Irradiation-induced hardening in fusion relevant tungsten grades with different initial microstructures

ChihCheng Chang[1,2*], Chao Yin[3], Dmitry Terentyev[1], Aleksandr Zinovev[1], Wouter Van Renterghem[1], Patricia Verleysen[4], Thomas Pardoen[2], Monika Vilemova[5], Jiri Matějíček[5]

[1]Belgian Nuclear Research Centre, SCK•CEN, Mol, Belgium; [2]Institute of Mechanics, Materials and Civil Engineering, UCLouvain, Louvain-la-Neuve, Belgium

[3]School of Nuclear Science and Technology, University of Science and Technology of China, Hefei, People's Republic of China;

[4]Department of Electrical Energy, Metals, Mechanical constructions & Systems, Ghent University, Ghent, Belgium [5]Institute of Plasma Physics, Prague, Czech Republic

E-mail: chih-cheng.chang@sckcen.be

Introduction

Tungsten (W) is considered the most promising plasma-facing material due to its high melting point, high thermal conductivity, low erosion rate, and other attractive properties. The commercial ITER specification tungsten is well developed and available on the market. The main concern of baseline tungsten is high ductile to brittle transition temperature (DBTT). To reduce DBTT, many efforts were devoted in recent years and several methods were proposed and implemented. However, all developments so far did not account for the degradation induced by neutron irradiation. Therefore, the investigation of neutron irradiation effects is important to validate the newly developed material grades. Given that the irradiation and post-irradiation examinations are cost and time-consuming actions, the applications of small specimen test technique (SSTT) are indispensable for research in the fusion field. In this work, the micro-hardness measurements are applied to extract the irradiation-induced degradation in four tungsten grades after the neutron irradiation in BR2. The study is made to understand the link between initial microstructure and the hardness change after the neutron irradiation.

Materials/Microstructure/Irradiation

To reduce DBTT, there are three mainstream ways

1. By cold/thermal mechanical processing
2. Prepare solid solution, for example by adding rhenium (Re)
3. Grain refinement and grain boundary strengthening by using strengthening particles (carbides, ODS)

In this work, we investigate four tungsten grades

Material	Composition	Manufacturing process	Provider
IGP 36×36 mm bar	Pure W (>99.97 wt%)	Two-side hammering	Plansee, Austria
ATW, 13 mm plate	Pure W (>99.94 wt%)	Rolling	AT&M, China
W-0.5ZrC 8 mm plate	99.5 wt% W + 0.5 wt% ZrC	Rolling + Thermal Mechanical Treatment	The Institute of Solid State Physics, China
FG 5 mm disk	Pure W (>99.97%)	Spark Plasma Sintering (SPS)	Institute of Plasma Physics, Czech Republic

Table 1. The information of studied tungsten grades

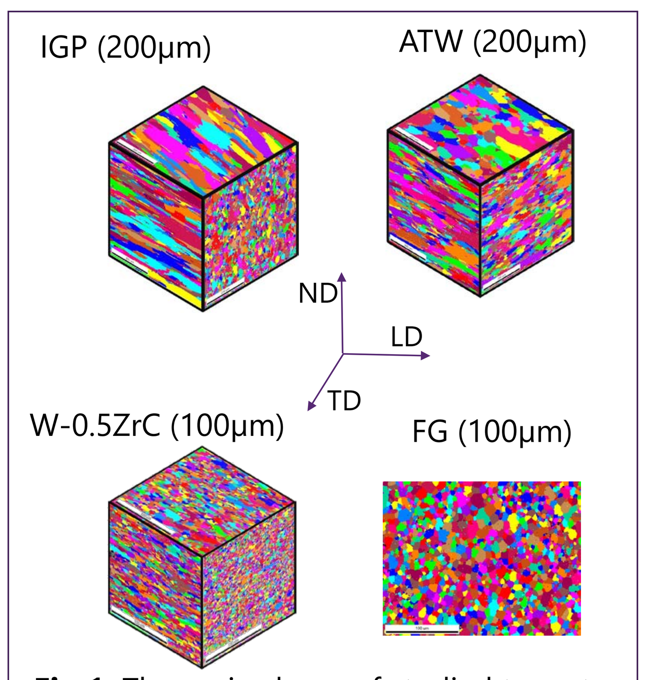

Fig 1. The grain shape of studied tungsten

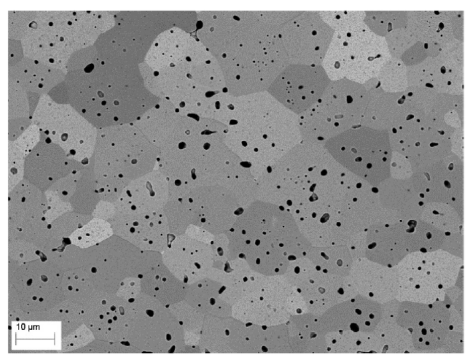
Fig. 2. SEM image of polished FG [1]

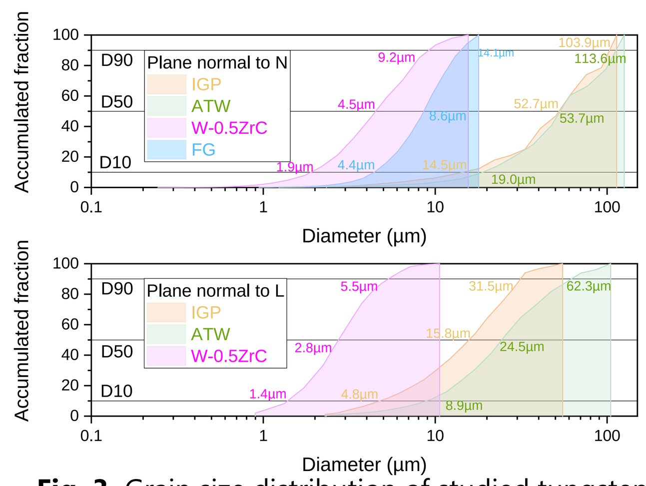
Fig. 3. Grain size distribution of studied tungsten

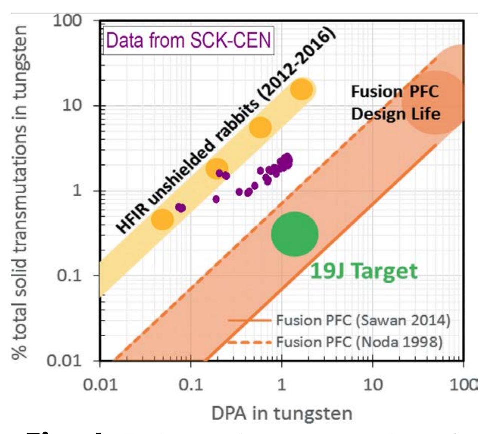

Fig. 4. Estimated transmutations for tungsten irradiated in different reactors[3]

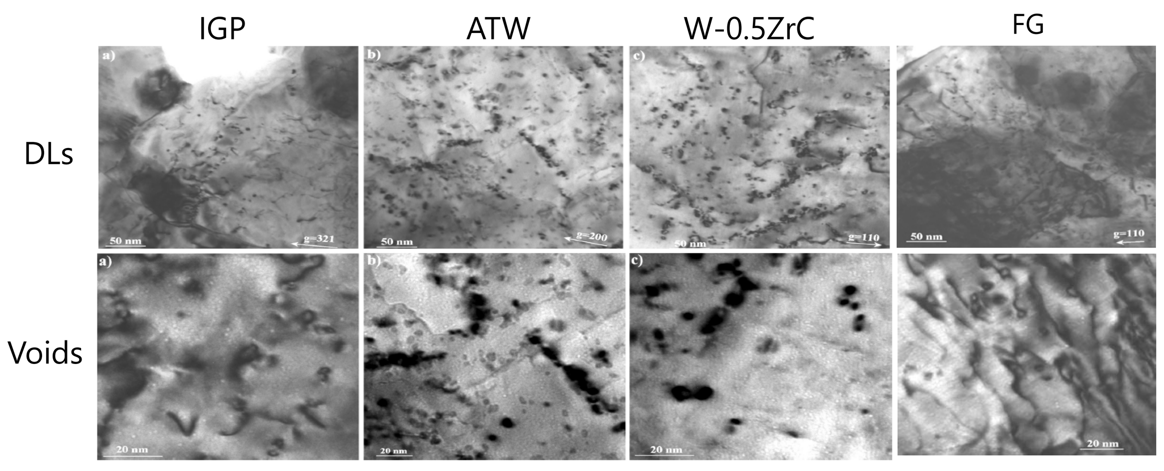

Fig. 5 The TEM images of dislocation loops (DLs) and voids induced by ion irradiation [4]

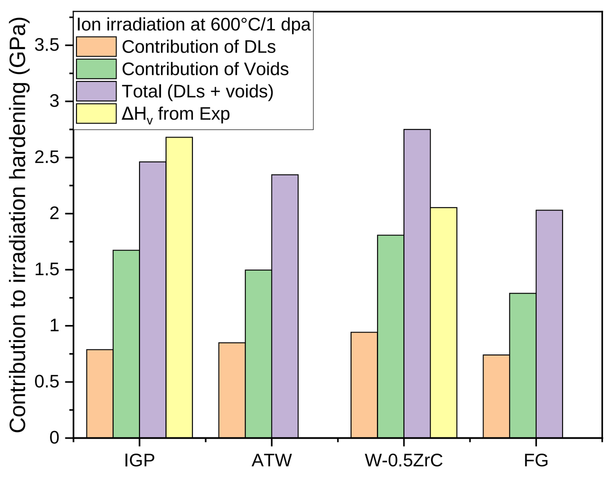

Fig. 6. The contributions of defects on irradiation hardening

Figure 1 From EBSD images, we can see that IGP, ATW, W-0.5ZrC have elongated grains due to mechanical processing while FG has equaxial grains due to SPS process without metalworking.

Figure 2 shows that FG has certain porosity (~4%) which was discover only after the material was taken for neutron irradiation[1].

Figure 3 shows the accumulated size distribution of studied tungsten grade. And elongated grains can be assumed as an ellipsoid, others – as spheres, so we can calculate the grain surface area and volume using D50 values and methodology presented in Yin et al[2].

Figure 4 shows the irradiation conditions for tungsten irradiated in different reactors earlier and in this study. The transmutation-per-dpa achieved in the present experiment is shown by purple dots on the figure.

Figure 5 shows the TEM micrographs obtained for each material. As expected, the damage consists of dislocation loops and voids, more info can be found in Van Renterghem, W. et al[4].

Figure 6 shows the individual contributions of loops and voids, calculated by the equation $\Delta H_v = 3.20 M\alpha\mu\beta(ND)^{0.5}$. The hardening coefficients α were taken from Hu et al[5]. The irradiation hardening estimated due to the ion irradiation defects is comparable to the experimentally measured value after the neutron irradiation, especially for the IGP materials.

Results

$S_{v,g}$ = StV ratio of grains which misorientation angle > 15°

$S_{v,sg}$ = StV ratio of grain which misoreintation angle 2° - 15°

$S_{v,particle}$ = StV ratio of ZrC precipitates

$S_{v,pore}$ = StV ratio of pores In FG

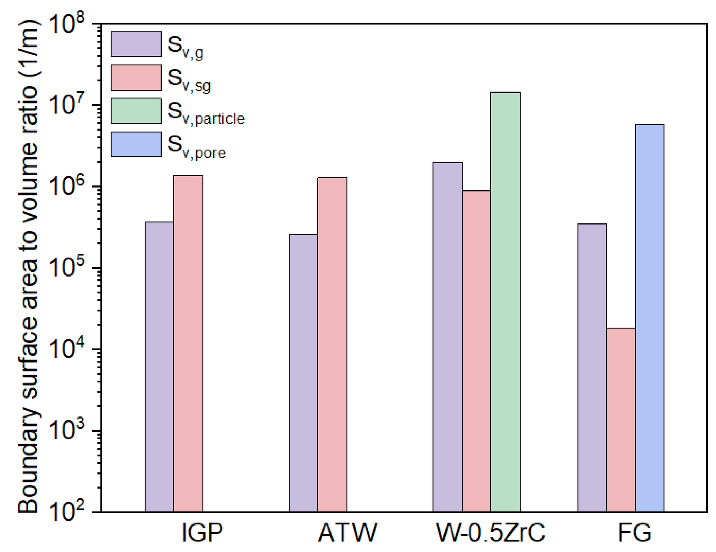

Fig 7. The boundary surface area to volume (StV) ratio of the studied tungsten grades.

- IGP and ATW, they have the roughly the same $S_{v,g}$ and $S_{v,sg}$. So it is hard to predict which one will be stronger.
- W-0.5ZrC, besides high $S_{v,g}$, $S_{v,sg}$ due to the manufacturing processing, it also has a high $S_{v,precipitate}$.
- FG has the lowest $S_{v,sg}$, it is because of the manufacturing process of FG. And FG has certain porosity. So we also calculate the $S_{v,pore}$.

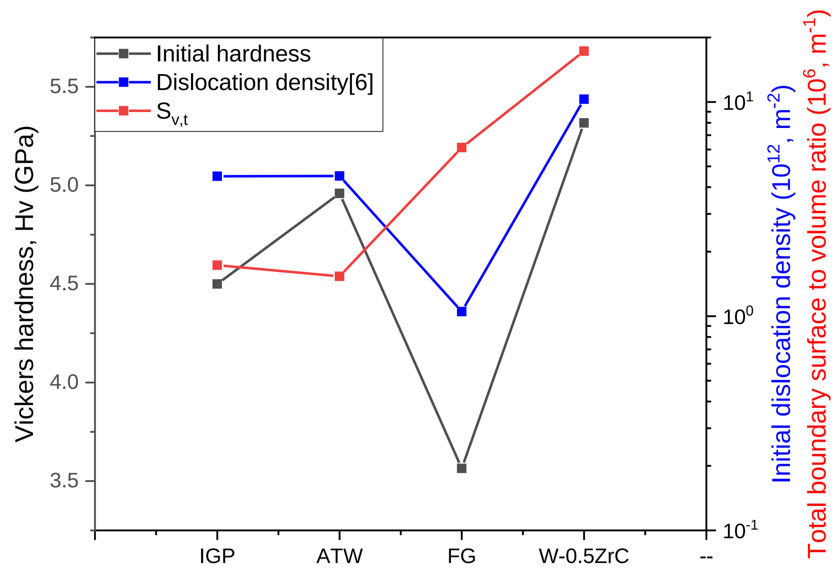

Fig 8. Hardness/dislocation density/$S_{v,t}$

- W-0.5ZrC has the highest hardness due to highest $S_{v,t}$ and highest dislocation density and small sub-grain size due to manufacturing process.
- FG has the lowest hardness due to lowest dislocation density, absence of sub-grains. However, $S_{v,t}$ is rather high due to the presence of pores.
- IGP and ATW exhibit comparable microstructure, hardness and $S_{v,t}$.

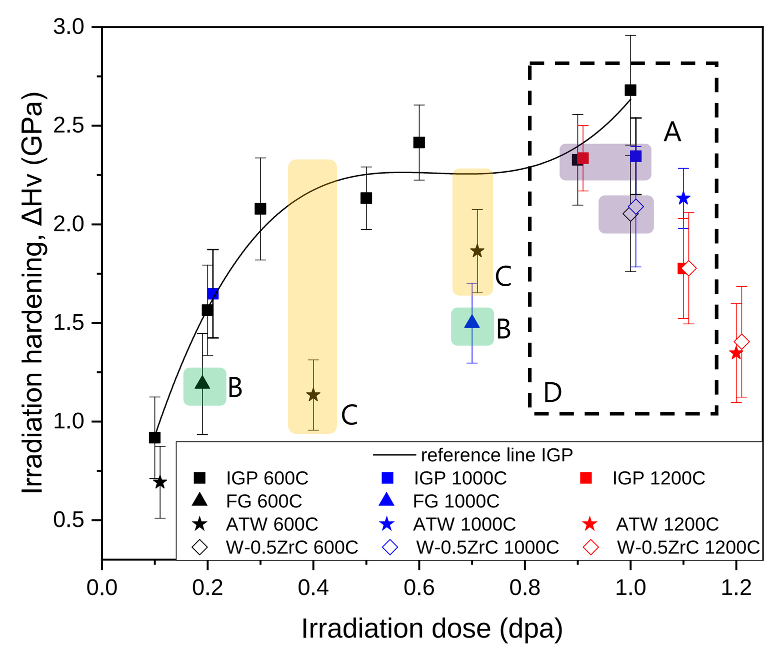

Fig 9. The irradiation hardening vs. irradiation dose.

- Region A : at ~(0.9-1) dpa, ΔH at T_{irr}=600°C is comparable to that at 1000°C given the error bar. At T_{irr}=1200°C, ΔH decreases below 2 GPa. Hence, the annealing of the irradiation damage is promoted at T_{irr} > 1000°C.
- Region B : FG tungsten has the lowest ΔH among the other tungsten grades. It might be due to high $S_{v,pore}$ demonstrating that pores can be considered as effective defect sinks.
- Region C : Although ATW and IGP have very similar microstructure and chemical composition, ΔH_{ATW} is much lower at T_{irr}=600°C/0.4 dpa and 0.7dpa. One possible reason for this difference is spatial distribution of impurities such as O, N, C (due to different heat treatment during the processing and stress relief).

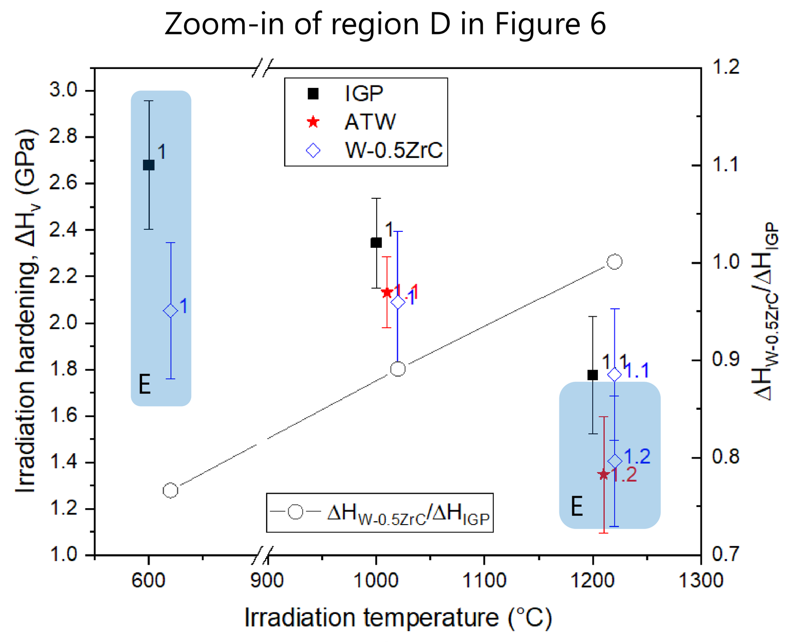

Fig 10. Comparison of irradiation hardening vs. temperature for the samples irradiated up to 1-1.2 dpa.

- Region D : At T_{irr}=600°C, ΔH in W-0.5ZrC is 20% lower compared to IGP, can be explained by a higher sink density in W-0.5ZrC. But at 1200°C, the hardening is comparable (within error bar). Hence, the effect of sinks is less important at high irradiation temperature as recovery of the damage occurs thanks to thermally activated processes (void dissolution, detrapping of the loops from dislocation lines, etc.).

Conclusion

1. ATW and IGP grades exhibit very similar microstructure and chemical purity (ITER specification), but the irradiation hardening is considerably higher in the IGP at T_{irr} =600°C at 0.4 dpa and 0.7 dpa. The reasons for this difference are not clear at the moment. Understanding of the spatial location of the main interstitial impurities (C, N, O) and their absolute concentration as well as comparative TEM study on the irradiation microstructure can help to reveal the reasons for the deviation in response of the hardness.
2. Comparison of the irradiation hardness measured at 600, 1000 and 1200°C shows that at T_{irr}=600°C the material with high sink density exhibits lower hardening increment. However, at T_{irr}=1200°C the hardening increase is comparable. This shows that the effect of microstructural sinks does not dominate at high irradiation temperature (~T_m/3), while high sink density evidently helps the suppression of the irradiation hardness at 600°C (~T_m/6).
3. FG tungsten exhibits low irradiation hardening compared to other studied grades. We ascribe this to the presence of a high density of voids (present due to the manufacturing) which also act as sinks for the irradiation defects.

[1]Matějíček, J., et al. (2020).DOI: 10.1016/j.jnucmat.2020.152518. [2]Yin, C., et al. (2021).DOI: 10.1088/1741-4326/abf417. [3]Garrison, L. M., et al. (2019).DOI: 10.1080/15361055.2019.1602390. [4]Van Renterghem, W., et al. (2021).DOI: 10.1016/j.jnucmat.2020.152710. [5]Hu, X., et al. (2016).DOI: 10.1016/j.jnucmat.2016.08.024. [6]Dubinko, A., et al. (2021).DOI: 10.1016/j.ijrmhm.2020.105409.

Exploring a better tomorrow

www.sckcen.be