HELIUM EFFECTS ON IASCC SUSCEPTIBILITY

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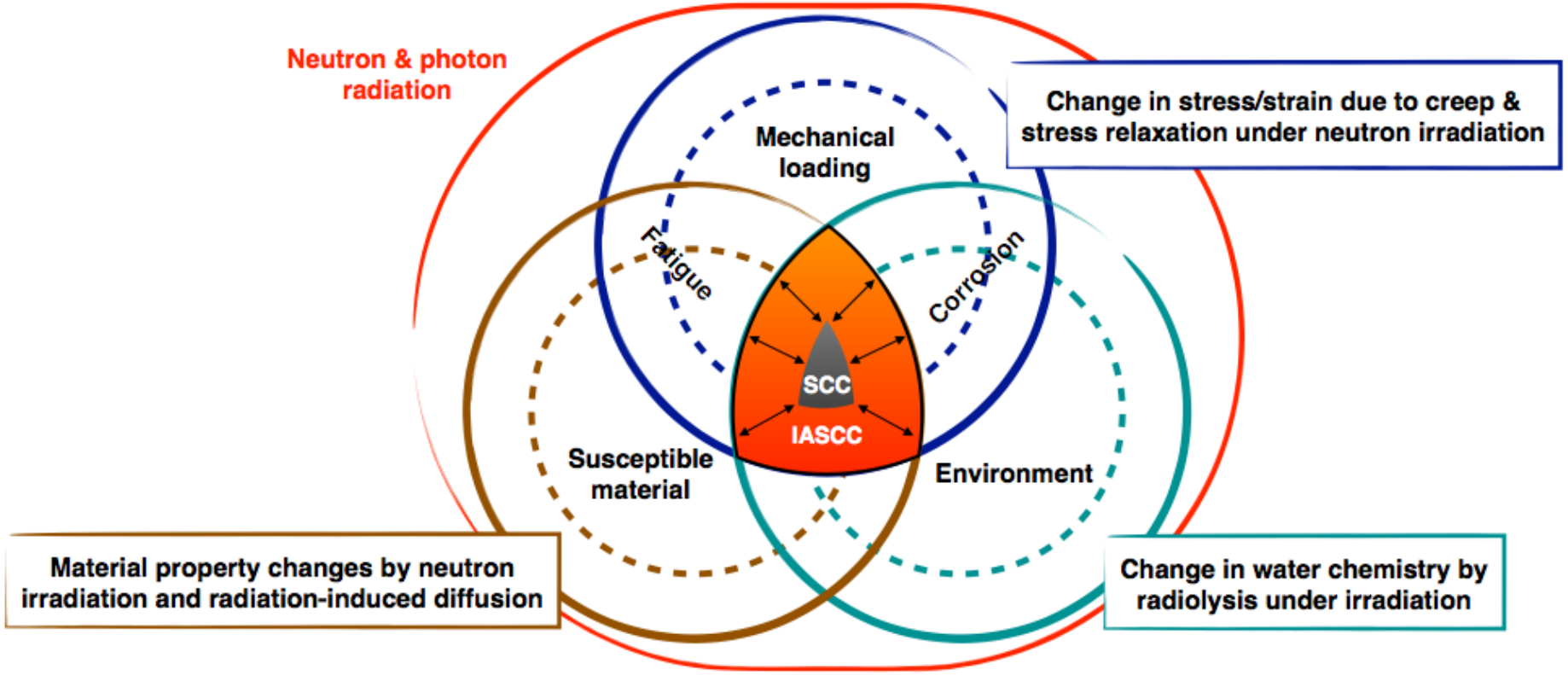
Content of the presentation

- Introduction
- Experimental
- Results & discussion
  - Bubble evolution after post implantation annealing
  - Helium effects on IASCC
- Summary, conclusions & perspectives

Introduction - What is IASCC?

By definition: IASCC is actually inter-granular SCC assisted by irradiation

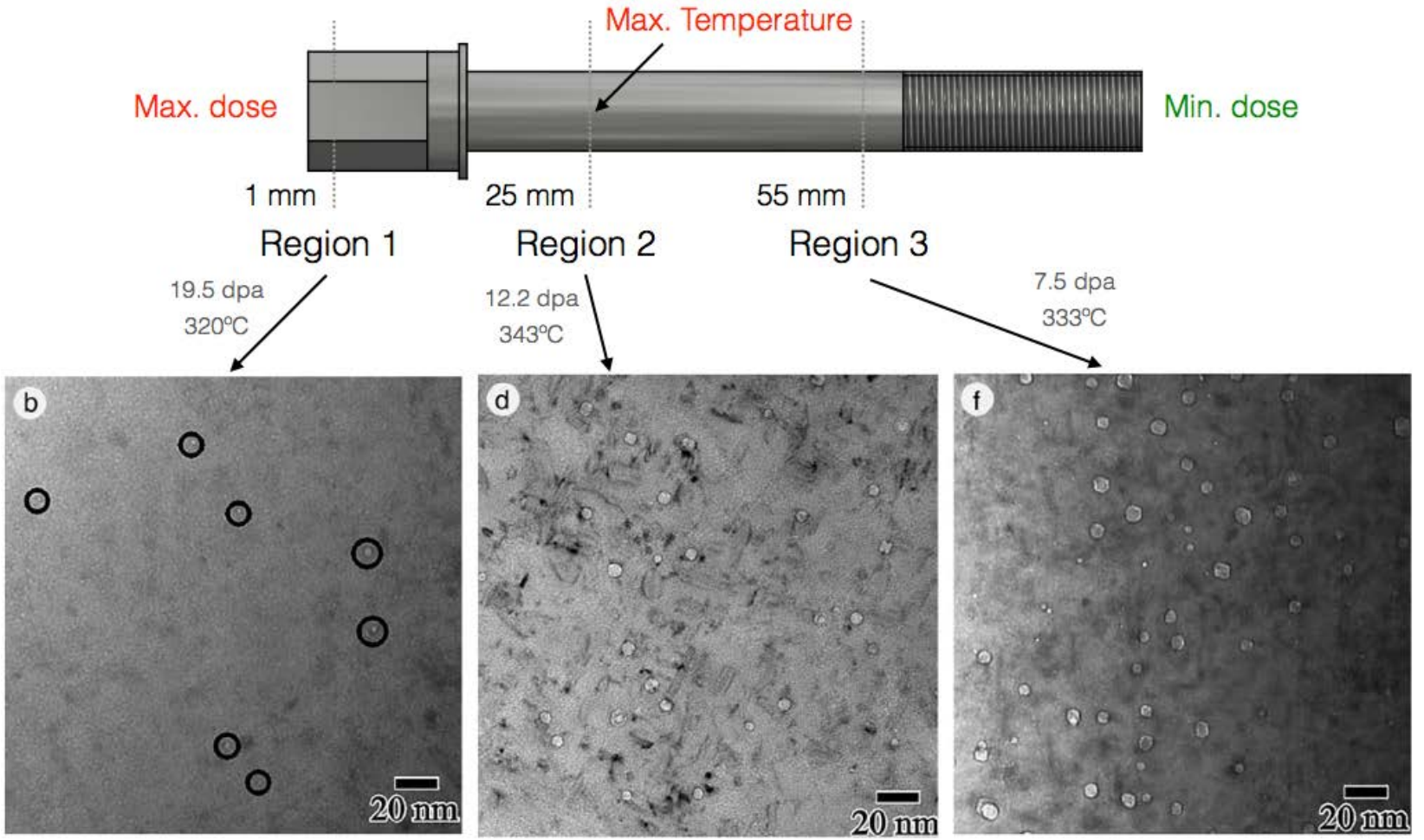


The susceptibility to SCC increase with irradiation

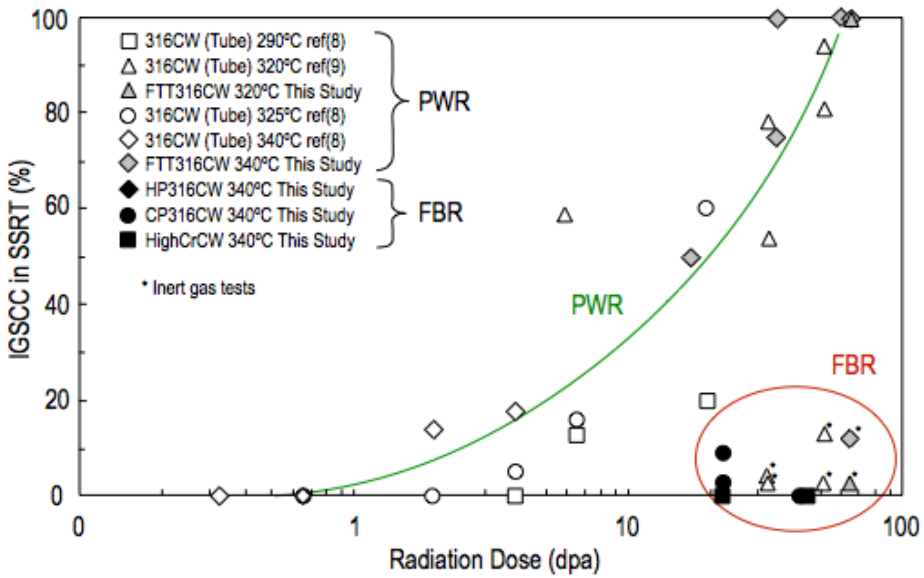
Introduction - Cavities in the BFBs Tihange 1

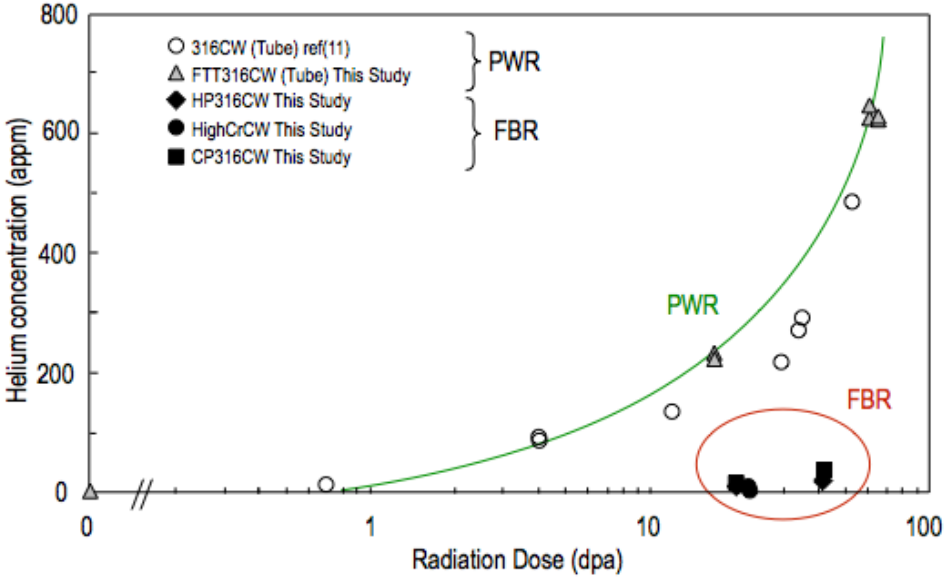
Cold worked 316SS **after service in PWR** conditions

Adapted from Edwards et al., 2003.



Introduction - Helium & IASCC





Good correlation between IGSCC susceptibility & He concentration evolution

- For given dpa, FBR-irradiated SS show much lower IASCC susceptibility than PWR-irradiated SS in spite of similar irradiation hardening and GB segregation
- He effect might be one of the main reasons for this large difference

PWR ~ 10 appm He/dpa >> FBR ~ 0.1 appm He/dpa

Introduction - Lab Investigations

- **Helium concentration for IGSCC (304 or 316)**

Artificially implanted HELIUM

SINGLE GB

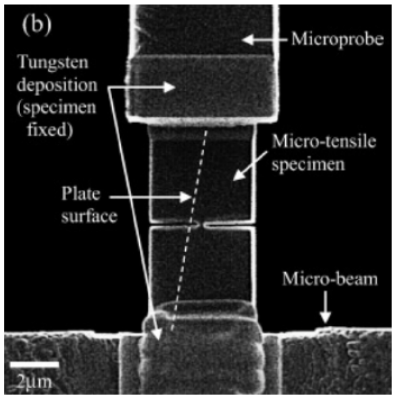
>20'000 appm He

Bubble spacing < 5 nm

GB coverage = 7 %

0.95 dpa

IG fracture



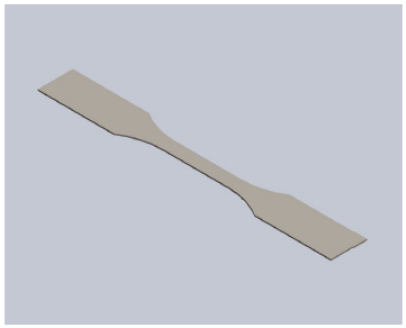
T. Miura et al., 2015.

POLYCRYSTAL*

>10'000 appm He

1 dpa

TG-C



H. Ullmaier, 2003

Irradiated in Halden reactor (BWR) 300°C

TEST IN HWC	TEST IN NWC
~few tenths appm He	~few tenths appm He
~1 dpa	>0.45-5 dpa
TG-C (<2%) + TG-D	IG (3-30%) + TG-D

Irradiated in BOR-60 (FBR) 320°C

TEST IN HWC	TEST IN NWC
~0 appm He	~0 appm He
~48 dpa	~5 dpa
TG-C (<2%) + TG-D	IG (0-50%) + TG-D

NO IG→ ↓ HE ?

Y. Chen et al., 2014

Approach

- Potential concern for some PWR internals & LTO > 50 years
- SA (baffle formers) and CW SS (baffle bolts)
- He & change in deformation mode at high dose
- Separation of He and displacement damage effects

  → He implantation (100 to 1000 appm, 0.016 to 0.16 dpa only)
- Simulation of He bubble structure in baffle bolts & variation of He bubble size and GB He bubble coverage

  → post implantation annealing study

  → critical He concentrations or GB coverage for IG (IA)SCC
- Characterization of IG (IASCC) susceptibility by SSRT tests in hydrogenated HTW

  → fracture & deformation mode by SEM & TEM

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Experimental - Material & Samples

- 316L

Cr	Ni	Mo	Mn	Si	Co	N	V	C	P	W	Al	Ti	Sn	Nb	S
17.61	12.32	2.379	1.768	0.466	0.164	0.0673	0.036	0.0275	0.024	0.023	0.018	0.007	0.006	0.003	<0.003

- SA at 1050ºC for 30' quenched in water
- SA or 20 % CW

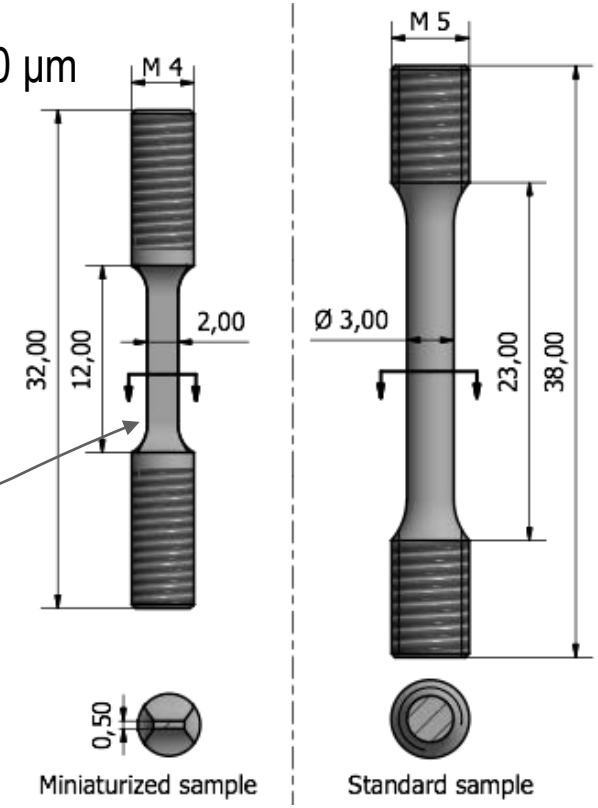
average grain size 52 µm

- Tensile specimens

45 MeV ≃ 250 µm

$$\frac{t_c}{s} = 7$$

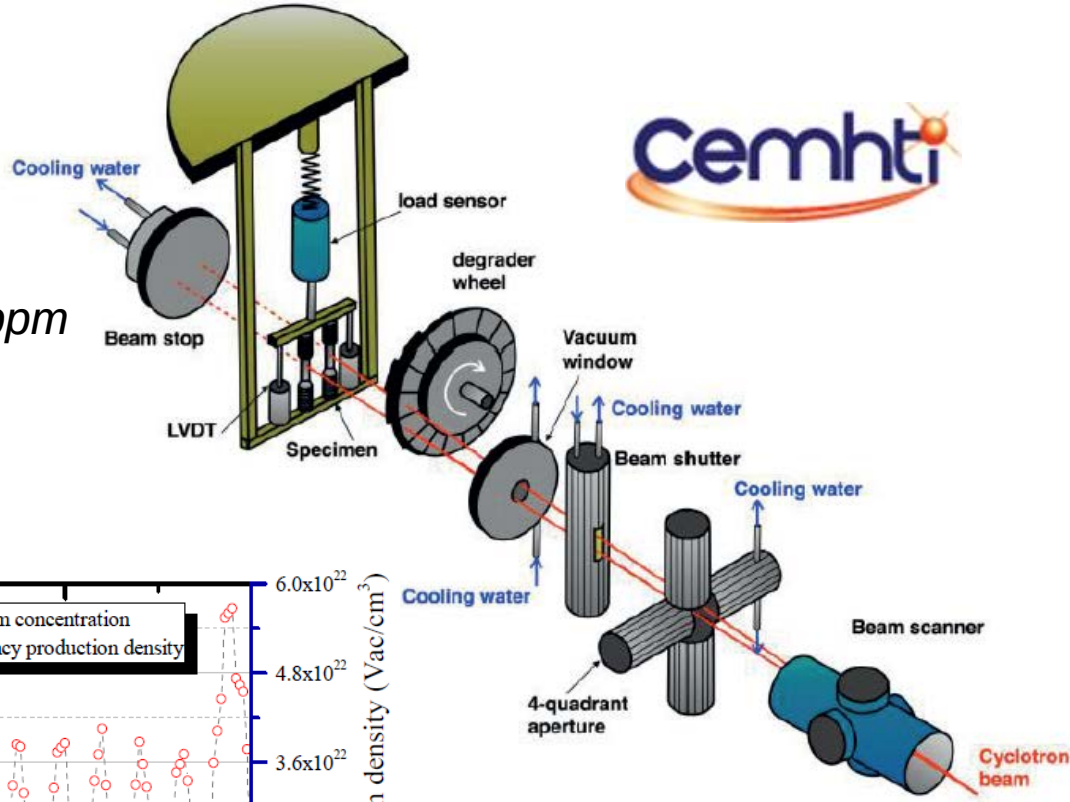
$$\frac{t}{w} \geq 0.2$$

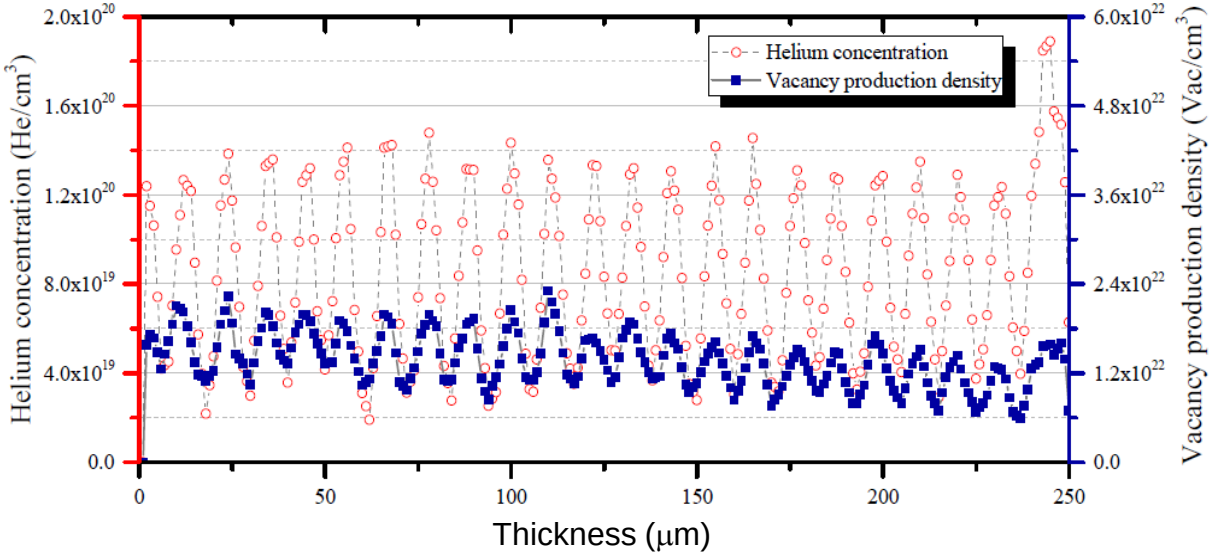


Experimental - Helium implantation

Irradiation parameters

α-energy = 45 MeV
T-Irradiation = 300 ºC
Fluence = 2.17 x 1018 He cm^{-2}
He-concentrations = 100, 300, 1000 appm
damage = 0.016, 0.05, 0.16 dpa





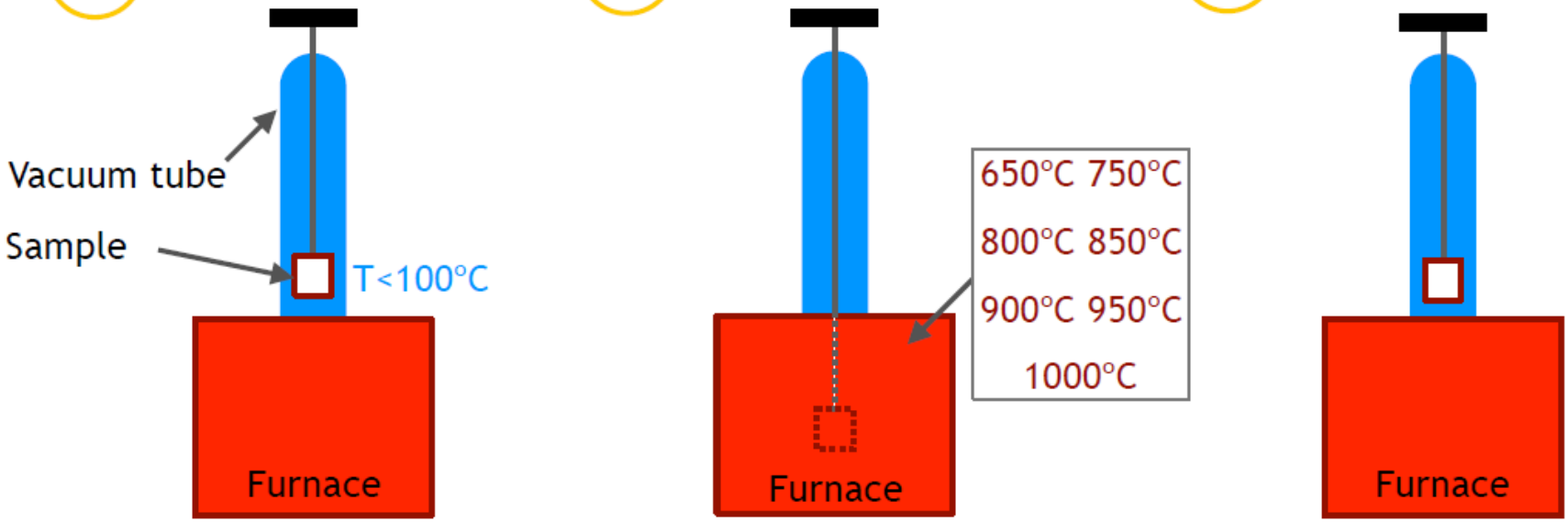
Experimental - Post implantation annealing

Objective: Reproduce BFB microstructure & increase GB coverage

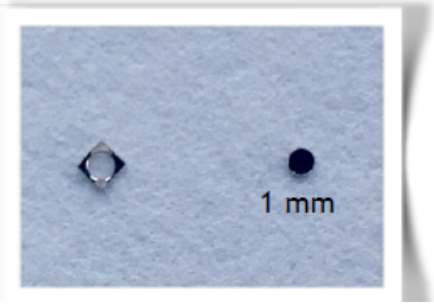
1 *1-2h furnace condit.*

2 *1h in furnace at T.*

3 *Cooling in vacuum*



Used for He implanted **plates** and miniaturised **samples**



Experimental - Mechanical testing

Tensile test machines and conditions

All tests are SSRT at 10^{-6} or 10^{-7} s^{-1}

- **Water loop.** Standard and miniaturized samples
  - High temperature water tests (HTW) at 288ºC
  - Normal water conditions (NWC) with 500ppb O_2
  - Hydrogenated water conditions (HWC) with 2.2 ppm H_2

- **Roell + Korthaus machine.** Only standard samples

  Air tests
  - Room temperature tests (RTA) at 25ºC
  - High temperature tests (HTA) at 288ºC

- **SSRT machine.** Only miniaturized samples

  Air tests
  - Room temperature tests (RTA) at 25ºC
  - High temperature tests (HTA) at 288ºC

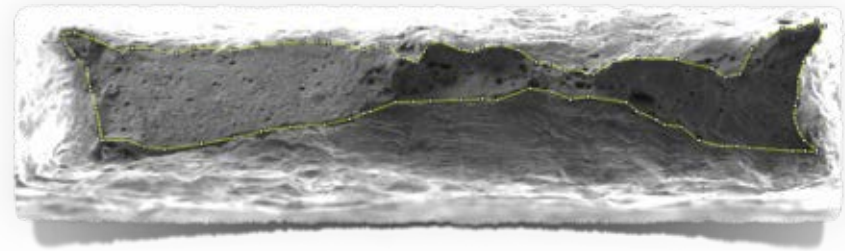
Effective gauge length is needed

Experimental - Microstructure observations

Metallurgical investigations

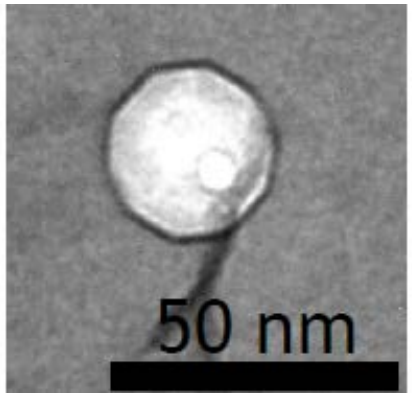
Using the “NVision 40 FIB-SEM” with 15 kV

- Characterize the fracture morphology and RA.
- crack initiation and propagation mechanisms.



Microstructure investigations by TEM

Using JEOL 2020



PIA 850°C

Δf≃500 nm

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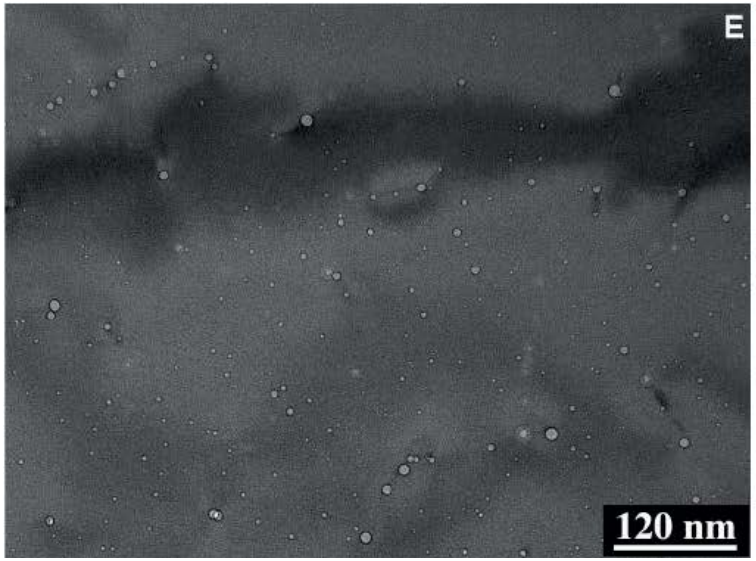
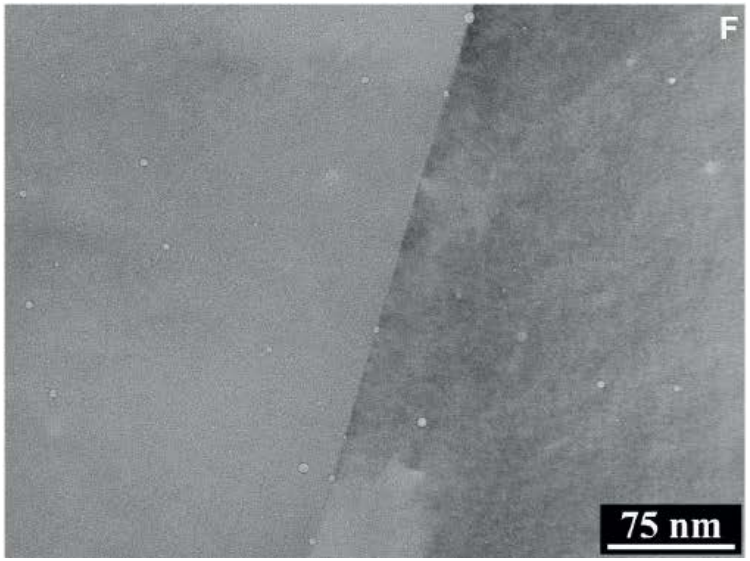
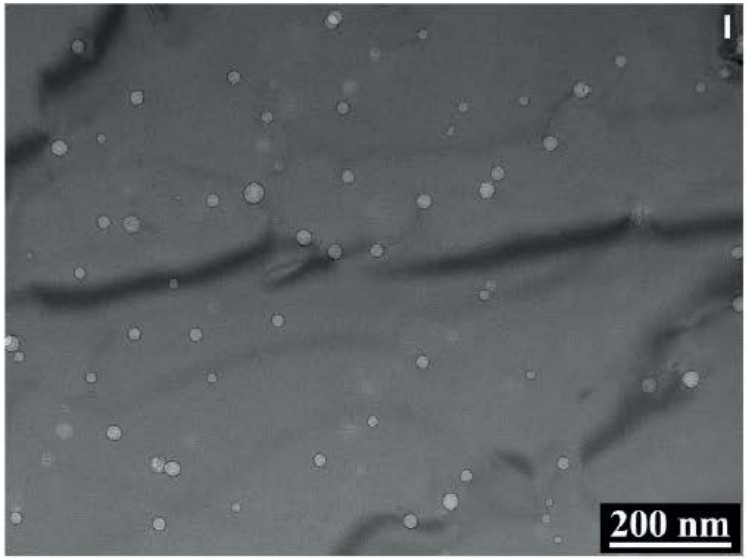
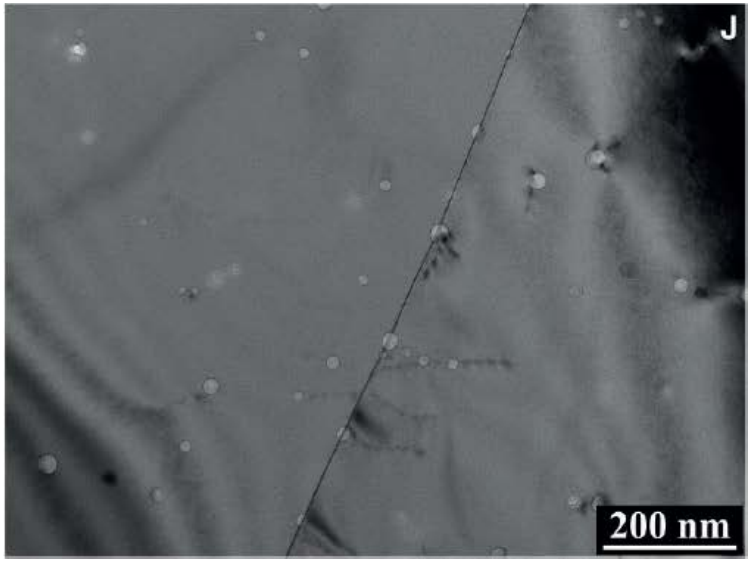
Results - Bubble distr. PIA (1000 appm)





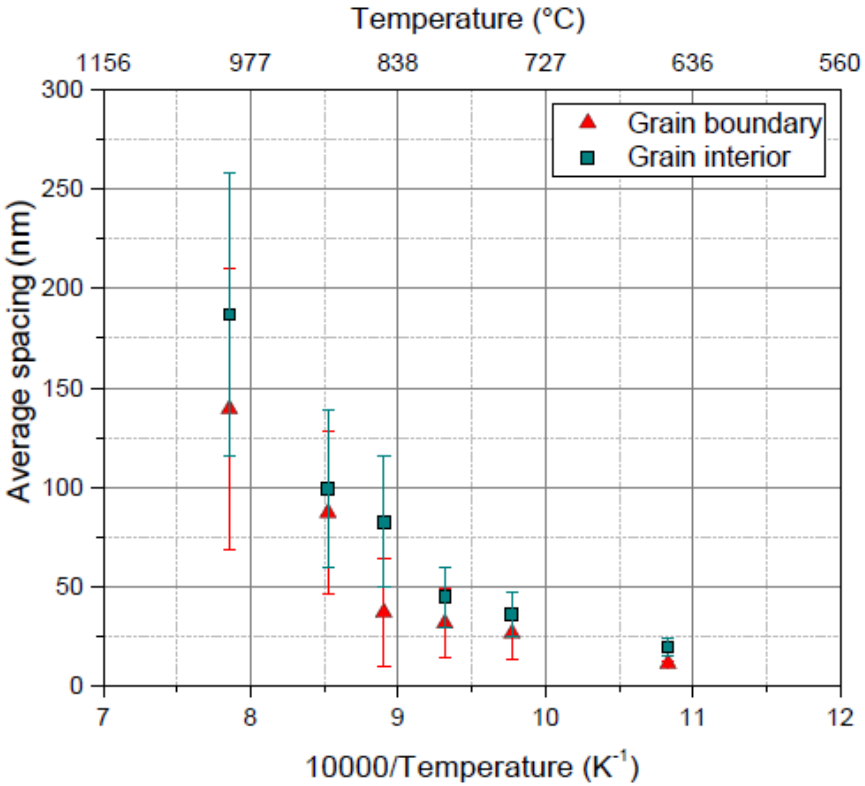
at 750°C

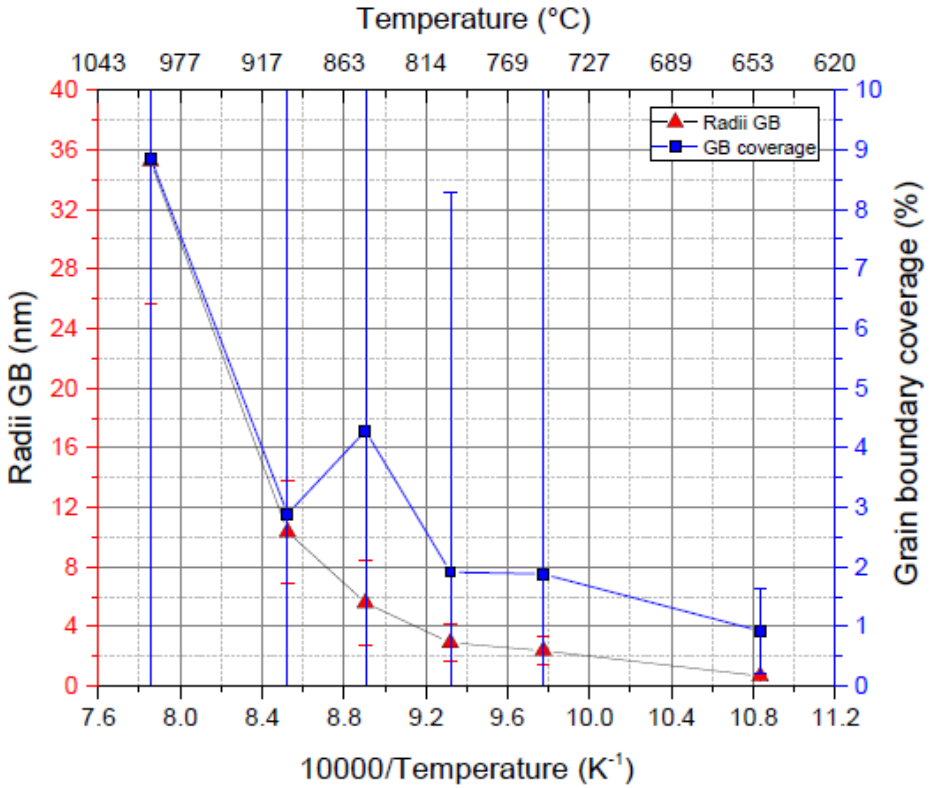




at 900°C

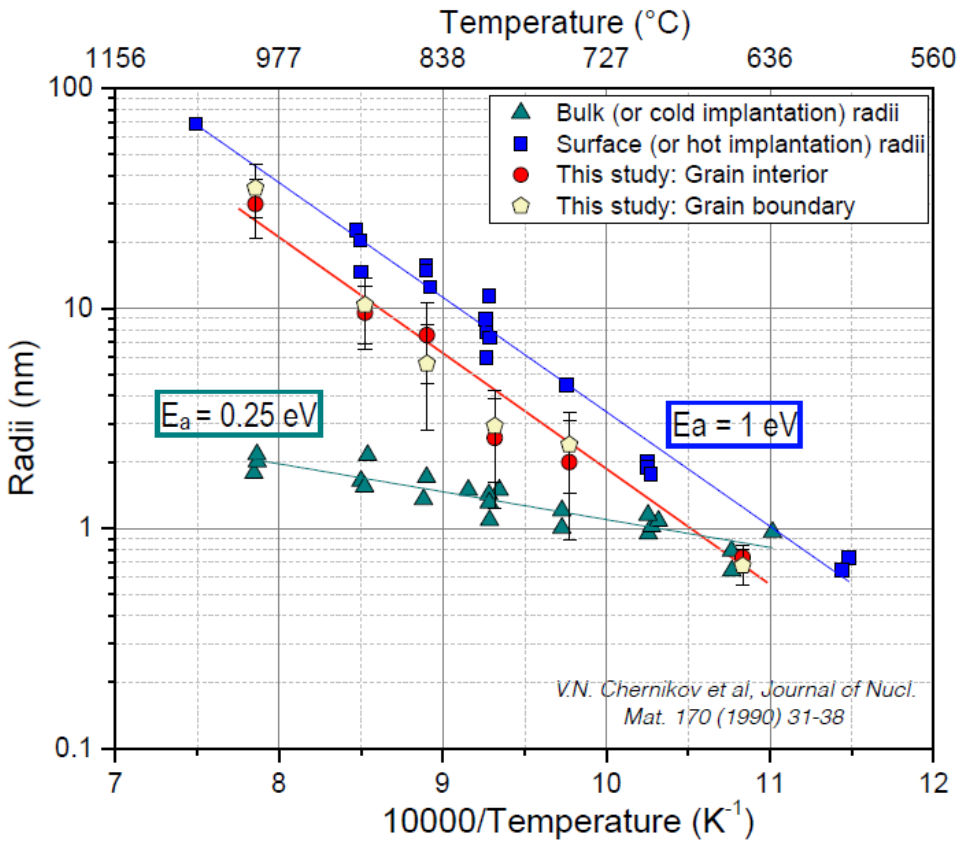
Results – Bubble average spacing and GB coverage





Limited of GB coverage, hence limited GB weakening

Results - Helium bubble evolution PIA



The thermal activation analysis shows that the He bubbles grow according to the **dissociative mechanism (Ostwald Ripening)** both, for GB and grain interior**.** This mechanism occurs **at least 300°C below** the one reported in RT implantation.

	Cold implant.	Hot implant.	Grain interior	Grain boundary
E_a (eV)	0.25	1.03	1.07	1.11
Q (eV, = E_an)	1.26 - 1.51	3.07 - 4.15	3.21 - 4.28	3.33- 4.44
Mechanism	Surf. diffusion	Dissociation	Dissociation	Dissociation

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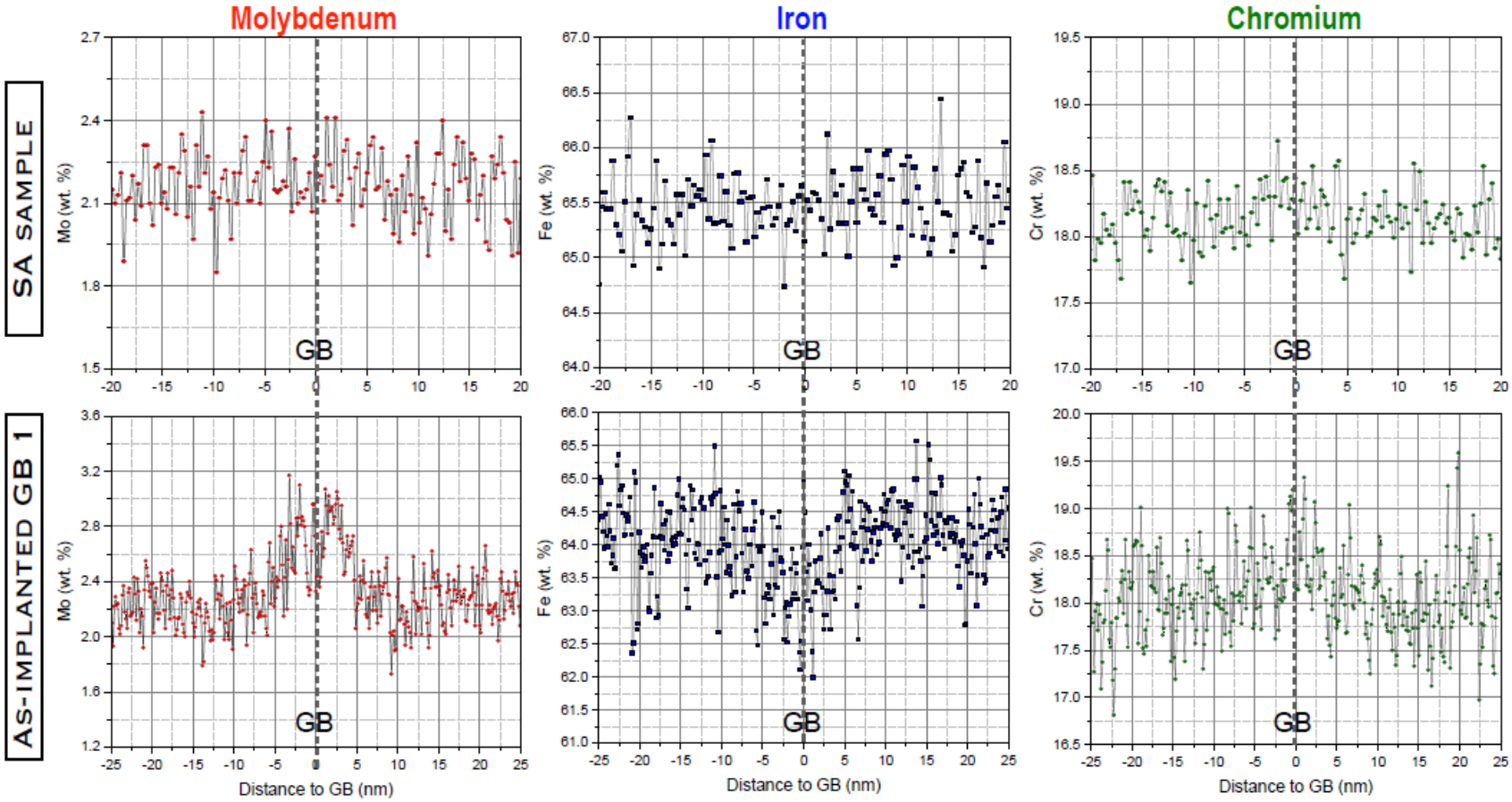
Results - RIS on Grain Boundary

- **GB RIS with FEI TALOS F200X (200 kV)**



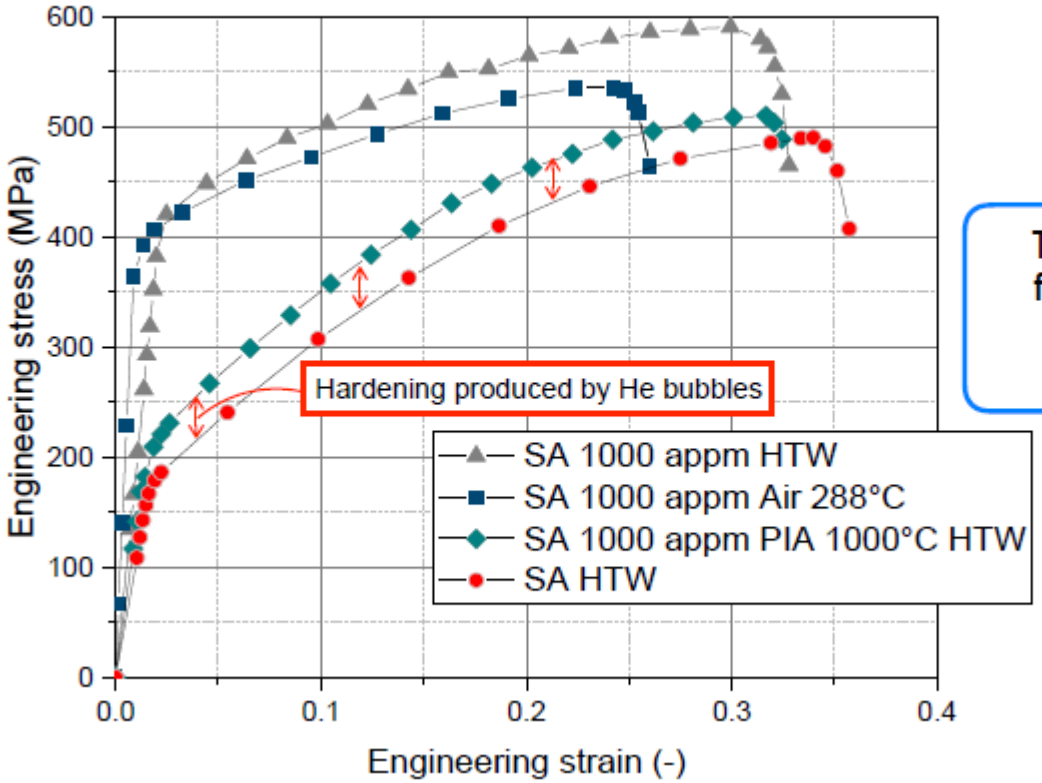
Enrichment
2.1⟶3 % wt.
Usually W shape

Depletion
64.5⟶63.5 % wt.
Usually V shape

No measurable effect

Results - SSRT to SA + 1000 appm samples

- 1000 appm with/out HT

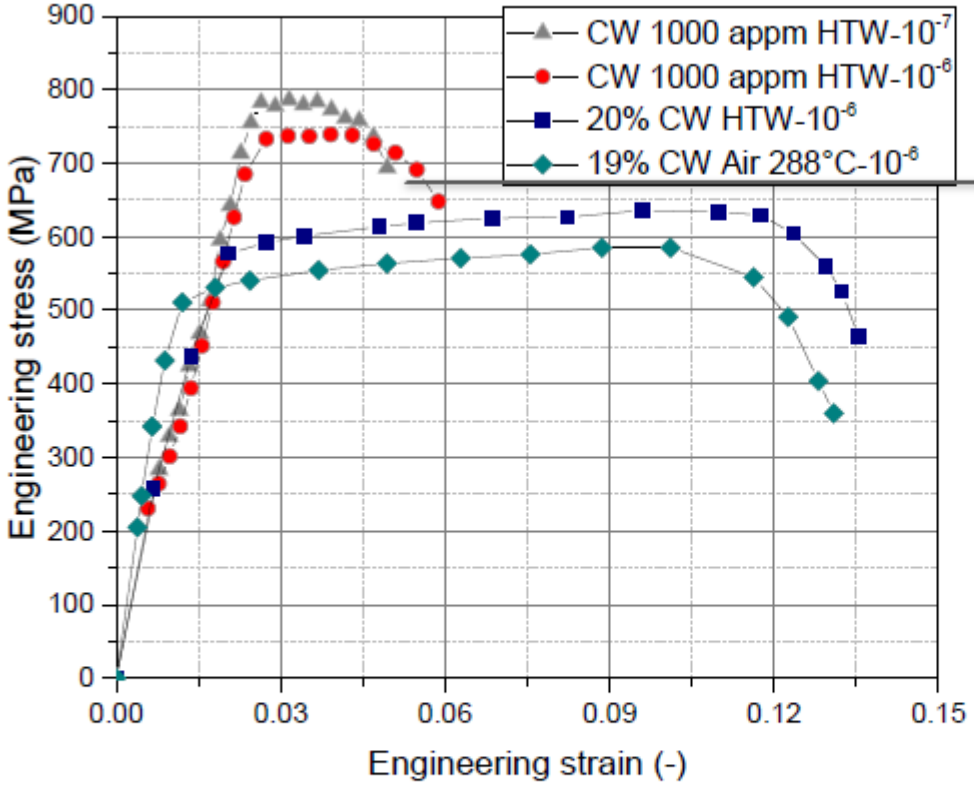


Transgranular dimple fracture is dominant, some transgranular cleavage in HTW

	SA HTW	1000appm Air 288°C	1000appm HTW	1000appm PIA 1000°C HTW
YS	178	383	418	198
UTS	492	547	594	513
ε_u (plastic)	0.28	0.23	0.26	0.27

Results - SSRT to CW + 1000 appm samples

- CW with/out 1000 appm



CW samples show tensile response similar to IASCC curves

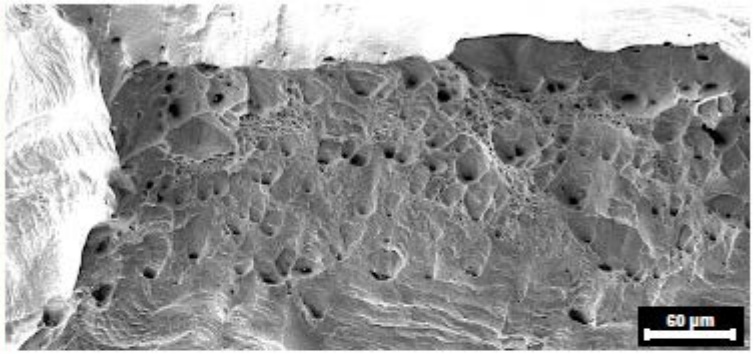
Transgranular dimple fracture is dominant, some transgranular cleavage in HTW

	CW HTA	CW HTW	CW-1000appm	CW-1000appm 10^{-7}
YS	579	576	734	787
UTS	631	641	740	790
ε_u (plastic)	0.08	0.08	0.013	0.001

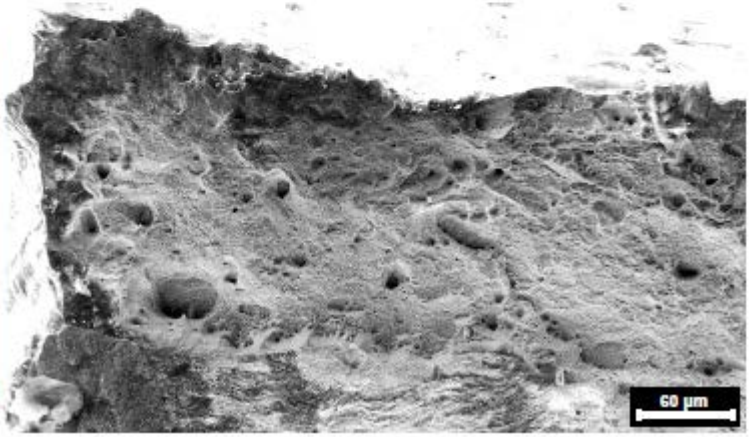
Results - Fracture investigations

- Fracture surface: 1000 appm with/out HT

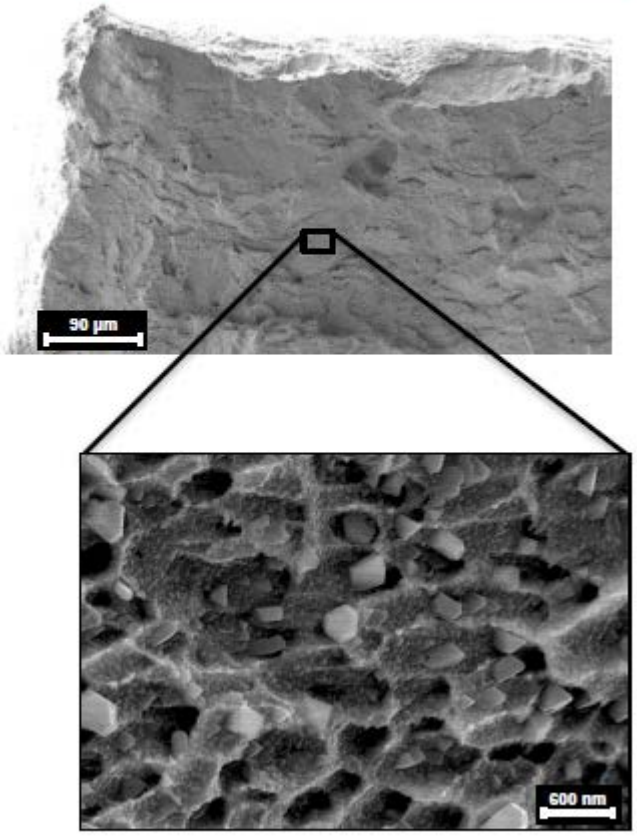
SA-1000 appm (Air-288°C)



SA-1000 appm (HWC)

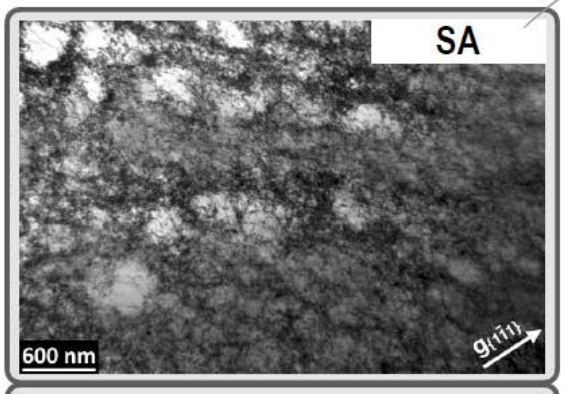


SA-1000 appm + PIA 1000°C (HWC)



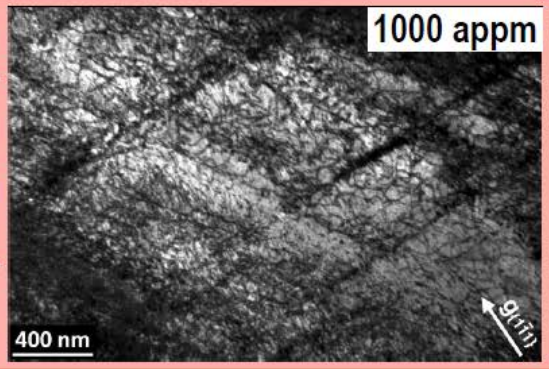
Results - Microstructure investigation

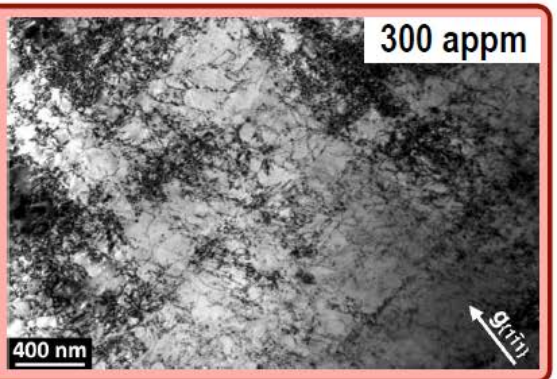
- **Deformation microstructure (all HTW)**



3D deformation cells typical for high Ni contents.

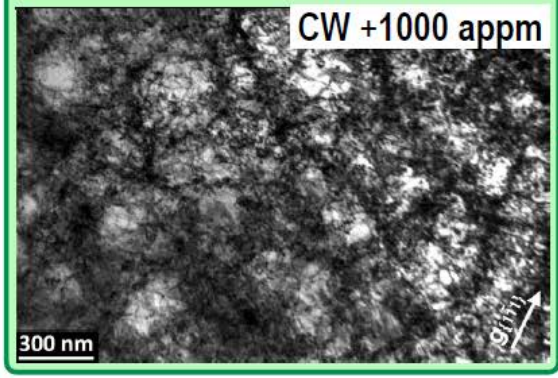
As-implanted samples





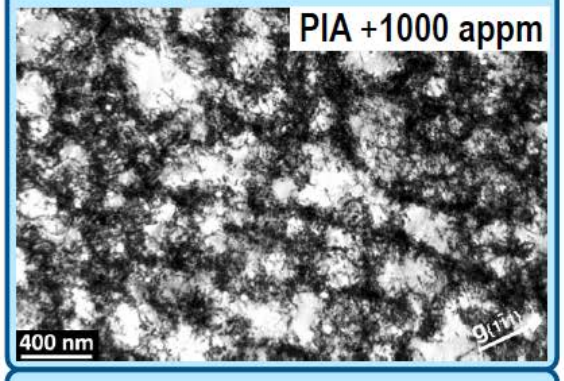
No apparent 3D deformation cells with random arrangement of dislocations. Microstructure that might be a precursor for SCC.

As-implanted CW



Prior 3D deformation microstructure still present. No evident increase of more planar deformation

Post-implantation annealed



Recover 3D deformation microstructure

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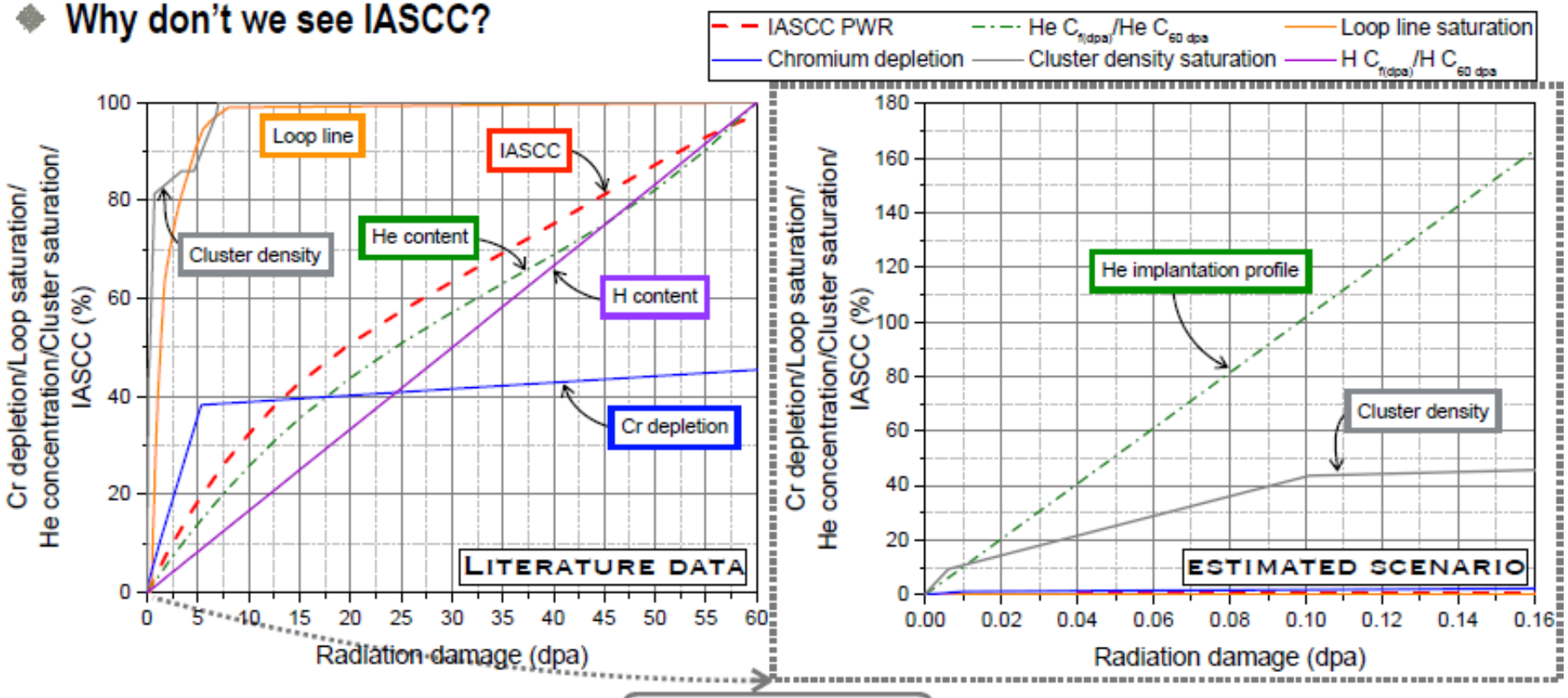
Discussion

- **Why don't we see IASCC?**



Main differences

- Chromium
- Black dots
- # of loops
- Hydrogen
- Hardening

Summary, conclusions & perspectives I

- Similar bubble size & distance in grain interior and on GB. PIA increases bubble size, but does only moderately increase GB He bubble coverage.
- The activation energy of bubble evolution for GB and Matrix shows that in both cases the bubble grows with dissociative mechanism (OR). This mechanism occurs 300ºC below the one reported in RT implantation. The coarsening mechanism might depend on both annealing T and bubble size.
- Homogenised He implantation in SA and CW at 300ºC up to 1000 appm has little impact on the macroscopic tensile properties and fracture morphology in air and HTW and results in very limited RIS only (only Mo).
- The deformation microstructure clearly changes from dislocation cells to random distribution of dislocations in SA & He implanted samples, respectively. The formation of deformation bands is enhanced in as-implanted condition.
- Accelerated SSRT (10-6 - 10-7 s-1) in HTW with 2.2 ppm DH at 290 °C did not induce IG(IA)SCC initiation in smooth tensile specimens with homogenized helium implantation at 300°C up to 1000 appm (<0.16 dpa) in SA, CW and PIA (≤ 1000°C) conditions.

Summary, conclusions & perspectives II

- However, the mechanically dominated short-term SSRT may be too short to exclude SCC initiation and could overlook other more time-consuming (e.g. corrosion-dominated) precursor and initiation processes.
- The absence of IASCC might be related to the lack of a well-marked deformation mode change between the non-implanted and the implanted materials. The number of dislocation channels in the implanted samples and the He enrichment on the GB & GB helium bubble coverage (~10%) are insufficient to induce IASCC in these conditions.
- These results suggest that a helium concentration ≤ 1000 appm alone cannot induce IASCC, therefore there has to be some synergy between dpa damage and helium concentration.
- The formation of irradiation-induced dislocation channels (at high dose) with high-stress concentration on grain boundaries, together with the current helium bubbles grain boundary coverage (~10%), could promote intergranular cracking.
- Further evaluations should thus include samples with high displacement damage (besides of high helium concentration) and crack growth experiments with pre-cracked specimens.

Acknowledgments

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