WP 4 (T4.1.1, T4.4.1 & T4.3.4) IASCC TESTING

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[1] Wood, [2] University of Manchester, [3] Framatome, [4] VTT

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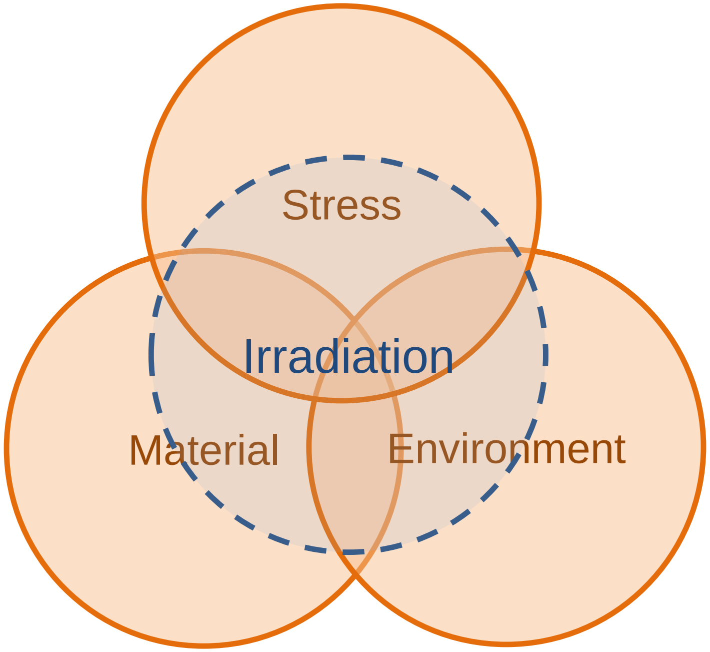
Content

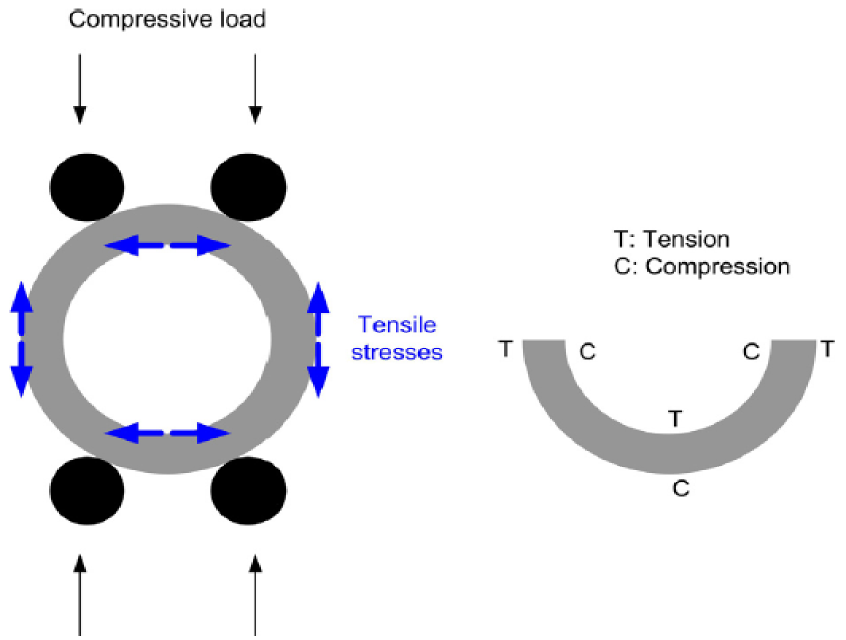
Introduction: IASCC

- IASCC is a phenomenon whereby a material that would not usually be susceptible to SCC in a particular environment becomes so, due to changes to the material and/or environment by the presence of neutron irradiation
- Both microstructural matrix damage and compositional segregation are considered important factors causing IASCC
- Many forms of testing have been carried out to understand the phenomenon further
- Static loading is often used for crack initiation tests, i.e. measuring time to initiation
- Active loading is often used to identify SCC susceptibility in a material and commonly takes the form of Constant Extension Rate Test (CERT)



Introduction: IASCC

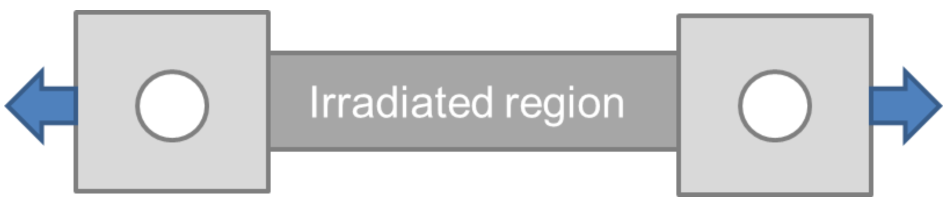
- Statically loaded C or O ring specimens are a simple and common way of IASCC testing material from service reactors
  - Sections of flux thimble tubes can be used as they are already in the correct shape
- Using surrogate irradiation, such as proton or ion, has many benefits such as cost and ease of handling the material
  - However, specimen geometry is more limited as the irradiation process requires a flat surface

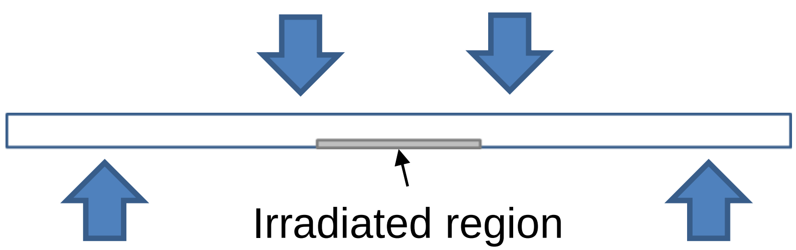


Introduction: IASCC

Proton Irradiated Testing

- Previous studies looking at IASCC initiation have commonly employed simple tensile loading of flat specimens to allow proton irradiation to be conducted on one surface[1, 2]
- Differing mechanical properties between the irradiated surface and the un-irradiated bulk material may affect the stress experienced within the irradiated layer, and therefore the IASCC initiation response
- A four point bend methodology has, therefore, been developed aiming to better control the stress experienced by the irradiated layer
  - It also has the benefit of reducing the irradiated surface area

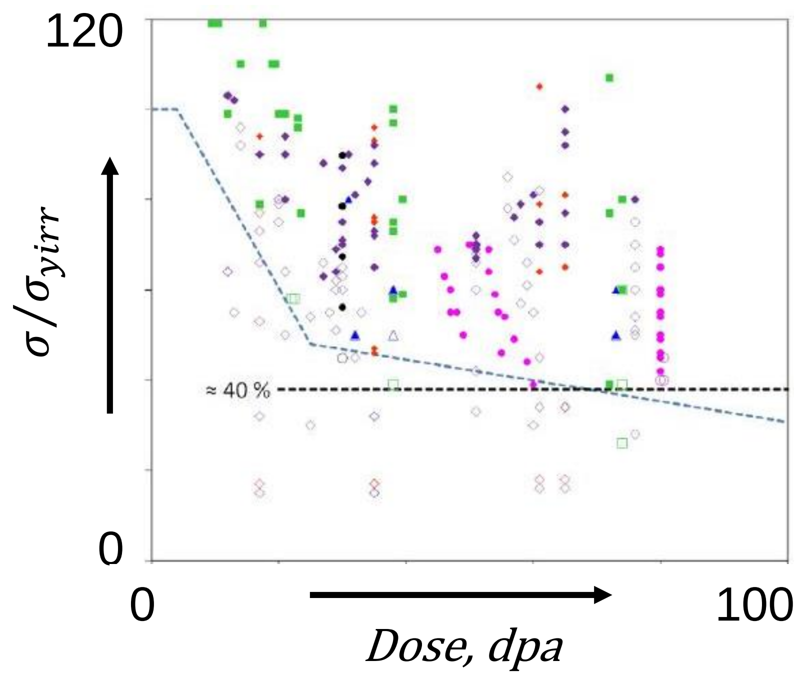


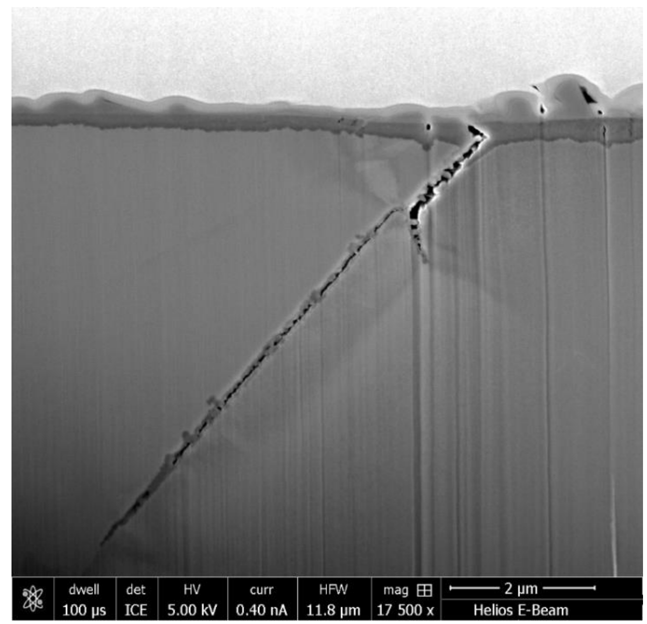


[1] W.J. Mills, et al, Effect of irradiation on the SCC behaviour of Alloy X-750 and Alloy 625, Proc. 6th Int. Symp, on Environmental Degradation in Nuclear Power Systems – Water Reactors, San Diego, August 1993, pp 633 – 642.
[2] M. Wang, et al, Irradiation assisted stress corrosion cracking of commercial and advanced alloys for light water reactor core internals, J. Nucl. Mat. 515 (2019), pp 52 – 70.

Introduction: IASCC

- The IASCC testing within WP4 has two key aims:
  - To produce SCC initiation data contributing to the database used for determining an IASCC threshold
  - To provide post-test material for characterisation to aid the mechanistic understanding of IASCC

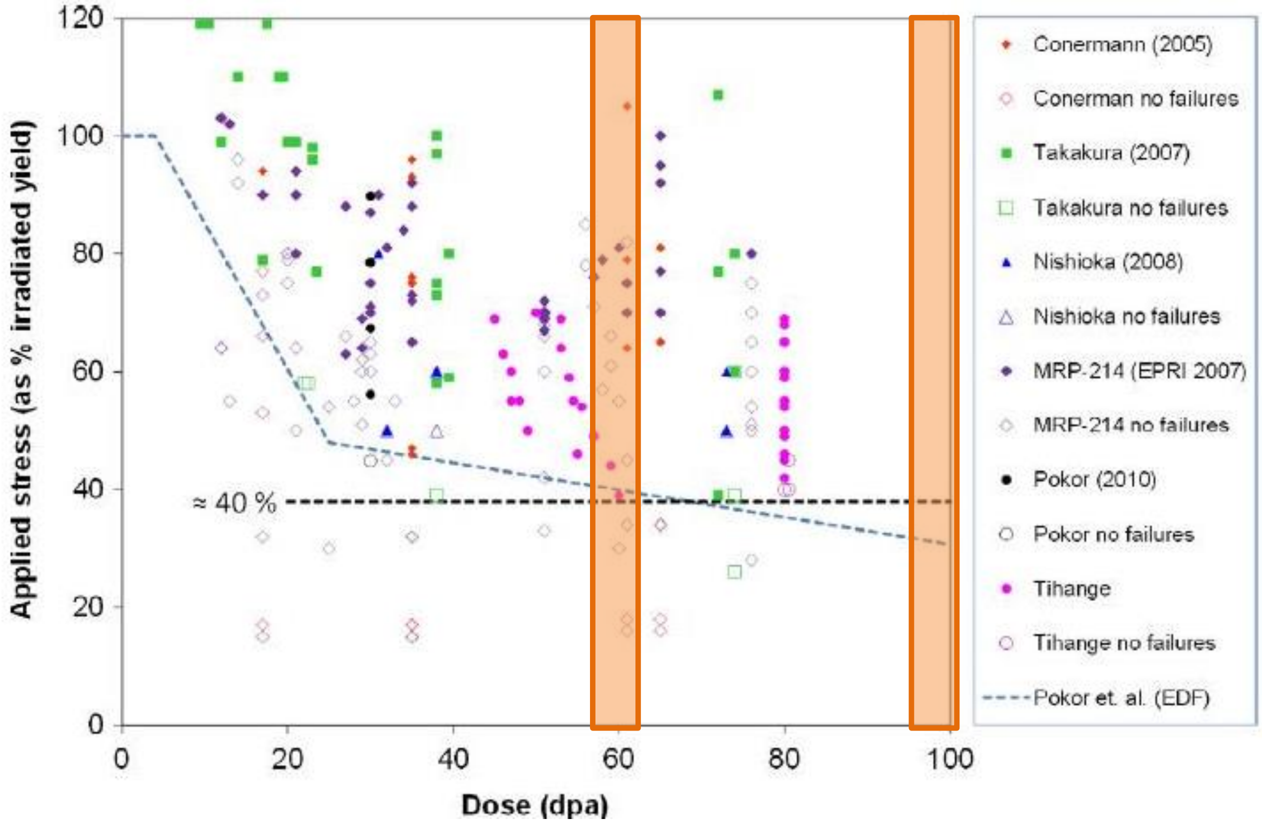




Cross sectional SEM image of SCC observed in an irradiated 316L SS specimen from this programme

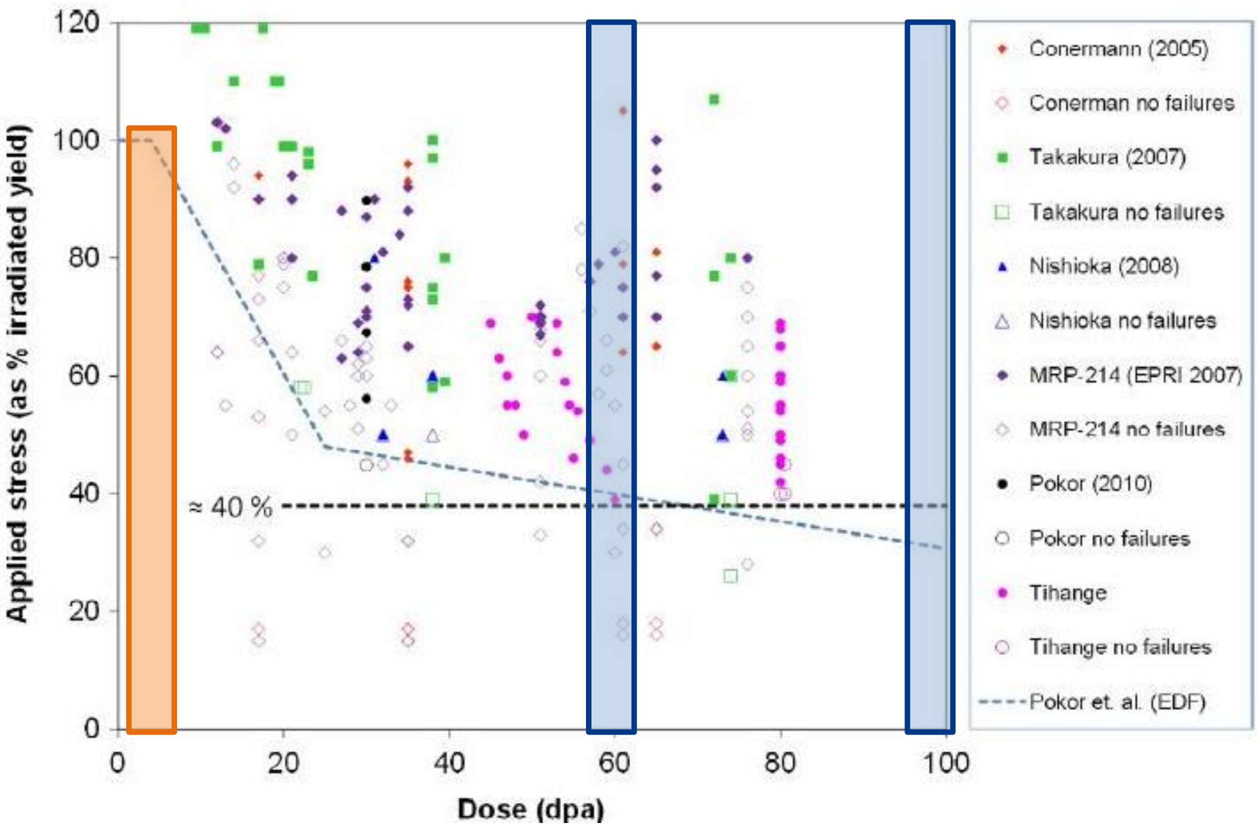
Introduction: VTT

- IASCC of neutron irradiated material = 65 / 100 dpa



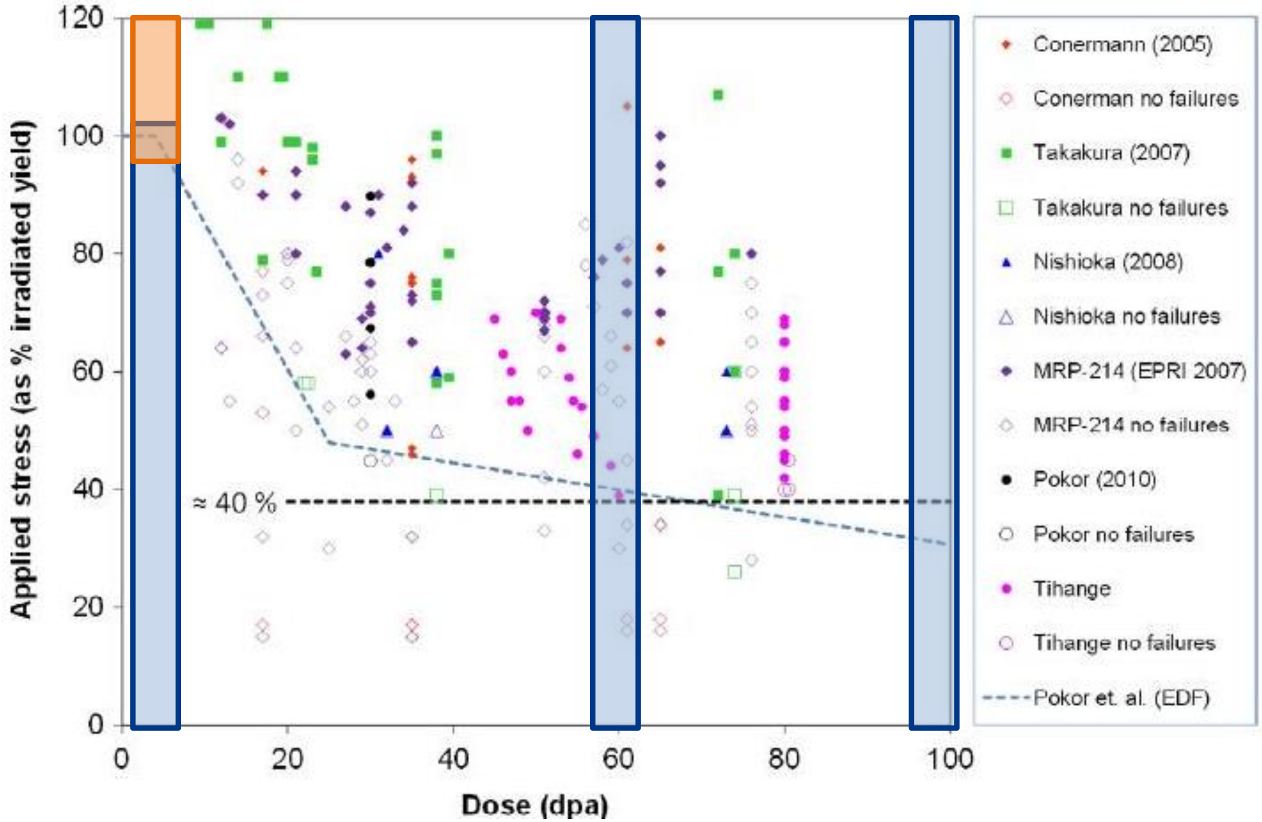
Introduction: Framatome

- IASCC of neutron irradiated material = 65 / 100 dpa
- IASCC of proton irradiated material = 5 dpa



Introduction: Wood/UoM

- IASCC of neutron irradiated material = 65 / 100 dpa
- IASCC of proton irradiated material = 5 dpa
- IASCC of proton irradiated material = 5 dpa



NEUTRON IRRADIATED IASCC TESTING

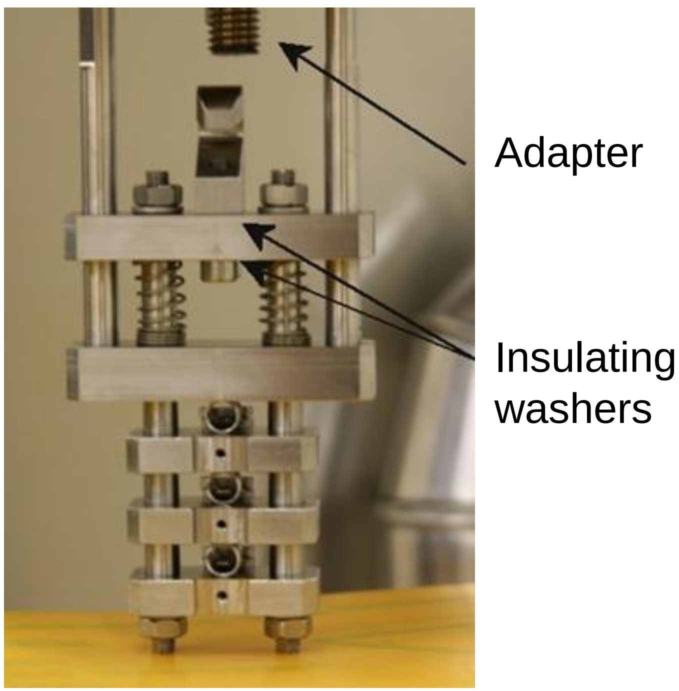
VTT: Neutron Irradiated Material

- Material
  - Type 316L stainless steel from two Flux Thimble Tubes (FTTs).
  - From Ringhals Unit 2: FTT-99 with a peak dose of 65 dpa, and FTT-10 with a peak dose of 100 dpa.
- Properties of the irradiated o-rings:

Specimen ID	FTT	Dose, dpa	Length, mm	σ_y (Mpa) @ 320°C	UTS (Mpa) @ 320°C	ε_u (%) @ 320°C	ε_{tot} (%) @ 320°C
LS011	FTT-99	65	8	937	999	0.7	7.0
LL012	FTT-99	65	8	937	999	0.7	7.0
HS011	FTT-10	100	8	982	995	0.5	6.0
HL01	FTT-10	100	8	982	995	0.5	6.0

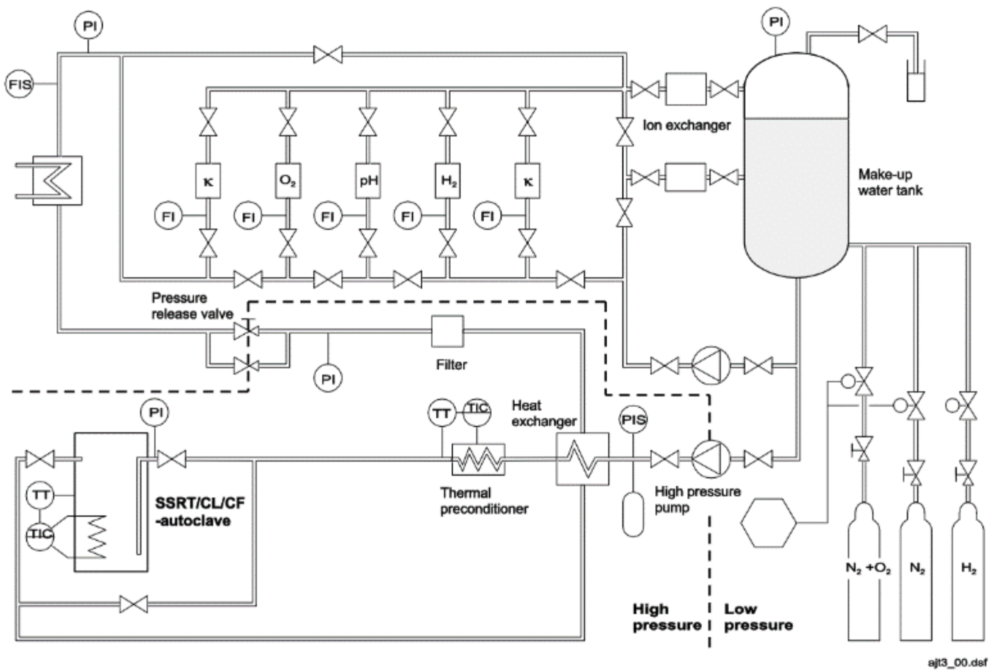
VTT: Methodology

- ❑ Operation of the loading system
  - Manipulator opens the rig
  - Tweezers with an integrated camera are used to place the specimens
- ❑ Two specimens included per test, to test two different dpa levels under each condition
- ❑ Load applied under test conditions



VTT: Methodology

- Recirculating water loop with online monitoring of temperature, pressure, flowrate, water conductivity, pH and dissolved H_2 & O_2



VTT: Progress

- O-ring cutting technique has been developed
- Autoclave and loop has now been successfully operated with non-irradiated 316 SS mock-up material
- Shielding set-up and remote autoclave loading system has been designed and components fabricated
- Loading of the specimens remotely has been demonstrated

VTT: Ongoing Work

- Two tests are to be carried out at 340°C in a simulated-PWR aqueous environment
  - One with standard H_2 and one with high H_2
- 2 Specimens are to be tested under each condition
  - One of each dpa level

Test	Specimen ID	L (mm)	Dose (dpa)	σ_y (MPa)	σ (MPa)	σ/σ_y	H_2 (cc.kg^{-1})
1	LS011	8	65	937	610	0.65	17-28
	HSO11	8	100	982	610	0.62	17-28
2	LS012	8	65	937	610	0.65	50
	HL01	8	100	982	610	0.62	50

PROTON IRRADIATED IASCC TESTING

Material

❑ Non-irradiated Material Properties:

Chemical Composition

Wt. %	C	S	P	Si	Mn	Ni	Cr	Mo	Cu
316L	0.023	0.008	0.025	0.488	1.73	12.31	17.52	2.45	0.132

Room Temperature Mechanical Properties

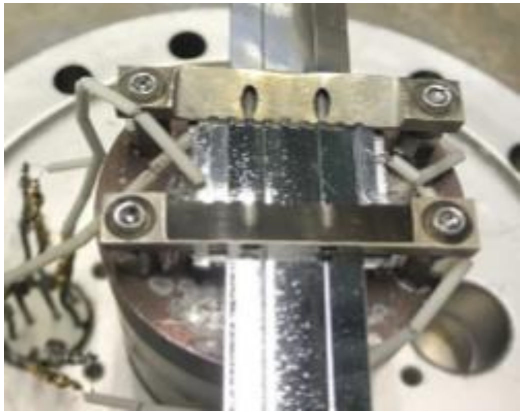
Temp.	σ_y (Mpa)	UTS (Mpa)	A%	Hardness (Hv)
RT	279	584	56.5	150

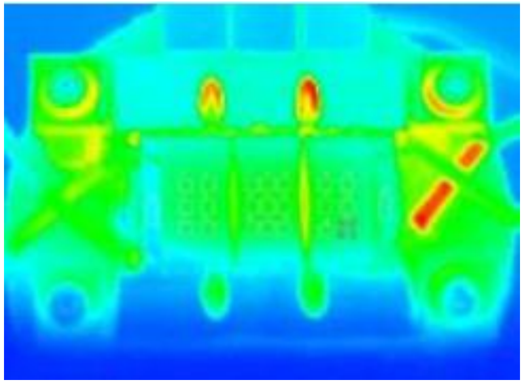
Microstructural Properties

Material	Heat	Ms (℃)	Md30 (℃)	Grain Size (µm)	Ferrite (%)	SFE (mJ/m2)
316L	T8289	-224	-7.2	125	3.3	30.4

Proton Irradiation

- Proton irradiation performed at the University of Michigan
- Both UoM and FRAMATOME specimens applied same method
- Damage at 5 dpa est. across central 10mm of specimens
- Irradiation carried out at 350°C

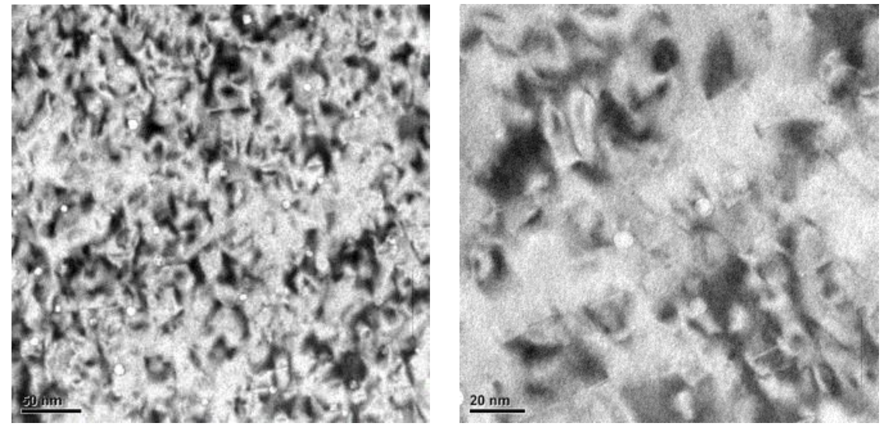


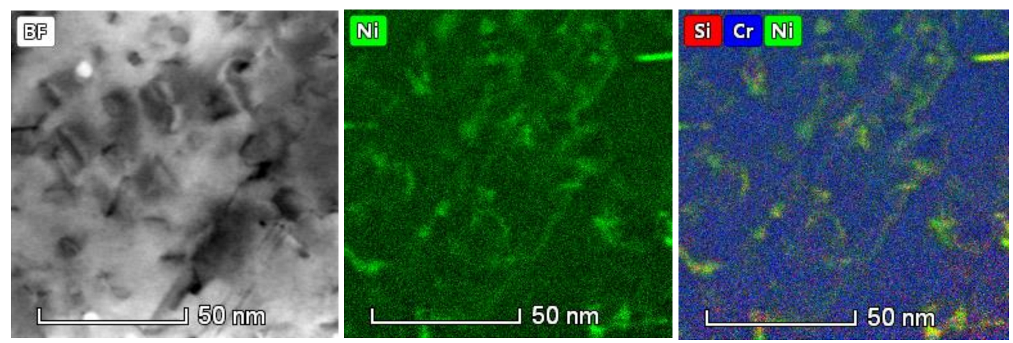


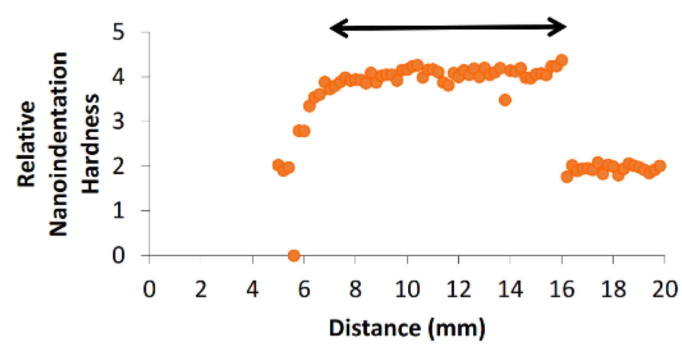
Proton Irradiation

UoM Characterisation

- Numerous very fine cavities (~5-10nm diameter)
- Numerous dislocation loops (~5-30nm)
- Ni and Si segregation to loops, cavities etc.
- Fe and Cr displaced from loops, dislocations and cavity/matrix interface
- Nano-hardness used to confirm the irradiated zone as 10mm along the samples

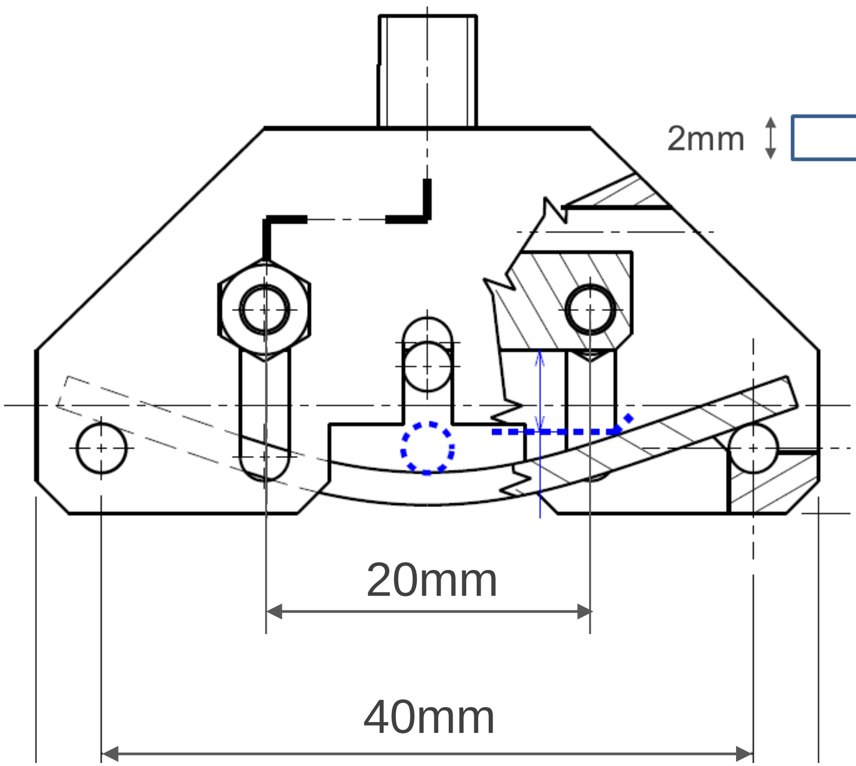


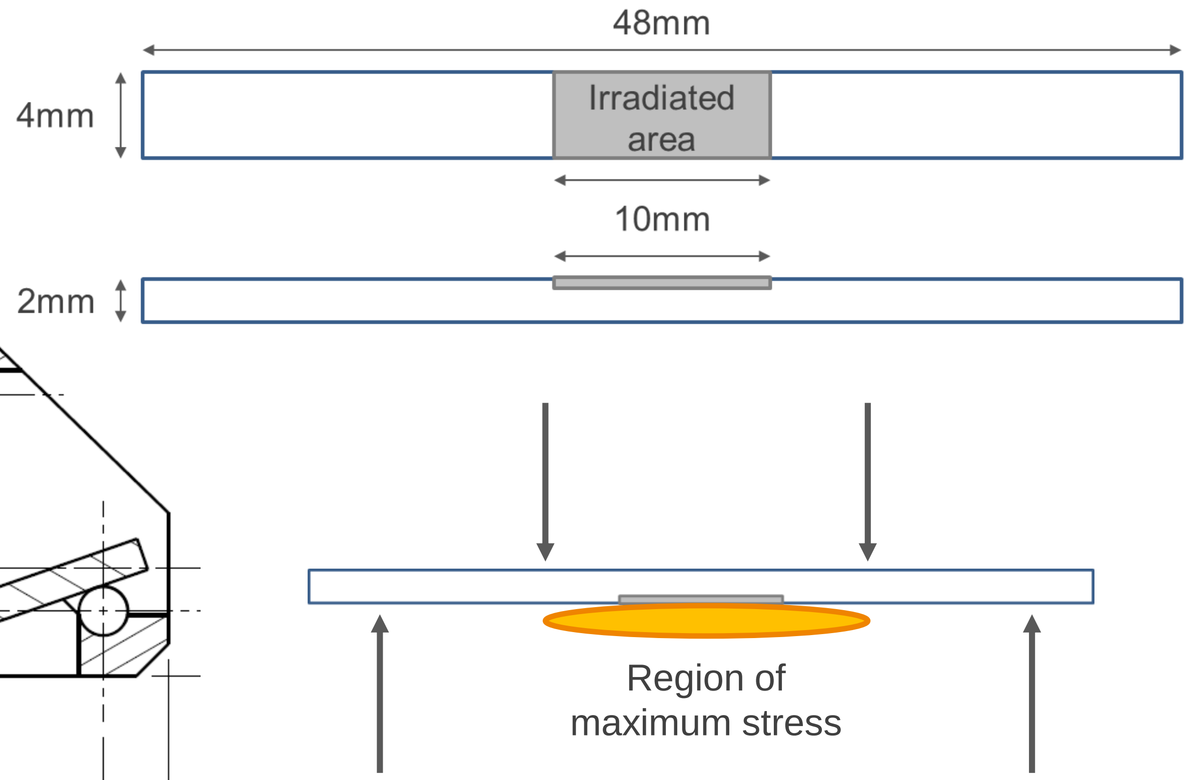




Framatome: Methodology

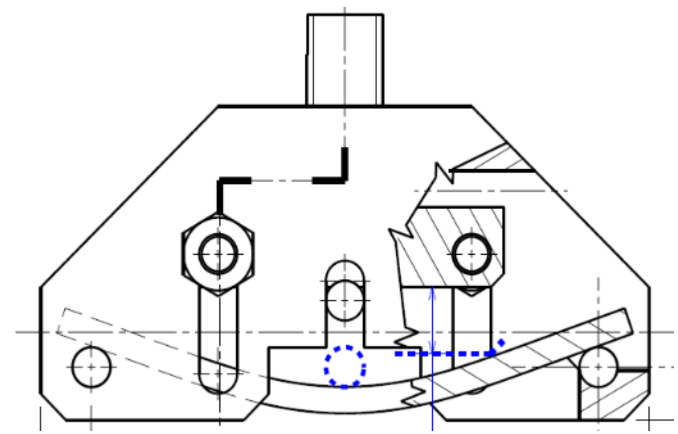
- Specimen and Loading Geometries:

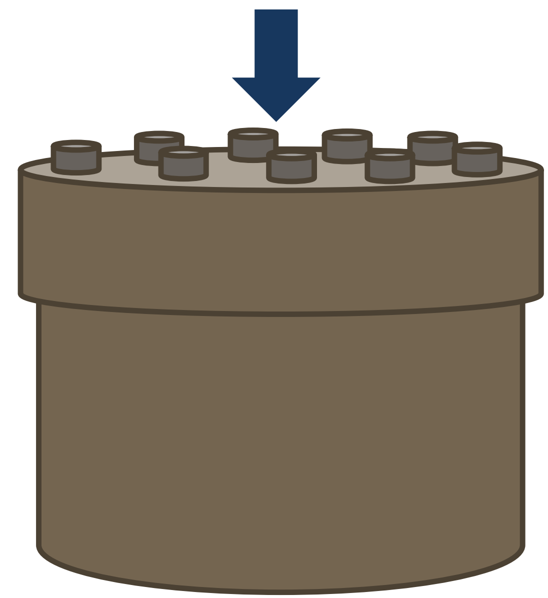




Framatome: Methodology

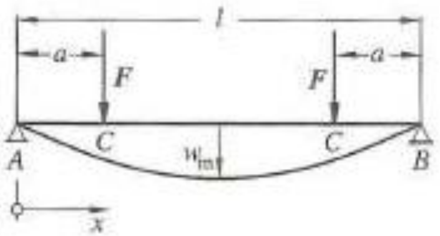
- Statically loaded in 4 point bend jig at room temperature to permit accurate control of specimen deflection
- Placed in autoclave
  - 340°C, Sim-PWR water chemistry
  - Recirculated water
  - Online monitoring of conditions
- Both base material (non-irradiated) and irradiated specimens tested at multiple deflections
- Tested for 500h, 1000h and 2000h





Framatome: Loading

- As the target applied stresses were at or below yield, simple elastic beam bending equations were used to calculate the required deflection at the central point of the specimen



$0 \le \xi \le \alpha$:

$$w = \frac{Fl^3}{6EI}\xi\left(3\alpha - 3\alpha^2 - \xi^2\right)$$

$\alpha \le \xi \le l/2$:

$$w = \frac{Fl^3\alpha}{6EI}\left(-\alpha^2 + 3\xi - 3\xi^2\right)$$

$$w_C = \frac{Fa^2}{6EI}(3l - 4a)$$

$$w_m = \frac{Fa}{24EI}\left(3l^2 - 4a^2\right)$$

$$-\varphi_A = \varphi_B = \frac{Fa}{2EI}(l - a)$$

$$\left|\varphi_C\right| = \frac{Fa}{2EI}(2a - l)$$

Specimen Type	Level 1 Central Deflection (mm)	Level 2 Central Deflection (mm)	Level 3 Central Deflection (mm)
Irradiated	-	0.550	0.733
Non-irradiated	0.275	-	0.733

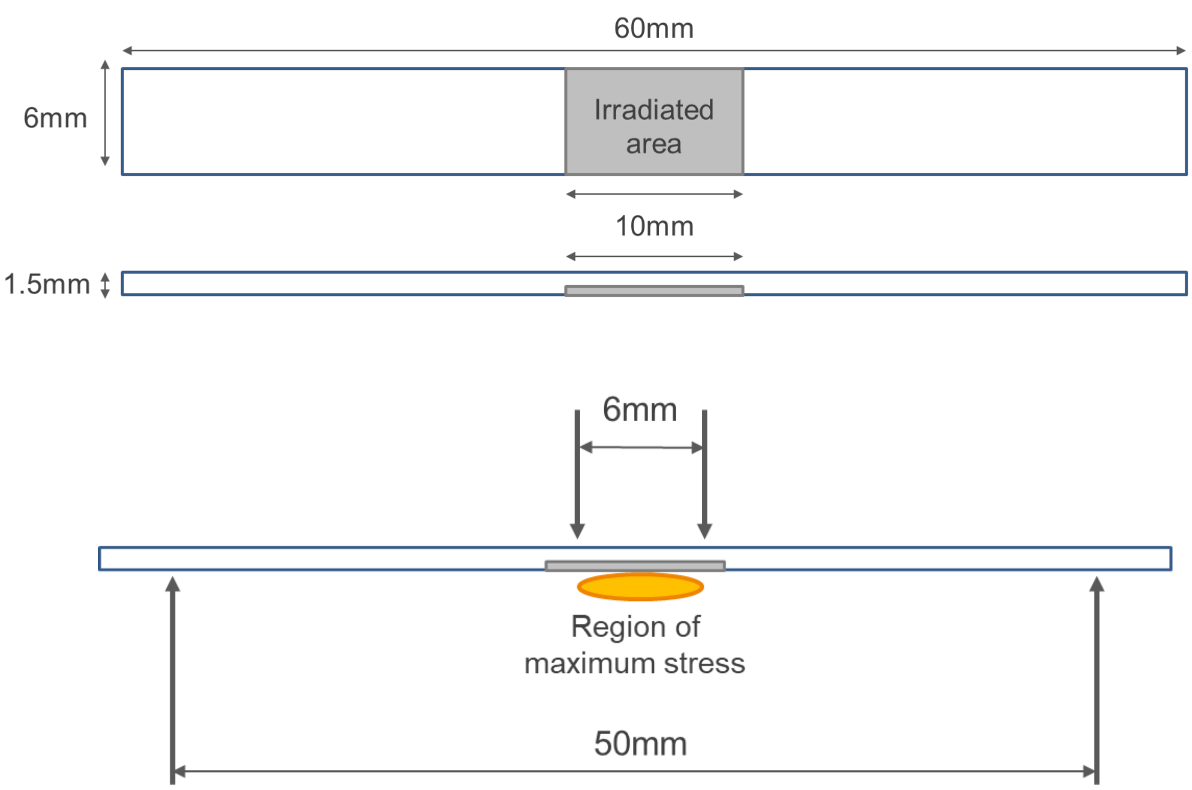
Framatome: Test Matrix

- All testing carried out at 340°C in a simulated-PWR aqueous environment
- 2 Specimens were tested under each condition

Specimen Condition	Applied Strain (%)	Test Duration (hours)
Non-irradiated	0.16	500, 1000, 2000
	0.43	500, 1000, 2000
Irradiated	0.32	500, 1000, 2000
	0.43	500, 1000, 2000

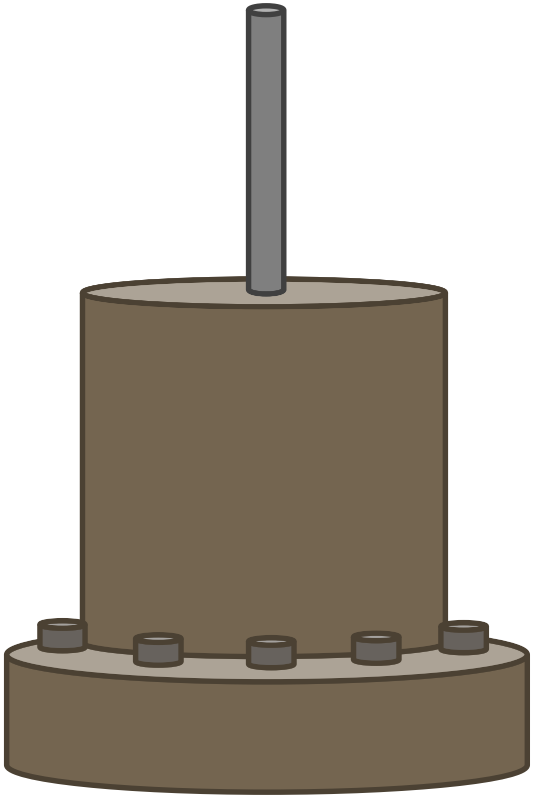
Wood/UoM: Methodology

- Specimen and Loading Geometries



Wood/UoM: Methodology

- Two stage loading technique applied
  - Pre-conditioning stage conducted to allow oxidation of specimen under low stress[1]
  - Final load applied at a slow strain rate of 10^{-6} s^{-1}



[1] L. Chang, J. Duff, G. Burke, F. Scenini, SCC Initiation in the Machined Austenitic Stainless Steel 316L in Simulated PWR Primary Water, Corrosion Science Vol. 138, (2018), p. 54-65.

Wood/UoM: Methodology

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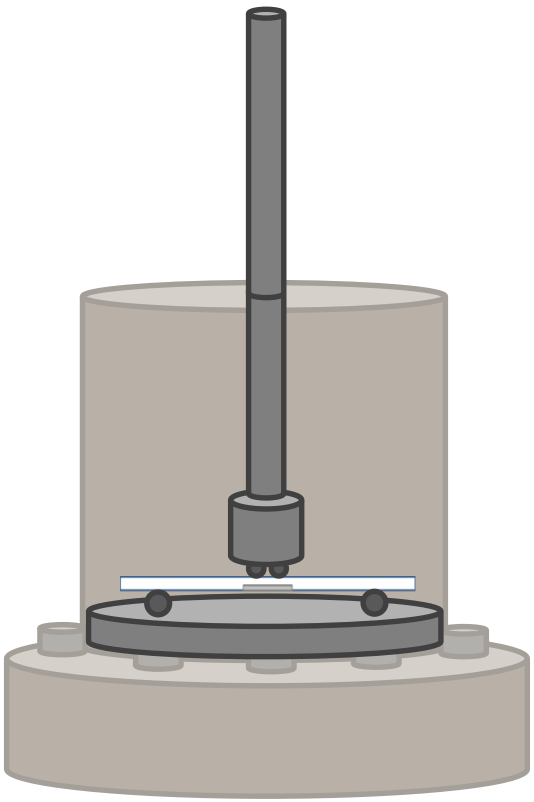
- Loading applied under test conditions within autoclave
  - 340°C, Sim-PWR water chemistry
  - Online monitoring of test conditions



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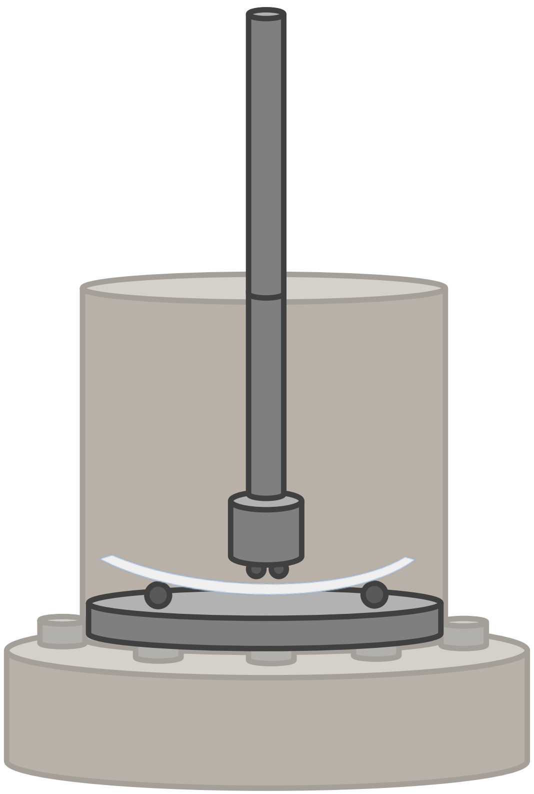
  - Final load applied at a slow strain rate of 10^{-6} s^{-1}
- Loading applied under test conditions within autoclave
  - 340°C, Sim-PWR water chemistry
  - Online monitoring of test conditions
- In Tests 1 & 2, specimens were inspected after holding at the final load for 1000 hours
- In Test 3, the specimen was inspected immediately after loading



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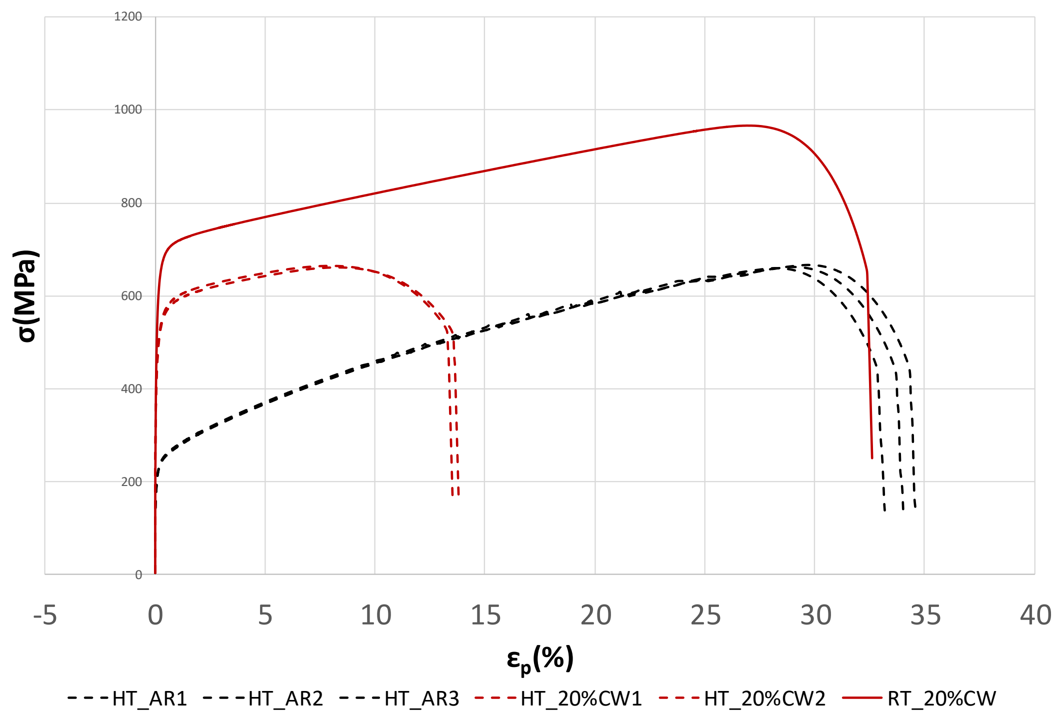
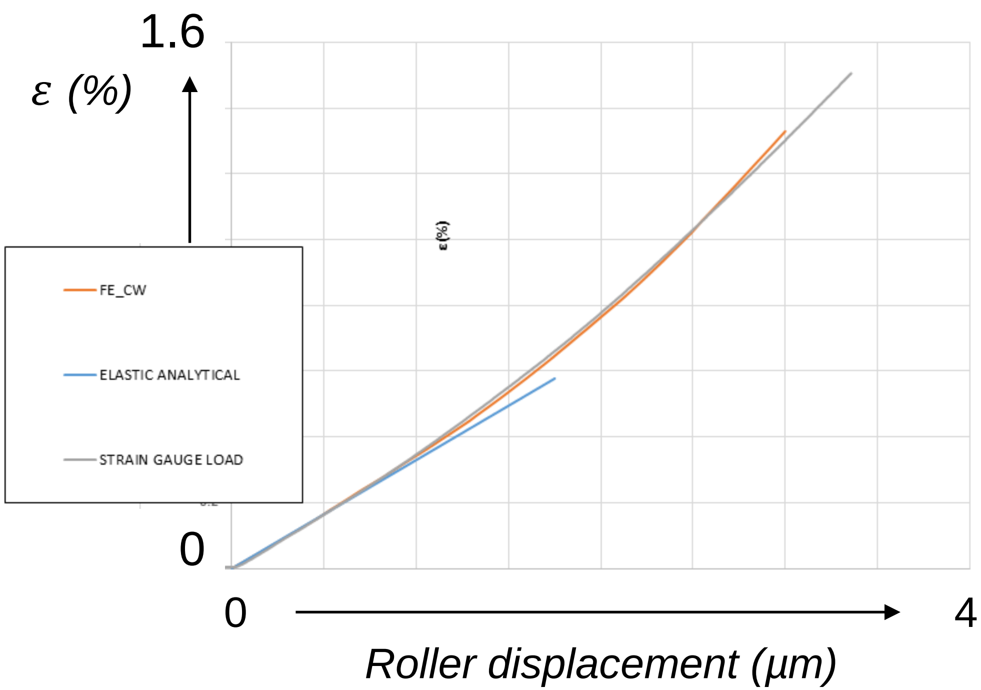
Wood/UoM: Loading Calculations

- Finite Element Analysis (FEA) was employed to model the stress/strain induced in the irradiated layer as a function of roller displacement
- Tensile data were available for a similar 316L SS at 300°C which were used to model the specimen bulk
- Hardness data were used to infer properties of the irradiated layer
  - Results indicated similar properties to 20% CW 316L SS
- Tensile data were available for a similar 20% CW 316L SS at 340°C which were used to model the irradiated layer



Wood/UoM: Loading Calculations

- To support the FEA, and to identify any compliance within the loading system, two strain gauge tests were performed on surrogate cold worked material (RT, air) and results compared with the model
- Data were in good agreement for the total strain induced on the specimens



- However, the results implied varying levels of compliance within the loading system between tests
- Compliance was subsequently calculated for all tests and post-test corrections applied to the test stress/strain

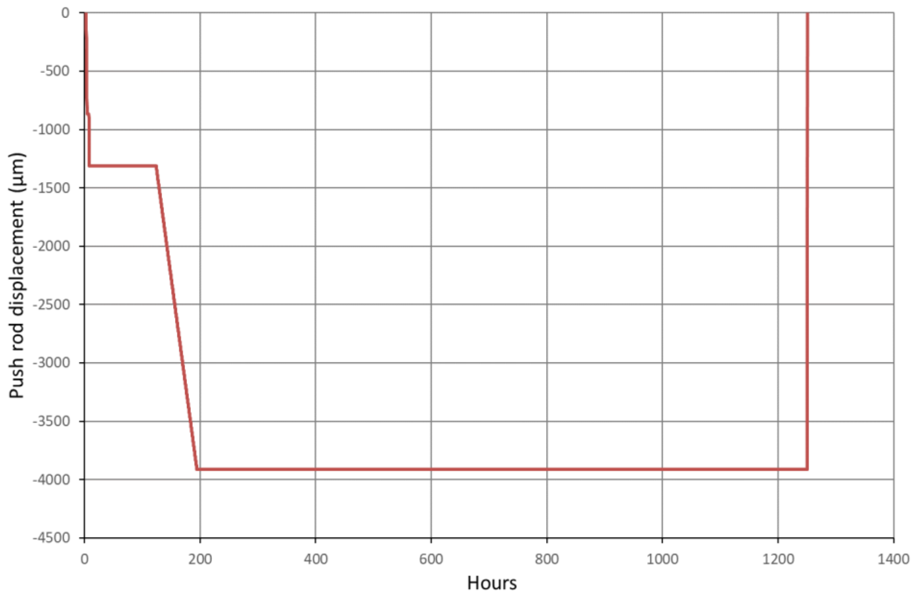
Wood/UoM: Test Matrix

- All testing carried out at 340°C in a simulated-PWR aqueous environment
- 1 Specimen tested under each condition

Specimen Condition	Loading Stage 1		Loading Stage 2	
	Nominally Applied Strain (%)	Hold Duration (hours)	Nominally Applied Strain (%)	Hold Duration (hours)
Irradiated	< yield	100	2	1000
Irradiated	< yield	100	1	1000
Irradiated	< yield	100	0.55	0

Post Test Analysis: FEA

- Both Laboratories recorded an accurate in-test specimen deflection
- These values were used in post-test FEA to determine the strain and subsequent stress achieved for each test



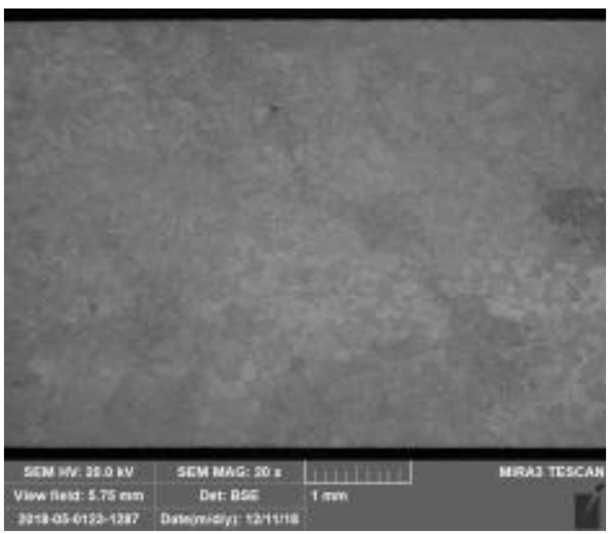
- Post test data from the Wood/UoM tests were used to incorporate compliance of the loading system
- The Wood/UoM pre-test loading models were used to incorporate the plastic stress/strain behaviour in the Framatome tests

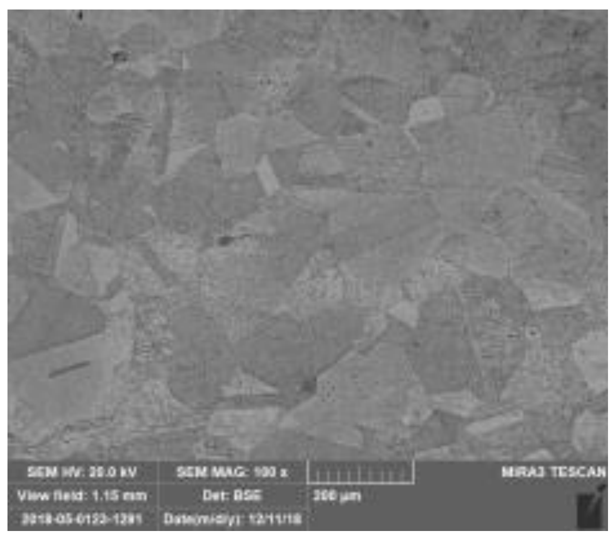
Post Test Analysis: Characterisation

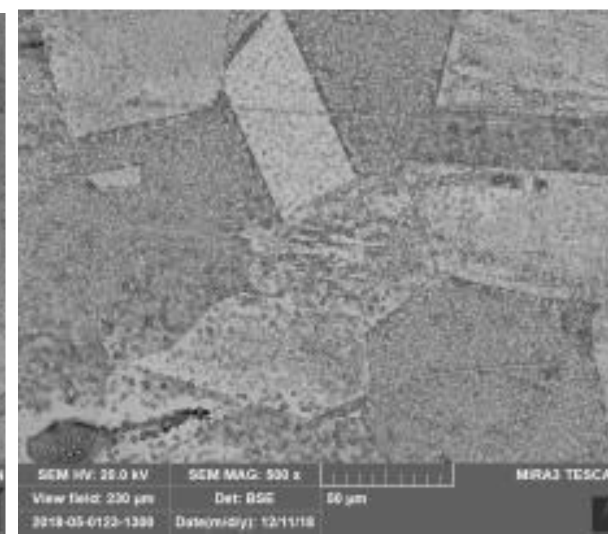
Framatome

- Framatome did not observe any evidence of SCC initiation during post-test inspection for all test conditions

Irradiated, 800MPa, 2000 hours





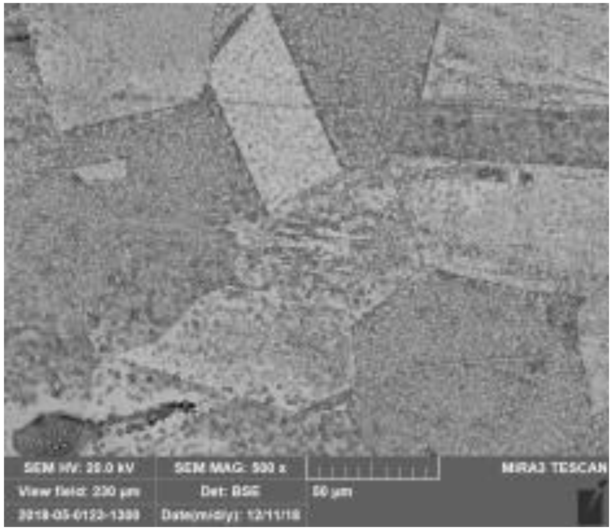


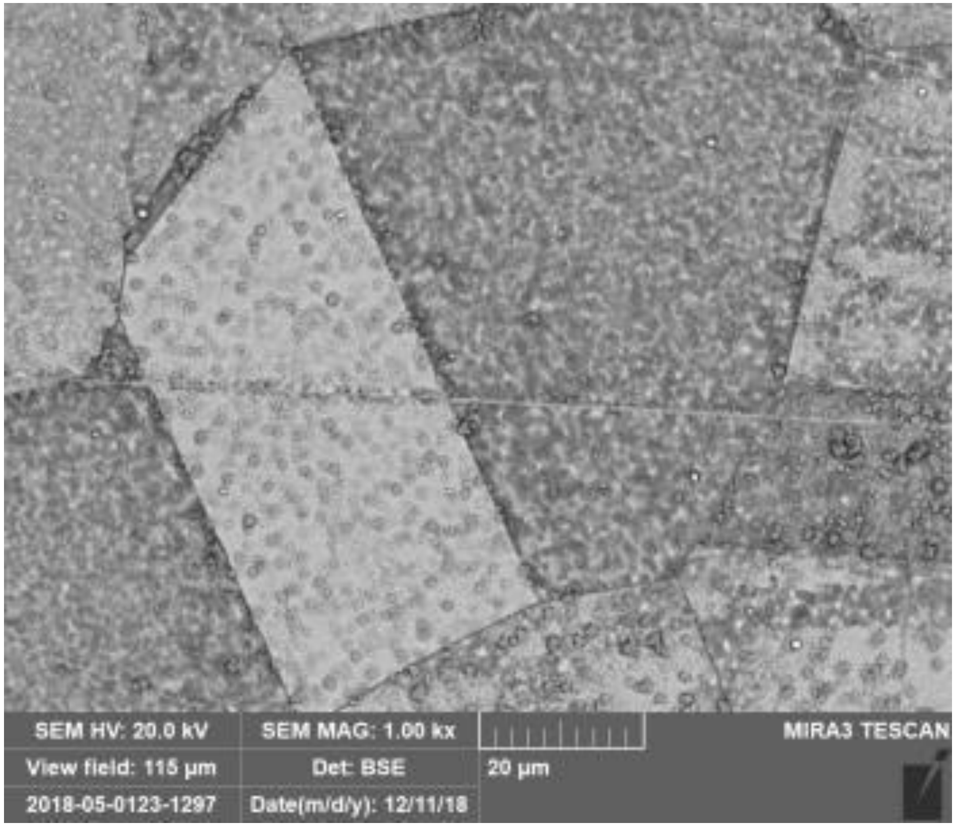
Post Test Analysis: Characterisation

Framatome

- Possible evidence of more favourable oxide growth at grain boundaries

Irradiated, 800MPa, 2000 hours

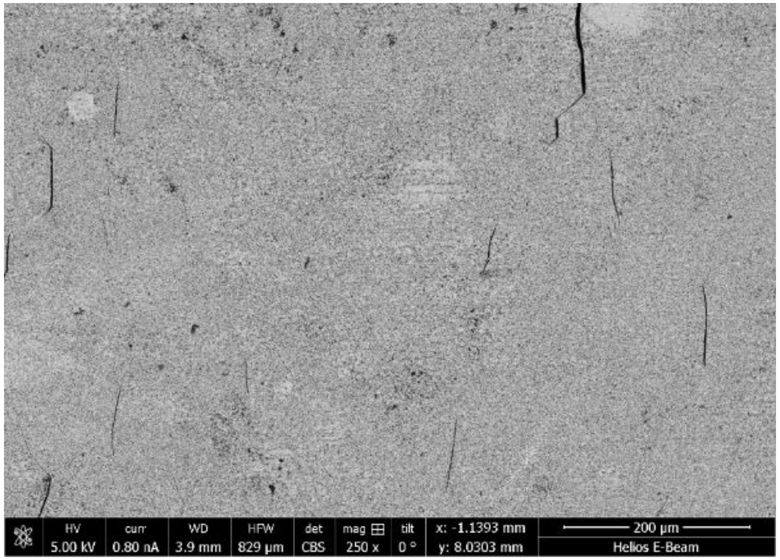


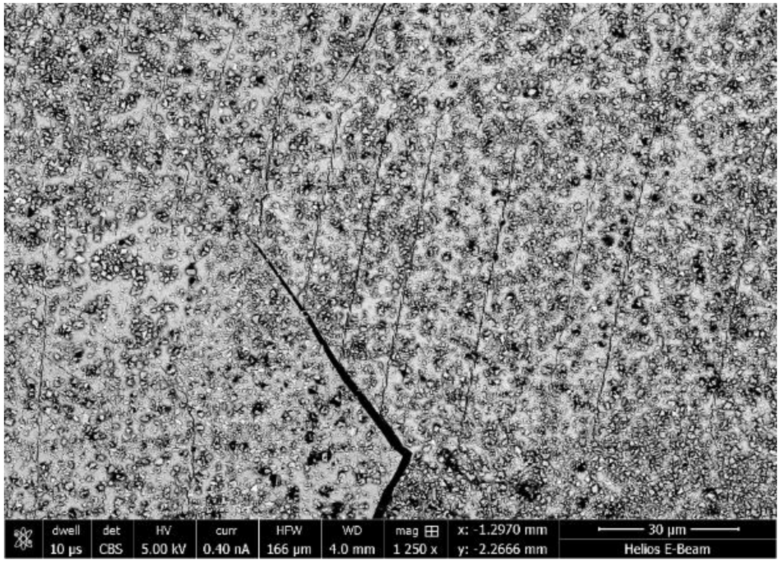


Post Test Analysis: Characterisation

Wood/UoM

- Post-test SEM of the irradiated surface of all the samples show intergranular cracking and surface emergent slip channels consistent with IASCC.

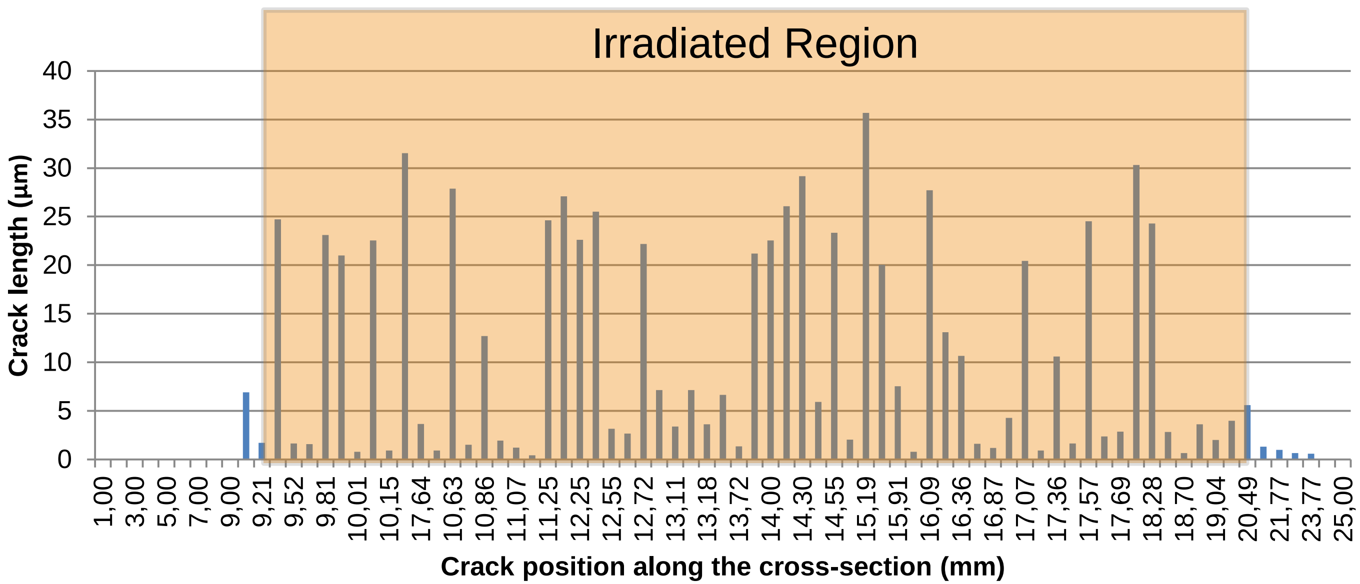




Post Test Analysis: Characterisation

Wood/UoM

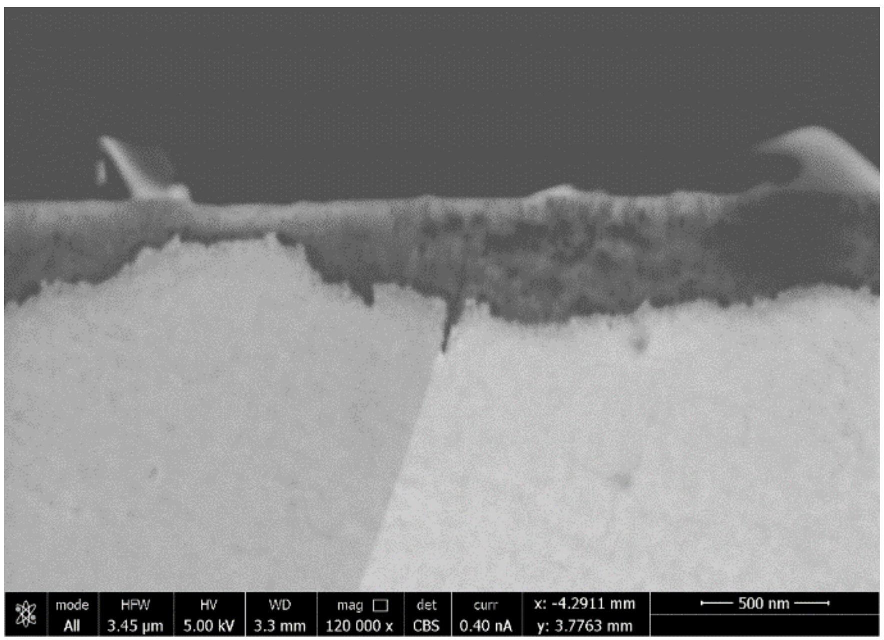
- Following surface examination, the proton irradiated specimen from Test 1 was cut by EDM along the longitudinal length
- 132 cracks along the length of the cross-section were identified,125 within the 10mm region corresponding to the proton irradiation

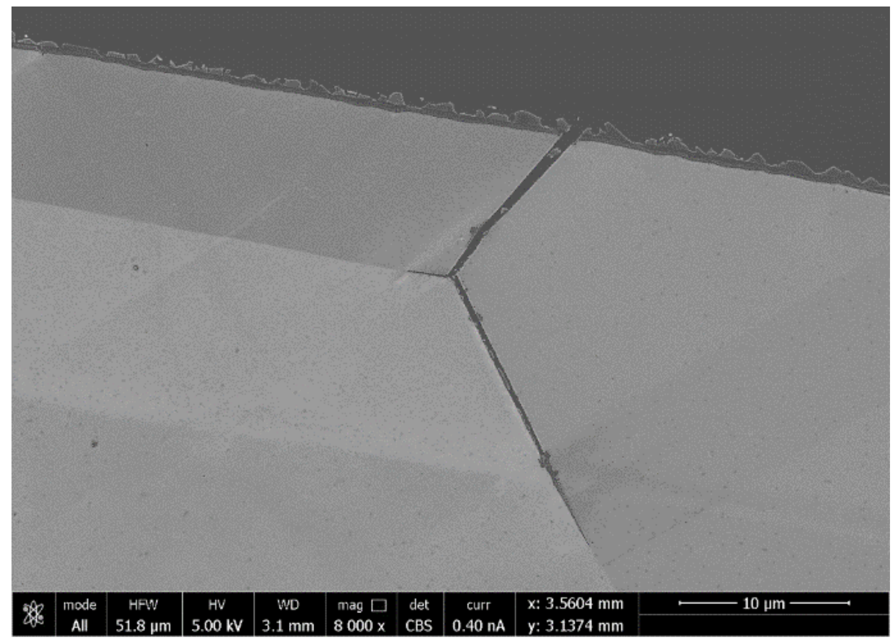


Post Test Analysis: Characterisation

Wood/UoM

- Cracks were intergranular with depths ranging between 1μm and 30μm

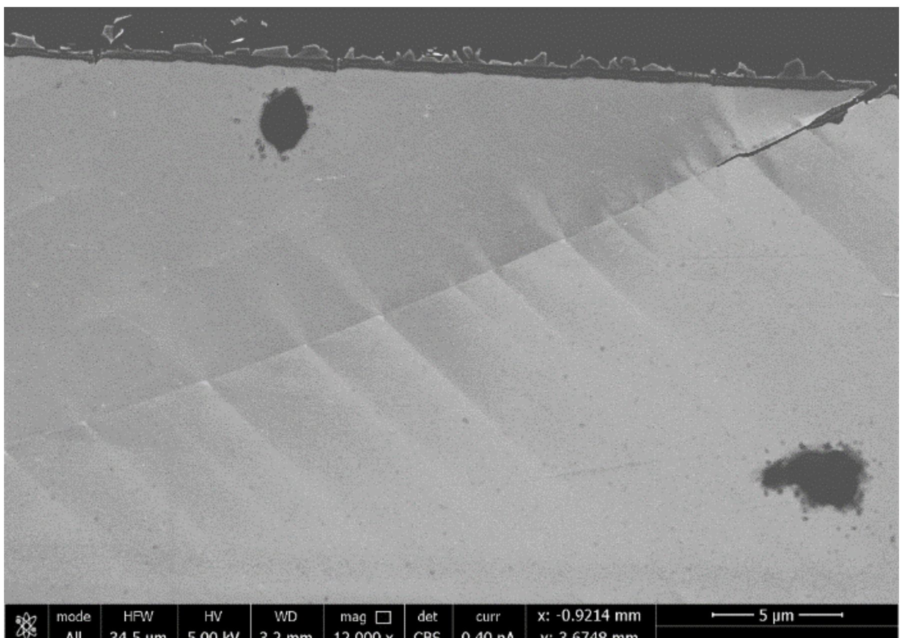


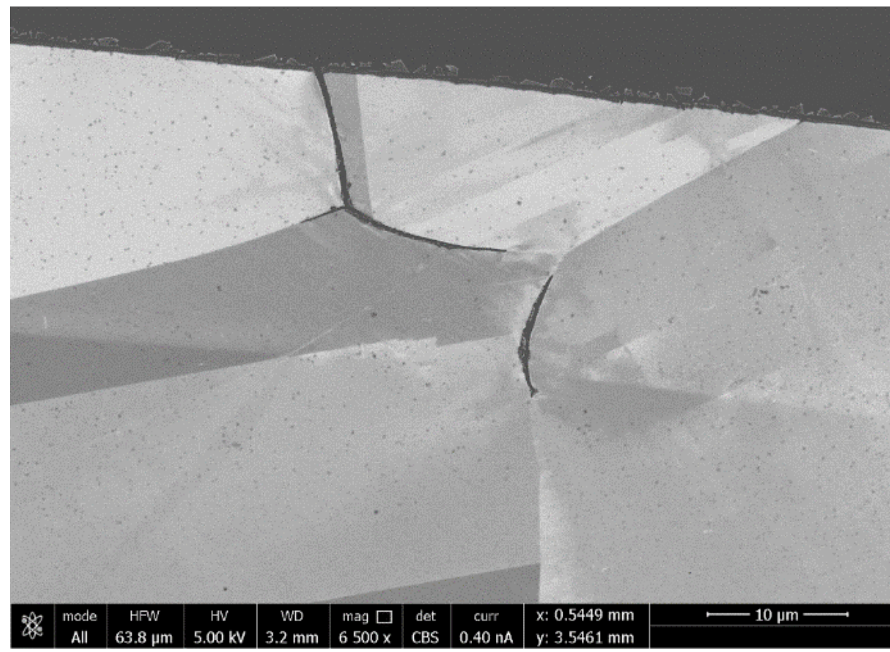


Post Test Analysis: Characterisation

Wood/UoM

- Grain boundary deformation without cracking as well as discontinuous crack paths consistent with crack bridging were seen.

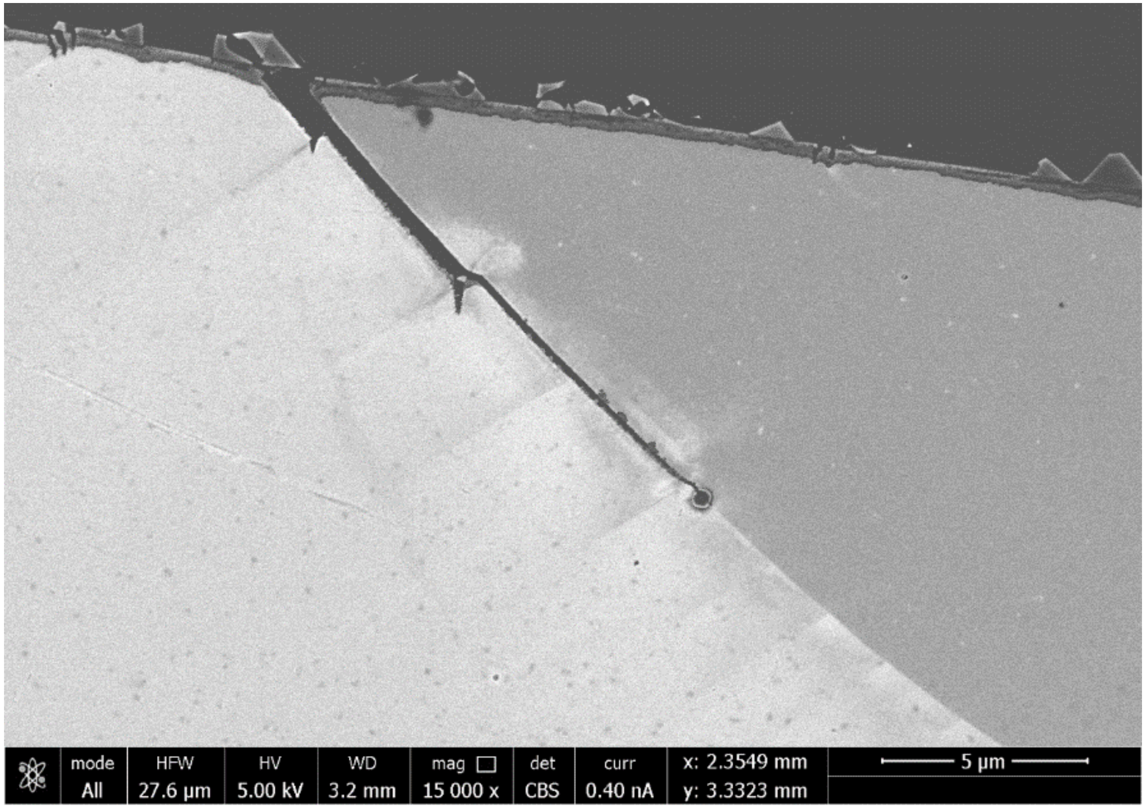




Post Test Analysis: Characterisation

Wood/UoM

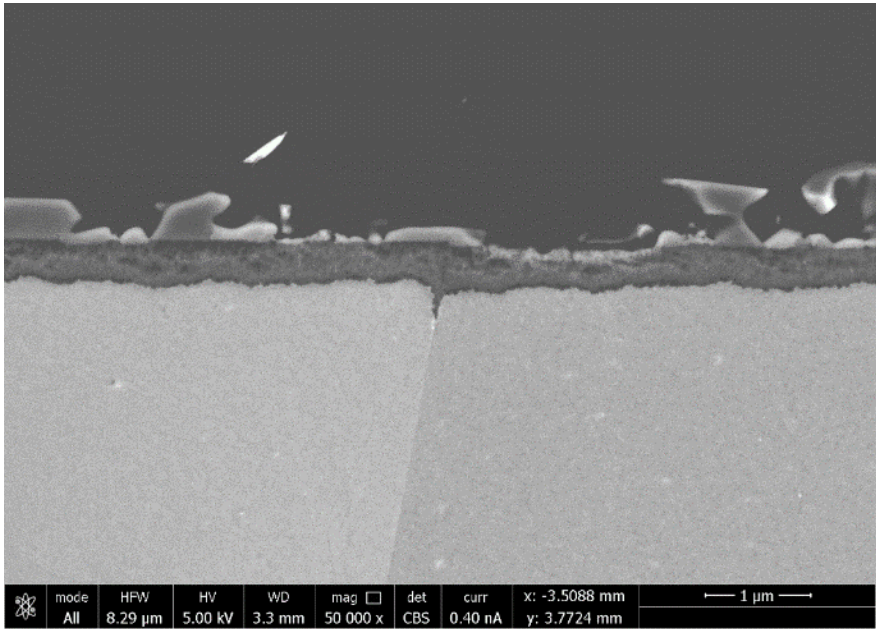
- The intersection of slip channels with a cracked grain boundary and short cracking of the slip channel



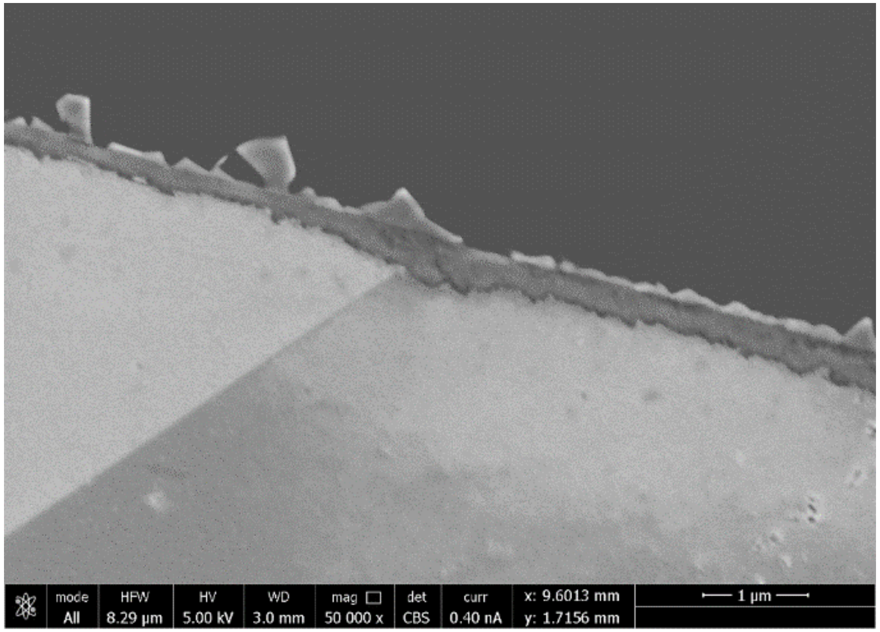
Post Test Analysis: Characterisation

Wood/UoM

- In the non-irradiated region, no grain boundaries with preferential oxidation were observed. In the proton irradiated zone, even some boundaries that had not cracked showed preferential oxidation.



Irradiated



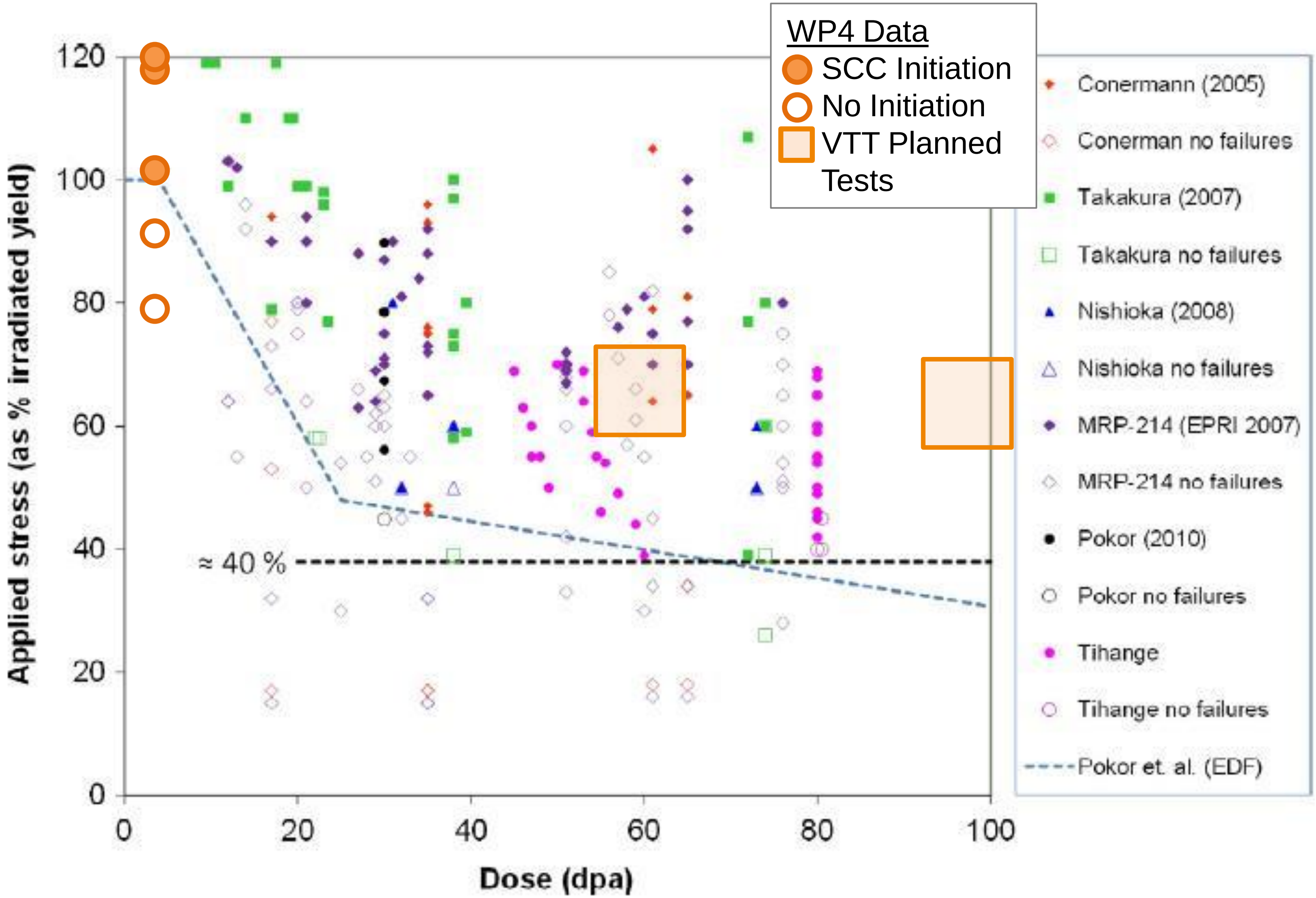
Non-Irradiated

Results

- Below is a summary of the IASCC tests completed to date with the post-test corrected stress and whether or not evidence of SCC was observed

Laboratory	Irradiated?	Stress, σ (MPa)	0.2 % Proof Stress, σ_y (MPa)	σ/σ_y	SCC?
Wood/UoM	Y	629	517	1.22	Y
	Y	609	517	1.18	Y
	Y	543	517	1.05	Y
Framatome	N	186	234	0.79	N
	N	246	234	1.05	N
	Y	416	517	0.80	N
	Y	480	517	0.93	N

Results


WP4 Data
SCC Initiation
No Initiation
VTT Planned Tests
Applied stress (as % irradiated yield)
Dose (dpa)
≈ 40 %
0
20
40
60
80
100
120
Conermann (2005)
Conerman no failures
Takakura (2007)
Takakura no failures
Nishioka (2008)
Nishioka no failures
MRP-214 (EPRI 2007)
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Tihange
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Framatome: Ongoing Work

- Performing an additional test to replicate conditions where SCC was observed by Wood/UoM
- Direct test comparison will assess the impact of differences in laboratory techniques and loading histories
  - Especially the significance of the SSR loading period used in the first 3 Wood/UoM tests
- Results aim to determine how their data can be used in conjunction with data produced by Wood/UoM

Laboratory	Irradiated?	Stress, σ (MPa)	0.2 % Proof Stress, σ_y (MPa)	σ/σ_y	SCC?
Wood/UoM	Y	629	517	1.22	Y
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	Y	543	517	1.05	Y

Wood/UoM: Ongoing Work

- Performing an additional test consisting of pre-conditioning stage only
- If no evidence of SCC is observed, the specimen will be placed back on test and loaded to 543MPa with a rapid loading regime
- Results aim to further elucidate impact of pre-conditioning and SSR loading

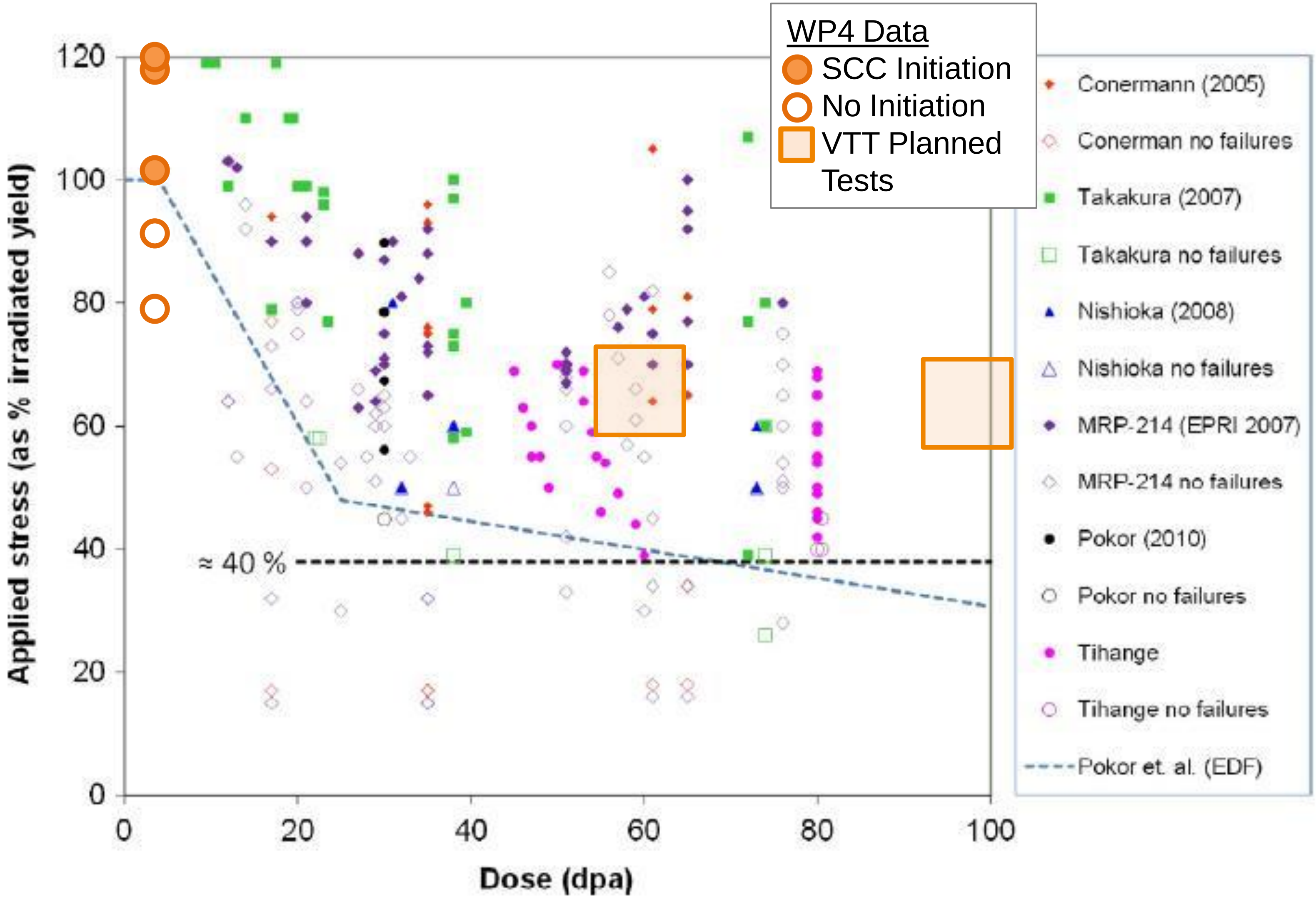
Laboratory	Irradiated?	Stress, σ (MPa)	0.2 % Proof Stress, σ_y (MPa)	σ/σ_y	SCC?
Wood/UoM	Y	629	517	1.22	Y
	Y	609	517	1.18	Y
	Y	543	517	1.05	Y

SUMMARY

Summary

- 2 tests to be carried out by VTT on neutron irradiated SS
  - One with standard PWR chemistry, one with high H_2 chemistry
  - Each test to contain 2 specimens of different dpa and load
- 4 SCC tests have been carried out by Framatome
  - Both irradiated tests were below yield and did not show evidence of SCC initiation
- 3 SCC tests have been carried out by Wood/UoM
  - All specimens were loaded above yield and have shown evidence of IASCC
- Post test characterisation shows evidence of preferential oxide growth at grain boundaries, IGSCC, slip channelling and crack bridging
- FE modelling of both Framatome and Wood/UoM test set-ups has allowed post test corrections to stress data
- Ongoing work to better understand differences in testing methodology
  - Pre-conditioning? SSR loading? Applying load at RT?

Summary



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