

Micro-structured tungsten: an advanced plasma-facing material

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ABSTRACT

A micro-structuring of the tungsten plasma-facing surface can strongly reduce near surface thermal stresses induced by ELM heat fluxes. This approach has been confirmed by numerical simulations with the help of ANSYS software. For experimental tests, two $10 \times 10 \text{ mm}^2$ samples of micro-structured tungsten were manufactured. These consisted of 2000 and 5000 vertically packed tungsten fibres with dimensions of $\varnothing 240 \mu\text{m} \times 2.4 \text{ mm}$ and $\varnothing 150 \mu\text{m} \times 2.4 \text{ mm}$, respectively. The 1.2 mm bottom parts of the fibres are embedded in a copper matrix. The top parts of the fibres have gaps about of $10 \mu\text{m}$ so they are not touching each others. The top of all tungsten fibres was electro-polished. A Nd:YAG laser with a pulse duration 1 ms and a pulse repetition frequency of 25 Hz was used to simulate up to 10^5 ELM-like heat pulses. No damage on either of the micro-structured tungsten samples were observed. Neon plasma erosion rate and fuel retention of the micro-structured tungsten samples were almost identical to bulk tungsten samples.

1. Introduction

Plasma facing materials (PFM) and components (PFC) are one of the main likely showstoppers for commercial fusion reactors, and still unsolved key components on ITER and DEMO road. PFCs have to fulfil many specifications, one of which ensures a power handling in the range of $10\text{--}20 \text{ MW/m}^2$ at the divertor region, an overall as low as possible fuel retention because of the safety restriction for tritium inventory, but also a long as possible life time, and reduced maintenance cost for commercial fusion viability. Tungsten has the highest melting temperature off all refractory metals tungsten and generally favourable material properties to sustain such high heat fluxes, of which the highest melting temperature. However, a major concern about tungsten is its ability to handle repetitive transient thermal loads or thermal shocks [1,2]. Almost all tungsten alloys based PFMs have been facing major damages (including cracks, microstructural changes and roughness increase) during repetitive thermal shock testing. ITER will have to face about 10^8 type I ELM events [3] that existing tungsten based PFM cannot withstand without cracking, even when mitigated to power densities below 1 GW/m^2 [1,2]. Several approaches have been intended to improve this aspect (by alloying tungsten or with specific grain microstructure), where one of them, is the promising tungsten fibre reinforced composite (W_fW) [4]. The proposed micro-structured tungsten

described in this paper is another approach and has been evaluated as potential PFM.

2. Micro-structured tungsten concept and experiment background

2.1. Micro-structured tungsten as PFM the concept explained

A critical issue of tungsten when used as PFM is its propensity to crack under thermal shocks. The rapid surface heating during rapid changes in the power load can create large stresses in the PFC because the temperature of the near surface heated region is increased in a shallow layer with a depth of about $\delta \approx 2 \cdot (\alpha \cdot \tau)^{1/2}$, where α is the thermal diffusivity and τ - the heat flux pulse duration. The temperature increase causes the thermal expansion of the heated layer, but the motion of the heated material is restrained by the colder surrounding material resulting in the stress development. This strain tends to accumulate after each thermal cycle until induced stress overcomes material ultimate tensile strength (UTS). In most cases, this UTS corresponds to its 0.2% yield strength (YS) when operating below ductile to brittle transition temperature (DBTT) (as tungsten in such case is brittle) [5]. Depending on the heat wave depth penetration and cycling frequency, the generated temperature gradient and its resulting stress can even induce delamination or detachment of upper layers or grains through

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crack propagation parallel to surface. While this mechanism is not specific to tungsten and can be found in almost any PFM candidate, this unwanted behaviours is also increased there by the fact that tungsten does not provide much (if any) ductility that would allow to dissipate thermal stress without failure, by plasticity deformation, in practical temperatures ranges (between 100 and 300 °C for tokamak first walls). While cracking is most pronounced at lower temperatures, surface degradation and crack formation is observed for repeated thermal cycling even with temperature higher than DBTT [1,2].

Implicitly, the ways to solve this issue, is to restrict the cracks propagation or to bring some increased ductility (or pseudo ductility) to the tungsten, and this possibility is investigated by alloying tungsten with other elements (e.g. Ta, La, Re, Ir) but with some limitations for each of them [6], by cold-rolling pure tungsten [7], by using fibre-reinforced composites (W_fW) [4] or tungsten laminated composites [8]. However, a remaining question of all these approaches is their stability over time and use. The most obvious is the transmutation of tungsten (into rhenium for example) due to neutron irradiation, which tend to severely complicate the possibility of keeping the achieved (pseudo-) ductility all along fusion reactor/tokamak operation time if relying on a specific alloying mix. It is also worth mentioning that the increase of the irradiation damage levels is accompanied by creation of gas bubbles and cavities formation. The same problem occurs for PFM solutions relying on specific grain structures (e.g. texture strengthening, ultra-fine grain material) to achieve some ductility when considering that recrystallisation cannot be excluded, especially in experimental tokamaks like ITER, which compromise such approaches.

The approach of micro-structured tungsten [9] tries to overcome such problems by removing the need of ductility by significantly reducing and mitigating the thermal stress by allowing the PFM to freely expand. This is done by creating a pattern having gaps normal to the PFC surface as shown on Fig. 1. This results in tungsten structured in 2D patterns facing the plasma and bounded to the structural material at the bottom. One of the simplest possible pattern is an arrangement of tungsten cylinders compactly stacked in a hexagonal lattice (Fig. 2a).

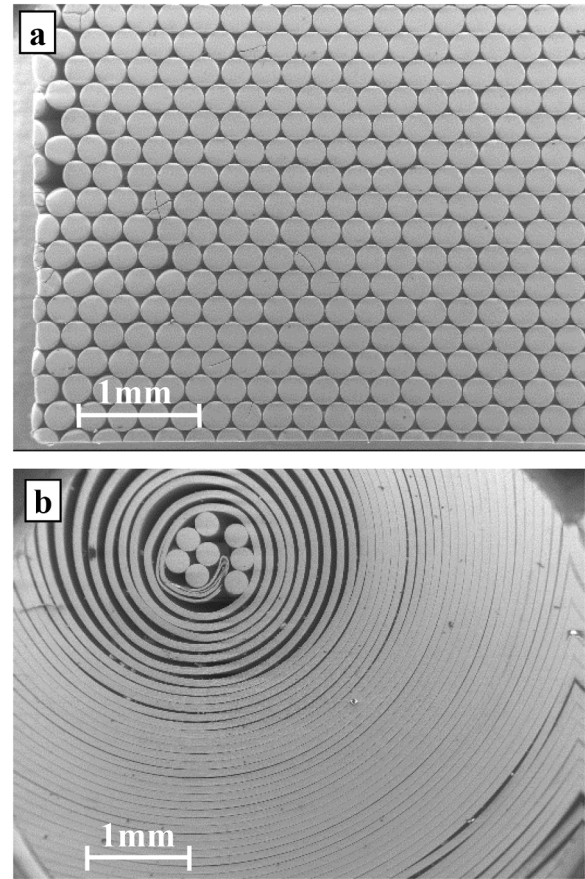


Fig. 2. SEM images of a) 1D and b) 2D realization of micro-structured tungsten concepts (top view).

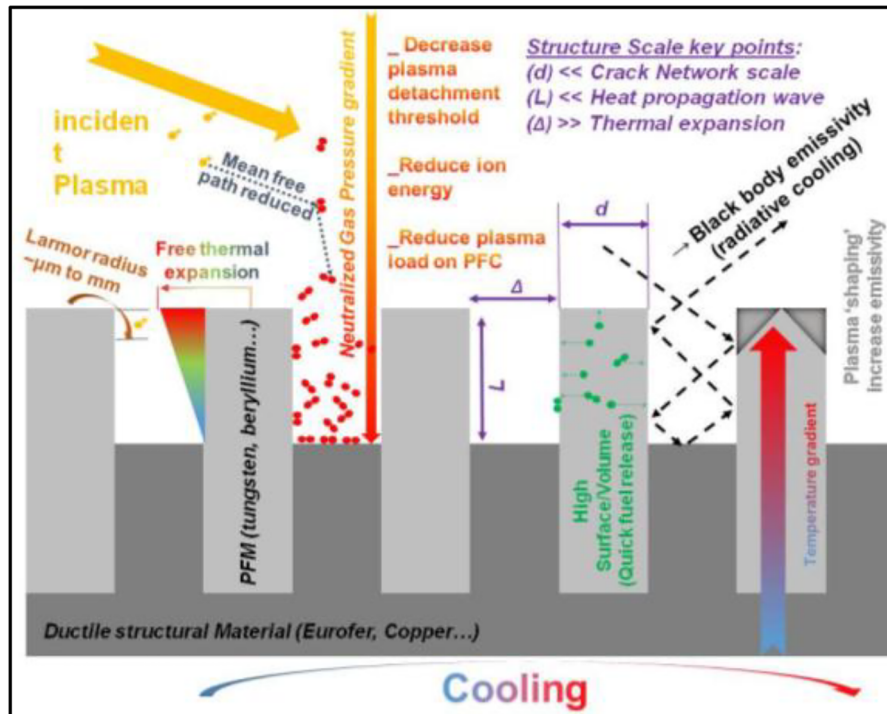


Fig. 1. Micro -structured tungsten concept principle (not to scale).

However it is also possible to explore different 2D geometries that would be applied to a laminated structure, for example by using a tungsten thin foil like shown below (Fig. 2b).

One of the key feature of micro-structured tungsten is to provide gaps for thermal expansion of a micro-structure elements having a typical scale along the plasma facing surface smaller than a mean size of typical thermal shock crack networks. Previous experiments [10] showed that, tungsten submitted to thermal cycling of 10–1000 cycles between 0.76 and 0.9 GW/m² was covered by islands, which were free of cracks with typical size between 0.5 and 1 mm [2]. The characteristic scale for low stress area can be lower as cracks tend to follow the grain boundaries. For this reason, the PFCs developed for the need of the experiments described in this paper, have been manufactured by using 240 μ m (Fig 2a) and 150 μ m diameter tungsten fibres, but also a 56 μ m thin foil (Fig 2b).

As shown in Fig 1, several other benefits would be expected from this structuring, beyond its intended crack resistance ability to withstand thermal cycling. The neutralised gas (hydrogen) density is expected to increase inside the proposed structure along the vertical axis compared to a flat PFC. This increase of the neutral gas density depends on many factors such as the fibre diameter, distance between fibres, the height between top of structure and structural material base, the incidence angle of plasma flux etc. Optimisation of these parameters should increase the relative pressure of neutralised gas in front of the PFC and as a consequence may reduce the effective power load on PFCs, and at the same time would improve the plasma confinement while lowering erosion. This phenomenon should be mostly effective in the plasmas with electron temperature of few eV [11].

The distance between top surface and beginning of the structural material has to be large enough since a) the incident particles should not reach the structural material (more prone to erosion), b) to take advantage of the increased emissivity (sufficient absorption happen in ‘gaps’), and c) to keep these advantages during the lifetime despite the inevitable erosion. However, this distance should be short enough for proper cooling to minimise the maximum temperature.

Spacing between structures behaves similar to a black body and increases the average emissivity of the PFC surface, improving the power handling at high temperatures. With a compact hexagonal lattice of tungsten fibres, one can expect an increase of emissivity from 0.24 for plain tungsten to 0.31, which represent about 29.5% of improvement (assuming black body radiation between fibres, gaps having multiple reflection like would in an Ulbricht sphere) which directly translate into a significant increase of the maximum heat flux that such a PFC could radiatively dissipate as shown in Fig. 3 (from 2.5 to 3.3 MW/m² at melting temperature). Last but not least, this emissivity should even increase over PFC lifetime due to the preferential sputtering of the edges of the fibres, which should lead to power handling

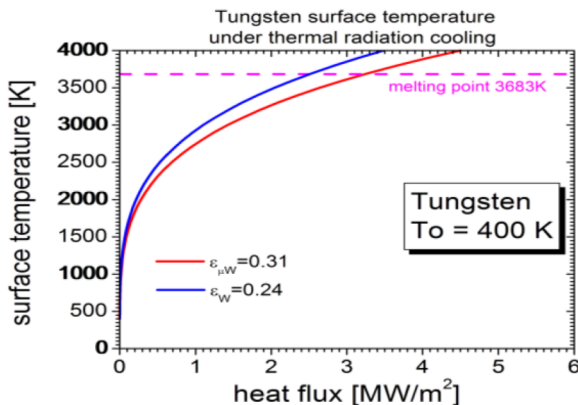


Fig. 3. Tungsten surface temperature versus radiative dissipation capability and emissivity.

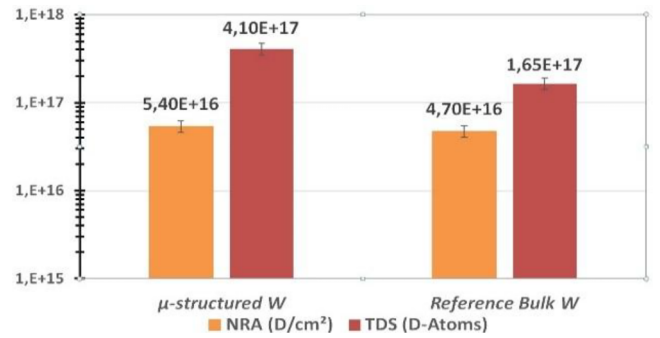


Fig. 4. Total and near surface (3.6 μ m thickness) fuel retention of micro-structured and bulk samples.

improvement over component lifetime, instead of degradation that one can expect from most other concepts and more common PFC.

The questions of sputtering and fuel retention remain to be discussed. Unlike traditional PFC using tungsten (tile, monoblock, etc.), the fuel retention in highly micro-structured tungsten is much less limited by diffusion of hydrogen isotopes in material as the PFM has much higher surface-to-volume ratio in comparison to a traditional PFM. This means that the average distance of trapped fuel to the surface is significantly reduced and implies a much quicker and easier desorption of fuel.

Sputtering itself needs to be also measured because it could annihilate any other benefits, as, because of the missing material, one could expect a shorter lifetime of a PFC using micro-structured tungsten. A question that authors are conscience of, and that, unfortunately, will not be answered in this paper, is whenever and how the eroded particles and dust, may fill gaps between fibres (co-deposits).

These two last points are critical when evaluating a PFC, as they are directly related to tritium inventory and PFC lifetime.

2.2. The prototypes

Two prototypes of micro-structured tungsten PFC were manufactured and tested. They were variations of the compact assembly of potassium doped tungsten fibres into a hexagonal lattice structure were investigated. The fibres used were 2.4 mm long in both cases having respectively 240 μ m and 150 μ m in diameter, to evaluate the influence of network lattice size on studied performances. Pure industrial grade copper was used to fill the gaps between the fibres (with 1.2 mm clearance from top) and to shape the samples to identical geometry to the reference bulk tungsten samples (PSI-2 compatible), resulting in an exposed surface of 10 \times 10 mm² for plasma interaction and about 2000 and 5100 respectively, carefully aligned fibres. The top parts of the fibres have gaps about of 10 μ m so they are not touching each others.

The reference sample was made from sintered and forged pure tungsten manufactured by Plansee according to ITER specifications with average (SEM measured) grain size between 1 and 10 μ m, slightly elongated perpendicularly to the exposed surface. A more detailed characterisation can be found in [12]. The average grain size in the tungsten fibres was slightly smaller, being spread in an interval of 0.2–2 μ m. The potassium doping might be responsible for some cracking observed during manufacturing and assembly, as it can be seen in Fig 2a). Finally, the top surface of all samples (micro-structured and bulk tungsten) was mechanically grinded, polished and finally electro-polished in 2 M NaOH solution in water to have identical mirror finished surfaces.

2.3. The experiments

These prototypes were exposed in the linear plasma facility PSI-2 to study plasma surface interaction but also for thermal shock

Table. 1
Experimental parameters relative to thermal fatigue evaluation.

Spot label	α	β	γ	δ	δ
Pulse number	10	100	1000	10,000	100,000
Absorbed power (GW/m ²)	0.62	0.62	0.562	0.62	0.50
Frequency (Hz)	0.5	0.5	0.5	0.5	25
Duration (ms)	0.93	0.93	0.93	0.93	0.93
Room temperature (°C)	28	24	26	25	24
Steady-state/end Temperature (°C)	64	116	140	194	207

investigation with a repetitively pulsed laser. The plasma exposed surfaces of the samples were perpendicular to plasma flow, and laser beam exposure was made sequentially but in same chamber [10,12,13]. The samples were then analysed by different available means: scan electron microscopy (SEM), focused ion beam (FIB), thermal desorption spectroscopy (TDS), nuclear reaction analysis (NRA) and metallography.

For the study of fuel retention, the samples were preliminary out-gassed for 2 h at 1000 °C and then exposed to deuterium plasma (as described in Section 3.3). For the erosion evaluation, neon plasma was used instead. The temperature measurement was performed by two type K thermocouples at the back side of the samples and was used to calibrate the infrared (IR) camera signal. Fast pyrometer having the 15 μ s time response and the viewing spot diameter of about 1 mm was used for the surface temperature measurement in the centre of the laser spot.

For the thermal shock tests a Nd:YAG laser (LASAG FLS 352 N) was used ($\lambda = 1064$ nm), with a pulse energies between 19 J and 23.4 J spread homogeneously over a 3 mm spot diameter. The pulse frequencies were between 0.5 Hz and 25 Hz and their duration of 1 ms (Table. 1). The base temperature of the samples (Fig. 3) was always below DBTT (230 °C maximum).

A partial mapping of each sample has been also made with SEM (resolution about 200 nm), while a complete mapping of the sample surface the identification of some fibre cracks was made with a metallographic optical microscope (resolution about 1 μ m, 100 million pixel full mapping pictures) and compared before and after experiment. The pre-characterisation allowed to identify some cracks in fibres before any plasma exposure, which appear owing to the manufacturing process mostly during the last step of preparation by electro-polishing. Generally, this allowed tracking global and local morphology changes before and after experiments, like dust formation, erosion, and melting, but also changes of tungsten grains.

3. Experimental results

3.1. Steady state power handling

The first result of this experiment was to compare the temperatures of the plain bulk tungsten sample and the 240 μ m micro-structured sample. Both samples showed during the experiment the same behaviour, same temperatures but the micro-structured sample appeared significantly brighter on the IR image which was expected from higher emissivity. However, the temperature range during experiments was clearly too low (25 °C to 350 °C) to see any benefits from higher emissivity by higher radiative cooling (i.e., no cooler temperature measured by thermocouple). It was also confirmed by Ansys simulations that in such configuration (steady state plasma), but even also in transient ones, both samples have almost the same temperatures (extremum and distribution): for example, 358 °C max and 198 °C min for bulk sample to compare with 335 °C max and 206 °C min for micro-structured sample (with copper base), both as steady state temperatures calculated for the transient δ case (Section 3.4).

3.2. Sputtering

Comparative samples (7 tungsten bulk reference samples and one 240 μ m micro-structured tungsten) were exposed to neon plasma with 95 eV impact energy of Ne⁺ ions, and under a total fluence of 5.4×10^{25} Ne⁺ m⁻². The exposition last about 100 min and, meanwhile, the steady state operating temperature was about 230 °C for all samples. All 8 of them showed about the same mass losses of around 4.5 μ g resulting to very similar sputtering yield within a standard deviation of 8.5% which is smaller than the reproducibility factor of sputtering experiments on the PSI-2 linear plasma device. It is also important to note that micro-structured tungsten using a hexagonal compact lattice of cylinders exposed a planar surface of $\pi/(2 \cdot 3^{1/2}) \approx 0.9069$ to normal incident plasma flux. This number expresses also the density relative to bulk material (cylinder stacked). However, the total surfaces of sample is larger by 7–10 times, that is important for desorption.

3.3. Fuel retention

Fuel retention needs to be carefully assessed for micro-structured tungsten as it potentially expose more surface for plasma interaction (depending of incident angle) which logically could result in higher hydrogen isotopes capture, but should also ease their release by thermal desorption. The key factor here is the temperature, because when the temperature increase the total fuel inventory stored is reduced in the PFC due to a quick diffusion of hydrogen isotopes to the surface and desorption. A first evaluation of the influence of micro-structuring on fuel retention was carried with low steady state temperature of about 180 °C.

Both reference and micro-structured samples were exposed simultaneously to deuterium plasma of 51 eV during about 150 min for a total fluence of 5.1×10^{25} D⁺ m⁻². All samples were exposed to the normal incident plasma flux. The total retention measured by TDS was by 59.6% higher for the micro-structured sample in comparison with the reference samples, though the total surface area is a factor 7 higher. This is due to the fact that D can be desorbed from a wider area than the bulk W surface. A moderate (+14.9%) increase of near surface retention in the micro-structured sample measured by μ NRA can be explained by a slightly smaller grain structure prone to some higher deuterium capture.

3.4. ELMs and thermal fatigue

The previous experiments demonstrated that micro-structured tungsten behaves very similarly to bulk tungsten in terms of sputtering and in same range concerning the fuel retention, but its main advantage over them concerning the thermal fatigue has to be verified. To evaluate it, micro-structured and bulk tungsten were exposed to different number of laser pulses (Table 1 for parameters), to simulate ELMs events.

These ELMs like simulation has been performed in the same protocol as described in [12,10,2]. In this particular case, the laser pulses were sequential to the plasma exposure, but without venting the PSI-2 chamber. The evolution of the surface temperature of the micro-structured tungsten sample due to laser pulse irradiation measured by fast pyrometer is shown on Fig. 6. The post experimental analysis shows that even 10^5 pulses of 0.50 GW/m² were not enough to reach the damage threshold of micro-structured tungsten (Fig. 5) as no single crack newly appeared. It is important to remind that, as it was previously determined and evaluated [2], but also again confirmed in this experiment with the reference samples, the damage threshold, at such ELMs representative power densities, of the bulk ITER grade tungsten, is below 10 pulses. This micro-structuring represents an improvement factor of at least 10^4 for given low temperatures. The exact positions of the laser spots on micro-structured sample can be identified by tracking

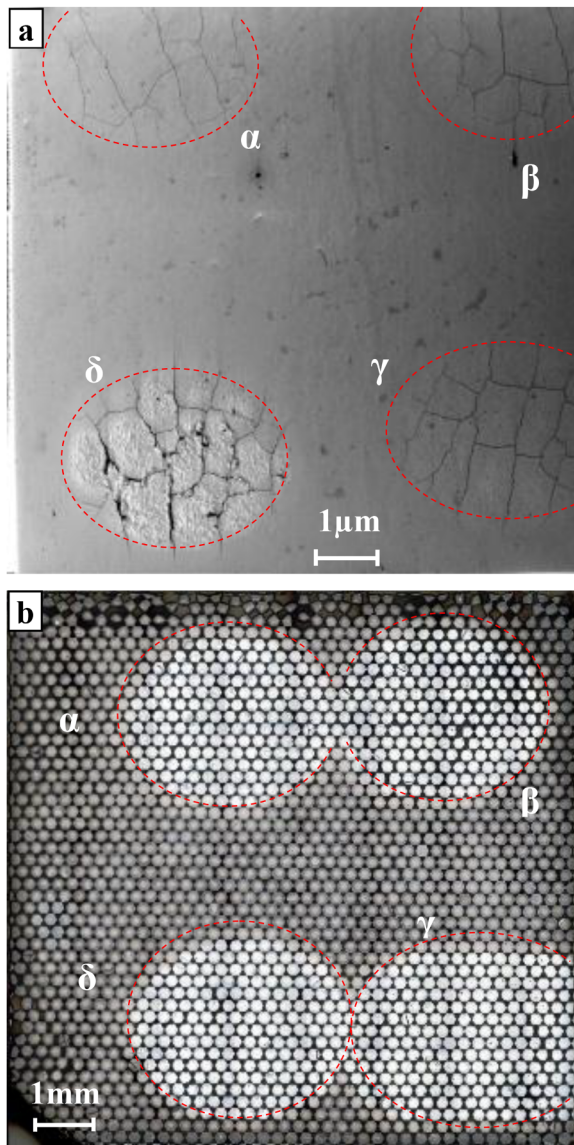


Fig. 5. Micrograph of samples after test irradiation at 10, 100, 1000 and 10,000 pulses.

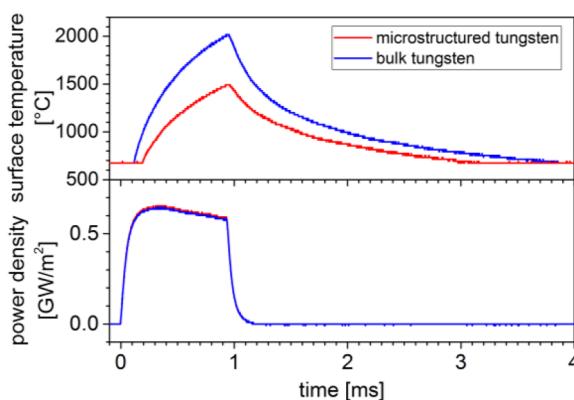


Fig. 6. Time traces of the surface temperature of micro-structured and reference tungsten sample and the laser power ($\sim 10^4$ pulse).

the molten particles in the gaps as well as by oxidation pattern, which appeared after a few days and spreading differently over exposed and un-exposed areas, likes shown Fig 5b).

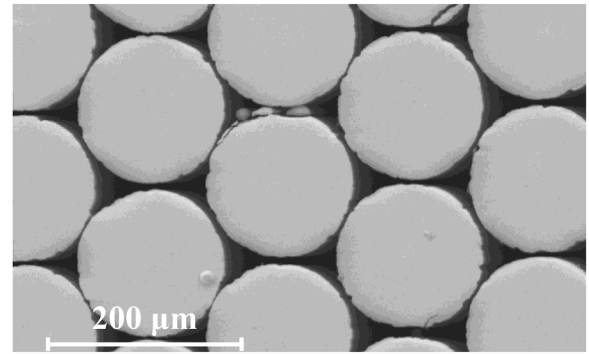


Fig. 7. SEM of micro-structured sample, with interstitial molten dust after 10^5 pulses.

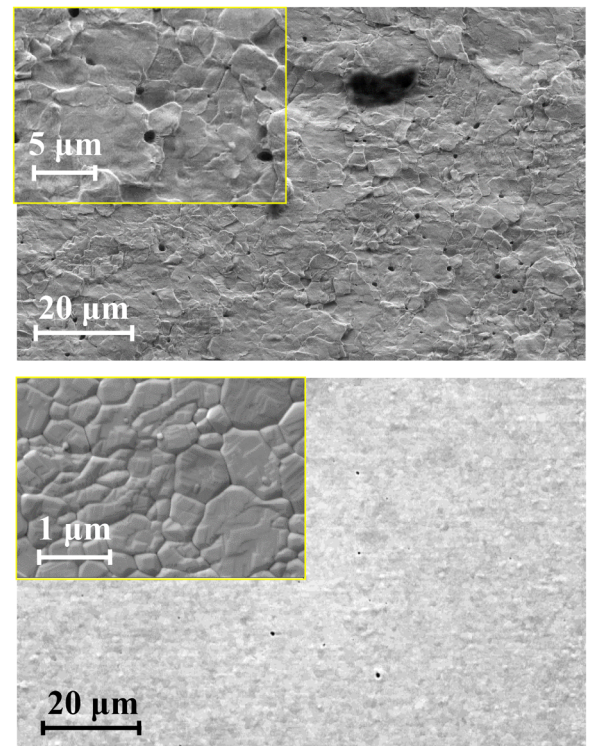


Fig. 8. SEM of bulk (top) and μ -structured (bottom) sample.

This can be affirmed as SEM of bulk sample reveal roughness increase and overlapping of thermal cycled tungsten grains but also an increase of their size (some grains reached $20\ \mu\text{m}$, with a starting size between 1 and $5\ \mu\text{m}$) while on micro-structured side, grains remains unchanged in their size (between 1 and $0.2\ \mu\text{m}$) and disposition (slightly larger in fibre centre than on edges) (Fig. 7).

4. Conclusions

The evaluation demonstrates that the micro-structured tungsten is a great improvement compared to bulk tungsten in the field of thermal fatigue caused by ELMs. Additionally, this improvement of more than a factor 10^4 , comes without any particular drawback over ITER grade bulk tungsten, which in other words, means that it would perform, at least as well, in the others important concerns (fuel retention, sputtering, power handling...). The increase measured in deuterium retention can appear significant but has to be considered in perspective with the magnitude of order of the increase found in thermal cycling tests ($+51\%$ vs $+10^6\%$). Additionally, this particular aspect can be significantly improved.

The current paper is only the preliminary evaluation of all required performances and it gives a general wide outlook of all aspects, even if each of these aspects (fuel retention, power handling, erosion/deposition ...) would need deeper and more accurate evaluation to completely characterise it. It is a concept validation, not a final product exhaustive evaluation.

Another interesting aspect of micro-structured tungsten is that its strength points are perfectly fitting for diagnostic mirrors applications, as it can withstand high number of laser pulses with high energy density (for example, Thomson scattering diagnostics [14]). This advantage could be also used for any diagnostics, which use the metallic optical mirrors, to recover its reflectivity, which was degraded due to the deposition, by means of the laser cleaning methods.

Additionally, the concept allows a wide range of optimisation parameters possibilities, as front and base material is not limited to tungsten or copper but can use others material (like smart alloys fibres to replace tungsten ones [15]), the geometrical exposed surface ratio for optimising emissivity or reflectivity, the sub-elements used, clearance height, possible sub-element pre-coating (e.g. yttrium coating for fuel retention mitigation like used in WfW [4]) etc...

Finally, the authors would like also to note that micro-structured tungsten uses current industry processes and do not require any expensive or exotic manufacturing process (but uses century old tungsten filament industrial processes), which is very important when considering the 800 m² of plasma facing material foreseen into ITER (of maybe only 140 m² of high heat flux divertor).

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