Radiation Response of Low-Activation Cemented Tungsten Carbide (cWC)

Radiation-dense materials studies with the Dalton Cumbrian Facility (DCF) with the NNUF

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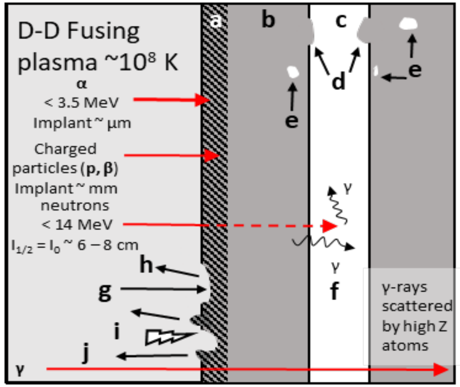
The cWC-RSB concept for compact radiation shielding

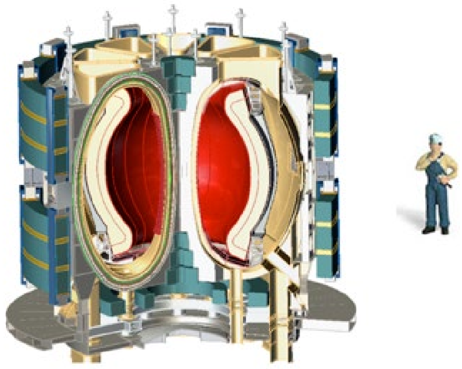
- Cemented Tungsten Carbides (cWC) are a well-established refractory material with a 100-year history but has only recently been considered as a radiation shielding candidate.[1]
- Reactive sintered borides (RSBs) are a novel material synthesised from boron additions to cWCs but were synthesized first in 2016[1]
- Combined cWC-RSB shields show considerable promise *in silico* but practical data on irradiation is in the earliest stages of acquisition
- Information on thin-film and bulk properties of irradiated cWC and RSBs in the earliest stages of acquisition.



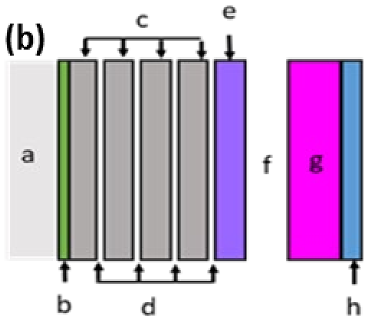
(a) PFC surface, (b) Outer cWC shield, (c) Coolant/moderator channel, (d) Expansion from internal void formation, (e) Voids and H/He bubbles, (f) γ-ray generation from absorption (g) plasma erosion, (h) ions, neutrals and impurities from sputtering, (i) plasma discharge (~GJ m^{-2}) (j) molten metal re-deposition and particle formation from (i).

1. Humphry-Baker, S. A. & Smith, G. D. W. *Philos. Trans.Royal Soc A:* **377**, 20170443 (2019).

The cWC-RSB materials



Design concept of a compact spherical tokamak reactor (After G.A. Cottrell 2006)



Schematic diagram of the central column from Figure 1(a) (black outline). (a) plasma, (b) PFC component, (c) cWC-based neutron shields, (d) coolant/moderator channels, (e) RSB inner shield[4-6], (f) vacuum gap, (g) HTS and (h) steel support.

- Magnetic confinement in a spherical tokamak has shown considerable recent progress.[2]
- Commercially available high-temperature superconductors (HTS) make high-field HTS magnets suitable for compact spherical tokamaks (cSTs)
- The smaller size of cSTs make for faster building and testing times than conventional ‘doughnut’ toroidal tokamaks.

Problems

- High heat and radiation fluences on the divertors and central column due to small size[3]
- Tight geometries make shielding around the central column a significant challenge.
- HTS magnets are significantly radiation sensitive

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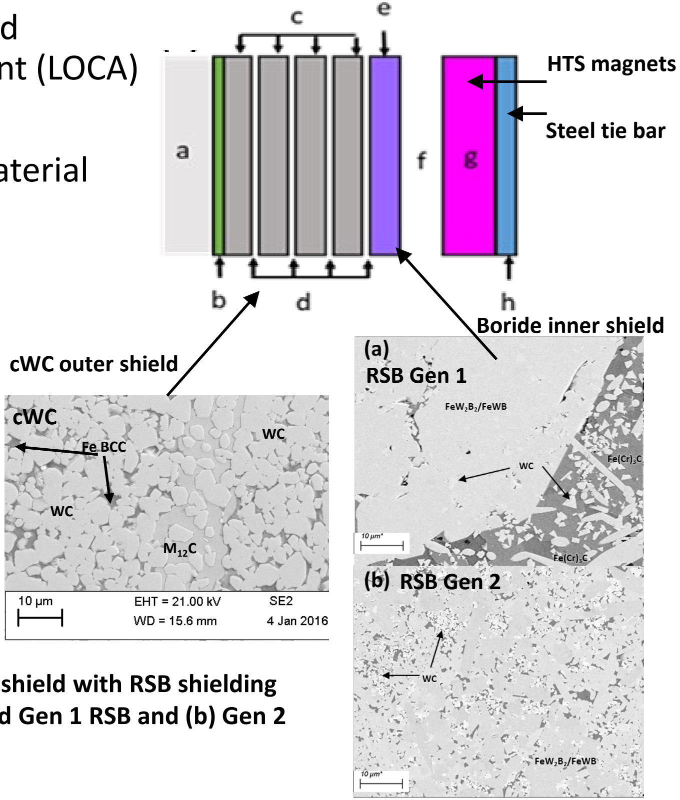
2. A.E Costley, J. Hugill and P.F Buxton *Nucl. Fusion* 55 (2015)
3. Windsor, C. G. *et al. Nucl. Fusion* **58**, (2018)
4. Windsor, C. G. *et al.* *Nucl. Fusion* **57**, 036001 (2017).
5. Windsor, C. G. & Morgan, J. G. *Nucl. Fusion* **57**, (2017).

cWC and RSB materials

What do cWCs and RSBs have to offer that's different to current materials

- W and W-based alloys are the main choices for fusion armour for ITER[6,7]
- W has issues with its high ductile-to-brittle transition temperature (DBTT) and difficulties in fabrication and accident tolerance in the case of a loss-of coolant (LOCA) accident).[8,9]
- cWCs have a 100-year history as a powder metallurgical (PM) engineering material
- cWCs are inherently less volatile in a LOCA
- RSBs are a novel tungsten iron boride PM composite material first processed in 2016 as a candidate slow neutron absorber
- Best RSB composition ~ 65 vol% iron tungsten boride; 15% WC with Fe BCC/$(Fe,Cr)_3C$ to balance ~ W : B : Fe = 1 : 1 : 1 at%
- Combined cWC-RSB configurations better than W metal for some cases[4,5]
- An RSB shield has an absorption coefficient 2 orders of magnitude higher than a cWC shield, hence its placement at the inner edge of the shielding stack.



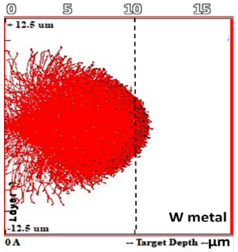
Hypothetical configuration of a combined cWC-RSB shield with RSB shielding for slower, thermal neutrons. (a) shows unoptimized Gen 1 RSB and (b) Gen 2 RSB optimized processing

6. D. Hancock et al , *J. Nucl. Mat.* **512** (2018)
7. C. Linsmeier et *al. Nucl. Fus.* **57**, (2017)
8. S.A. Humphry-Baker and W. Lee. *Scripta. Mat*. **116** (2016)
9. S.A. Humphry-Baker et al. *IJRMHM.* **93** (2020)
10. J.M.Marshall and G.Singh. *Mat.Today.Comms.* **36** (2023)

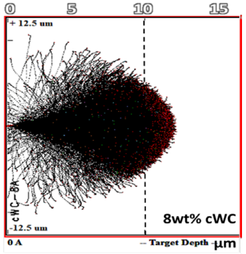
First test radiation response of cWCs and RSBs

Why proton and gamma irradiation?

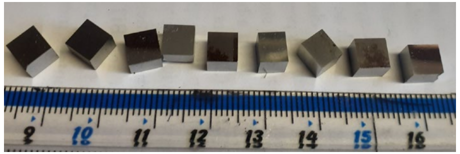
- 1.5 MeV proton and ^{60}Co γ-irradiation at the Dalton Cumbrian Facility (DCF)
- Proton irradiation at steady-state (410K) and high-temperature (823K)
- ^{60}Co γ-irradiation at ambient (293K) and cryogenic (77K)
- Proton irradiation at 4 hours for a maximum dpa ~ 2 at the Bragg peak as calculated by SRIM.
- γ-irradiation total maximum irradiation dosage: 10 MGy at 293K and 90 kGy at 77K
- Energies are below activation threshold for cWC and RSB materials



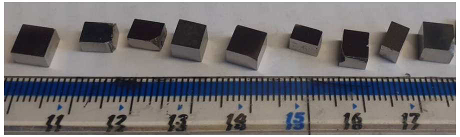
SRIM model of 1.5 MeV protons on W metal.



SRIM model of 1.5 MeV protons on cWC with an 8wt% binder content.



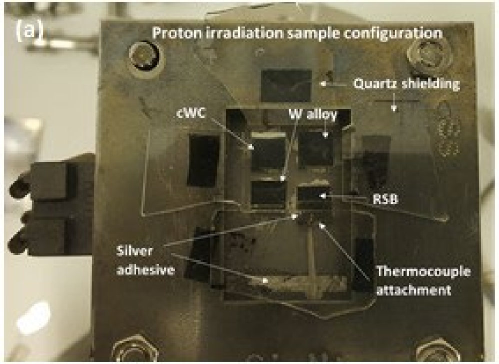
Cut and polished cWC samples

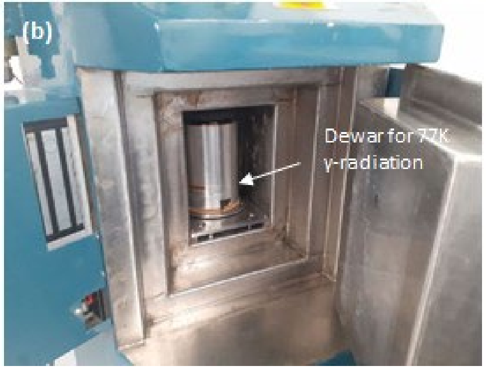


Cut and polished RSB Gen 1 samples

DCF experimental configurations

Proton irradiation commenced at ‘ambient’ (410K) and 550°C (823K)
Gamma irradiation at ambient (293K) and LN_2 (77K)





Proton Irradiation beamline setup:

- cWC and RSB samples mounted with W alloy control samples. Final samples were 6 mm x 6 mm x 2 mm cuboids
- Average beam current 20 μm
- Total proton dosage ~ 0.3 C, equivalent to ~ 5 x 10^{22} protons[11]

γ-irradiation setup:

- Radiation fluence 5 kGy per hour for ambient samples 3.3 kGy per hour for 77K samples in LN_2 dewar.
- 10 MGy samples irradiated over 3 months.
- 90 kGy 77K samples required pausing every 5kGy to remove ozone from dewar

[11] J.M.Marshall and G.Singh. *Fus. Eng. Des.* **193** (2023)

Post-Irradiation Evaluation: Microhardness

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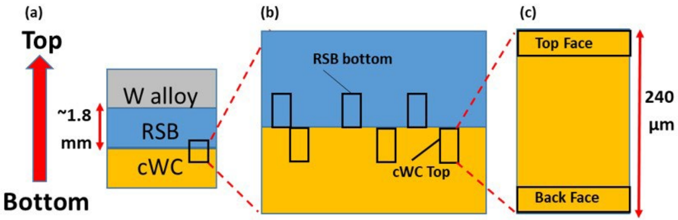
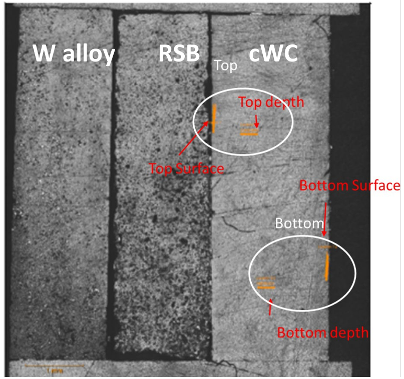
Sample configuration and microhardness

All samples are placed in mounts with cWC designated as the bottom and W-alloy as the top.
This enables identification of incident and back-faces of irradiated samples.

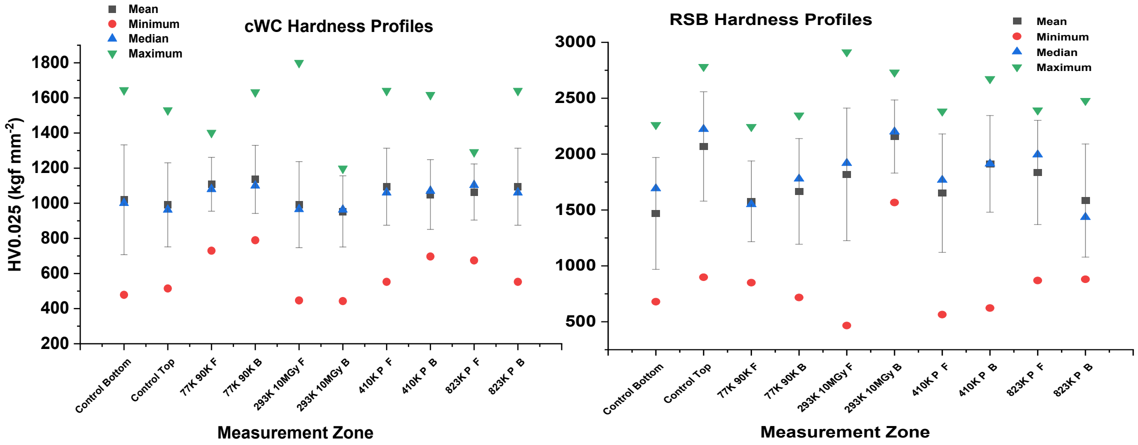




Microhardness measurements were made (a) < 20 microns of surface and (b) near the middle of samples .

(a) **Mean HV = 1000 kgf mm^2**

(b) **Mean HV = 1750 kgf mm^2**

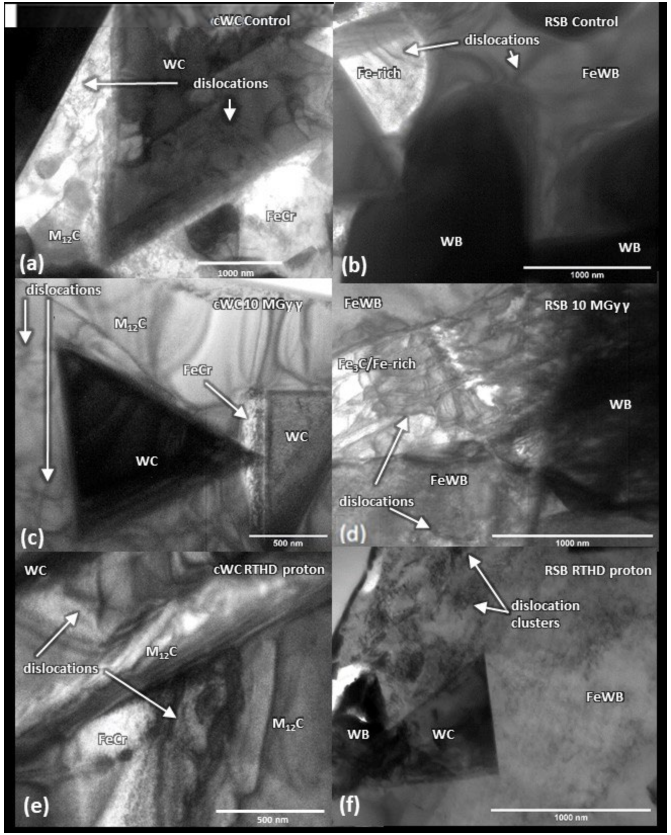


(a) and **(b)** Compilations of surface and depth from incident (F) and back (B) face from **(a)** cWC and **(b)** RSB Gen 1 sample.

Radiation-induced changes not overly significant for surface + depth compilations compared to differences between control bottom and top, particularly for RSBs.

The general trend is that irradiated samples tend to have a higher hardness but a lower SD than control samples.

Evaluation of irradiated materials – TEM analysis



TEM samples were taken at 10 µm from the incident face of ambient temperature irradiated samples for both proton and γ-irradiated materials.

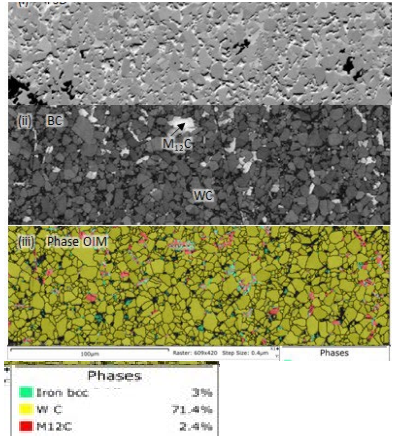
- 10 MGy irradiated cWC sample shows fewer dislocations relative to control sample but with larger, coalesced dislocations.
- Significant increase in dislocations observed in RSB sample post ambient 10 MGy irradiation.
- Larger, more frequent dislocations observed in ambient (RTHD) proton irradiated cWC sample including the dislocation-resistant $M_{12}C$ phase
- Numerous, fragmented dislocations in RSB materials post proton irradiation

Current work is on quantifying dislocation concentrations

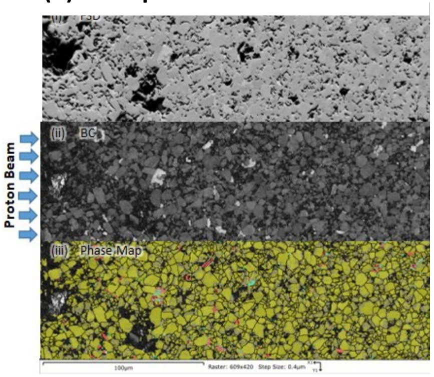
Evaluation of irradiated materials – EBSD from cWC

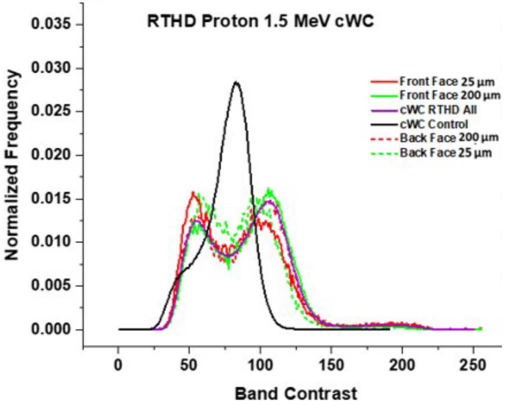
EBSD as a method of evaluating radiation-induced stress

(a) Control cWC



(b) 410K proton beam

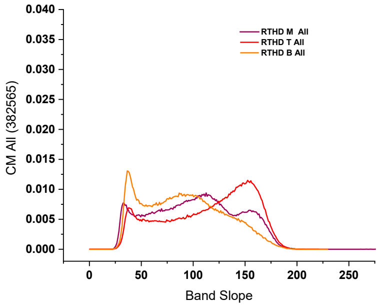




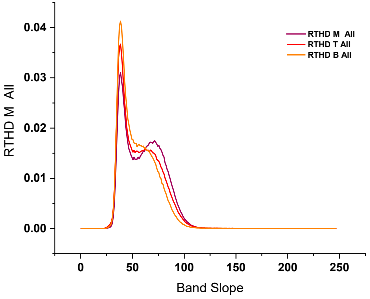
BS and BC is phase-independent

Band contrast profiles from control and 410K proton irradiated cWCs

(a) Control cWC Band Slope (BS)



(b) 410K proton beam Band Slope (BS)



Band Contrast: Dependant on internal strain, grain boundaries and preferred orientation

Band Slope: Dependant on internal strain, mosaicisity and dislocation presence

Caution is required when determining which EBSD parameter is used for evaluating radiation-induced changes in materials

Evaluation of irradiated materials – EBSD from cWC

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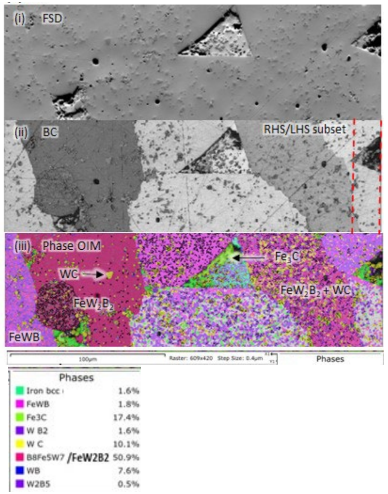
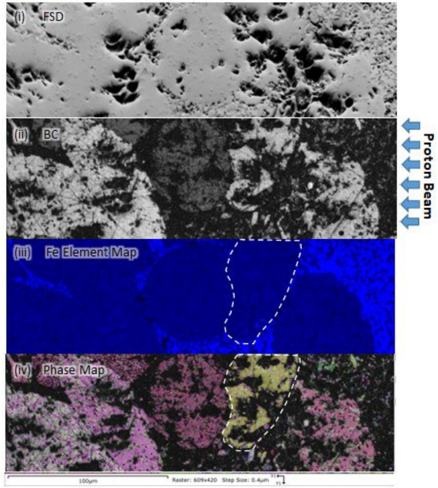
EBSD as a method of evaluating radiation-induced stress

(a) RSB control



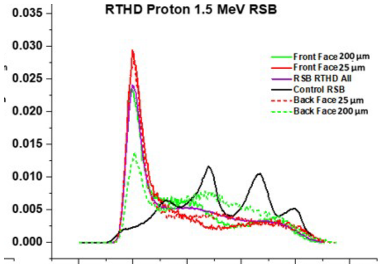
(b) 410K



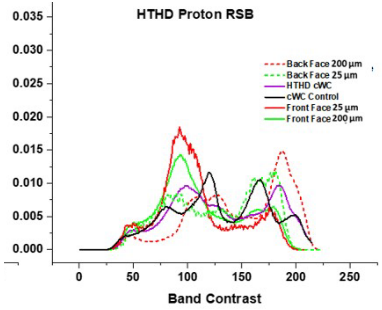
Combined EBSD-EDX maps enable mis-identification of phase regions from EBSD models.

In (b) the region initially identified as WC is actually FeW$_2$B$_2$.

(c) 410K Proton RSB



(d) 823K Proton RSB



Band Contrast profiles from **(c)** 410K proton irradiation and **(d)** 823K proton irradiation.

From **(a)** and **(b)** it is observed that preferred orientation has a significant impact on BC contrast alongside inter-granular stress.

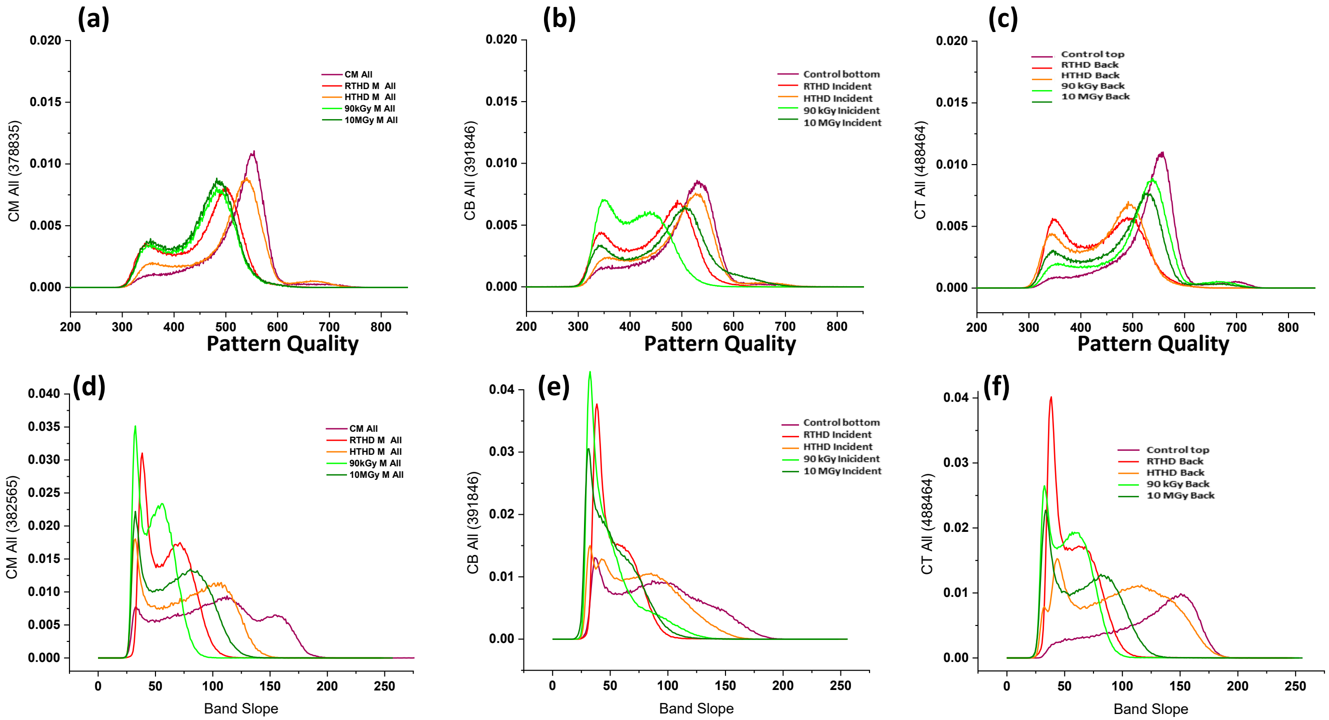
This can give spurious results if BC is the main metric for assessing radiation damage if the preferred orientation has a low BC contrast

Band Contrast and Band Slope more accurate than BC alone

Radiation Response in cWC materials by EBSD

Pattern Quality (PQ) and Band Slope depth profiles from cWCs

Pattern Quality related to BC but averaged over a wider sets of parameters



Pattern Quality (a-c)

(a) **All sample (front + back + middle)**

(b) **Incident faces**

(c) **Back faces**

Band Slope (d-f)

(d) All sample (front + back + middle)

(e) Incident faces

(f) Back faces

Edge effects are significant for cWC and RSBs in BS plots

Irradiation significantly enhances edge effects and reduces the maximum PQ and BS values

Next stage: Correlating changes in BS and PQ with quantitative TEM

Next: Research at the BUFFF

Thermal properties of cWC and RSB materials at the Bangor University Fuel Fabrication Facility (BUFFF)

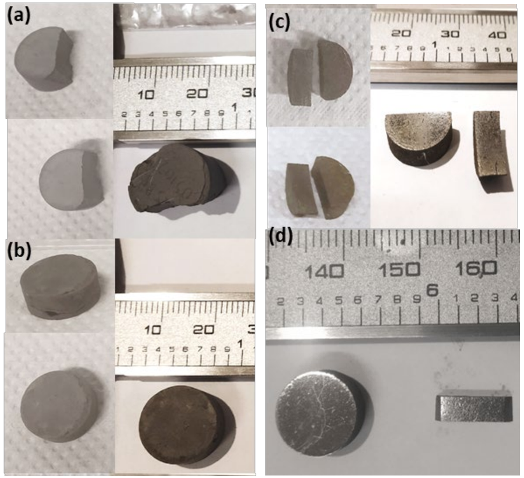
Preliminary investigations of cWC and RSB in March 2023

Plans include thermal capacity, dilatometry and live video of *in situ* sintering studies of cWC and RSBs

Dilatometry aims to investigate cWC, RSBs and joined cWC/RSB – steel samples.

In-situ imaging of RSBs through the sintering process will enable exact determination of swelling and shrinkage at different stages of the sintering process and how these differ for cWC and RSB materials

Feedback of updated sintering data to industrial partners at Hyperion MT for small-scale industrial sintering



Interrupted sintering studies on RSBs: (a) 1300°C; (b) 1350°C; (c) 1400°C and (d) 1450°C

RSBs swell up to >200% of original volumes

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Biggest RSB sample at > 98% density (12.2 g cm^{-3}) 30 mm diameter x 13 mm height.

Working with the NNUF

NNUF access has been instrumental in accelerating radiation studies of cWC and RSB materials

- First ambient and non-ambient study of irradiation on cWC and RSB materials
- γ-irradiation (^{60}Co) appears to consolidate dislocations in cWCs but creates new stress-induced dislocations in RSBs
- Evidence of dislocation (stress-induced) migration from harder to softer phases in cWCs and RSBs.
- More stress-induced dislocations observed in the 77K 90 kGy samples ≤ 50 µm from incident face than for 10 MGy 293K sample
- First experimental determination of attenuation (1.5 MeV proton) and HVL for cWC and RSB

Material	**Proton depth (µm)**	**γ-ray attenuation coefficients (1.5 MeV)**				**γ-ray $I/I_0 = 0.5$ (cm)**	
	1.5 MeV	**μ_{en}/ρ (cm^2 g^{-1})**	**μ/ρ (cm^2 g^{-1})**	**μ_{en}(cm^{-1})**	**μ(cm^{-1})**	**HVL_{en}**	**HVL**
cWC8D	10.58	0.0248	0.0504	0.35	0.71	1.99	0.98
RSB (B5T522W)	10.78	0.0241	0.0491	0.29	0.58	2.39	1.18

Working with the NNUF: Next Stage

He-Fe bombardment and in-situ stress-testing at DCF and TANIST

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- Combined heavy-ion and He-bombardment on cWC and RSB materials to simulate neutron degradation.
- Ion-beam irradiation at the DCF at 400°C, 550°C and 700°C to determine the temperature where ion-induced swelling peaks.
- TANIST facility at the University of Manchester enables EBSD on stress-testing *in-situ* to build up a time-resolved evolution of tensile stress over time.

This work will be the first time-resolved study of stress-induced evolution of ion-bombarded cWC and RSB candidate samples under fusion-simulated conditions

Working with the NNUF

Thank you all for your attention!

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My team at Warwick:

Left to right: Suresh Srinivasen, Joe Gillham, myself and Gurdev Singh

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