern on possible higher erosion upon applying porous materials as plasma facing components, another major worry is the fuel retention in the porous structure [40, 41]. To measure the D retention in the PoMa- W_f/W materials, samples were exposed to D plasma together with a reference W, followed by a TDS measurement up to 1000 K.

The D-release spectra measured by TDS for PoMa- W_f/W and reference W are shown in figure 7. All three spectra exhibit multi-peak structure and span over a large temperature range. D desorption spectrum of the PoMa- W_f/W samples start at temperature of $\sim 400 \text{ K}$ while the reference W starts at $\sim 450 \text{ K}$. Typically, the release-starting temperature in TDS signal can

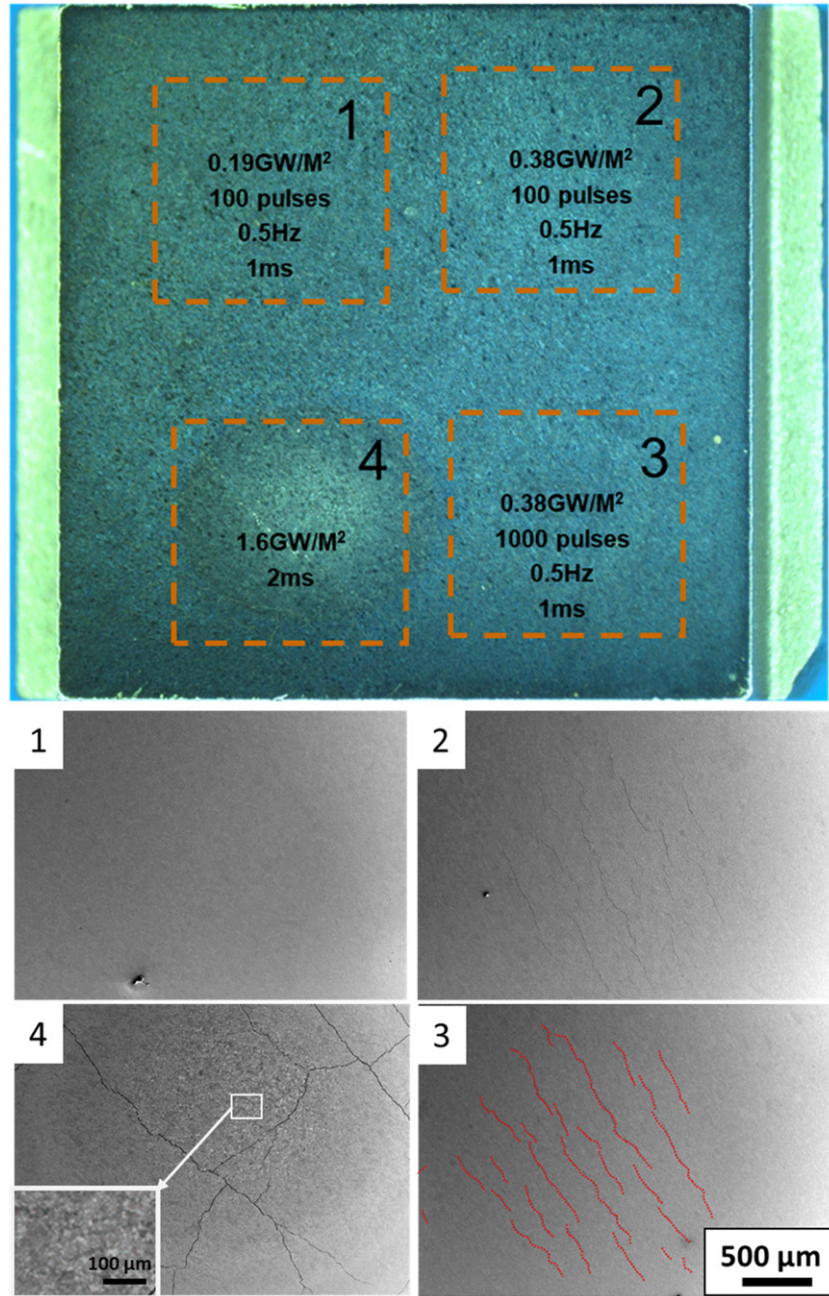


Figure 8. Surface morphology of reference W after laser thermal shock testing.

be corresponded to the sample temperature at the end of exposure to hydrogen. In our case, once the plasma beam is switched off, the sample starts cooling. But D_2 gas injection has not been ceased immediately. It is highly probable that samples can absorb some D_2 gas at the surfaces and the pores for the cases of PoMa- W_f /W. We attributed the D release at temperature slightly below the exposure temperature (~ 470 K) to the absorbed part during the cooling process. Our focus via the TDS measurements are on comparing the total amount of retained D in the samples. As labelled in the legend of figure 7, the total amount of retained D in W_f /W with 85% (5.1×10^{20} D m $^{-2}$) and 92% (3.9×10^{20} D m $^{-2}$) density are comparable to that in reference W (4.5×10^{20} D m $^{-2}$). Given the fact that there are many pores in both W_f /W samples, the

comparable D retention can only be attributed to the openness of these pores (i.e., pore networks) in the matrix [31]. With open pore networks, the implanted D particles will easily be reemitted or they can penetrate throughout the sample [42]. Therefore, the high porosity will not cause higher D retention compared to the dense bulk W. This is stressed as the most important finding in the present work.

3.3. Laser thermal shock

Apart from erosion and fuel retention, another worry when using PoMa- W_f /W as plasma facing materials is its relatively lower thermal conductivity due to high porosity. As reported in [2], the thermal conductivity of PoMa- W_f /W is considerably

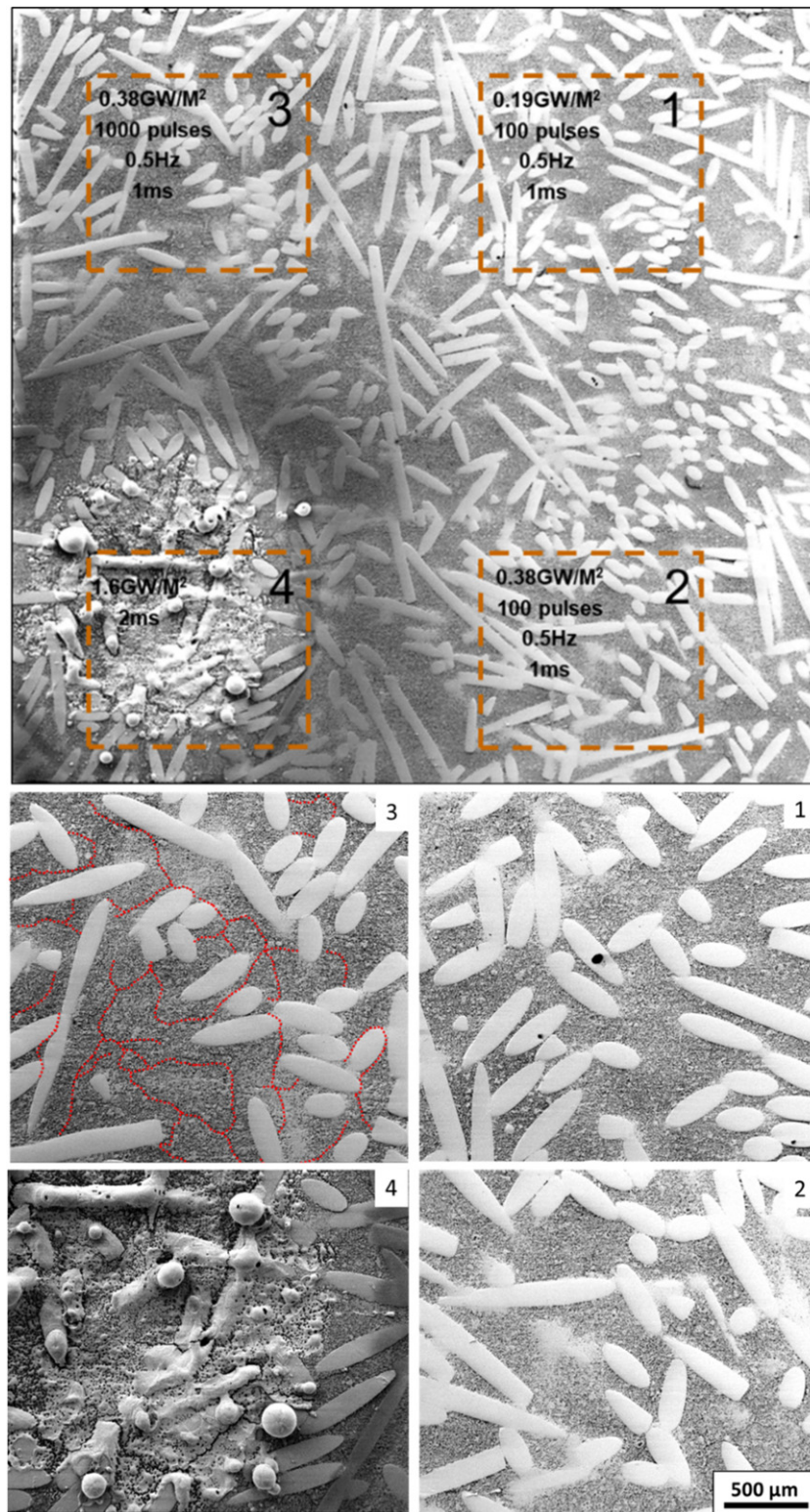


Figure 9. Surface morphology of PoMa-W_f/W (80% relative density) after laser thermal shock testing.

lower than the bulk W. This lower thermal conductivity could lead to higher thermal stress [43] and, in very high loading condition, surface melting, especially for the case of transient heat load condition in future fusion operation.

To study the performance of PoMa-W_f/W materials under high thermal loads/stresses, pulsed laser beam shock was

applied. During each laser pulse, the locally heated spot tends to expand due to thermal expansion and shrink during cooling. On the other hand, the unheated adjacent area around shocked spot restricts the expansion and shrinkage, which leads to high stress and fatigue for cycled loading [44]. This could cause crack initiation in tungsten as reported in [45]. The test aims

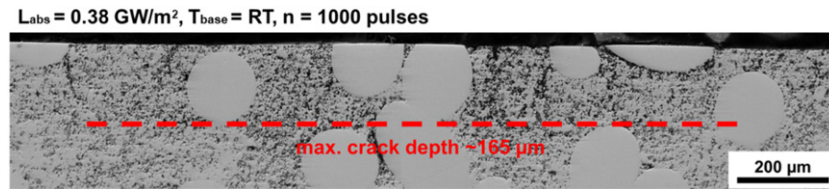


Figure 10. Cross section of W_f/W (85% relative density) after laser beam shock with a loading condition of 0.38 GW m^{-2} for 1 ms with a pulse number of 1000 (spot 3).

to simulate the material behavior under high thermal stress and fatigue under cycled exposures in future fusion operation. After pulsed laser shock, the surface morphologies of reference W and typical PoMa- W_f/W are shown in figures 8 and 9. In both figures, the four shock spots are magnified to display the crack formation. To emphasize the typical crack profile, the cracks are marked by red dash lines for spots 3.

For reference W, for spot 1 (figure 8(1), 0.19 GW m^{-2} , 100 pulse), no cracks or any surface morphology changing are observed. With a power density of 0.38 GW m^{-2} for 100 cycles (spot 2, figure 8(2)), cracks appear in a straight parallel pattern. After 1000 cycles with the same power density (spot 3, figure 8(3)), no significant change can be observed in terms of cracking morphology in comparison with that after 100 cycles (spot 2, figure 8(2)). When the sample is loaded with an extremely high power of 1.6 GW m^{-2} for a long duration of 2 ms (figure 8(4)), large cracking networks were built-up instead of the parallel pattern. A careful comparison with that in spot 2 & 3 (figures 8(2) and (3)) gives rise to an enhanced surface roughness and also larger grain size, indicating the occurrence of recrystallization. According to previous thermal fatigue study on W [46], the damage threshold for ITER-grade W under heat load is between 0.19 GW m^{-2} and 0.38 GW m^{-2} . The observed results in terms of crack formation and surface morphology evolution after laser thermal shock testing are consistent with the previous study.

For PoMa- W_f/W (figure 9), the damaging behavior of PoMa- W_f/W under laser thermal shock is in general very similar to reference W. Similar damage threshold (between 0.19 to 0.38 GW m^{-2}) in terms of crack appearance is found (see figures 9(2) and (3)). The number of cracks is also comparable for PoMa- W_f/W and reference W. However, the cracking pattern at the exposed surface is rather a network in PoMa- W_f/W than straight lines in reference W sample. This is a clear sign of crack deflection that can be attributed to tungsten fibers. Namely, cracks appear only within the matrix, no crack can pass through any W-fiber by a careful search. These aspects demonstrate our proposal on extrinsic strengthening, when applying short W fibers in porous matrix. Further testing on PoMa- W_f/W materials with much higher power density and pulse duration (1.6 GW m^{-2} , 2 ms) gives rise to surface melting (see spot 4, figure 9(4)). This phenomenon is not observed for the reference W sample with the same loading condition (figure 8(4)). It is attributed to the lower thermal conductivity of PoMa- W_f/W .

In order to further demonstrate the cracking behavior of PoMa- W_f/W , cross section of the laser shock spot (0.38 GW m^{-2} for 1 ms with a pulse number of 1000) was

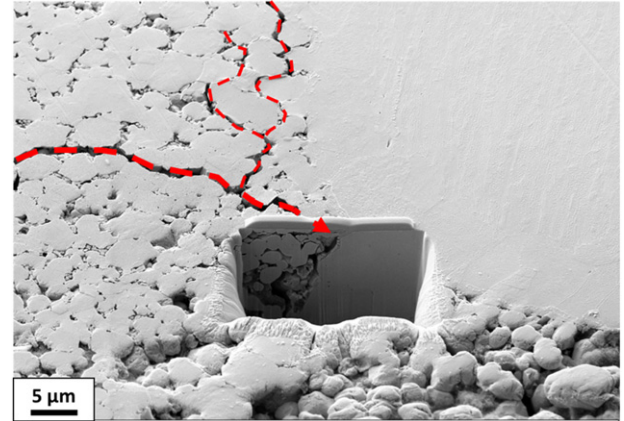


Figure 11. SEM image showing crack stopped by the fibers for W_f/W (92% relative density), sample preparation prepared by FIB cut.

observed, as shown in figure 10. Additionally, the cracking path is traced in figure 11. Here a small crack cross section is prepared by focused ion beam (FIB).

From figure 10, the maximum crack depth is measured as $\sim 165 \mu\text{m}$ for this sample, which is in the same range compared to pure W in previous studies [46, 47]. Additionally, it can be seen that the cracks are not in a sharp manner. They are passivated due to the porous matrix. Compared to a sharp crack in a normal tungsten, the stress concentration at the passivated crack tip is much lower [48], hindering the further crack propagation. From figure 11, one can see that the cracks are deflected by a fiber. This mechanism is often reported in the fiber-reinforced composites. The deflected cracks lead to larger cracking surface, consuming higher fracture energy.

To sum up, although PoMa- W_f/W has to suffer a higher thermal stress due to lower thermal conductivity, apart from under the extremely high loading condition, the cracking behavior is still comparable to ITER-grade W, as both cases give a similar cracking threshold with similar numbers of cracks. This can be attributed to two effects: (a) the crack tip passivation due to the porous matrix and (b) the crack deflection by the tungsten fibers. Considering the much higher high fracture toughness of PoMa- W_f/W , the initiated surface cracks cannot easily propagate through the material causing unexpected failure, which gives a promising advantage when using PoMa- W_f/W as the plasma facing materials.

4. Conclusion

Our previous work demonstrated the much higher fracture toughness of W_f/W compared to pure W [1], especially after

neutron irradiation [49]. The present work further studies the performance of the developed PoMa-W_f/W materials under fusion-relevant exposure conditions. Our testing experiments of PoMa-W_f/W materials on erosion resistance during Ne irradiation, D retention after D plasma exposure and cracking threshold by laser thermal shock show better or at least similar results in comparison with those from reference bulk W samples. The generally positive results have largely eliminated the worries possibly induced by the applied porous matrix, such as particle flaking, rather high hydrogen fuel retention, etc. The severe melting of PoMa-W_f/W materials under extreme loading condition (1.6 GW m⁻² for 2 ms), being the solely observable drawback under the applied testing conditions, is attributed to the lower thermal conductivity due to the porous matrix. However, since such extreme heat load is also rare and should be avoided during the operation of future fusion reactors, we deem that the observed melting is less relevant to future fusion application compared with other tested properties. Therefore, the developed PoMa-W_f/W, allowing for large-scale production, is a promising candidate for application in a future fusion reactor.

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