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Effects of Helium on IASCC Susceptibility

SOTERIA FINAL WORKSHOP

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Contributors

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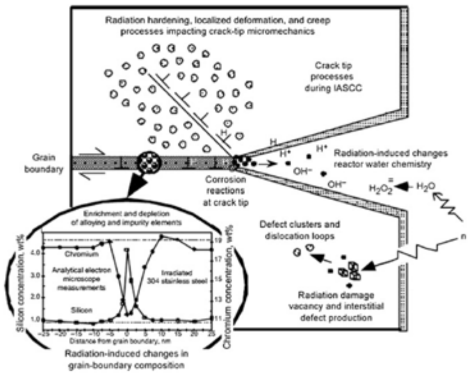
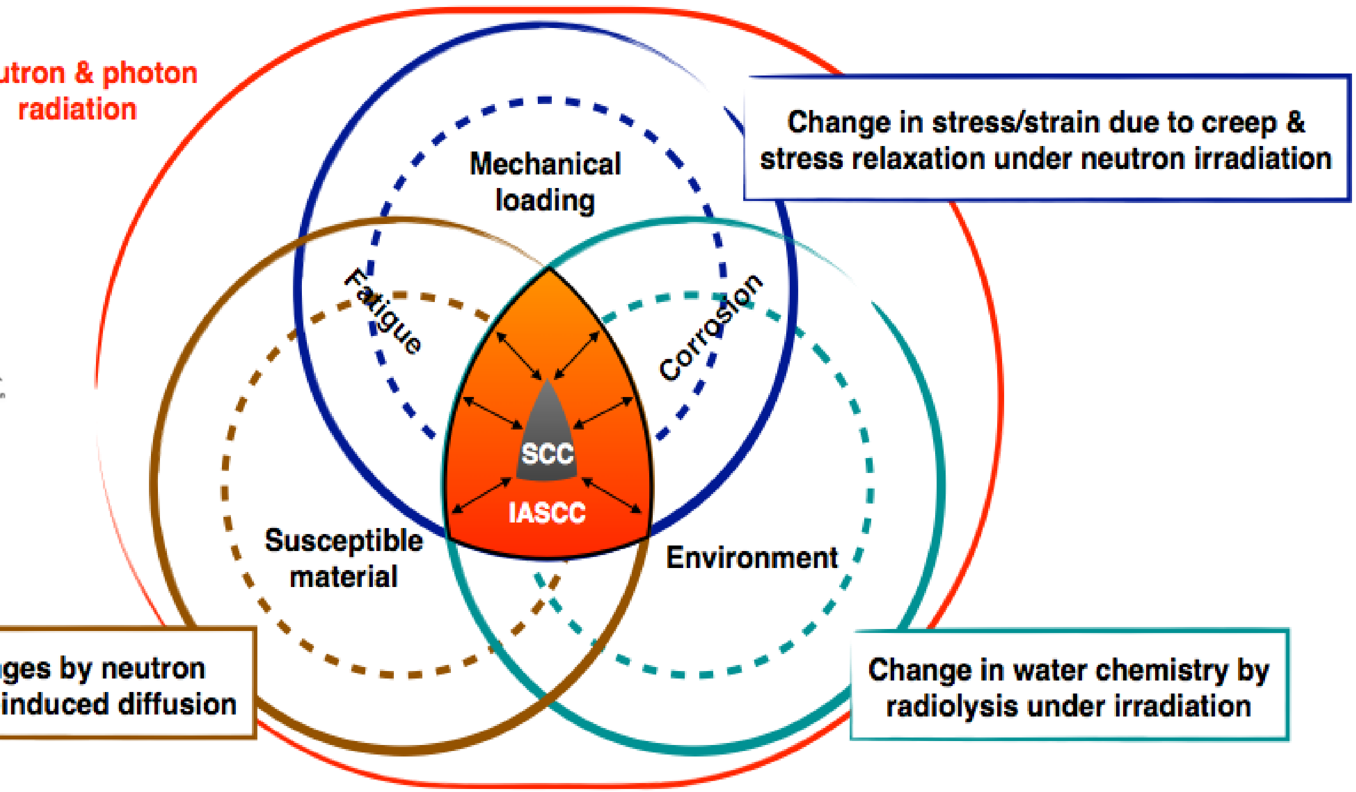
What is IASCC?

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

http://dx.doi.org/10.1016/S0022-3115(99)00075-6

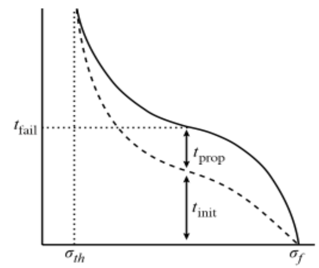




How is SCC measured?

Constant load

To smooth samples

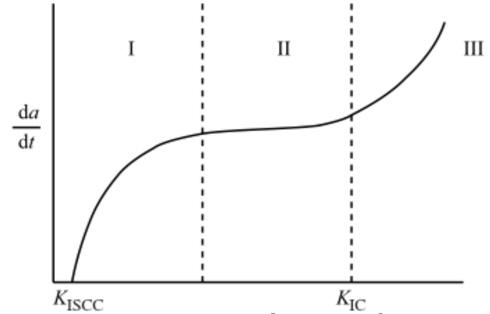


Measure the time to failure as a function of the applied stress.

To pre-cracked samples

Measure the crack length as a function of time. CGR in function of K.

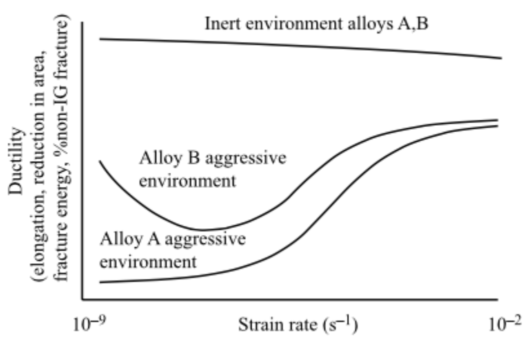
CGR
(Crack growth rate)



K   Stress intensity

SSRT

Smooth or pre-cracked



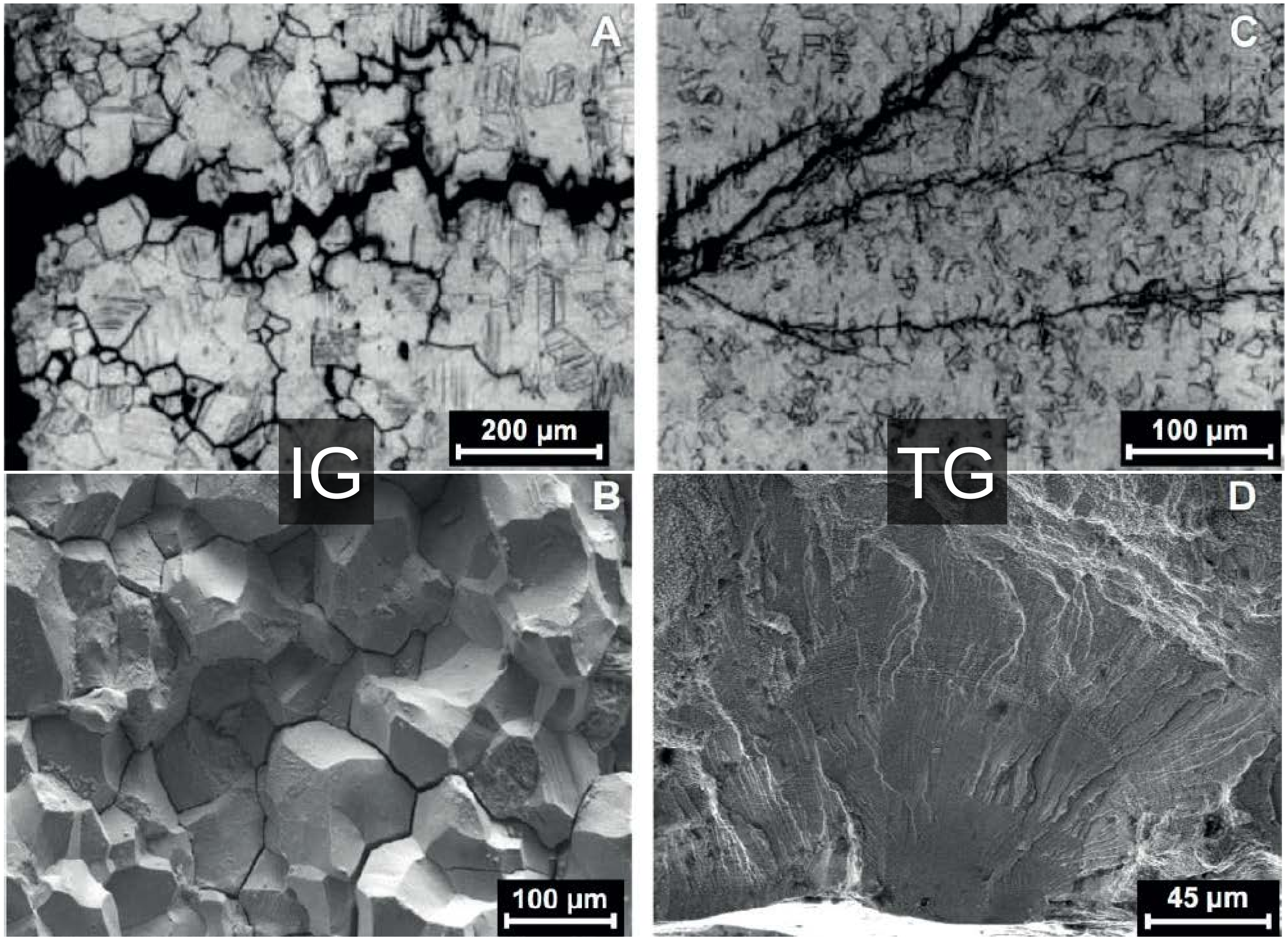
Measure of elongation, reduction of area (ductility) and TG/IG fracture.

SCC morphology (316 AuSS)

Alkaline env. @ 80 °C | Chloride cont. env. @ 100 °C

optical
D. Landolt
(2007)

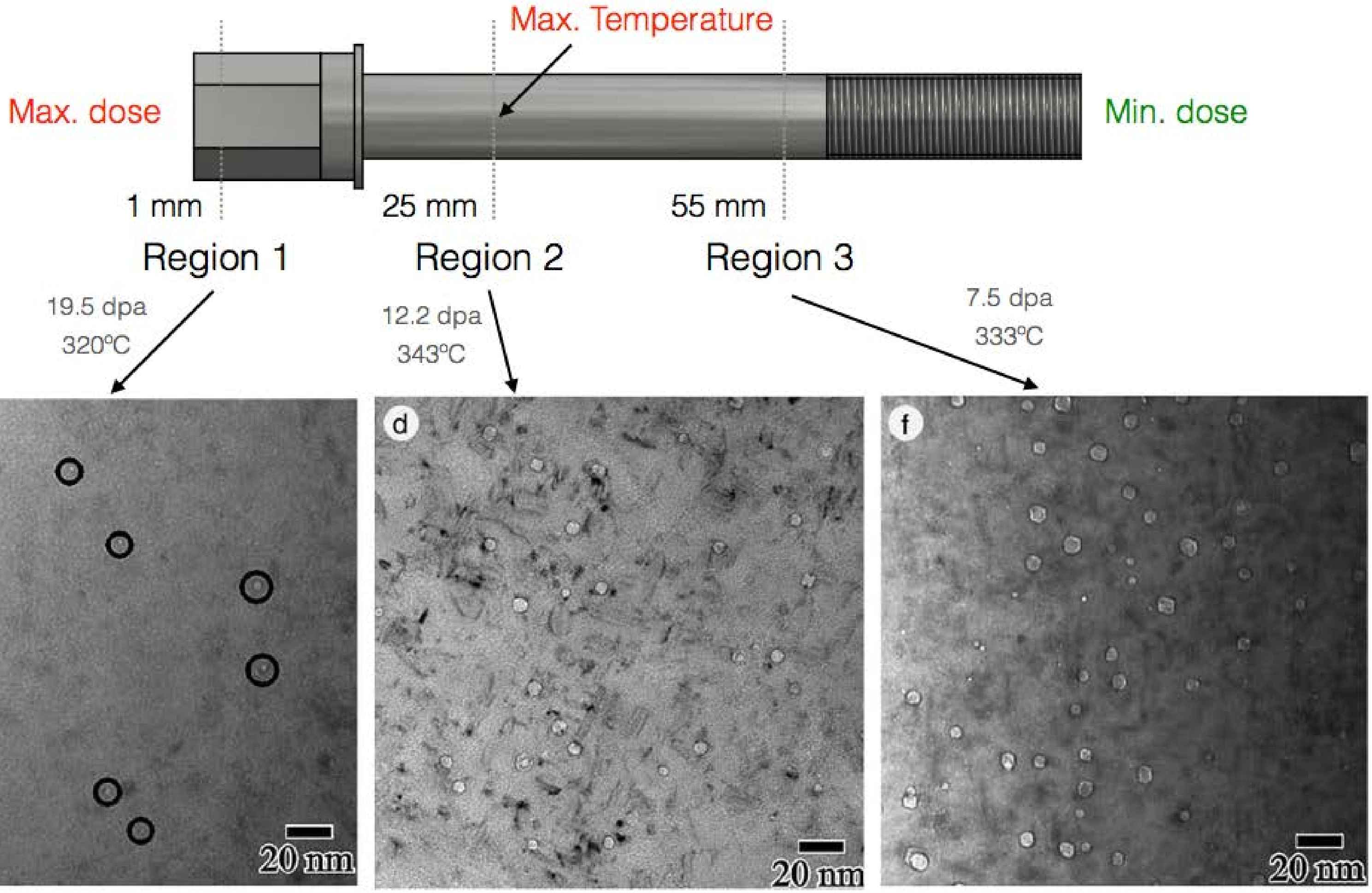
SEM
D. Du et.al
JNM456(2015)228



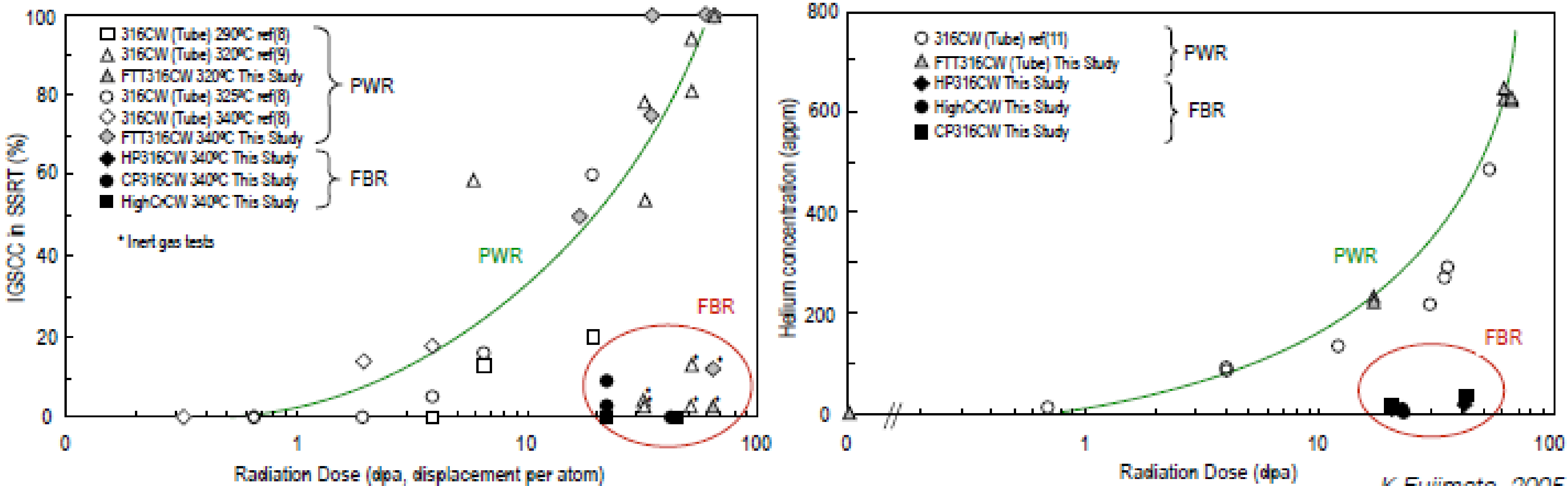
Cavities in the BFBs Tihange 1

Baffle former bolts (BFB):

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



Helium & IASCC



K.Fujimoto. 2005.

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

Selected studies on helium effects

Artificially implanted HELIUM

Tested in vacuum

Single GB

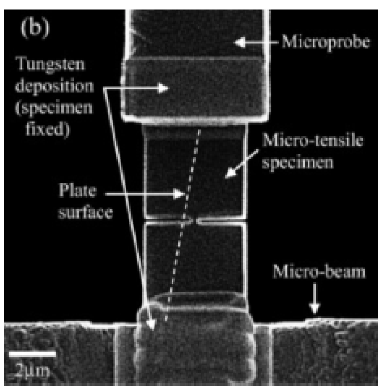
>20'000 appm He

Bubble spacing < 5 nm

GB coverage = 7 %

0.95 dpa

IG fracture



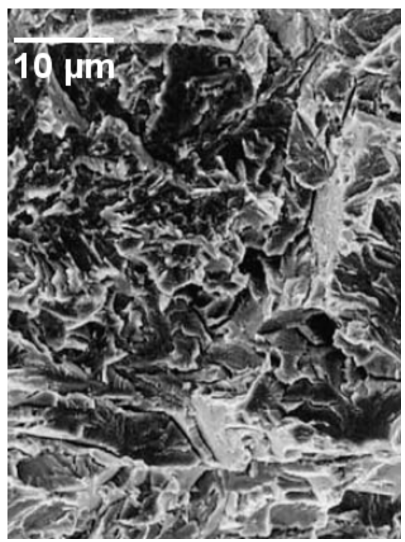
T. Miura et al., 2015.

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 a**
- **SA (baffle formers) and CW SS (baffle bolts)**
- **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 with smooth sample in hydrogenated HTW**
  - **→ fracture & deformation mode by SEM & TEM**

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Methodology – Materials & sample

316 L

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

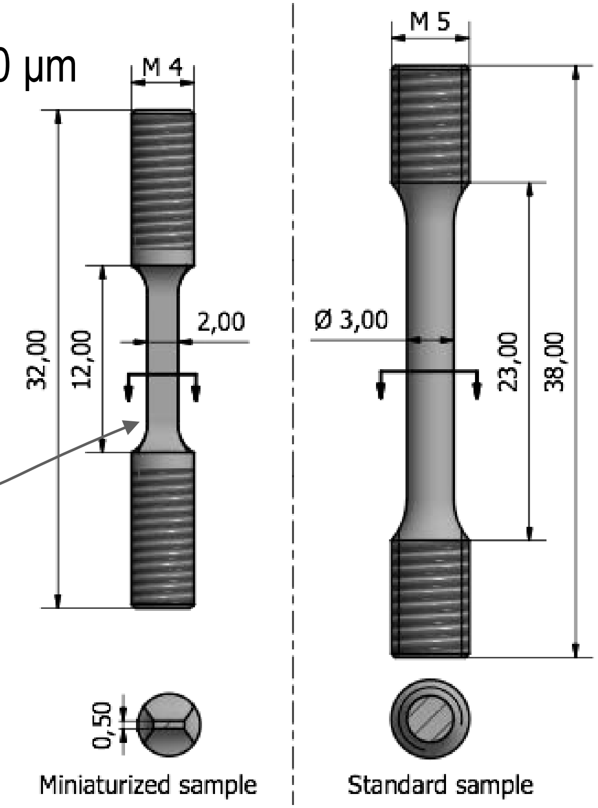
average grain size 52 µm

- Tensile specimens (ASTM standard)

45 MeV ≃ 250 µm

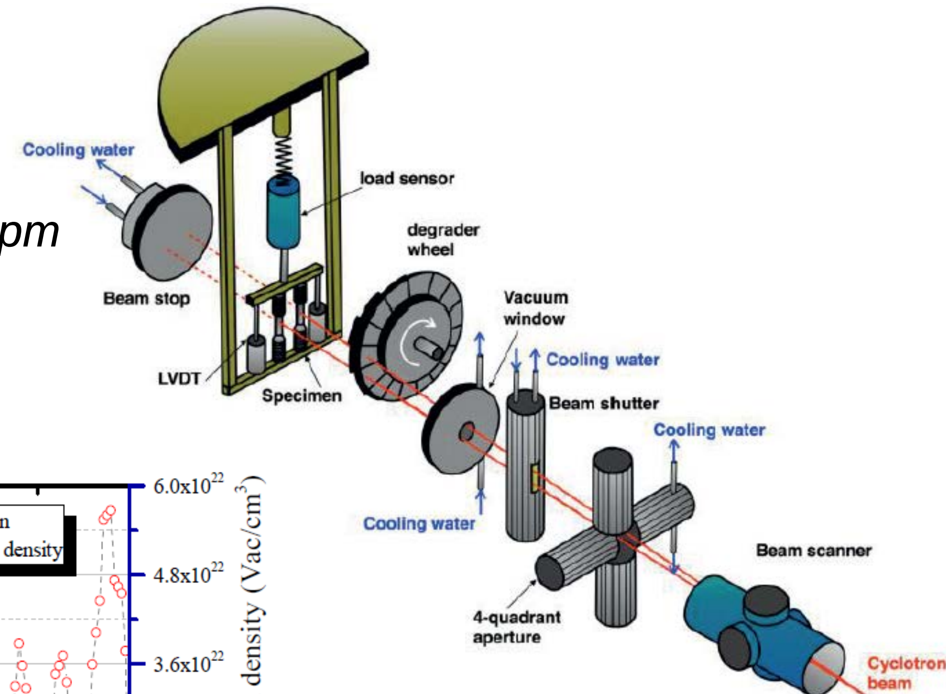
$$\frac{t_c}{s} = 7$$

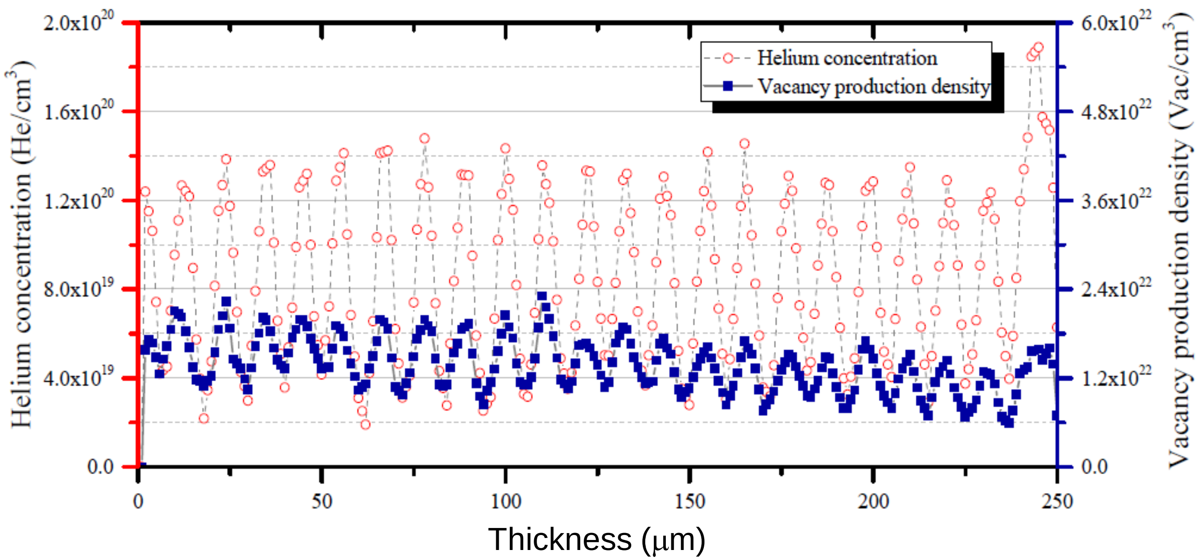
$$\frac{t}{w} \geq 0.2$$



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

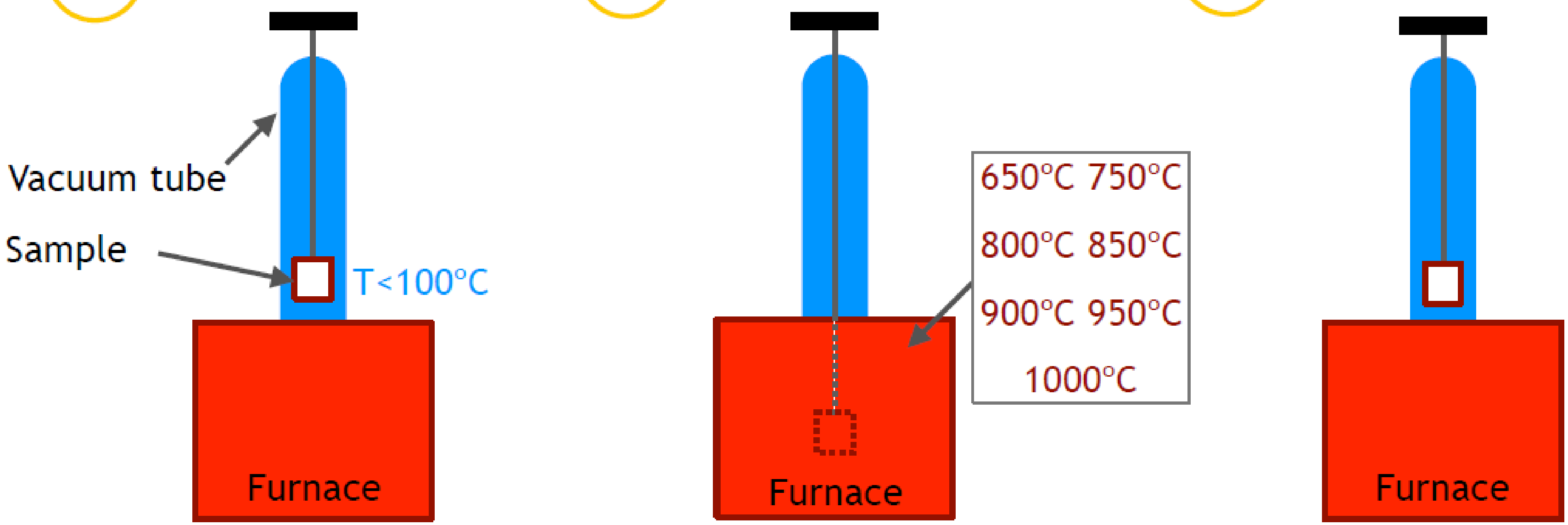




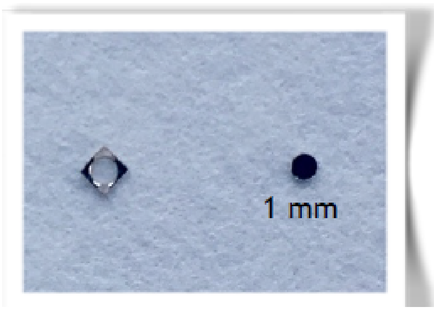
Methodology - Post implantation annealing

Objective: Reproduce BFB microstructure & increase GB coverage

1 *1-2h furnace condit.* 2 *1h in furnace at T.* 3 *Cooling in vacuum*

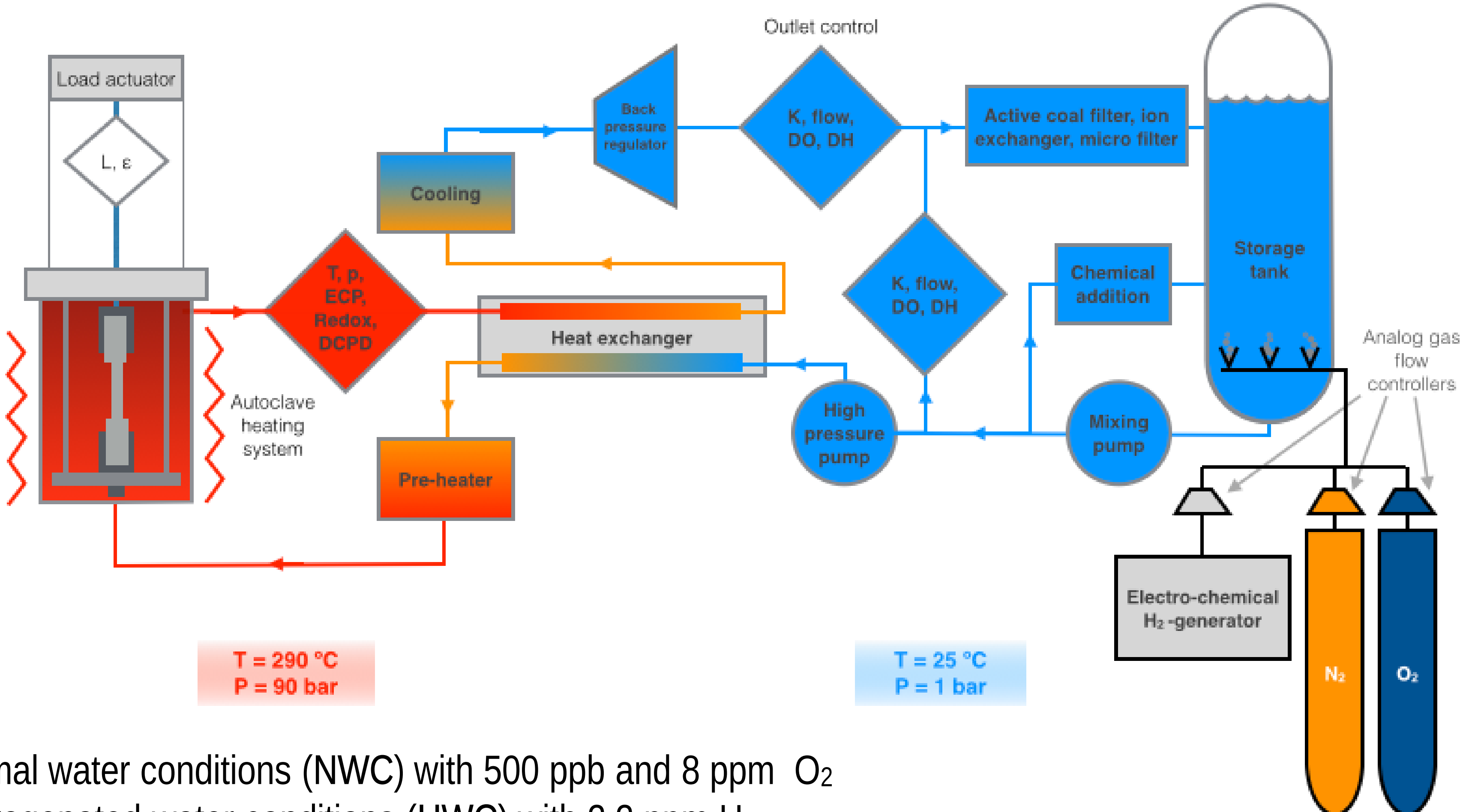


Used for He implanted **plates** and miniaturised **samples**



Methodology - Mechanical testing

Water loop Diagram

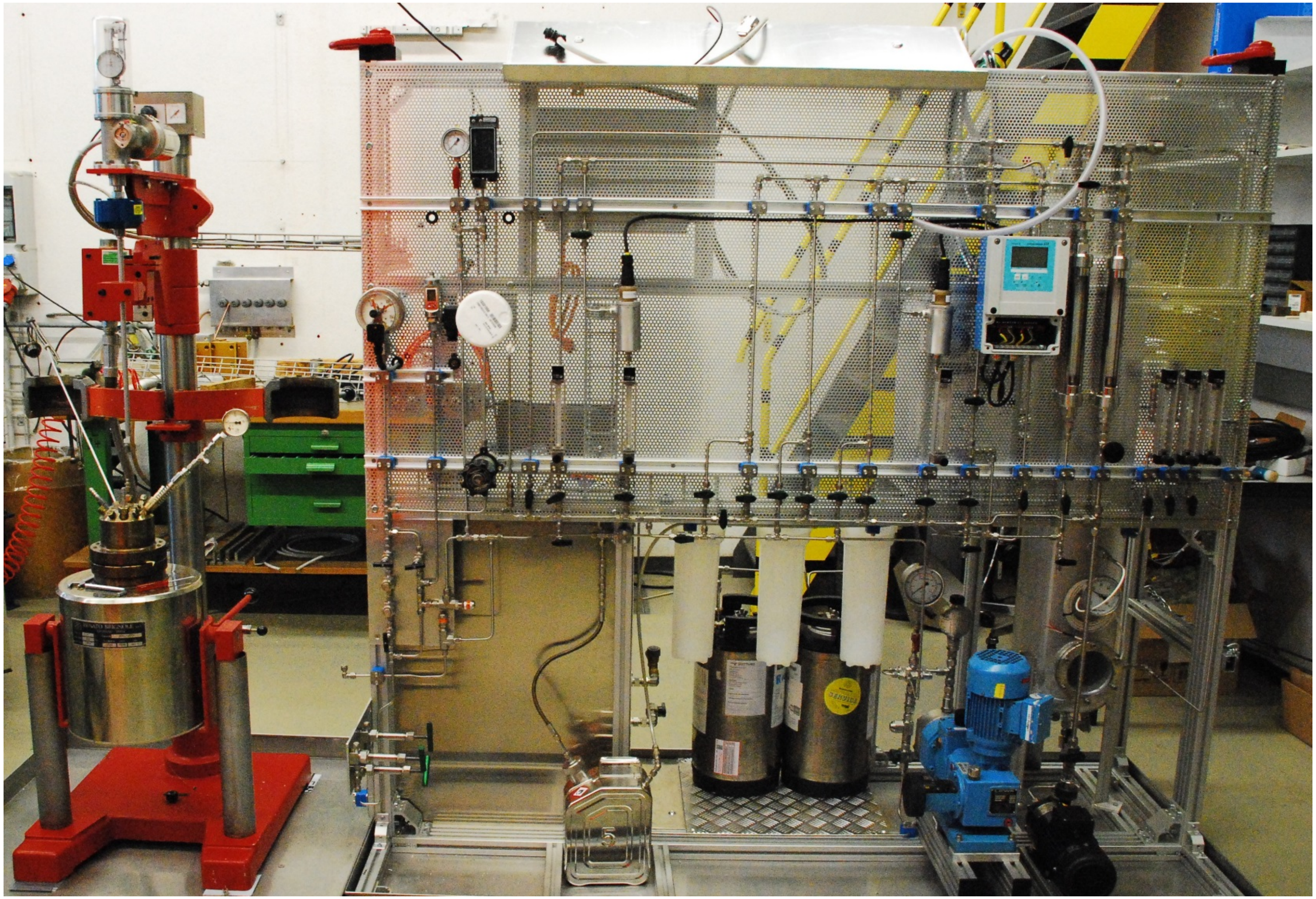


Normal water conditions (NWC) with 500 ppb and 8 ppm O_2

Hydrogenated water conditions (HWC) with 2.2 ppm H_2

Methodology - Mechanical testing

Water loop device located at Hot lab at PSI



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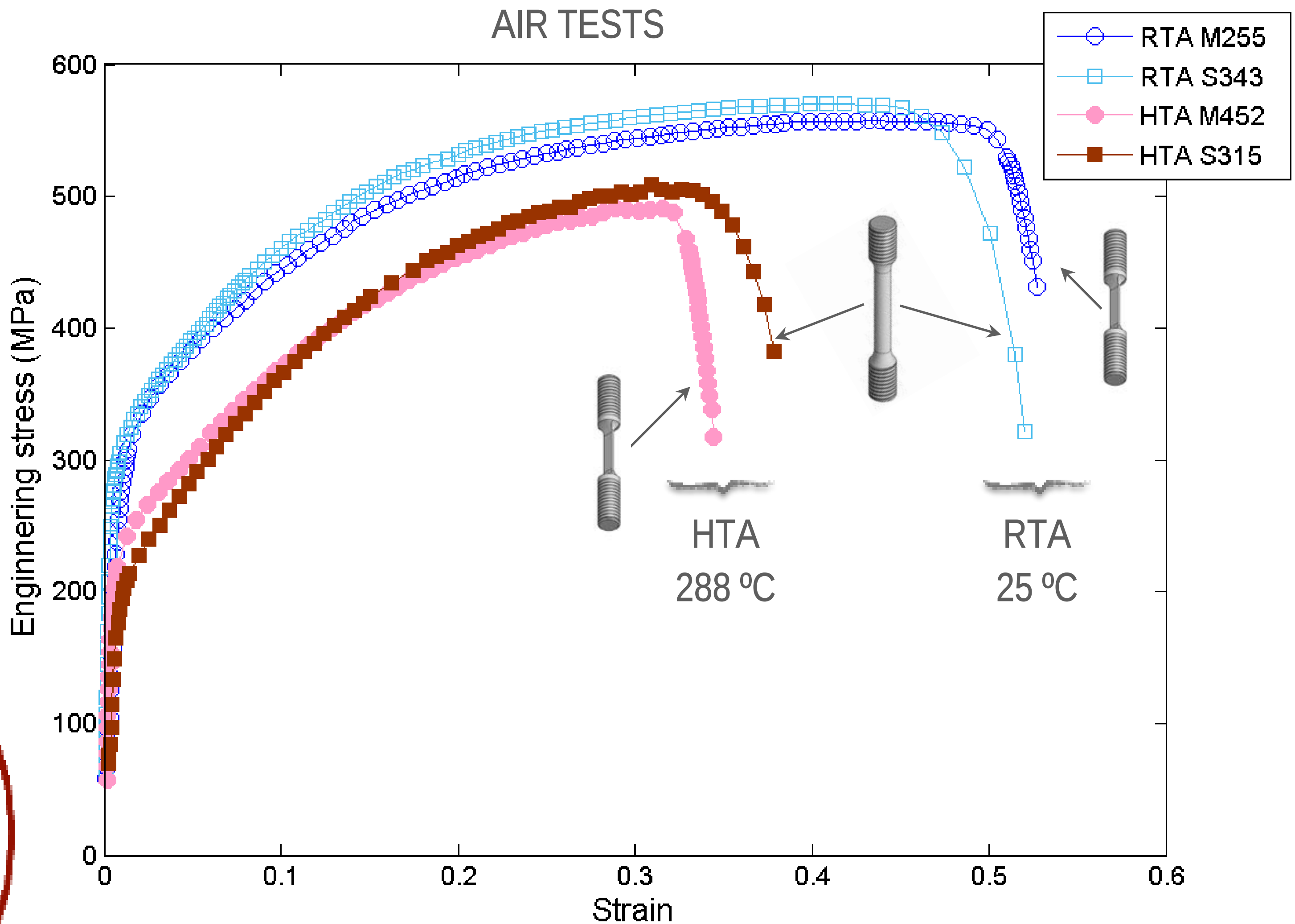
Representative engineering stress-strain curves

Different tensile machines

↓

Effective gauge length

For all conditions, similar YS, UTS & US



Overview of engineering stress-strain results

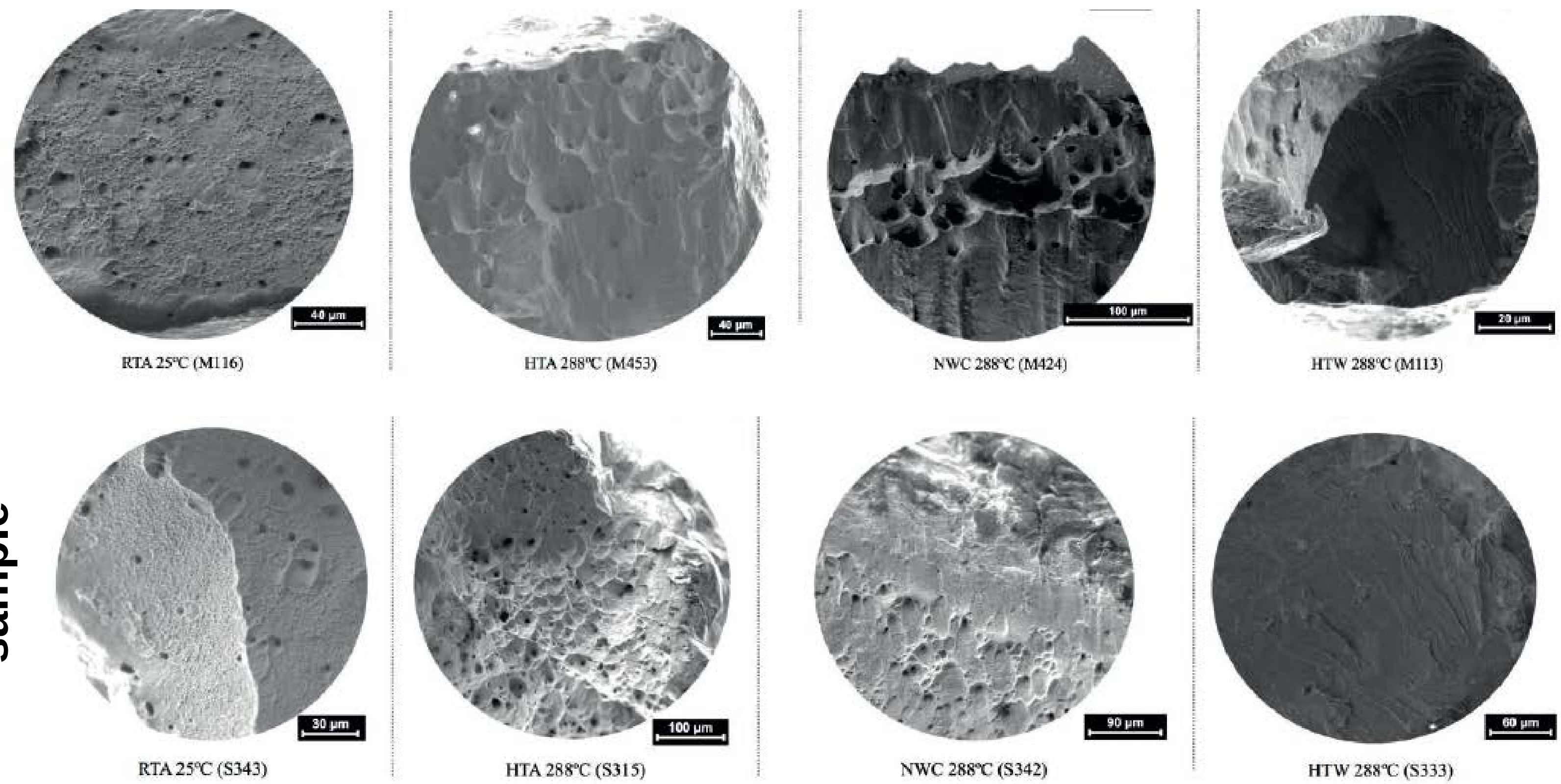
Sample type	Temperature (ºC)	Environment	$Rp_{0.2\%}$ (MPa)	UTS (MPa)	US	RA
Miniaturised (5)	25	RTA	281	558	0.41	0.82
Standard (4)	25	RTA	290	577	0.41	0.83
	REL. ERROR RTA		3.0%	3.3%	0.85%	1.42%
Miniaturised (7)	288	HTA	216	500	0.29	0.79
Standard (5)	288	HTA	204	509	0.32	0.70
	REL. ERROR HTA		5.9%	1.7%	8.2%	12%
Miniaturised (1)	288	NWC (500 ppb O_2)	224	499	0.34	0.71
Standard (1)	288	NWC (500 ppb O_2)	208	527	0.38	0.68
	REL. ERROR NWC 500 ppb O_2		7.8%	5.4%	8.9%	5.1%
Miniaturised (2)	288	HWC (2.2 ppm H_2)	-	478	0.32	0.67
Standard (2)	288	HWC (2.2 ppm H_2)	234	501	0.29	0.51
	REL. ERROR HWC		-%	4.6%	11%	30%
Miniaturised (1)	288	NWC (8 ppm O_2)	192	498.8	0.29	0.72
Standard (2-3)	288	NWC (8 ppm O_2)	203	512	0.38	0.67
	REL. ERROR NWC 8 ppm O_2		5.3%	2.6%	24%	6.4%

standard and miniaturized flat dog-bone samples are almost identical !

Fracture surface

Miniaturized sample

Standard sample



RTA 25ºC (S343)

HTA 288ºC (S315)

NWC 288ºC (S342)

HWC 288ºC (S333)

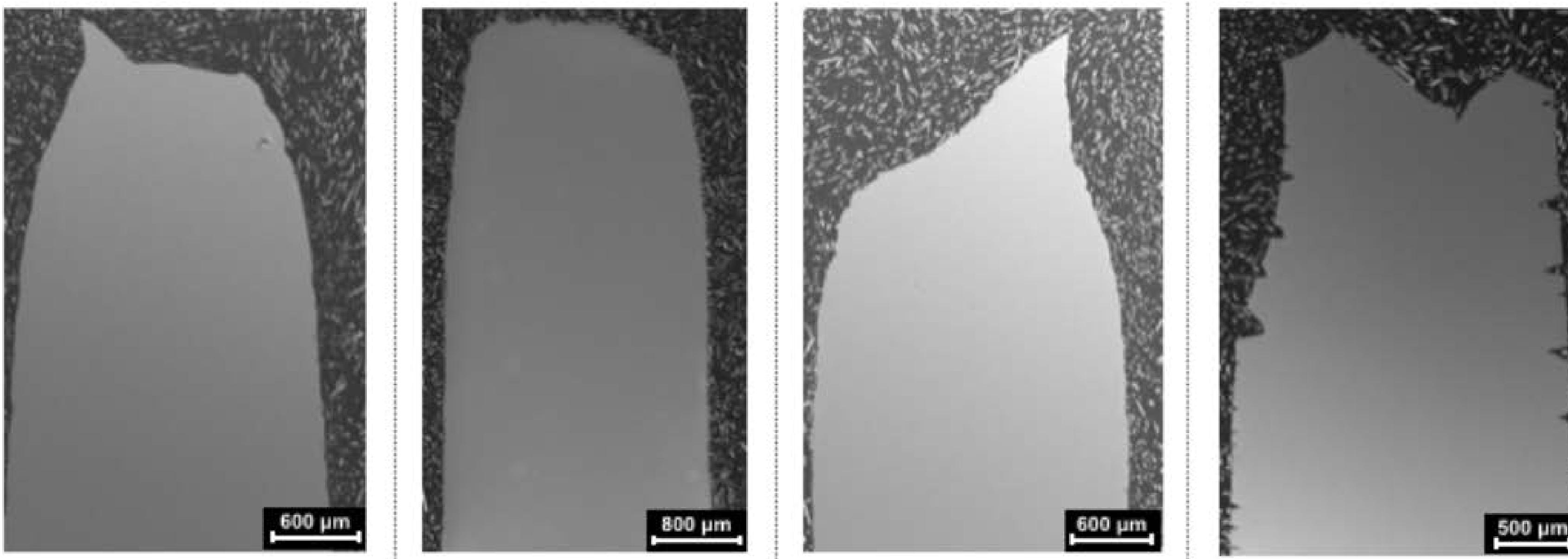
TG-D fracture

TG-D & TG-C fracture (< 3%)

Metallography investigation

Axial cuts

- Standard sample



RTA 25ºC (S343) | HTA 288ºC (S315) | NWC 288ºC (S342) | HWC 288ºC (S333)

Increasing temperature → *Decrease of the reduced area*

No oxygen effect

Hydrogen effect → *Cracks initiating in the sample surface*

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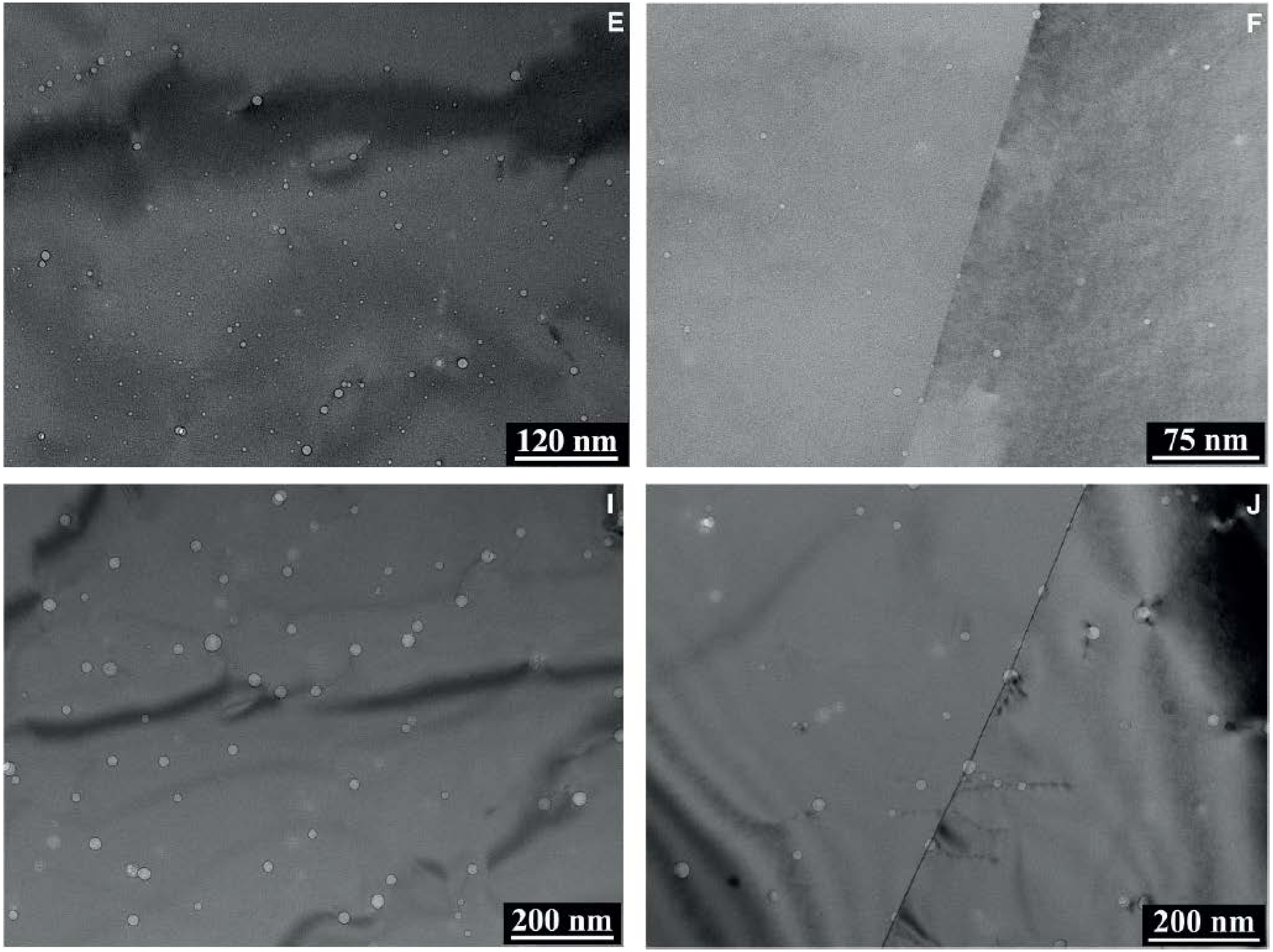
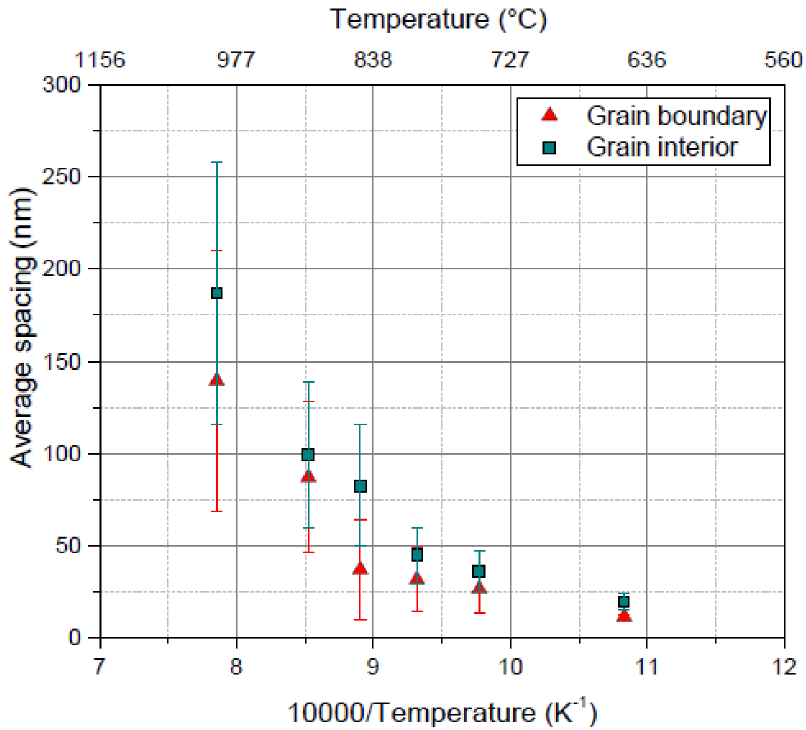
Bubble distribution after PIA (1000 appm)

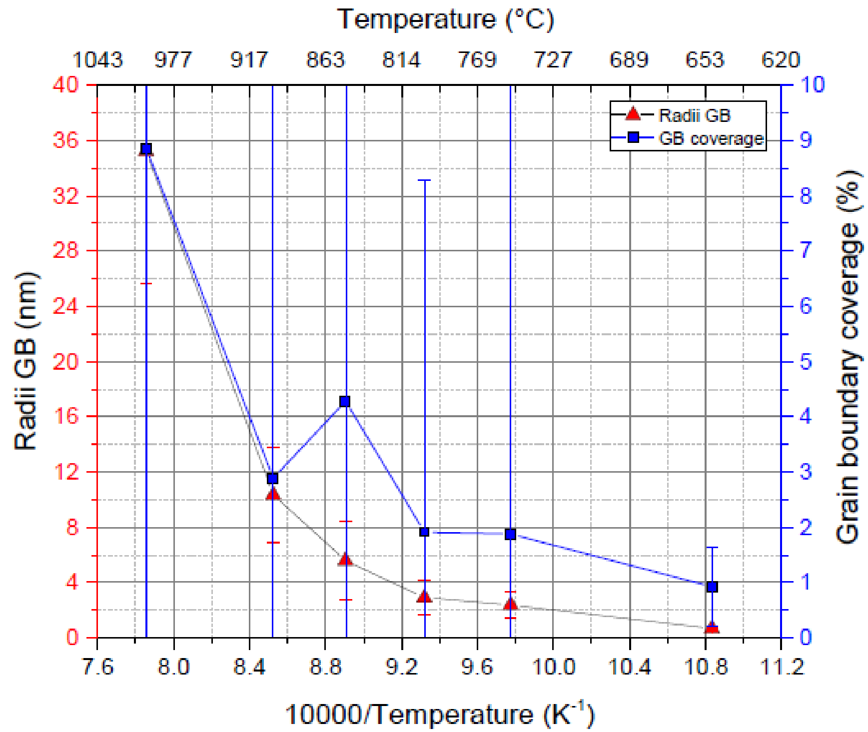
at 750°C

at 900°C



Bubble average spacing and GB coverage

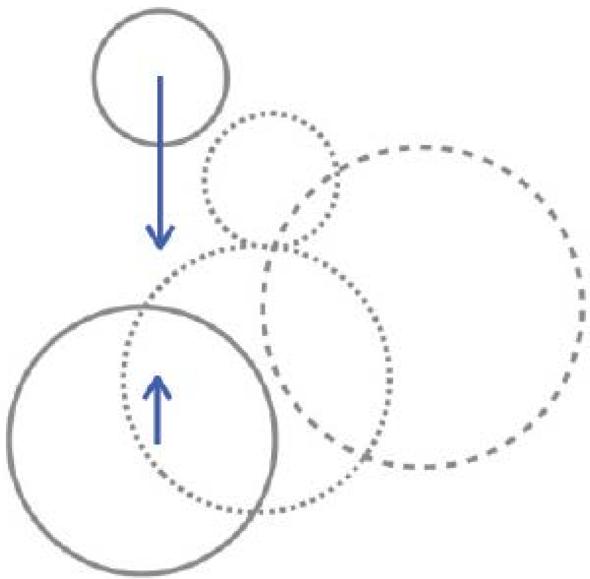




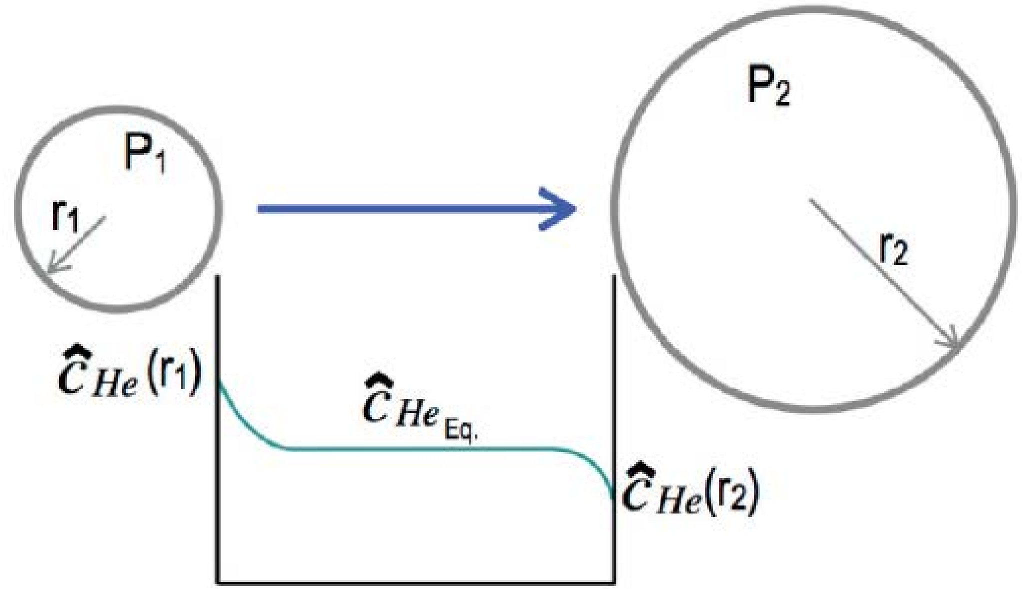
Limited of GB coverage, hence limited GB weakening

Bubble growth mechanism during PIA

Migration & Coalescence



Ostwald Ripening (dissociation)



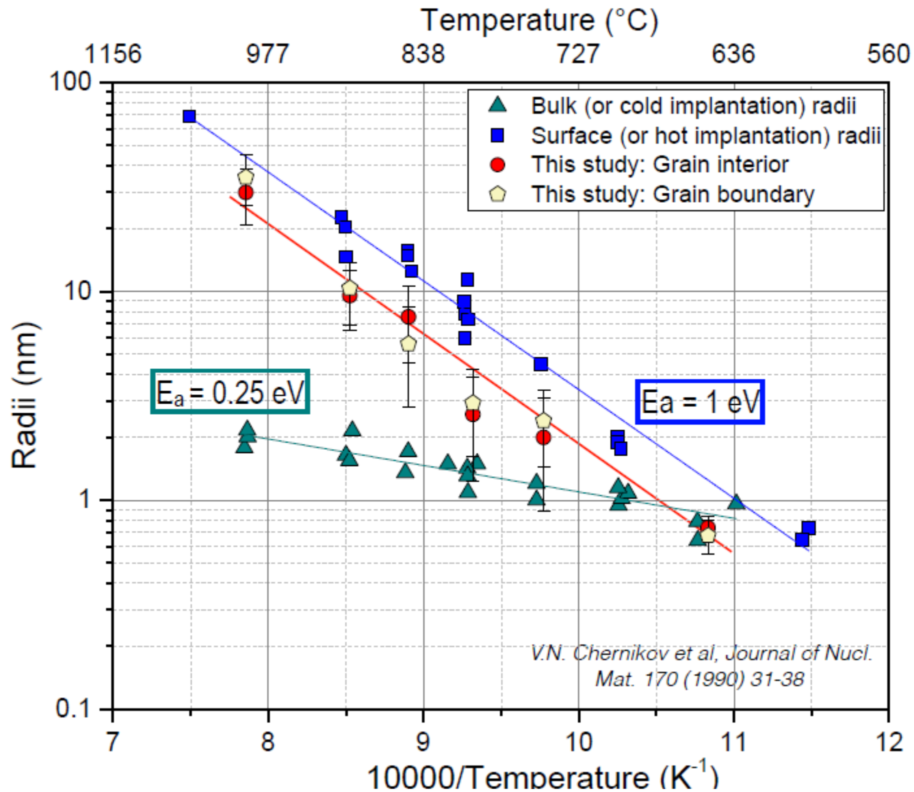
$$\bar{r}_b^n \propto D_X c_{He} t$$

surface diffusion (sd), n=5-6
volume diffusion (vd)
vapor transport through the bubble (g)

$\bar{r}_b^2 \propto kTD_{He}K_{He}t$ Helium dissociation

$\bar{r}_b^3 \propto (\gamma\Omega/kT)D_V t$ Vacancy dissociation

Helium bubble evolution



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, = $E_a n$)	1.26 - 1.51	2.06 – 3.07	2.14 – 3.21	2.22 – 3.33
Mechanism	Surf. diffusion	Dissociation	Dissociation	Dissociation

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Helium bubble hardening

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- **Models for YS increase**

1. DBH $\Delta\sigma_y = \alpha_i M \mu b (N_i d_i)^{1/2}$
2. FKH $\Delta\sigma_y = \alpha_i M \mu b r_i (N_i)^{2/3}$
3. BKS $\Delta\sigma_y = \alpha_i M \mu b (N_i d_i)^{1/2} \frac{1}{2\pi}\left[\ln\left(\frac{l}{b}\right)\right]^{-1/2}\left[\ln\left(\frac{D'}{b}\right)+0.7\right]^{3/2}$

α - hardening coefficient ≃ ?

M - Taylor factor ≃ 3

μ - shear modulus ≃ 76 GPa

b - Burgers vector ≃ 0.255 nm

N_i - Density of defects

d_i - diameter of defects

r_i - radius of defects

D' - Effective diameter

- **Tensile & microstructural data**

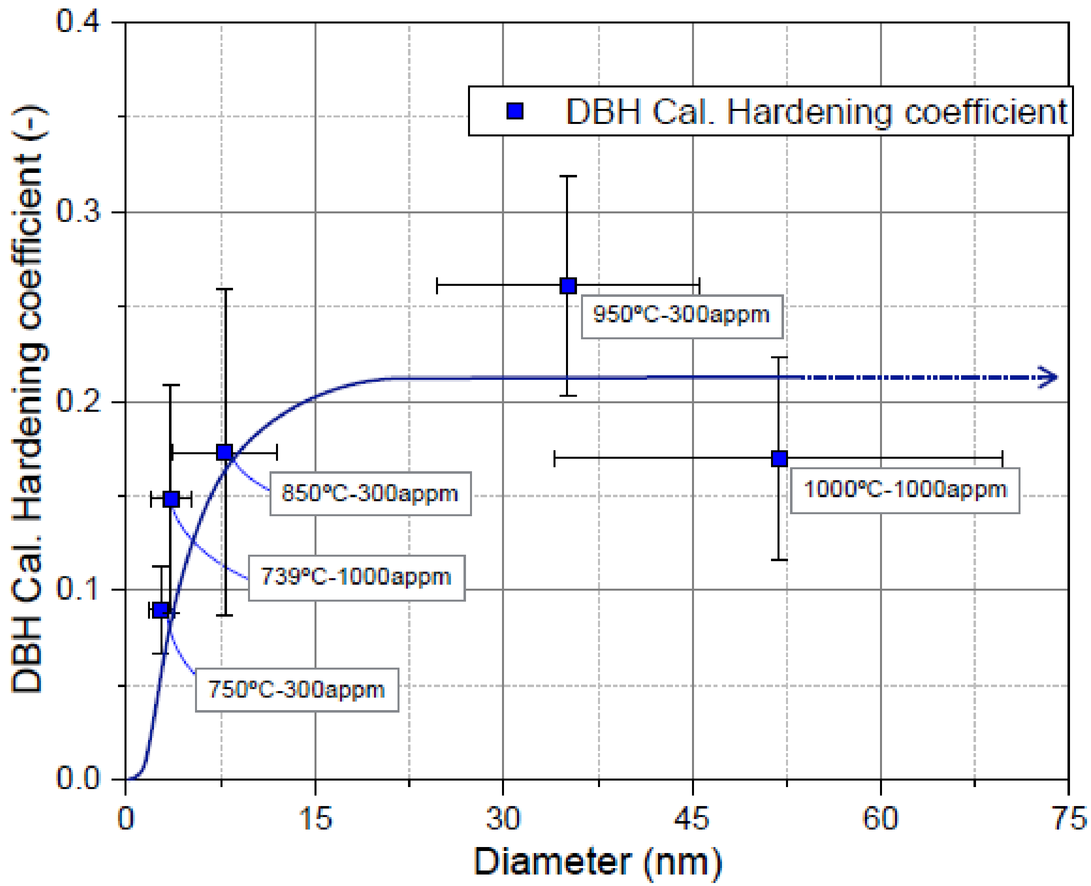
	1000 appm		300 appm		
	750°C	1000°C	750°C	850°C	950°C
Delta YS tensile (MPa)	123	24	45	37	29
AVG. Radii (nm)	1.8	27.5	1.4	3.9	17.6
AVG. Density (bubble/nm^3)	5.6×10^{-5}	1×10^{-7}	2.6×10^{-5}	1.6×10^{-6}	1×10^{-7}

We can determine the hardening coefficient (α)

DBH Model

Dispersed Barrier Hardening

$$\Delta\sigma_v = \alpha_i M \mu b (N_i d_i)^{1/2}$$

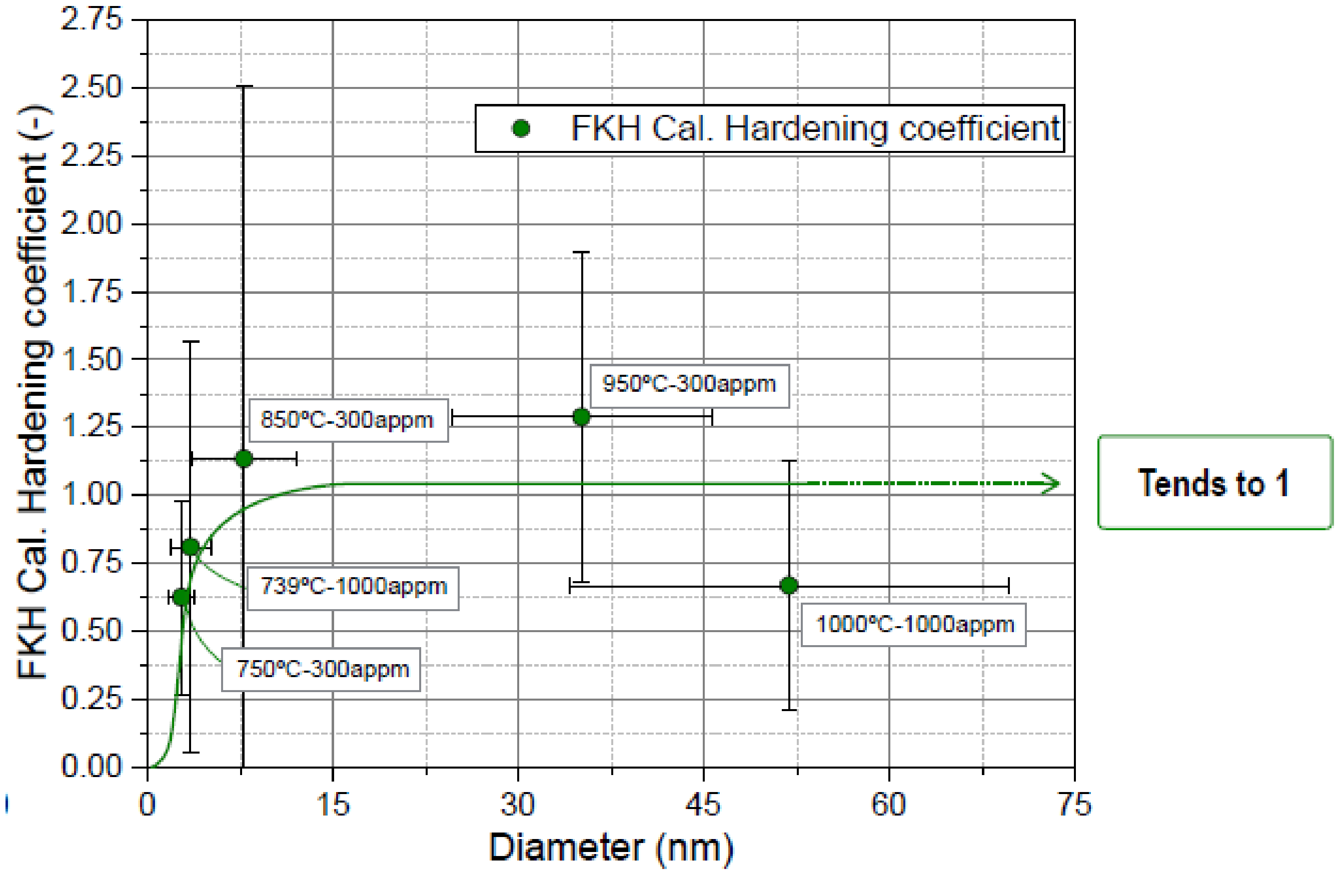


Tends to 0.2

FKH Model

Friedel-Kroupa-Hirsch

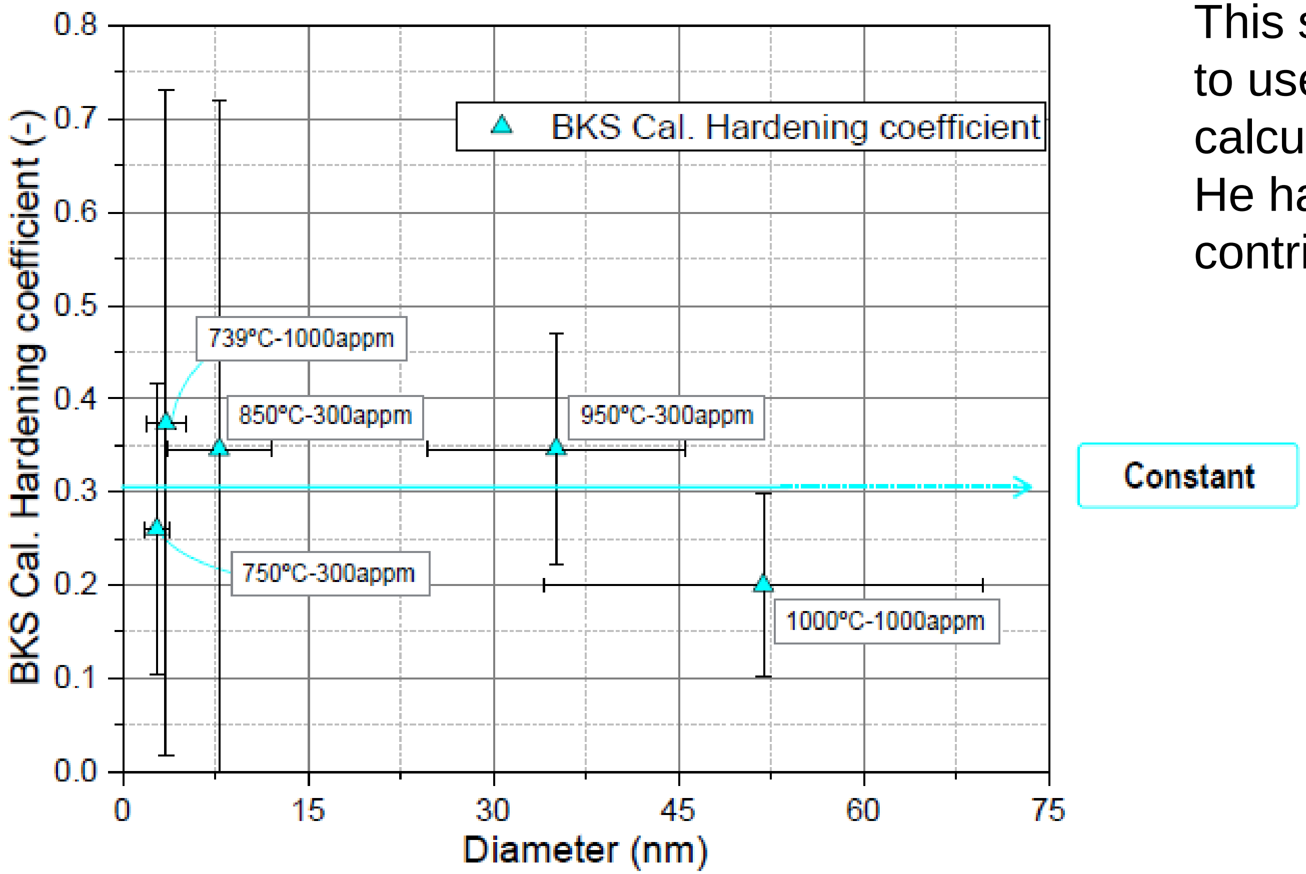
$$\Delta\sigma_y = \alpha_i M \mu b r_i (N_i)^{2/3}$$



BKS Model

Bacon Kocks Scattergood

$$\Delta\sigma_y = \alpha_i M \mu b (N_i d_i)^{1/2} \frac{1}{2\pi} \left[\ln\left(\frac{l}{b}\right) \right]^{-1/2} \left[\ln\left(\frac{D'}{b}\right) + 0.7 \right]^{3/2}$$



This suggests to use BKS for calculating the He hardening contribution

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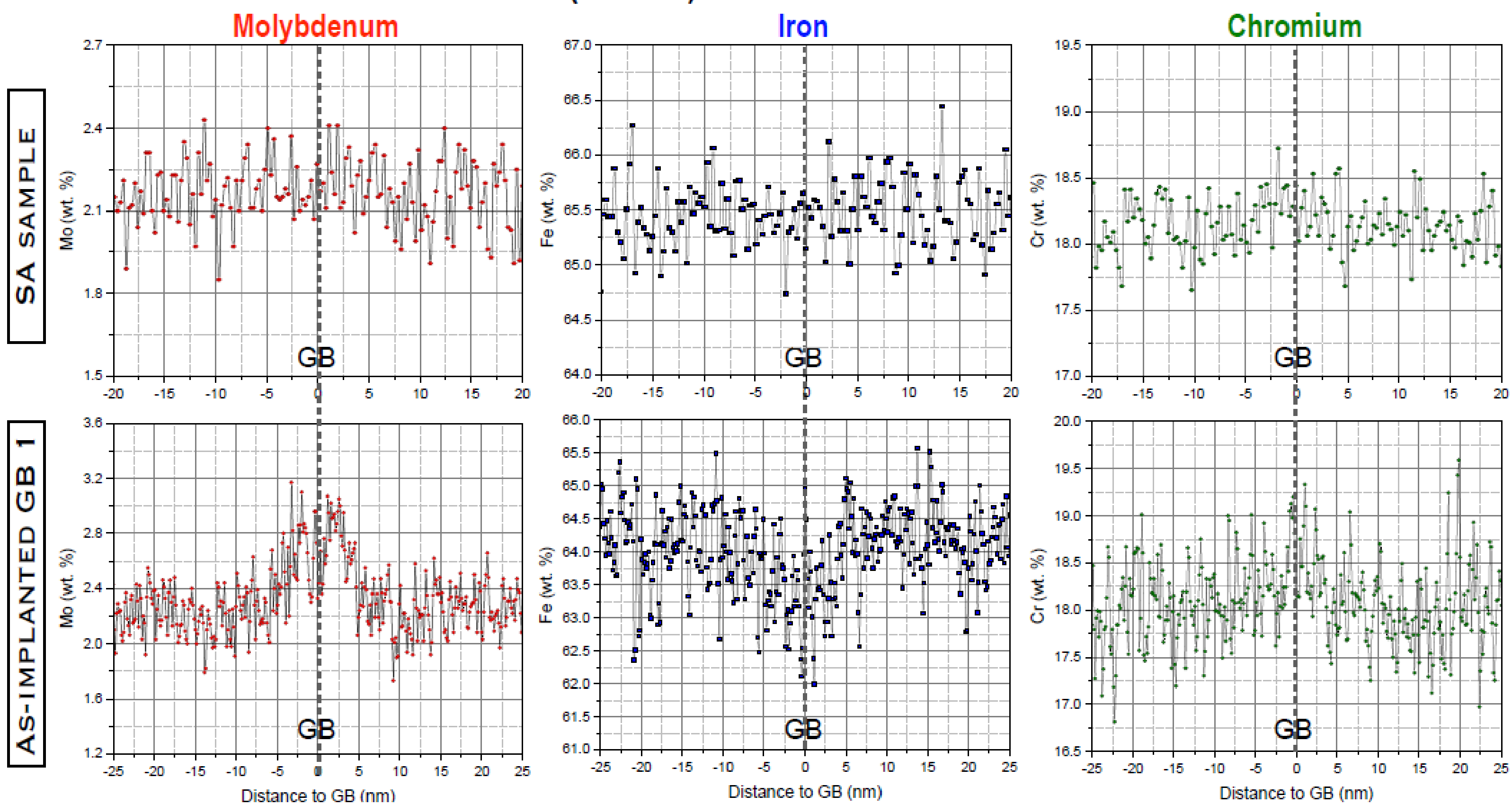
RIS on Grain Boundary

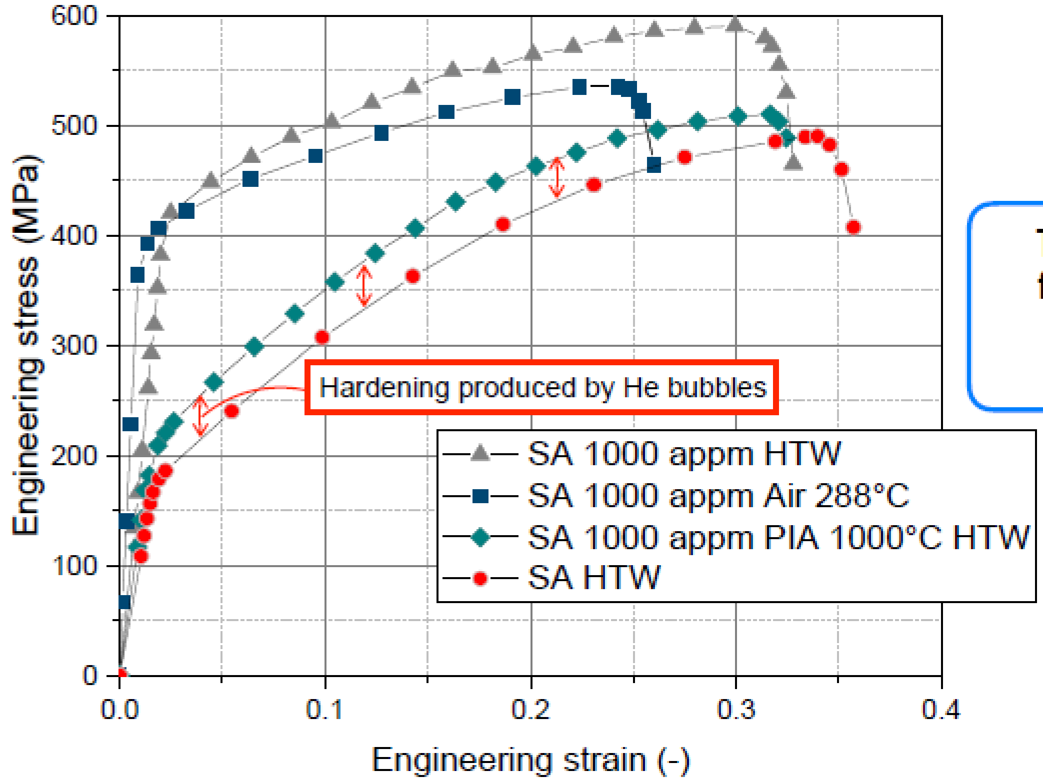
- GB RIS with FEI TALOS F200X (200 kV)



Enrichment	Depletion	No measurable effect
2.1⟶3 % wt.	64.5⟶63.5 % wt.	
Usually W shape	Usually V shape	

SSRT to SA + 1000 appm samples

- 1000 appm with/out HT

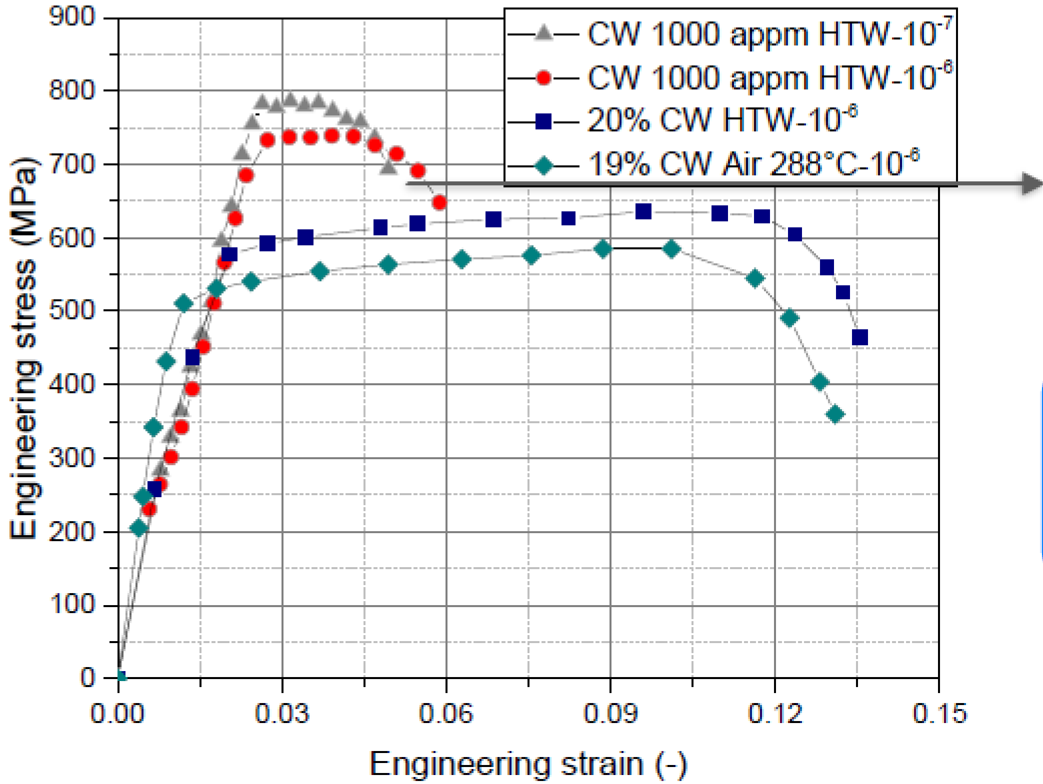


Transgranular dimple fracture is dominant, some transgranular cleavage in HTW

	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

SSRT to CW + 1000 appm samples

- CW with/out 1000 appm



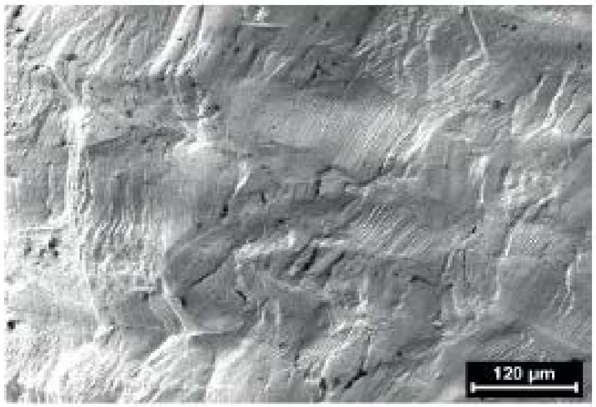
CW samples show tensile response similar to IASCC curves

Transgranular dimple fracture is dominant, some transgranular cleavage in HTW

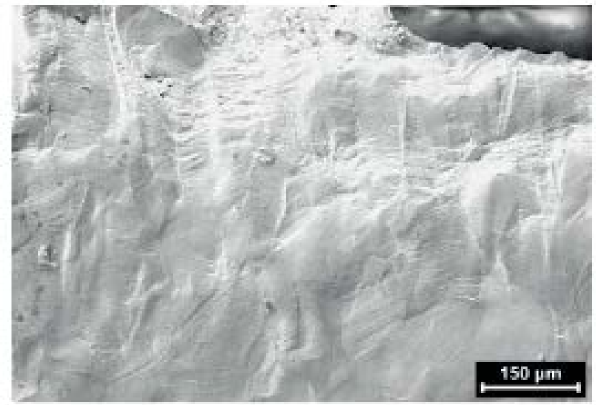
	CW HTA	CW HTW	CW-1000appm	CW-1000appm 10^{-7}
YS	579	576	734	787
UTS	631	641	740	790
ε_u (plastic)	0.08	0.08	0.013	0.001

Metallography investigations

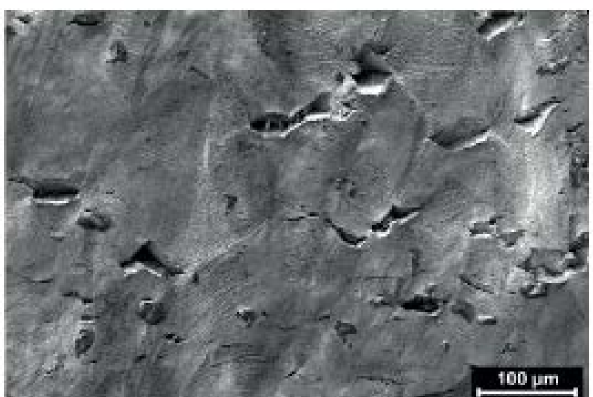
Surface of the samples tested in different environment and material conditions



HTA SA

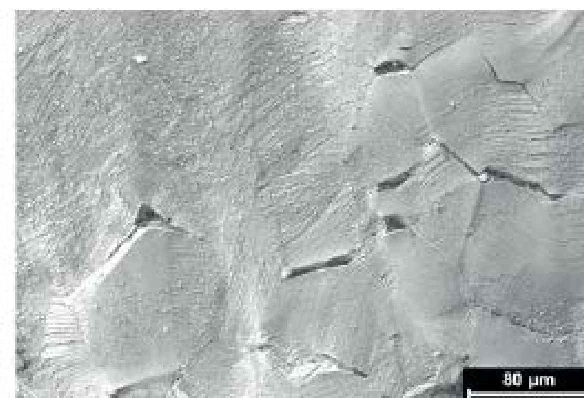


HTA $He_{1000\ appm}$



HTW SA

HTW $He_{300\ appm}$

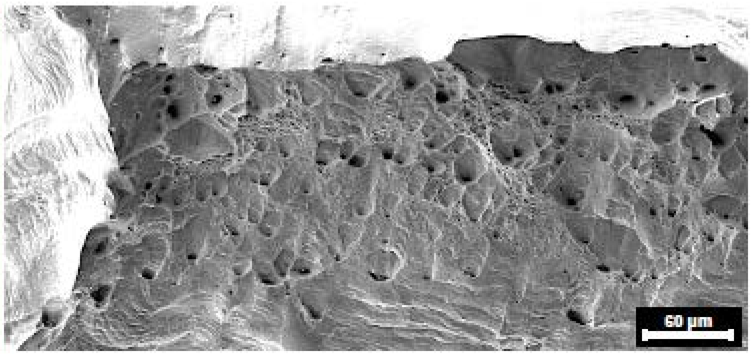


HTW $He_{1000\ appm}$

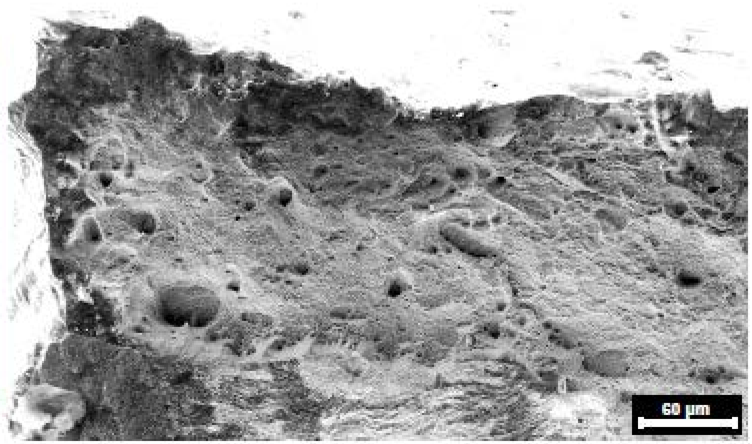
Fracture investigations

- **Fracture surface: 1000 appm with/out HT**

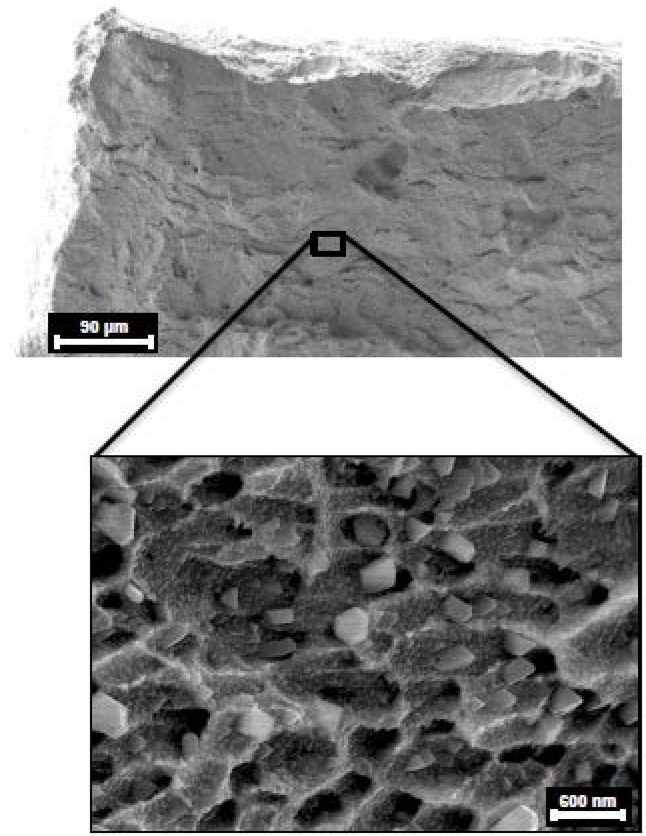
SA-1000 appm (Air-288°C)



SA-1000 appm (HWC)

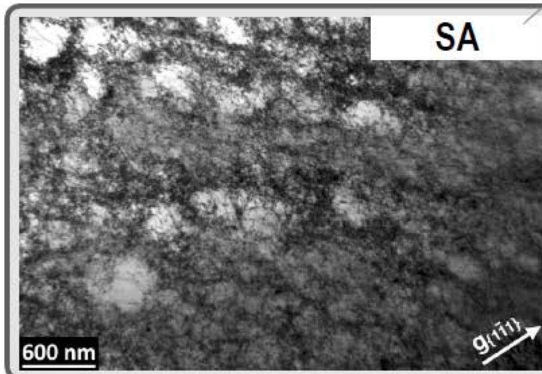


SA-1000 appm + **PIA** 1000°C (HWC)



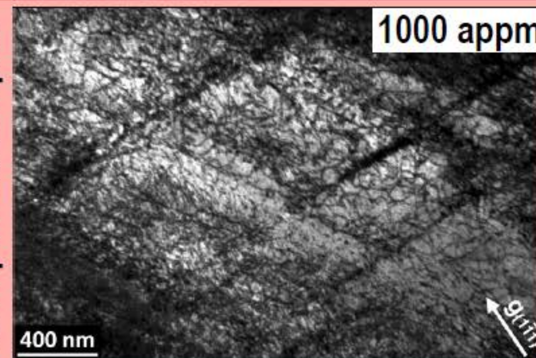
TEM investigation

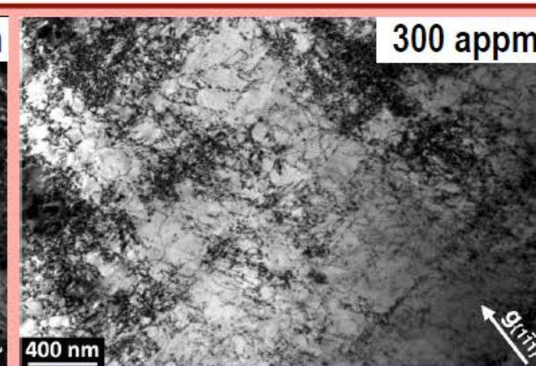
- **Deformation microstructure (all HTW)**



3D deformation cells typical for high Ni contents.

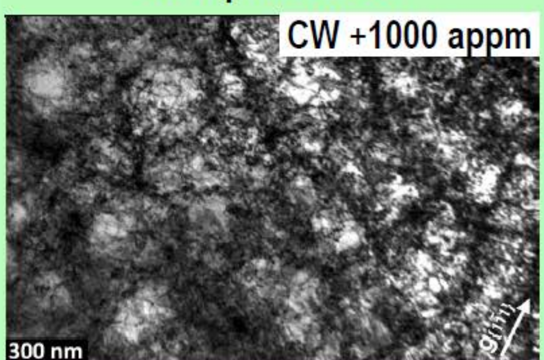
As-implanted samples





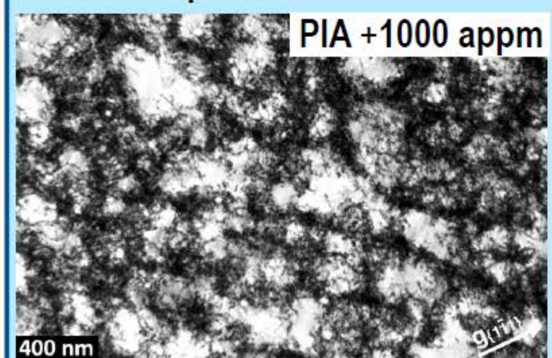
No apparent 3D deformation cells with random arrangement of dislocations. Microstructure that might be a precursor for SCC.

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?**

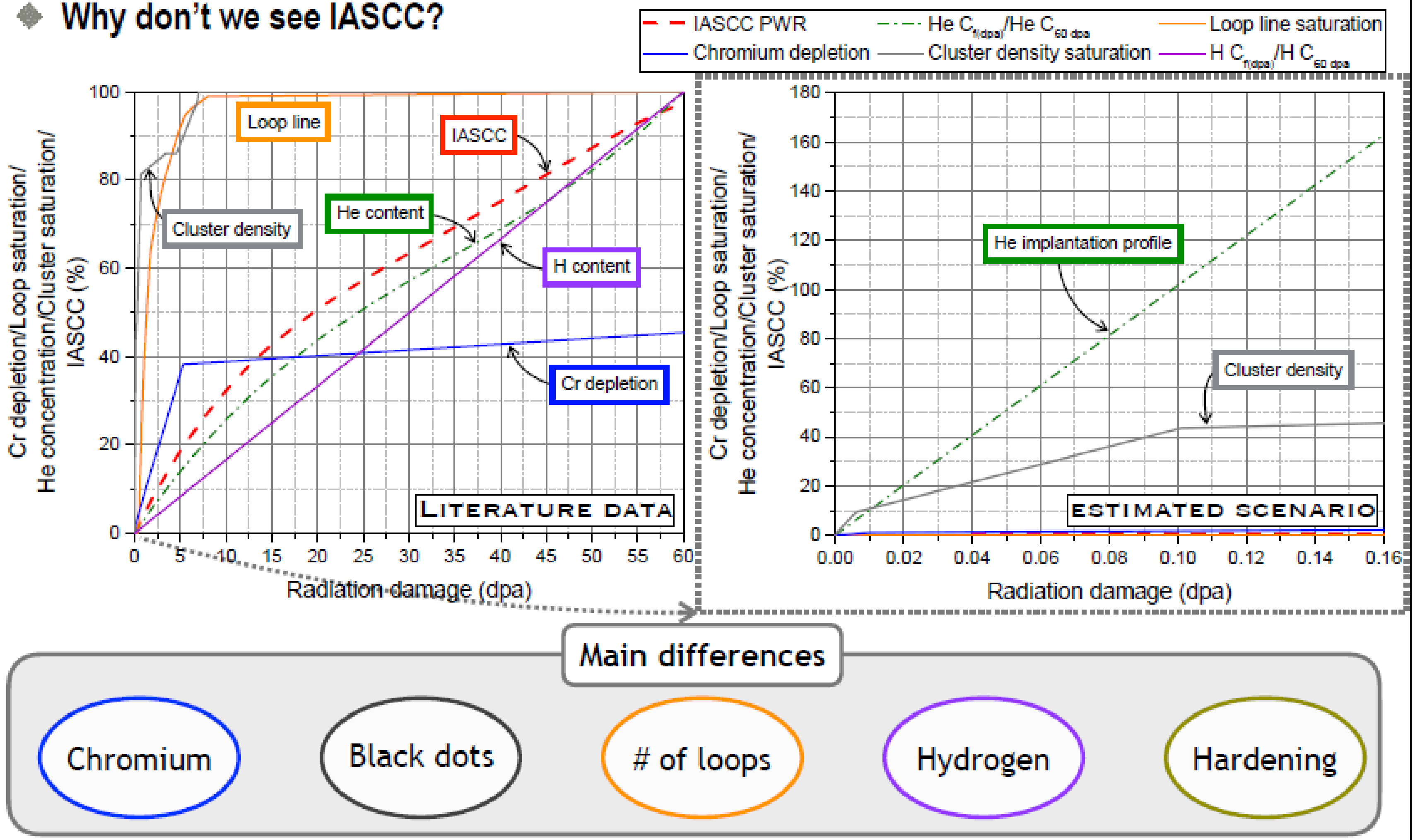

Table of contents

Summary, conclusions & perspectives I

- Results of SSRT of 316L sample in air and different hot water (different chemical water conditions) for standard and miniaturized flat dog-bone samples show that the mechanical properties and fracture mode are almost identical for both sample types.
- Optical microscope and SEM observations show 100% ductile fracture mode after SSRT test for RT and HT in air, normal hot water conditions but 2% cleavage appearances at hydrogenated hot water condition (288°C)
- 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.
- The hardening coefficient increases with the bubble size in the FKH and DBH models but not in BKS model. This suggests to use BKS for calculating the He hardening contribution.
- Homogenized He implantation in SA and CW at 300ºC up to 1000 appm 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.

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Summary, conclusions & perspectives II

- 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.
- 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.
- These results suggest that a helium concentration ≤1000 appm alone cannot induce IASCC, therefore there has to be some synergy between irradiation 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.

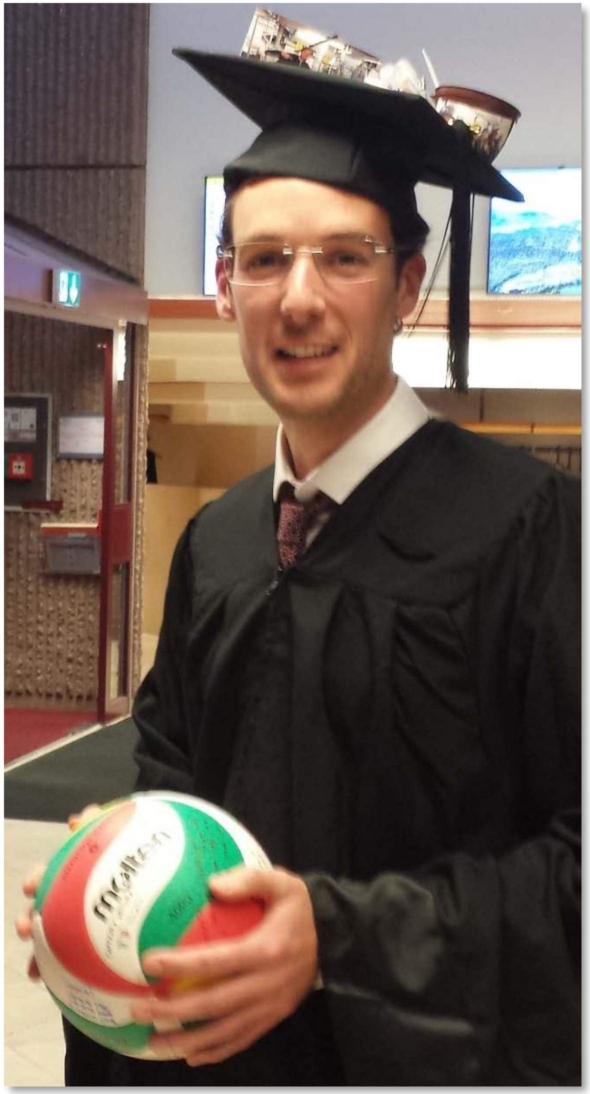
Defense of Ignasi Villacampa on 12.1.2018

infoscience.epfl.ch/record/233705

Helium effects on irradiation assisted stress corrosion cracking susceptibility of 316L austenitic stainless steel

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

Ignasi VILLACAMPA ROSES



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Thank you for your attention