Nuclear Materials and Energy 12 (2017) 200–204

Contents lists available at ScienceDirect

Nuclear Materials and Energy

journal homepage: www.elsevier.com/locate/nme

Self-castellation of tungsten monoblock under high heat flux loading and impact of material properties

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ARTICLE INFO

Article history:
Received 12 July 2016
Revised 3 October 2016
Accepted 22 October 2016
Available online 27 December 2016

Keywords:
Fracture
Tungsten
ITER
Divertor
High heat flux
Recrystallization

ABSTRACT

In the full-tungsten divertor qualification program at ITER Organization, macro-cracks, so called self-castellation were found in a fraction of tungsten monoblocks during cyclic high heat flux loading at 20MW/m^2. The number of monoblocks with macro-cracks varied with the tungsten products used as armour material. In order to understand correlation between the macro-crack appearance and W properties, an activity to characterize W monoblock materials was launched at the IO. The outcome highlighted that the higher the recrystallization resistance, the lower the number of cracks detected during high heat flux tests. Thermo-mechanical finite element modelling demonstrated that the maximum surface temperature ranges from 1800 °C to 2200 °C and in this range recrystallization of tungsten occurred. Furthermore, it indicated that loss of strength due to recrystallization is responsible for the development of macro-cracks in the tungsten monoblock.

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1. Introduction

Among the components constituting the ITER divertor, the plasma-facing units (PFUs) of vertical targets are designed to withstand the highest surface heat fluxes, i.e. 10 MW/m^2 during steady state operation and 20 MW/m^2 during slow transients [1]. To meet these requirements, the PFUs employ a monoblock technology, made of pure tungsten armor joined to the copper alloy pipe via a pure copper interlayer [2,3,4], see Fig. 1(a). In order to validate and demonstrate the performance of available technology, the full-tungsten divertor qualification program was launched [2]. The main part of the program was to examine the performance of tungsten monoblock components in high heat flux (HHF) tests. As a result, monoblocks made out of several different tungsten products showed rather frequently macro-cracks, so-called self-castellation. The macro-cracks developed at the loaded surface, propagating through the tungsten armor toward the copper interlayer [3,5,6], but not impaired the monoblock integrity and thermal capability. An extensive modelling [7] was performed to understand the fracture modes, i.e. causes of the macro-crack initiation. In parallel, various tungsten monoblock materials were characterized to investigate the correlation between HHF test performance and tungsten properties.

In this paper, the summary of HHF test results and essential results of the tungsten material characterization program are presented. The correlation between HHF test performance and tungsten material properties is also discussed.

2. High heat flux test results of W monoblock components

Small-scale mock-ups, made of 5 or 7 monoblocks, and full scale prototype PFUs were tested under high heat fluxes in two electron beam facilities: FE200 in France [8] and IDTF in Russia [9]. In these tests, 5000 cycles at 10 MW/m^2 followed by 300 cycles at 20MW/m^2 were applied on the actively cooled monoblocks and full-scale prototype PFUs [10]. Each cycle had a duration of 20 s, including 10 s of heating followed by a 10 s dwell time. At least 280 monoblocks were successfully tested [3,5,11,12]. Interestingly, in average 30% of them showed macro-cracks. These macro-cracks were not observed after the cycling at 10 MW/m^2 and they were observed on a fraction of the monoblocks after a few tens up to a few hundreds of cycles at 20 MW/m^2 [3]. The cracks all exhibit the same features: initiated at the loaded surface (yz plane; see Fig. 1(a)), oriented along the cooling tube axis, and propagating

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http://dx.doi.org/10.1016/j.nme.2016.10.025
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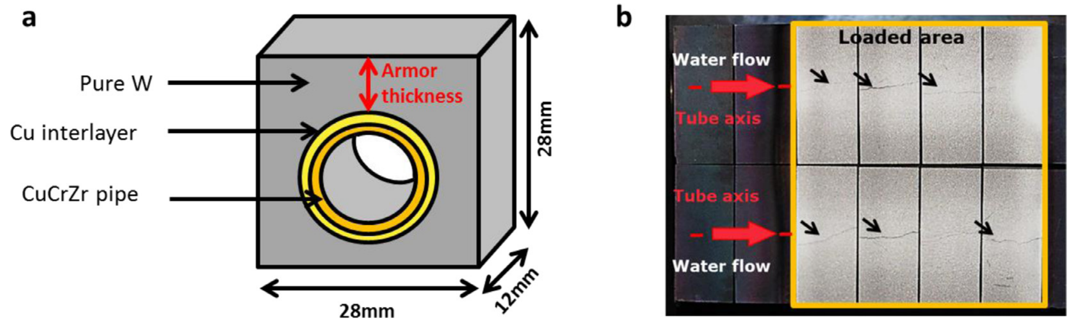

Fig. 1. (a) monoblock geometry, made of a rectangular tungsten armour, a copper interlayer (typically OD/ID = 17/15) and a CuCrZr tube (OD/ID = 15/12). (b) top view of the loaded area of 2 tested mock-ups showing 6 self-castellations marked by black arrows after 5000 cycles at 10 MW/m^2 followed by 300 cycles at 20MW/m^2[16].

Table 1
Summary of ~280 monoblocks tested in the frame of the full-tungsten divertor qualification program [3,5,11,12]. Each tested component is described with the tungsten product, the tungsten production process, the final surface finish of the loaded surface, the component manufacturing process (HIP = Hot Isostatic Pressing [15], HRP = Hot Radial Pressing [13], brazing [14]), the electron beam used, and the tungsten armor thickness (mm). The percentage of monoblocks with macro-cracks after 5000 cycles at 10 MW/m^2 and 300 cycles at 20MW/m^2 is given as well as the total number of loaded monoblocks.

Tungsten product	Production process	Surface finish	Bonding technology CuCrZr to W+Cu	Device	Cracking (300 cycles 20MW/m^2)	Number of monoblocks	Armour thickness
P5	forged Bar	grinded	HIP	FE200	**0%**	5	5.5
P5	forged Bar	grinded	HIP	IDTF	**0%**	6	5.5
P4	rolled Plate	grinded	Brazing	IDTF	**0%**	6	7.7
P1	rolled Plate	grinded	Brazing	IDTF	**0%**	3	7.7
P3	rolled Plate	grinded	Brazing	IDTF	**0%**	3	7.7
P1	rolled Plate	grinded	Brazing	IDTF	**0%**	6	7.7
P2	rolled Plate	EDM	HRP	IDTF	**0%**	4	6
P2	rolled Plate	EDM	HRP	IDTF	**0%**	4	6
P1	rolled Plate	grinded	Brazing	IDTF	**0%**	120	7.7
P3	rolled Plate	grinded	Brazing	IDTF	**0%**	10	7.7
P4	rolled Plate	grinded	Brazing	IDTF	**0%**	5	7.7
P5	forged Bar	grinded	HRP	IDTF	**0%**	6	6
P3	rolled Plate	grinded	HIP	IDTF	**8%**	24	6
P5	forged Bar	grinded	HIP	IDTF	**33%**	3	7.5
P5	forged Bar	grinded	HIP	IDTF	**66%**	6	7.5
P2	rolled Plate	EDM	HRP	IDTF	**66%**	3	6
P5	forged Bar	grinded	HIP	FE200	**75%**	4	7.5
P5	forged Bar	grinded	HIP	FE200	**100%**	10	5.5
P5	forged Bar	grinded	HIP	IDTF	**100%**	3	5.5
P5	forged Bar	grinded	HIP	IDTF	**100%**	4	5.5
P5	forged Bar	grinded	HIP	FE200	**100%**	8	7.5
P2	rolled Plate	EDM	HRP	FE200	**100%**	4	6
P2	rolled Plate	EDM	HRP	FE200	**100%**	4	6
P2	rolled Plate	EDM	HRP	FE200	**100%**	12	6
P5	forged Bar	grinded	HRP	FE200	**100%**	8	6
P5	forged Bar	grinded	HRP	FE200	**100%**	4	6
P5	forged Bar	grinded	HRP	FE200	**100%**	4	6

into the tungsten armour perpendicular to the y-direction, in the x-direction (see Fig. 1(b)).

The summary of the high heat flux test results, including the number of cracks detected after 5000 cycles at 10 MW/m^2 and 300 cycles at 20 MW/m^2 is shown in Table 1. It should be noted that the tests were aimed at examining the performance of the armor heat sink joints under cyclic heat loads but not at studying the behavior of the tungsten armor under high heat flux loading. Therefore, these high heat flux test results include many variables (Table 1), which makes the interpretation of the results more difficult:

- Variations in monoblock manufacturing: tungsten production route (rolling / forging), loaded surface grinding after EDM cutting, joining technology.
- Variations in monoblock geometry: armor thickness, copper interlayer thickness.
- Variations in the high heat flux test facility: electron-beam spot size and energy.
- Number of tested monoblocks for each manufacturer and tungsten product.

Typically, each manufacturer is associated to a specific tungsten supplier, a bonding technology, and a copper interlayer thickness. Two main tendencies were extracted:

- First, results are electron-beam dependent [16]. FE200 (spot size between 2 and 10 mm and an acceleration voltage of 200 keV [8]) has tendencies to damage more than IDTF (spot size approximately 50 mm and acceleration voltage of 60 keV [9]). Indeed, FE200 has a more focused beam; as a consequence a larger local power density is deposited in FE200 than in IDTF yielding in more damages due to local thermal shock loads. This was illustrated by HHF test results: for P2 tungsten with 6 mm tungsten armor only ~20% of the monoblocks tested in IDTF showed macro-cracks while this was the case for 100% of the monoblocks tested in FE200.
- Focusing on the comparison on IDTF results for the reasons explained above, results show that the tungsten product used in the monoblock armor plays a key role. While all materials are fulfilling the requirements of the ITER tungsten material specification [3,4], rolled plate tungsten products show cracks on

Table 2
Number of macro-cracks in different tungsten products after HHF test for 5000 cycles at 10 MW/m^2 and 300 cycles at 20MW/m^2.

Tungsten product	Production process	Cracking (Number of macro-cracks)	number of monoblock
P1	Rolled plate	**0% (0)**	129
P4	Rolled plate	**0% (0)**	11
P3	Rolled plate	**5% (2)**	37
P2	Rolled plate	**18% (2)**	11
P5	forged Bar	**43% (12)**	28

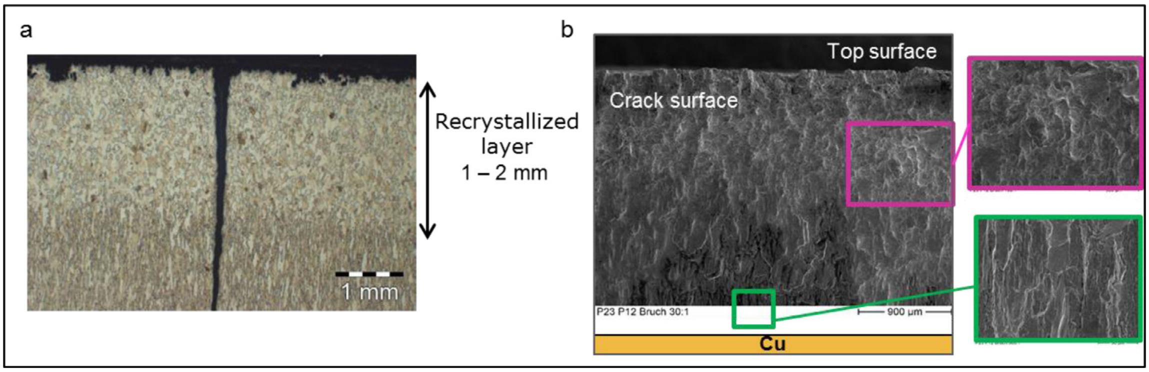

Fig. 2. Micrographs of macro-cracks appeared in cracked tungsten monoblocks after HHF tests at 5000 cycles at 10 MW/m^2 and 300 cycles at 20 MW/m^2. (a) cross-section at the vicinity of loaded surface, (b) fracture surfaces. [6].

in average 2% of the tungsten monoblocks (4 monoblocks over 188), while in average 43% of the forged tungsten product P5 do. Besides, among rolled plate tungsten products there are also differences in cracking probability, ranging between 0% and 18% of macro-crack appearance for product P1 and P2, respectively, see Table 2.

3. Fracture mode analysis

To determine the involved fracture mode, the thermo-mechanical behavior of the tungsten monoblock under cyclic high heat flux loading was studied by means of finite element (FE) modeling. Surface temperature was calculated to be in the range of 1800 °C to 2200 °C at 20 MW/m^2 depending on the distance from the cooling pipe. At such high temperature, the tungsten is likely to recrystallize, because recrystallization temperature ranges between 1000 °C and 1700 °C depending on its production route [17,18]. As a consequence, the tungsten material properties may change during cycling from the stress-relieved material properties to recrystallized, i.e. Yield strength and ultimate tensile strength decrease while reduction of area increases [19]. Due to decrease of strength after recrystallization, the plastic strain under cyclic heat loads increases for recrystallized tungsten compared with the as-received tungsten product. Consequently, recrystallized tungsten would exhibit a shorter life time from the view point of ductility exhaust. The details of the analysis are reported elsewhere in [7] and in [20]. Accordingly, recrystallization resistance was found to be one of the key properties.

These results have been confirmed by experimental evidences. The loaded monoblocks, which showed macro-cracks, were examined by metallography. As described in [6], microscopic analyses showed that, in the vicinity under the loaded surface (1 to 2 mm), tungsten grains started to grow due to recrystallization, see Fig. 2(a). In addition, in the same range the top part of the cracked surface showed deformed grains and intergranular fracture while in a distance > 2 mm from the loaded surface the crack surface showed brittle trans-granular fracture; see Fig. 2(b).

The deformed plastic grains observed in the recrystallized zone of the tungsten armor confirmed the results of the thermo-mechanical modeling [7] namely, the tungsten material would accumulate plastic deformation in the middle of the loaded surface of the monoblock once recrystallized. The crack would then develop when the ductility of the material exhausts.

4. Results of tungsten material characterization program

Although the macro-cracks did not impair the performances, it is still preferable to avoid them not to cause significant impact on plasma operation such as melting due to the formation of leading edges. This is why a characterization of monoblock tungsten products [3,16] was launched to study their mechanical properties and correlation to their HHF test performance.

In the material specification [4,16], the requirements are set for the as-received material properties. However, the mechanical properties and the resistance to recrystallization are not specified. The requirements are summarized in the following:

- Minimum tungsten content: 99.94%
- Maximal impurity content of carbon, oxygen, nitrogen, nickel and silicon: 0.01wt%
- Minimum density, as defined in ASTM B311: 19.0 g/cm^3
- Minimum hardness HV30, as defined in ASTM E92: 410 HV
- Grain size number as defined in ASTM E112 should be 3 or finer in the yz plane, with elongated grains in the x-direction, see Fig. 1

Five tungsten products used in the manufacturing of the monoblock components in the frame of the full-tungsten divertor qualification program [2,3], i.e. P1 to P5, were characterized. As already mentioned in Table 2, P1 to P4 are rolled tungsten plates and P5 is a forged tungsten bar. These products were characterized in their as-received state; i.e. stress relieved, and after 1 h annealing at 1300 °C, 1500 °C and 1800 °C. Tungsten products annealed at 1800 °C were considered to be fully recrystallized. Tests include (see Fig. 1 for plane orientation and directions):

- Recrystallization sensitivity test: hardness measurements HV30 at room temperature on stress-relieved and annealed tungsten after 1 h annealing at 1300 °C, 1500 °C and 1800 °C, in the xy and yz planes;

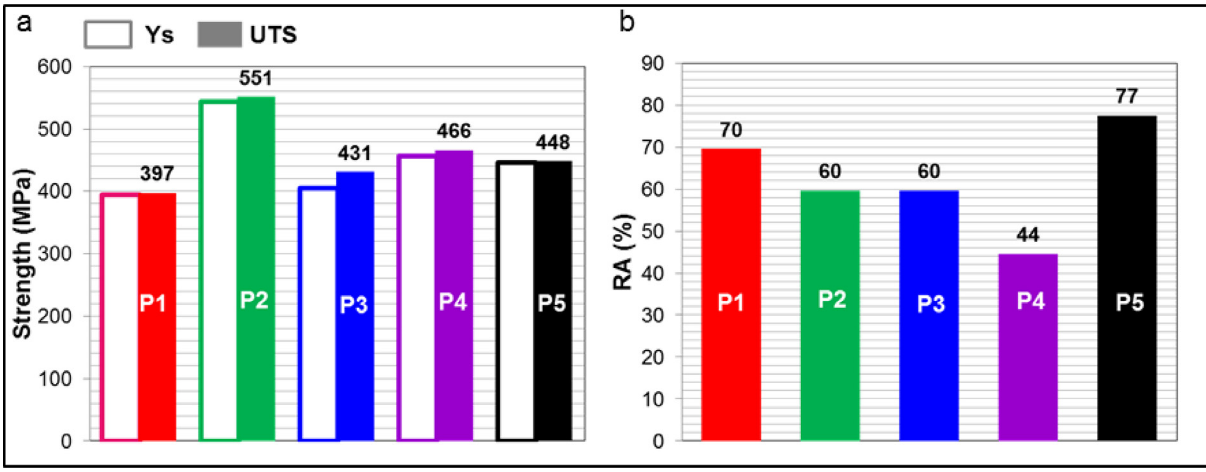

Fig. 3. (a) Ys and UTS and (b) reduction of area (in %) obtained from tensile test on as-received tungsten in y-direction at 800 °C.

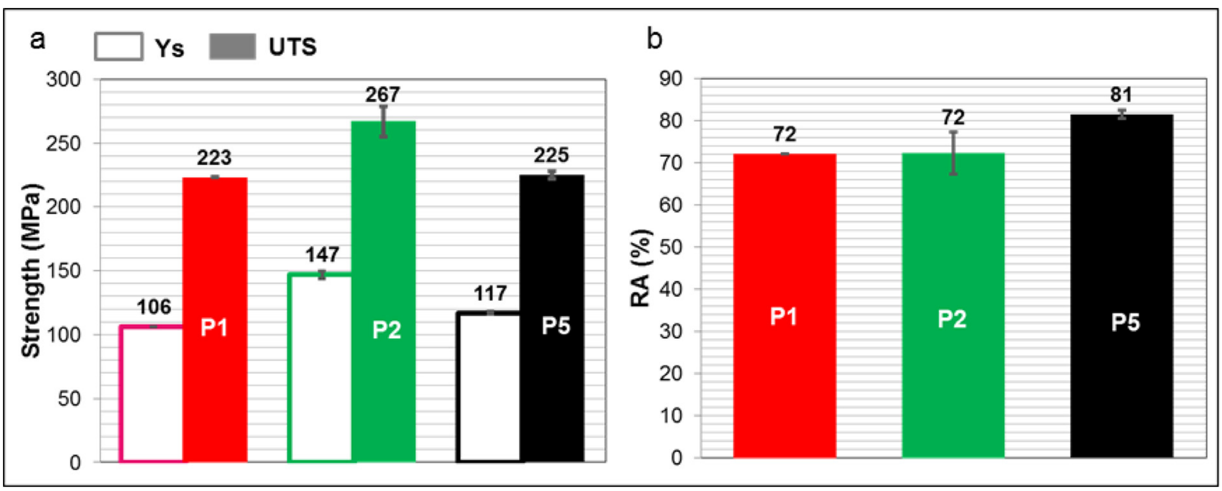

Fig. 4. (a) Ys and UTS and (b) reduction of area (in %) obtained from tensile test on recrystallized tungsten in y-direction at 800 °C.

- Tensile test: at 800 °C on all 5 stress-relieved products, tensile test on 3 out of the 5 products (P1, P2 and P5) after recrystallization (1 h annealing at 1800 °C) along the x and y directions. The tensile test geometry of the dog bone specimen is described in [16];
- Microstructure analysis through metallography, EBSD, and SEM on stress relieved and annealed samples at 1300 °C, 1500 °C and 1800 °C, in the xy and yz planes.

The tensile test results on as-received tungsten in the y-direction at 800 °C are shown on Fig. 3. Two to three samples per product were tested. P2 had the highest yield strength (Ys) and Ultimate Tensile Strengths (UTS), while P1 had the smallest Ys and UTS (see Fig. 3(a)). Fig. 3(b) shows the reduction of area, from which ductility is defined. The results show that P5 was the most ductile material.

The tensile test results on recrystallized tungsten in the y-direction at 800 °C are shown on Fig. 4. After 1 h annealing at 1800 °C, the three products P1, P2 and P5 showed softening and exhibited a decrease of Ys of about 75%. Reduction of area increased by 3%, 20% and 5% for P1, P2 and P5 respectively, after annealing at 1800 °C. The material properties of recrystallized tungsten showed less scatter between the different products. Accordingly, the effect of the production route seems to have been less noticeable as a result of recrystallization.

Fig. 5 shows the results of the hardness tests in the yz plane at room temperature on as-received samples, and after 1 h annealing at 1300, 1500, 1800 °C. Each product for each annealing temperature has been tested 3 to 6 times. With increasing annealing temperature, the hardness decreased from, in average, 439 HV30 for as received tungsten to 352 HV30 after annealing at 1800 °C.

As seen on Fig. 5, the sharp decrease of hardness with increasing annealing temperature varied for each tungsten product, highlighting that recrystallization occurs at a temperature specific to each tungsten product. The comparison of each product showed that the behaviour of the tungsten products varies even though they all fulfil the ITER material specification, and that some tungsten products were more resistant to recrystallization compared to others. P1 recrystallizes at the highest temperature ranging between 1500 °C and 1800 °C, P2 and P3 at a temperature ranging between 1300 °C and 1500 °C, P4 and P5 at a temperature below or equal to 1300 °C. Rolled tungsten showed a better recrystallization resistance than forged tungsten. A complete description of the experimental conditions and of the results is detailed elsewhere [21].

5. Correlation between HHF test performance and properties of tungsten monoblocks

Tensile properties of the five as-received tungsten products show no obvious correlation with their HHF test performance, nei-

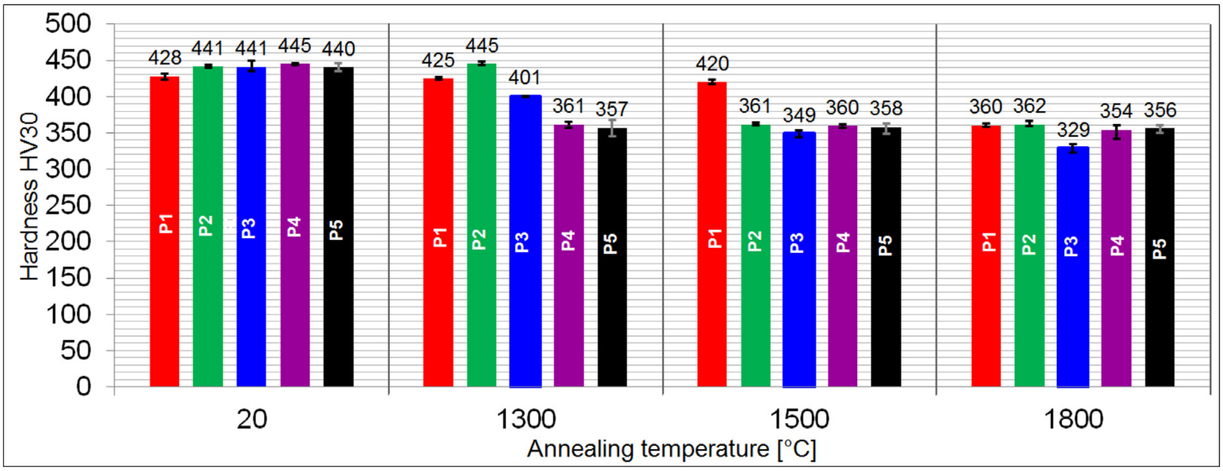

Fig. 5. Hardness HV30 measured on as-received tungsten and tungsten annealed at 1300 °C, 1500 °C and 1800 °C for 1 h.

ther in terms of strength nor ductility. Similarly, tensile properties of recrystallized materials show no obvious correlation with high heat flux test results. Among the five tungsten products tested, P1 showed the highest recrystallization temperature between 1500 °C and 1800 °C and also the best HHF test performance of the PFUs. In contrast, P5 showed low recrystallization temperature below 1300 °C and also the worst HHF test performance. The comparison of the recrystallization resistance test with the HHF test results indicated that the higher the recrystallization temperature the less frequent macro-cracks appeared.

Based on the data presented above, P4 would be the only Tungsten product not following the above mentioned correlation. However, HHF test results at the Japanese electron beam test facility JEBIS have shown that monoblocks from P4 tungsten showed macro-cracks in 3 out of 5 monoblocks after HHF test for 300 cycles at > 20 MW/m^2, peak power density at around 23 MW/m^2 due to the JEBIS electron beam configuration [22]. Thus, the results concerning P4 may be underestimated due to limited number of tested monoblocks (lack of statistics) at 20 MW/m^2.

In the frame of the full-tungsten divertor qualification program, additional HHF test could allow:

- Increasing confidence in the current results by increasing statistics
- Cross testing of material products using different bonding technologies in the tungsten armor to pipe
- Investigating the impact of tungsten armor thickness and surface-finish.

6. Conclusion

In the full-tungsten qualification program, the tested mock-ups and full scale prototypes (280 monoblocks in total) successfully demonstrated their performances. However, some of the tested monoblocks, in average 30%, showed macro-cracks so-called “self-castellation” in the tungsten armor. The number of macro-cracks varies with the tungsten product used for the manufacturing of the monoblock, although all the products used fulfilled the ITER material specification.

During cycling at 20MW/m^2, the top surface temperature ranges between 1800 °C and 2200 °C. At such temperature tungsten is likely to recrystallize. Thermo-mechanical FEM indicated that macro-cracks resulted from exhaust of ductility of recrystallized tungsten due to cyclic heat loads at 20MW/m^2. This result was corroborated by metallographic examinations showing plastic deformation on the crack surfaces at the vicinity of the loaded surface.

The tungsten characterization program highlighted that despite the differences in mechanical properties between all the products the main property that seems to have an effect on the HHF test performance is their resistance to recrystallization. The higher the recrystallization resistance, the higher the HHF test performance of the PFUs, i.e. lower the number of macro-cracks after 300 cycles at 20MW/m^2.

7. Disclaimer

The view and opinion expressed herein do not necessarily reflect those of the ITER Organization.

References

[1] R.A. Pitts, et al., J. Nucl. Mater. 438 (2013) S48–S56.
[2] T. Hirai, et al., Phys. Scr. T159 (2014) 014006.
[3] T. Hirai, et al., J. Nucl. Mater. 463 (2015) 1248–1251.
[4] T. Hirai, et al., Fusion Eng. Des (2016) in review.
[5] P. Gavila, et al., Fusion Eng. Des. 98–99 (2015) 1305–1309.
[6] G. Pintsuk, Fusion Eng. Des. 98–99 (2015) 1384–1388.
[7] S. Panayotis, Fusion Eng. Des. (2016) in review.
[8] I. Bobin-Vastra, et al., Fusion Eng. Des. 75–79 (2005) 357–363.
[9] V. Kuznetsov, et al., Fusion Eng. Des. 89 (2014) 955–959.
[10] T. Hirai, et al., Fusion Eng. Des. 88 (2013) 1798–1801.
[11] K. Ezato, et al., Fusion Eng. Des. 98–99 (2015) 1281–1284.
[12] P. Gavila, et al., Fusion Eng. Des. 86 (2011) 1652–1655.
[13] E. Visca, et al., Fusion Eng. Des. 84 (2009) 309–313.
[14] K. Ezato, et al., Fusion Eng. Des. (2016) In press, doi:10.1016/j.fusengdes.2015.12.049.
[15] T. Huber, et al., Plansee Seminar-17 International Conference on High Performance P/M Materials, 2009 25–30 May 2009.
[16] T. Hirai, et al., ICFRM-17 Proceeding, Nuclear Materials & Energy, 2016.
[17] I. Smid, et al., J. Nucl. Mater. 258–263 (1998) 308–312.
[18] J.W. Davis, et al., J. Nucl. Mater. 258-263 (1998) 160–172.
[19] G. Sanazzaro et al., Development of design Criteria for ITER In-vessel Components, Fusion Eng. Des. 88 (9-10):2138–2141
[20] M. Li, J.-H. You, Interpretation of the deep cracking phenomenon of tungsten monoblock targets observed in high-heat-flux fatigue tests at 20 MW/m2, Fusion Eng. Des. 101 (December 2015) 1–8.
[21] M. Wirtz, et al., Material properties and their influence on the behavior of tungsten as plasma facing material, 26th IAEA Fusion Energy Conference, 17-22 Oct 2016.
[22] ITER Private Communication, ITER_D_NVXJ4K.