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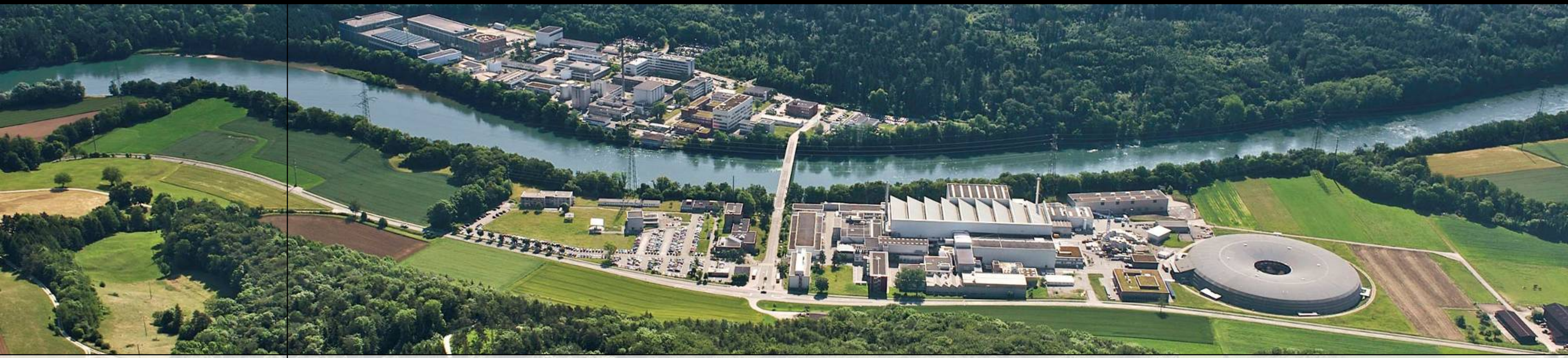


Wir schaffen Wissen – heute für morgen

Paul Scherrer Institut

The hardening effect of He-bubbles in F82H irradiated in the Swiss spallation neutron source

Christiane Vieh

Laboratory for Nuclear Materials

Nuclear Energy and Safety

Motivation based on PSI (STIP) research achievements

Comparison radiation damage fission/fusion to spallation

↑ primary knock-on energies / ↑ displacement cascades / ↑ in generation rate of He & H

He/dpa (in FM steels): fission: < 1 appm / **fusion:** ~ 10 appm / **spallation:** up to 100 appm

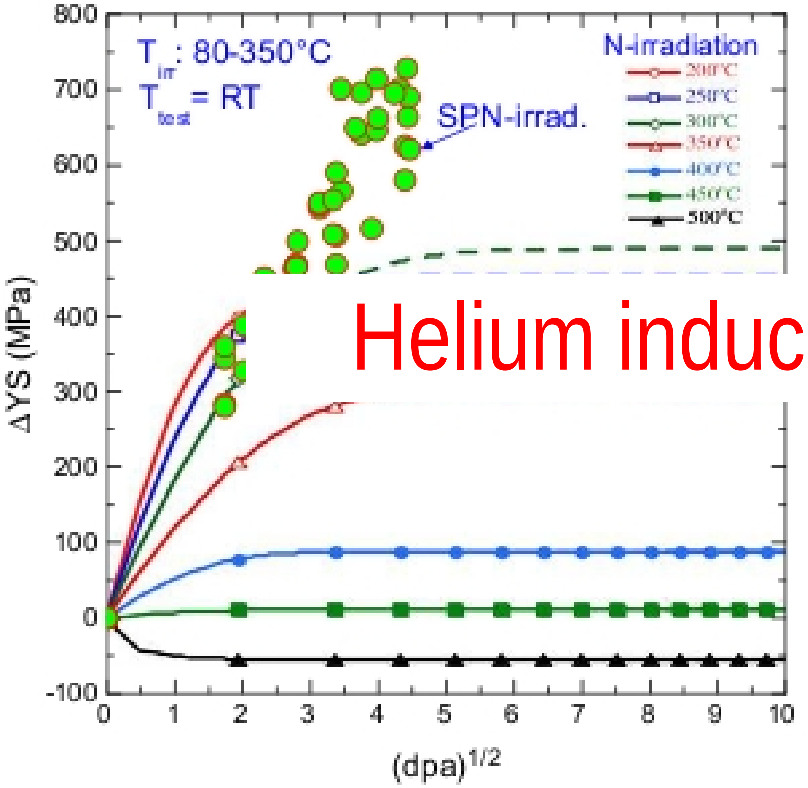
FM steels: 50- 350 ° C doses < 20 dpa

- high density of clusters or loops were observed

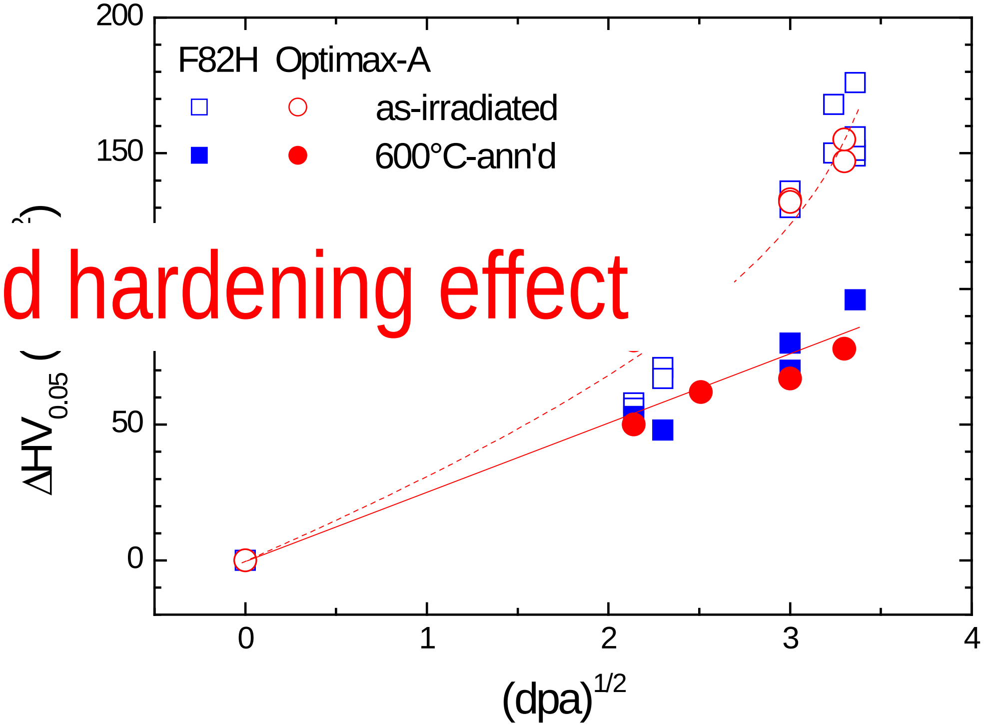
FM steels: T > 400° C

- fewer clusters or loops were observed
- high density of He bubbles

Y. Dai et al. / JNM 415 (2011)



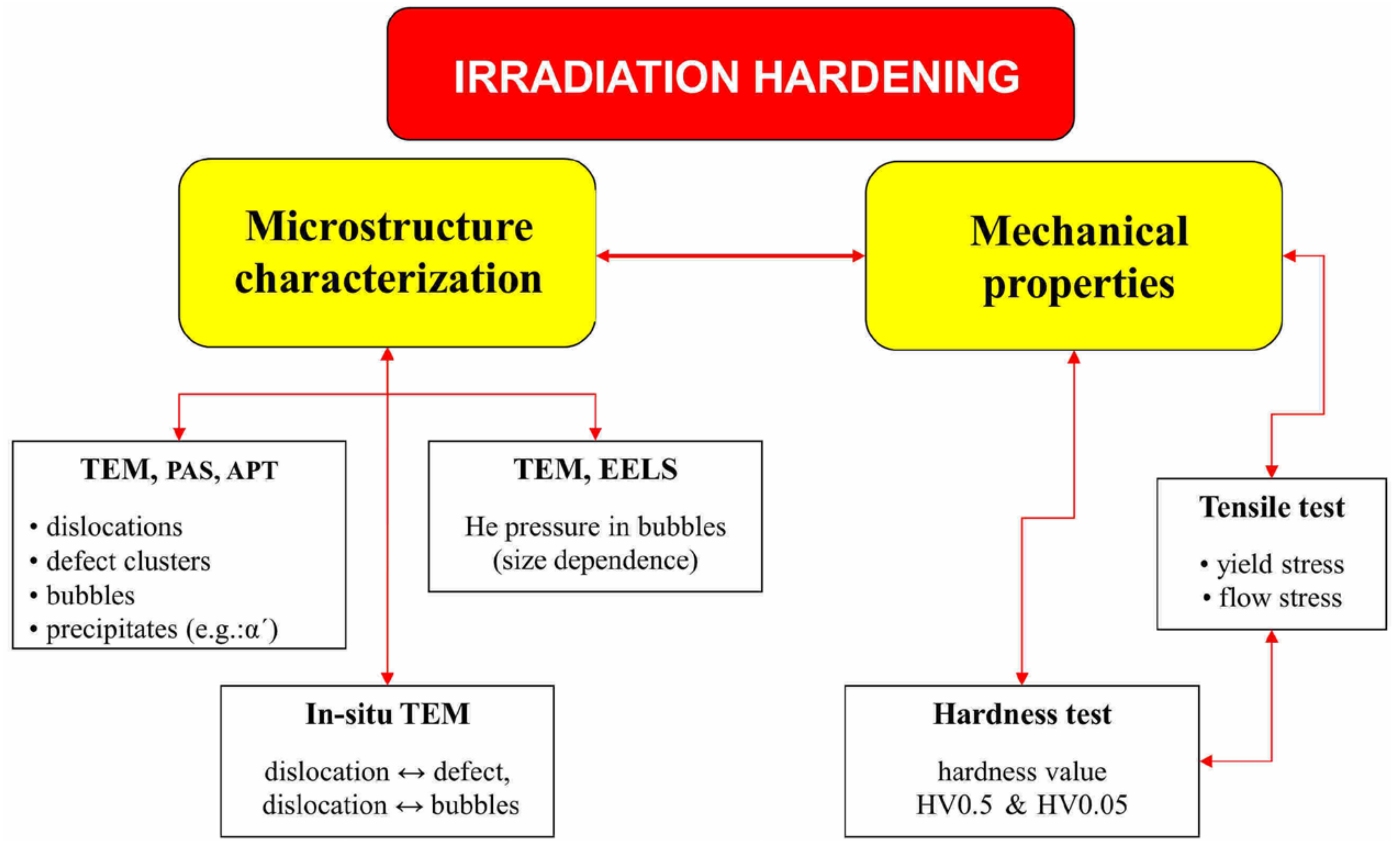
L. Peng et al. / JNM 398, (2011)



Helium induced hardening effect

Motivation

Unsolved: effect of He (size & pressure) on mechanical properties!



Are He-bubbles strong barriers to mobile dislocations
=> contribution to the hardening effect?

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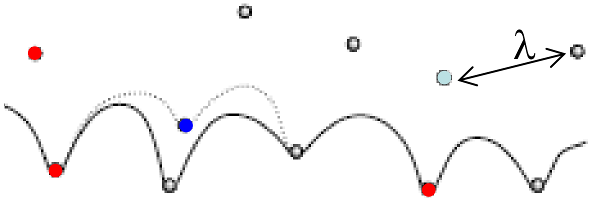
Statement of the problem

Irradiation induced hardening: several types of defects



$$\Delta\tau = \alpha\mu b\sqrt{\sum_i N_i d_i}$$

α = ????

α_i… strength of defect i

μ... shear modulus

b… Burgers vector

λ… mean distance between defects

N_i… volume number density of defects i

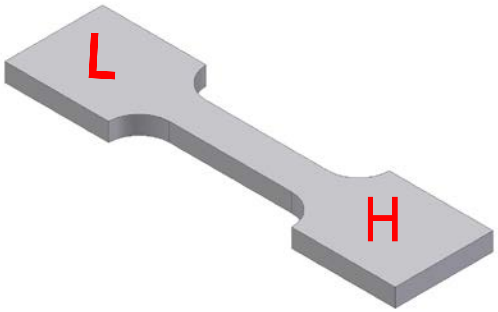
d_i… mean size of the defect i

$$\lambda = \frac{1}{\sqrt{\sum_i N_i d_i}}$$

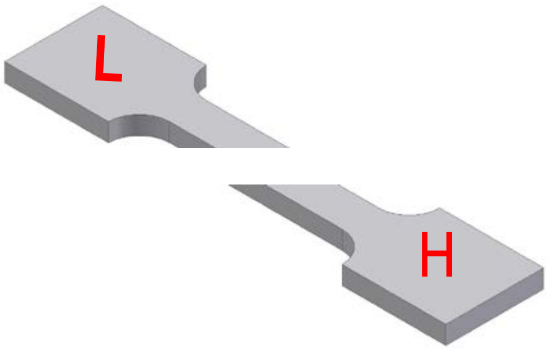
In the center of interest

- distinction of the individual contribution from different hardening features
- perform annealing treatments to annihilate the irradiation-induced defect step-by-step but the He bubbles, which will coarsen, become visible

Mechanical properties: tensile tests of F82H (STIP-II)

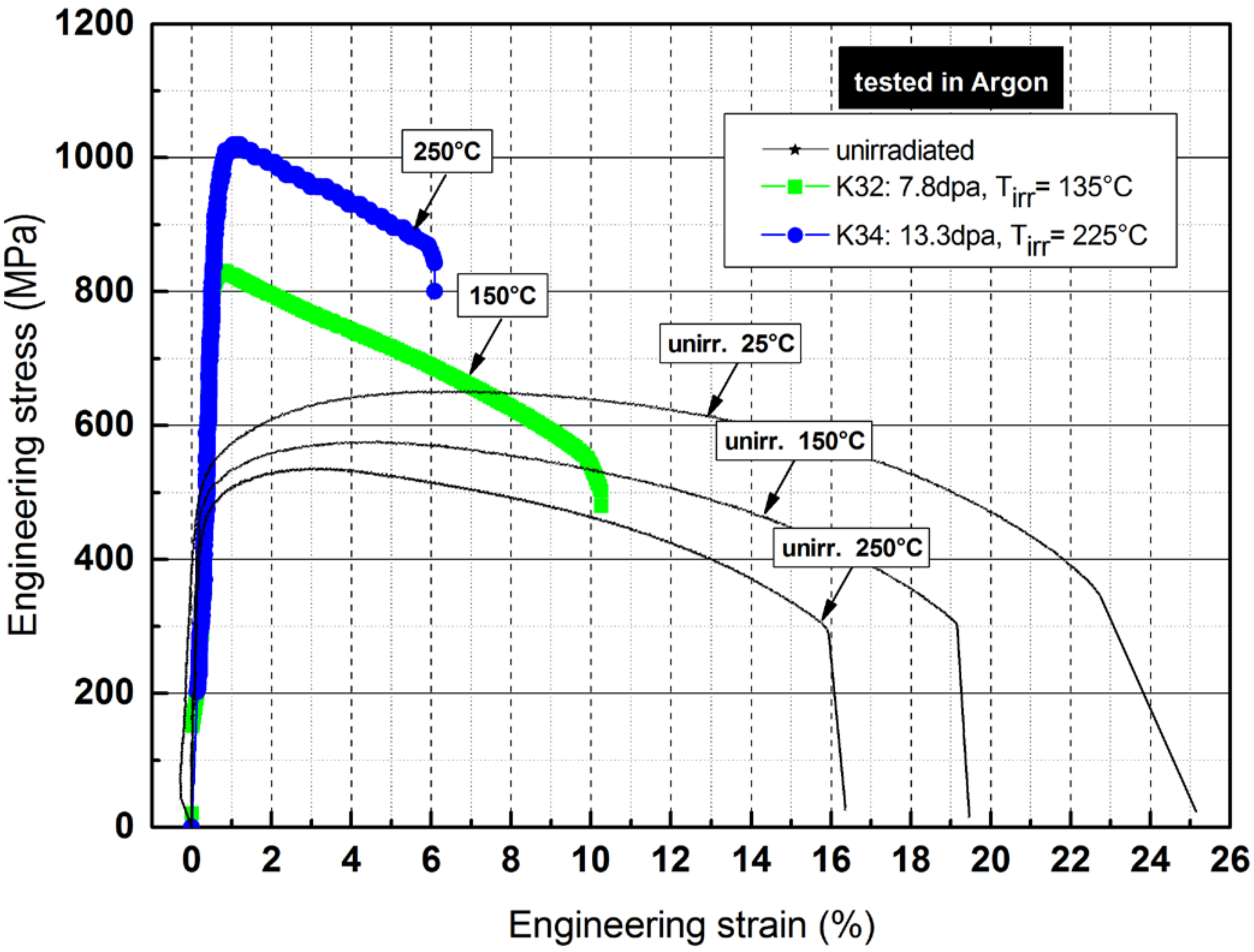


before tensile test



after tensile test

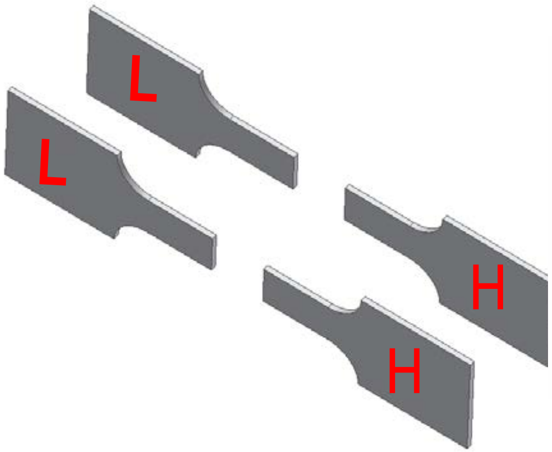
hardening: increase in (yield) strength, depends strongly on irrad. T, irrad. dose & He concentration



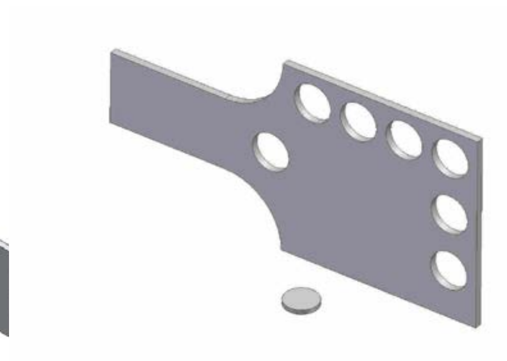
Specimen preparation of radioactive FM steel

Material	Cr	Ni	Mo	Mn	V	W	Ta	C	Si	P	N
F82H	7.71	0.02	0.003	0.16	0.16	1.95	0.02	0.09	0.11	0.002	0.006

HISTORY of a Large Tensile (LT) – specimen

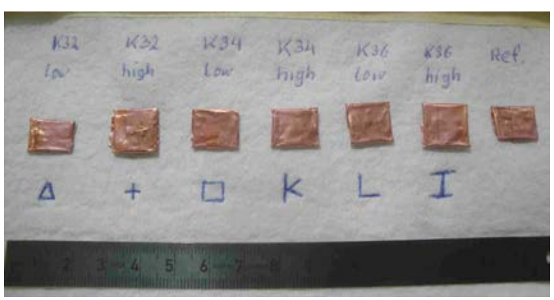


4 pieces after cutting

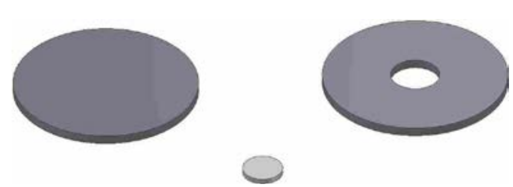


0.8 mm discs

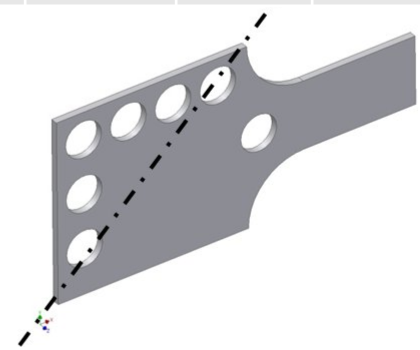
F82H			
Specimen description	Dose (dpa)	Tirr (° C)	He conc. (appm)
K32 low dose	6.2	115	465
K32 high dose	9.6	162	785
K34 low dose	11	185	900
K34 high dose	15.9	268	1430
K36 low dose	17.5	296	1600
K36 high dose	20.3	344	1825



Annealing of 0.8 mm discs

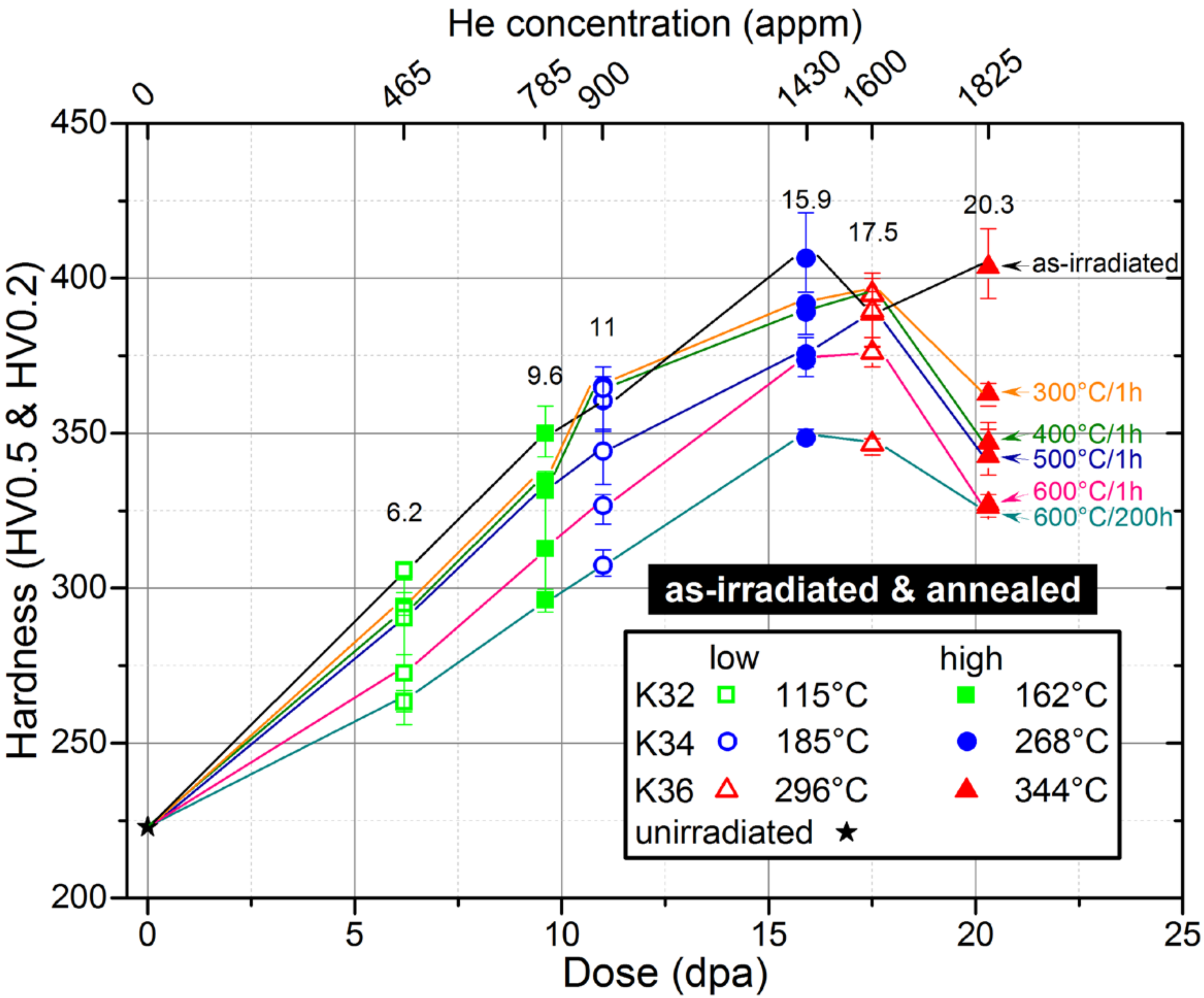


After annealing for TEM



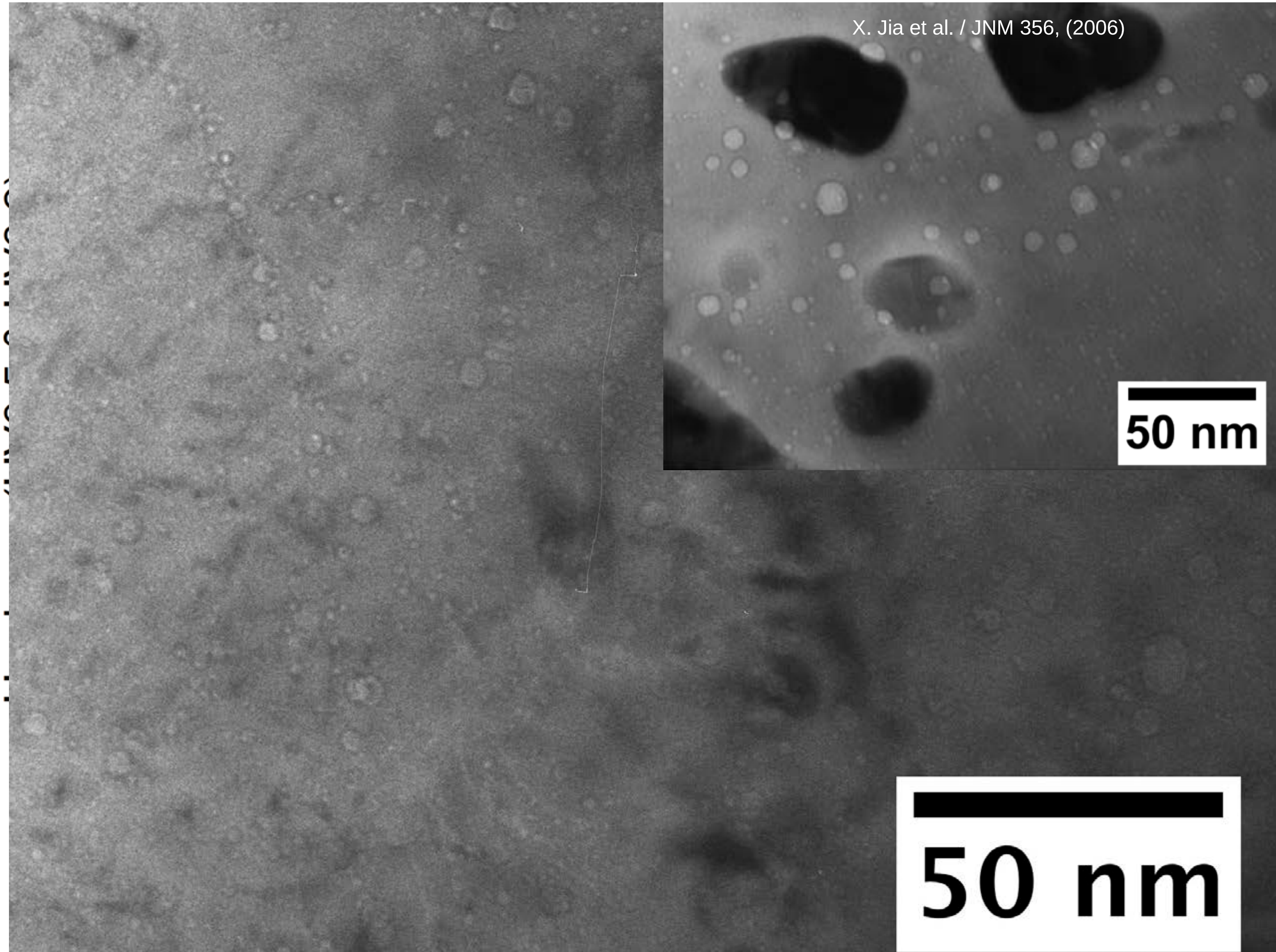
Annealing & HV tests

Mechanical properties: hardness tests



significant ↑hardness following irradiation

Mechanical properties: hardness tests

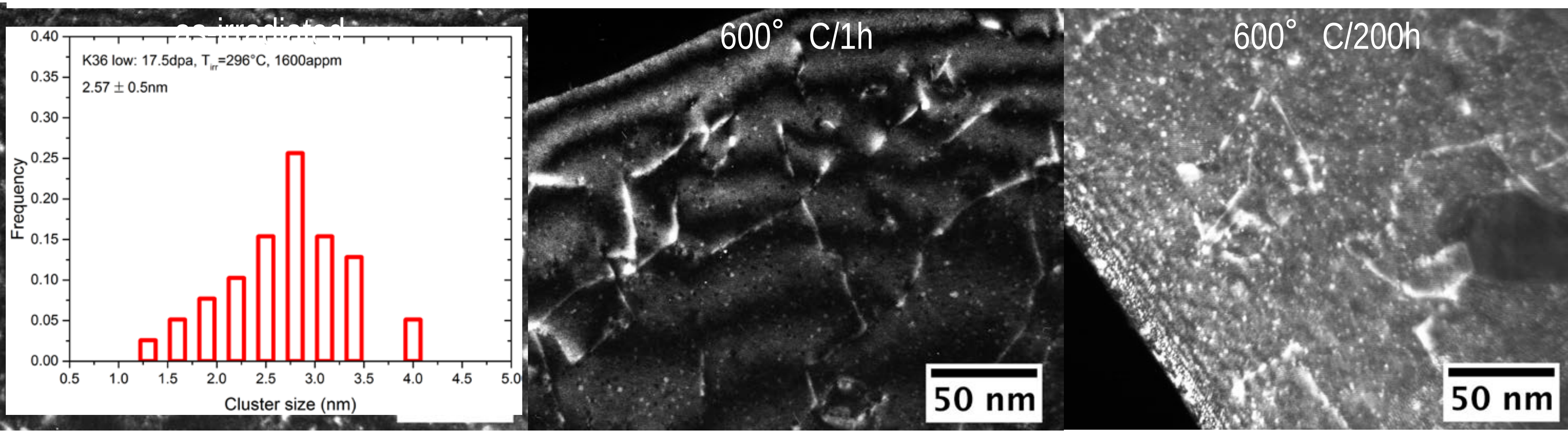


decrease with ↑ T

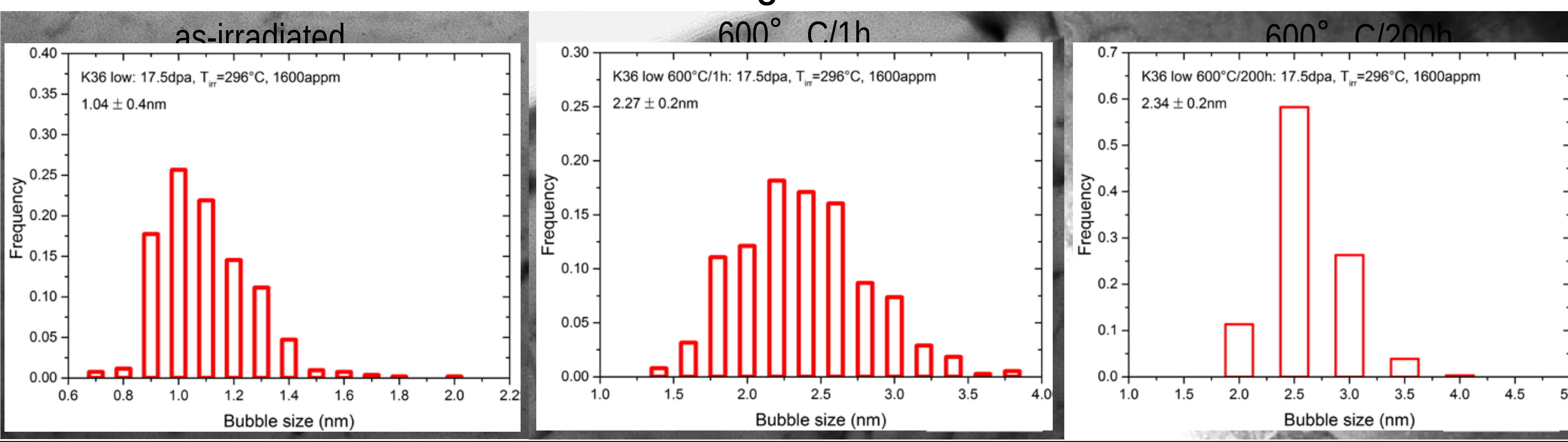
Microstructural investigation by TEM

F82H (K36 low dose): 17.5 dpa, Tirr = 296° C, 1600 appm => $\Delta\tau = \alpha\mu b\sqrt{\sum_i N_i d_i}$ => $\alpha_{He} = 0.15$

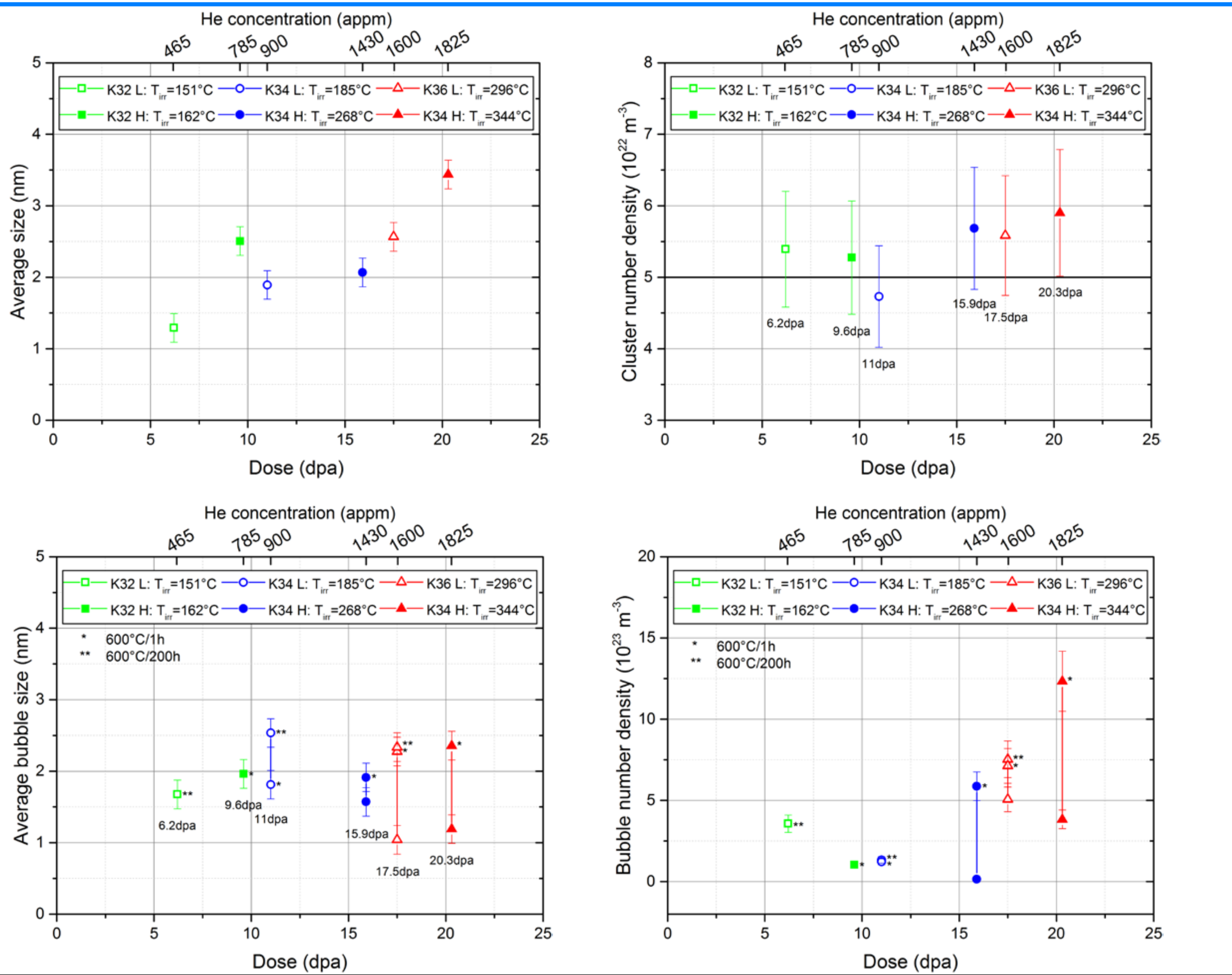
Dark Field



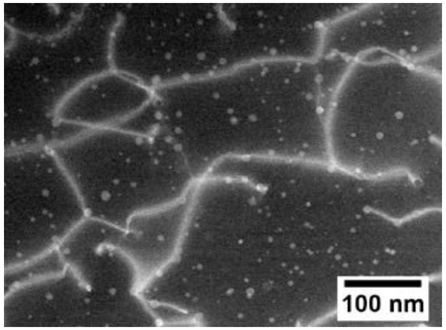
Bright Field

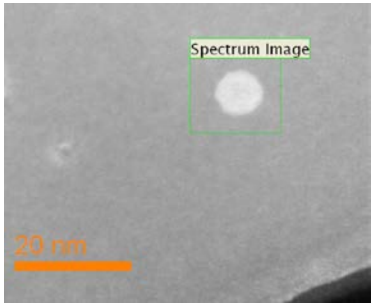


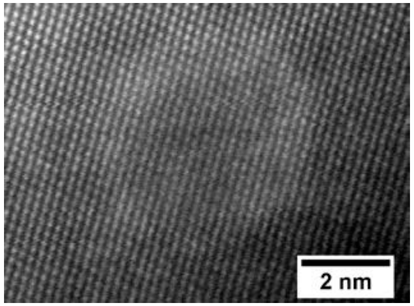
Characterization of the evolution of defects

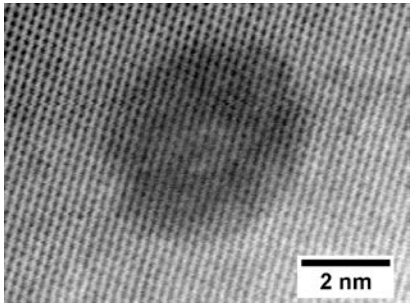


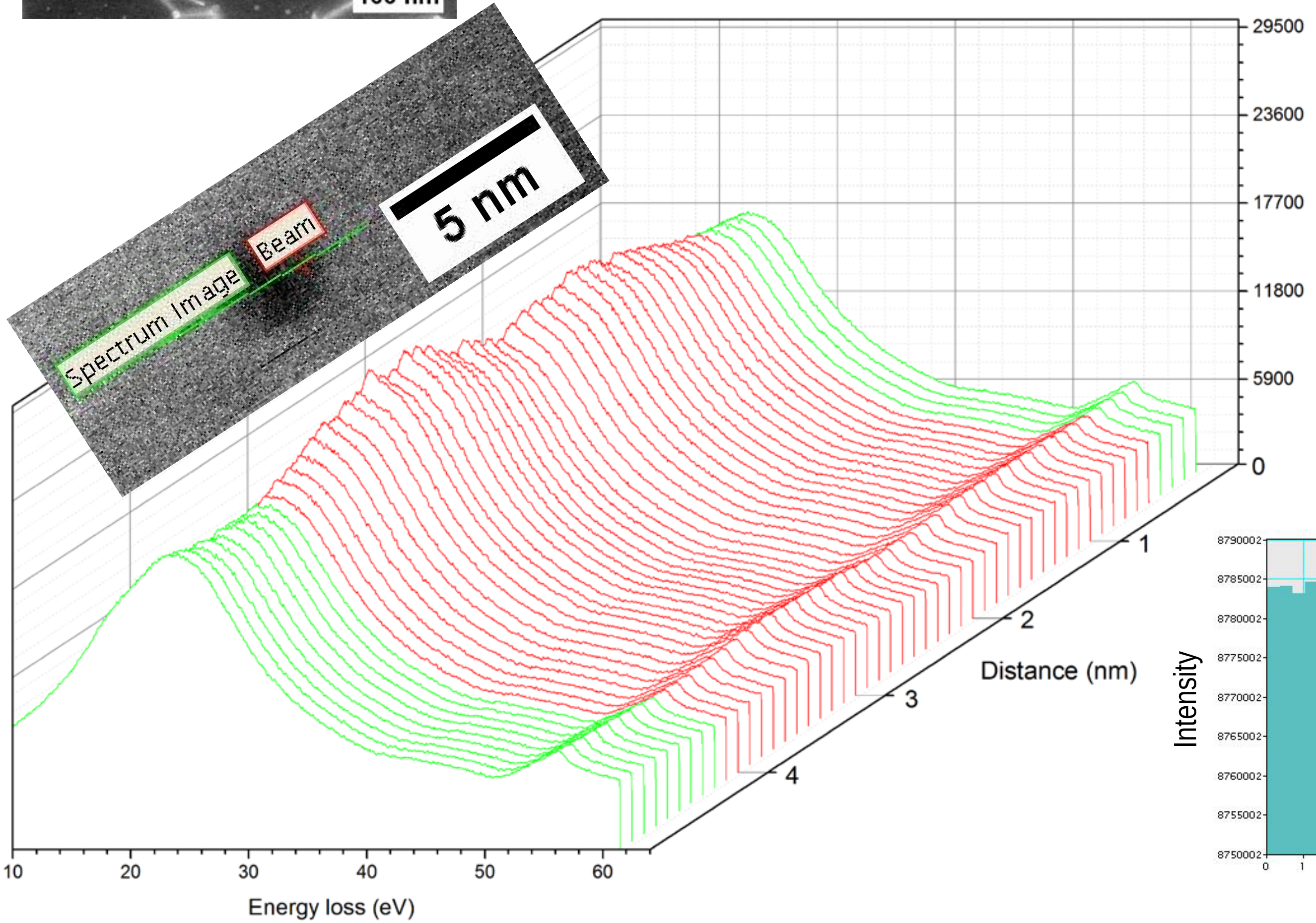
STEM/EELS: Measurement

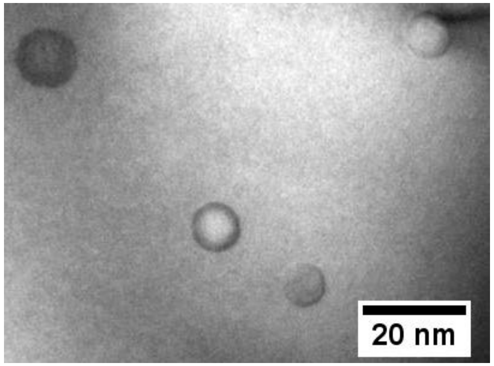


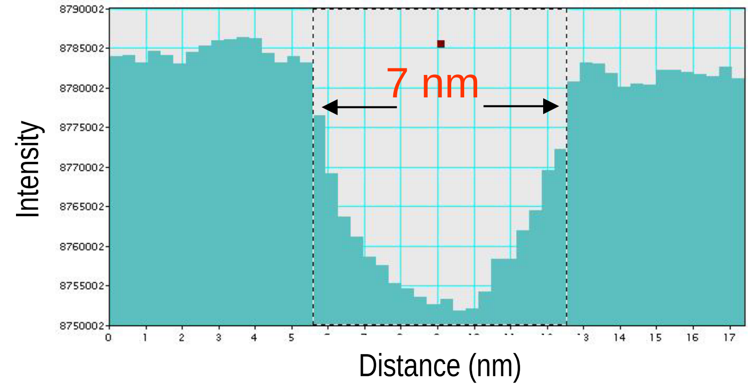






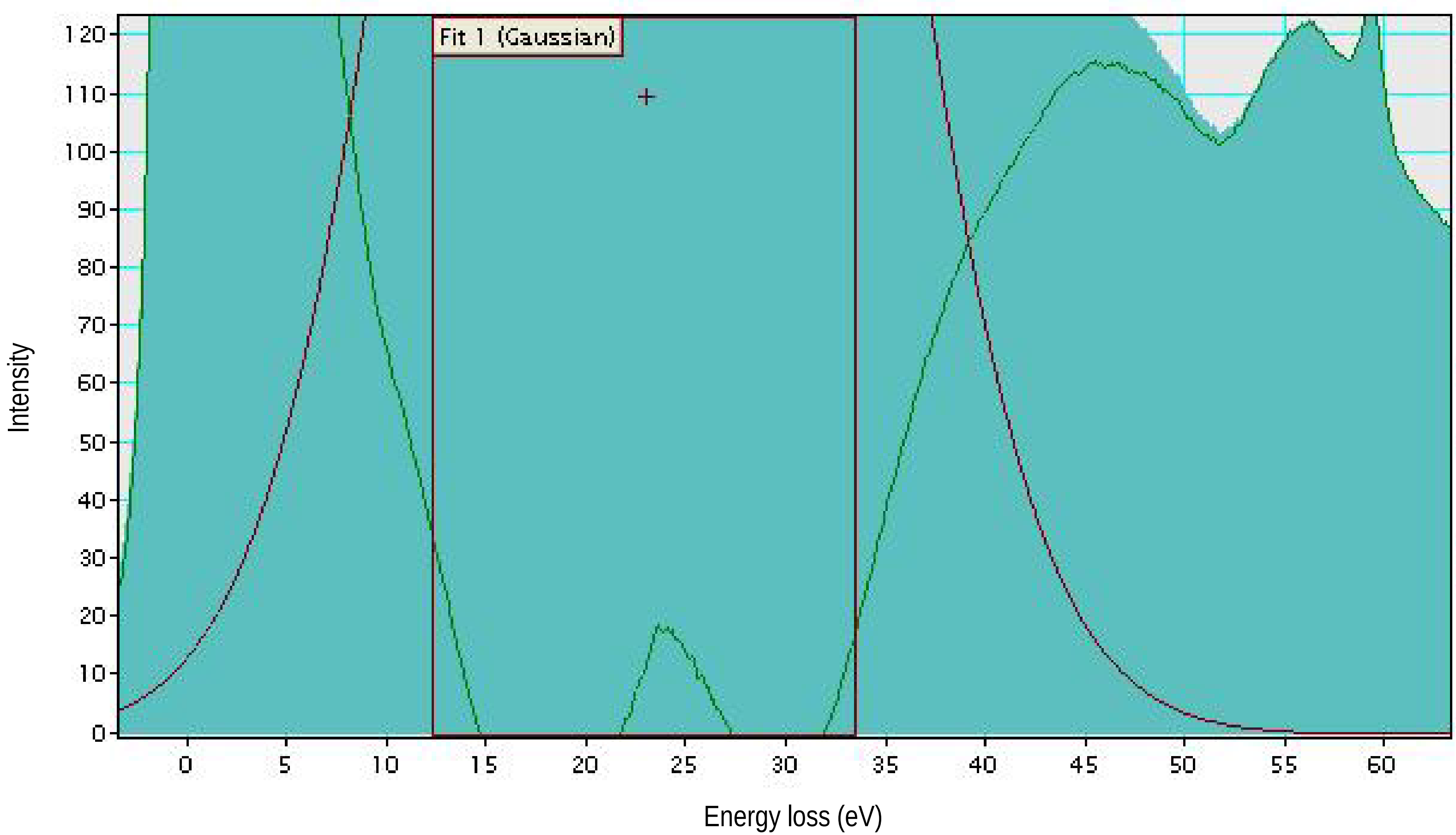






STEM/EELS: Evaluation of the He number density

Number density of Helium: 1s – 2p transition



He number density n (atoms/nm³):

$$n_{He} = I_{He} / (\sigma_{He} \; I_z \; d)$$

Iz ...intensity of the elastic peak

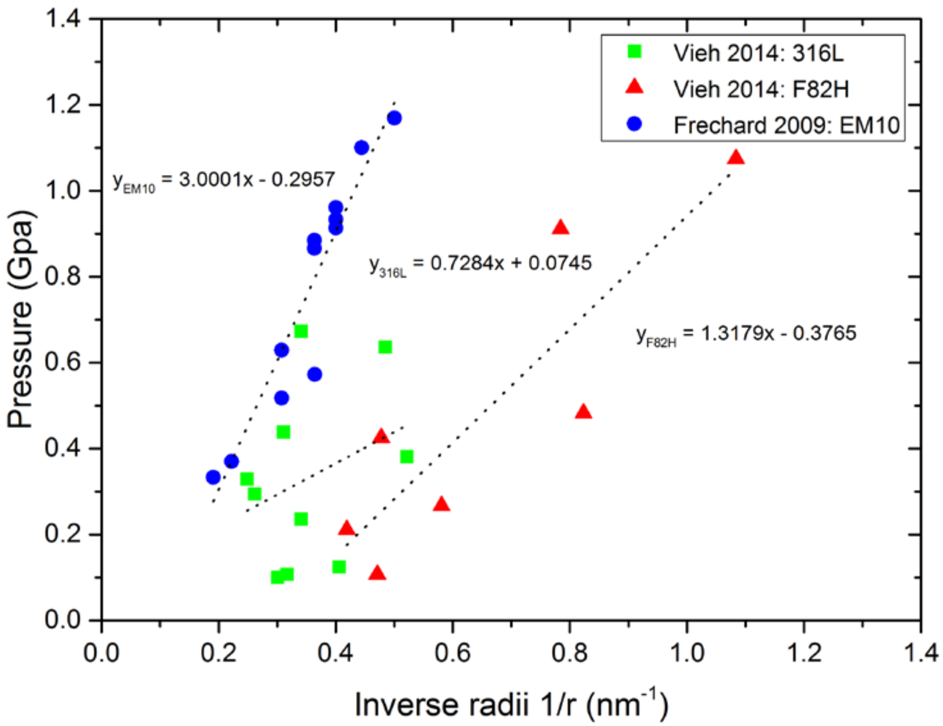
I_{He} ...intensity of He 1s-2p transition

d ...corresponds to the bubble thickness

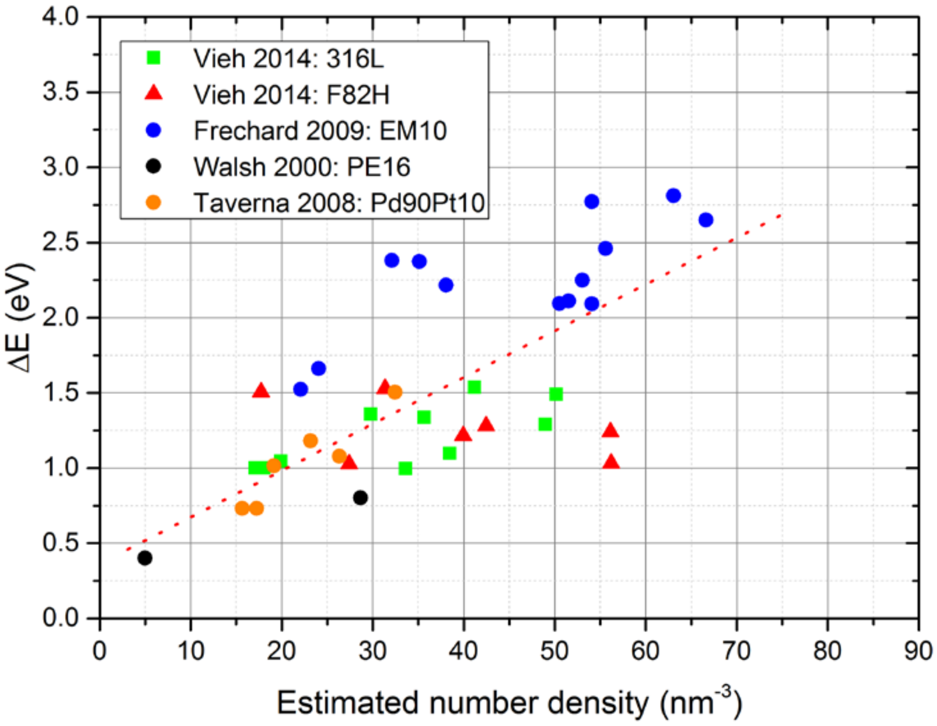
σ_{He} ...cross section of the helium 1s-2p transition

Pressure estimation of fcc & bcc steel

Pressure vs. Inverse radius



E-shift vs. Estimated number density



	Pressure [GPa]	Radius [nm]	Matrix material
Walsh (2000)	0.3- 1.5	5- 10	PE16
Fréchard (2009)	0.3- 1.2	2- 6	EM10
Vieh (2014)	0.1- 1.1	0.9- 4	316L, F82H

Summary & Conclusion

STIP-II: F82H (6.2- 20.3dpa, 151-344° C, 465-1825 appm He)

Microstructural investigations

- Irr. damage - nanometric defect cluster (sizes: 1.29-3.44 nm)
- Small amounts of produced He (< 900 appm) no He-bubbles (limit in resolution => PAS)
  - After annealing (600° C/1h and 200h), no defect clusters anymore
- High density of small cavities already at 6.2dpa after annealing (600° C/200h)
- He-bubble size ↑ with dose and He-content but mainly with T (size mainly less than 2 nm).

Helium induced hardening

- Dispersed Barrier Hardeing (DBH) model
- Barrier strength of He-bubbles $\alpha_{He} = 0.15$
- Linear Superposition for barrier strength of defect clusters $\alpha_C = 0.41$.
- He bubbles are weak barriers but their contribution cannot be neglected due to their high density

Pressure in He-bubbles

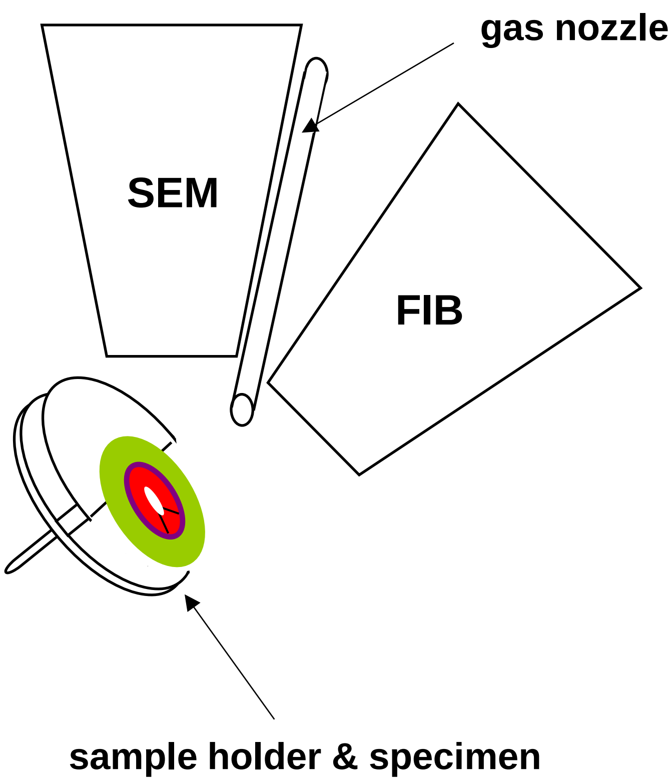
- Calulated from the estimated number density (20-60 nm^{-3}) using an EOS
- Pressure: 0.1- 1.1 GPa
- He- bubbles are underpressurazed

Many thanks too

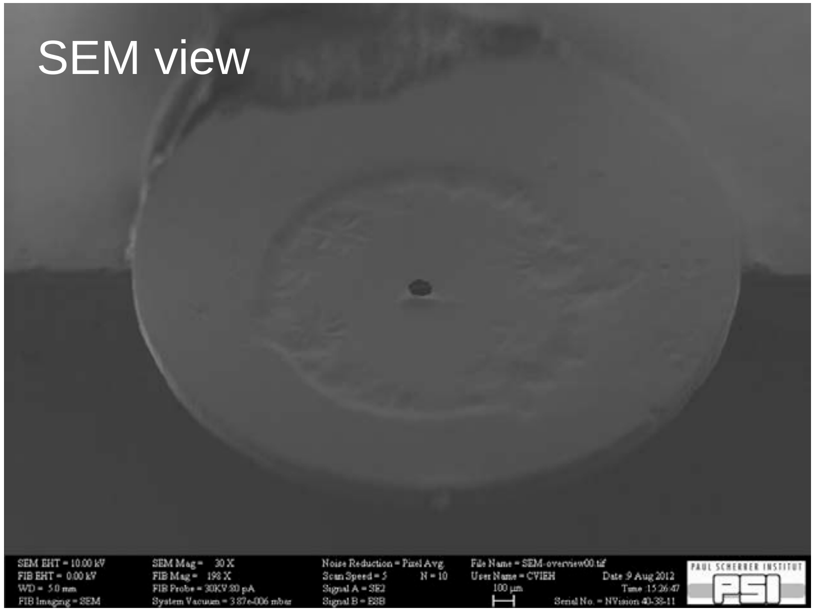
B. Roger, V. Krsjak, C. Gspan, G. Lucas, D. A. Alexander, C. Hébert, P. Spätig, Y. Dai

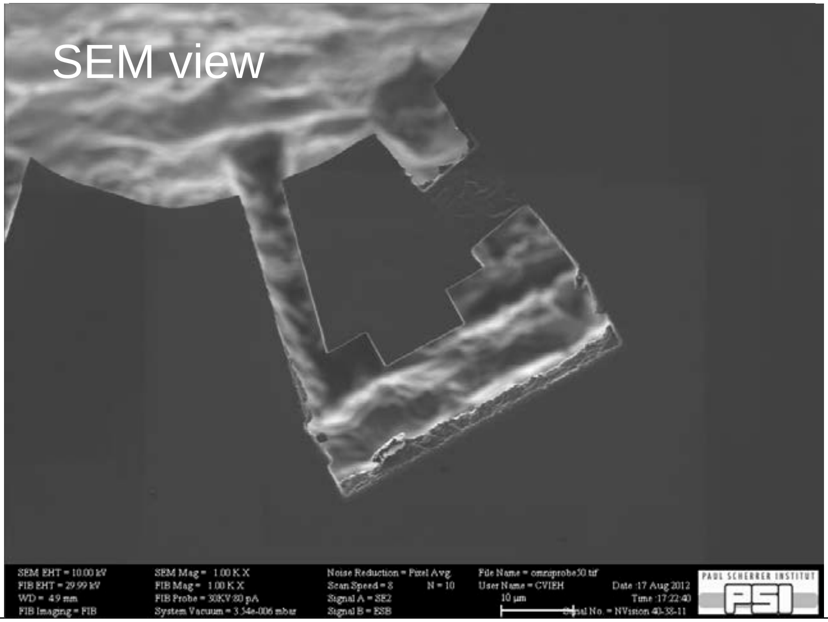
In-situ compression test in TEM

Specimen preparation via SEM/FIB



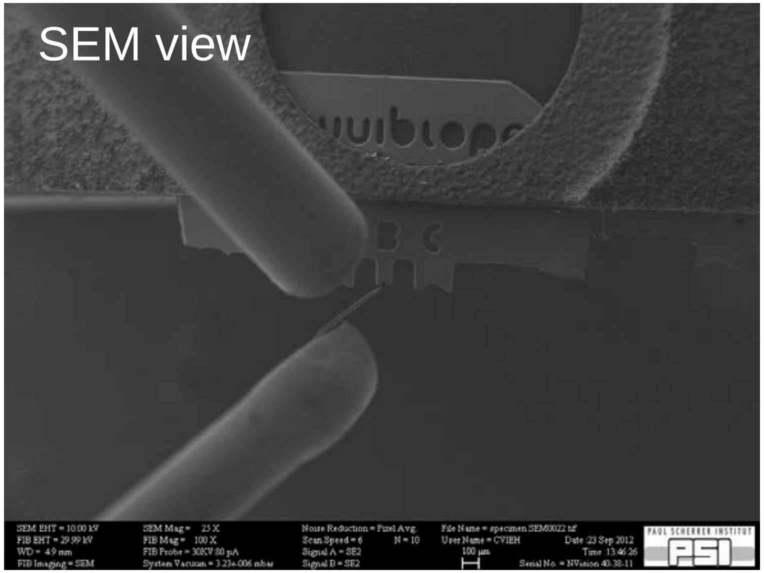
Sample position within SEM/FIB

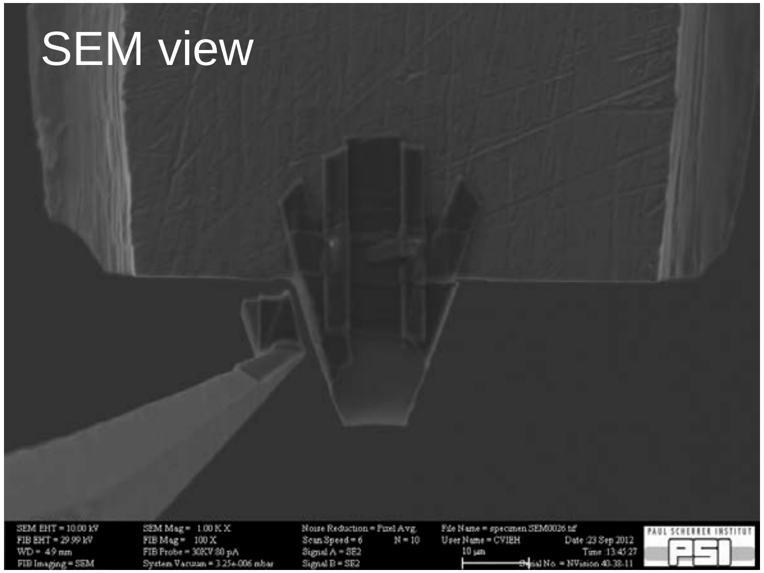


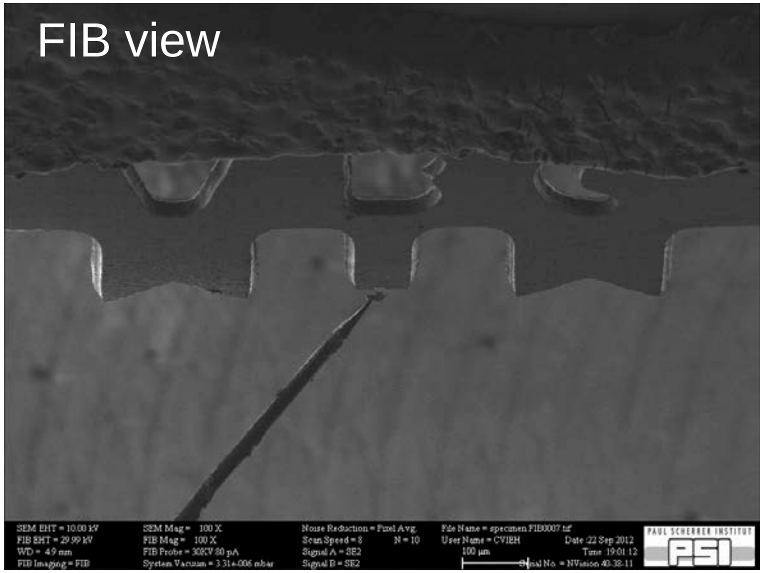


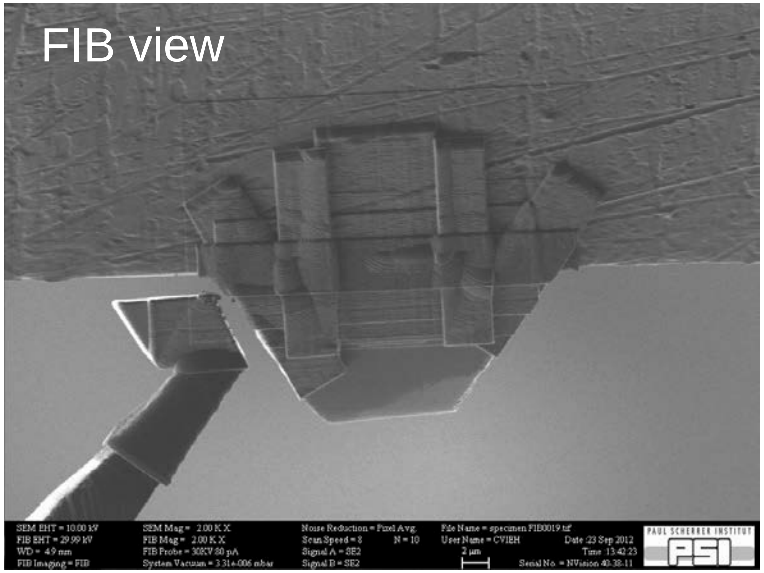
In-situ TEM: compression test

Specimen preparation via SEM/FIB







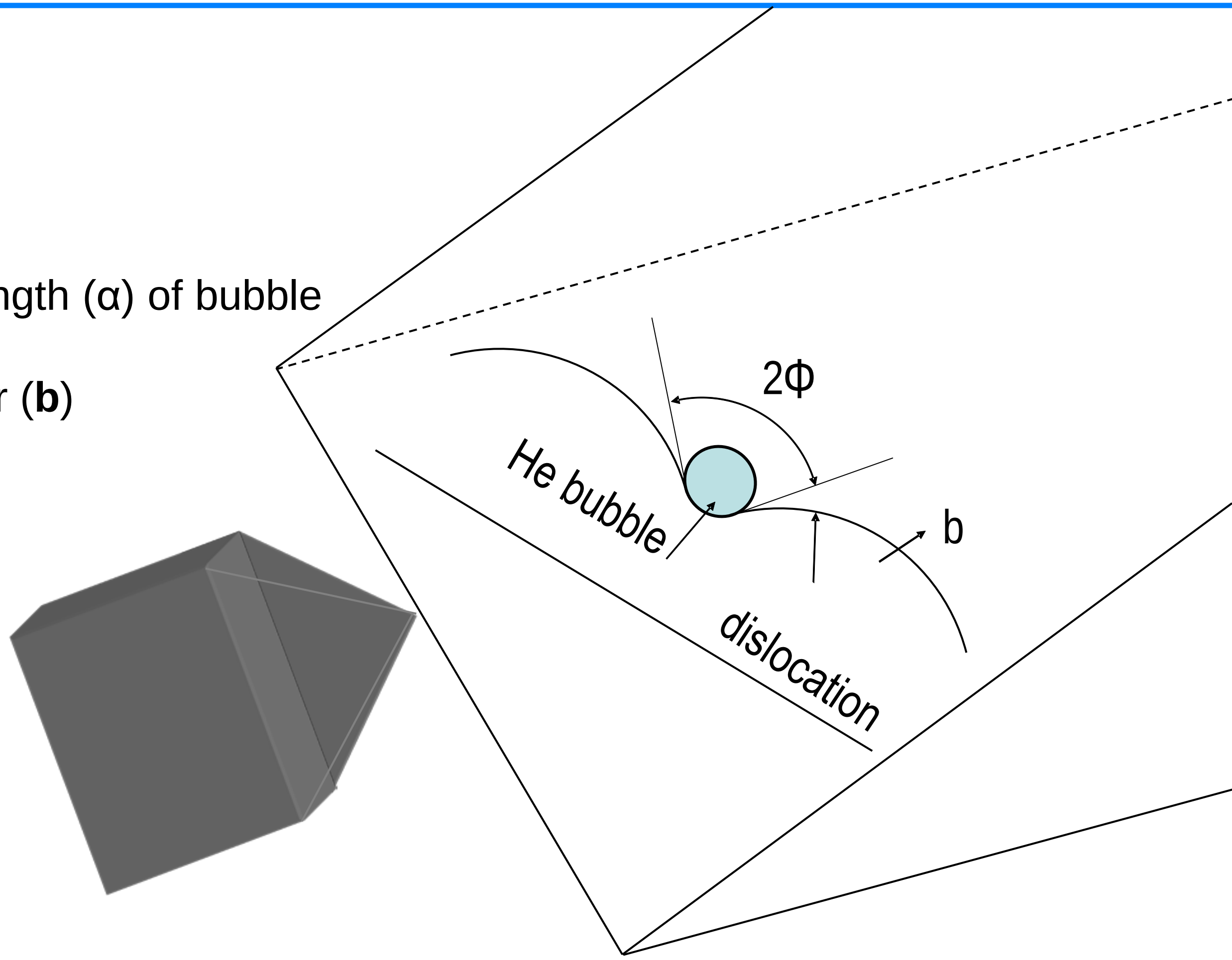


In-situ TEM: compression test

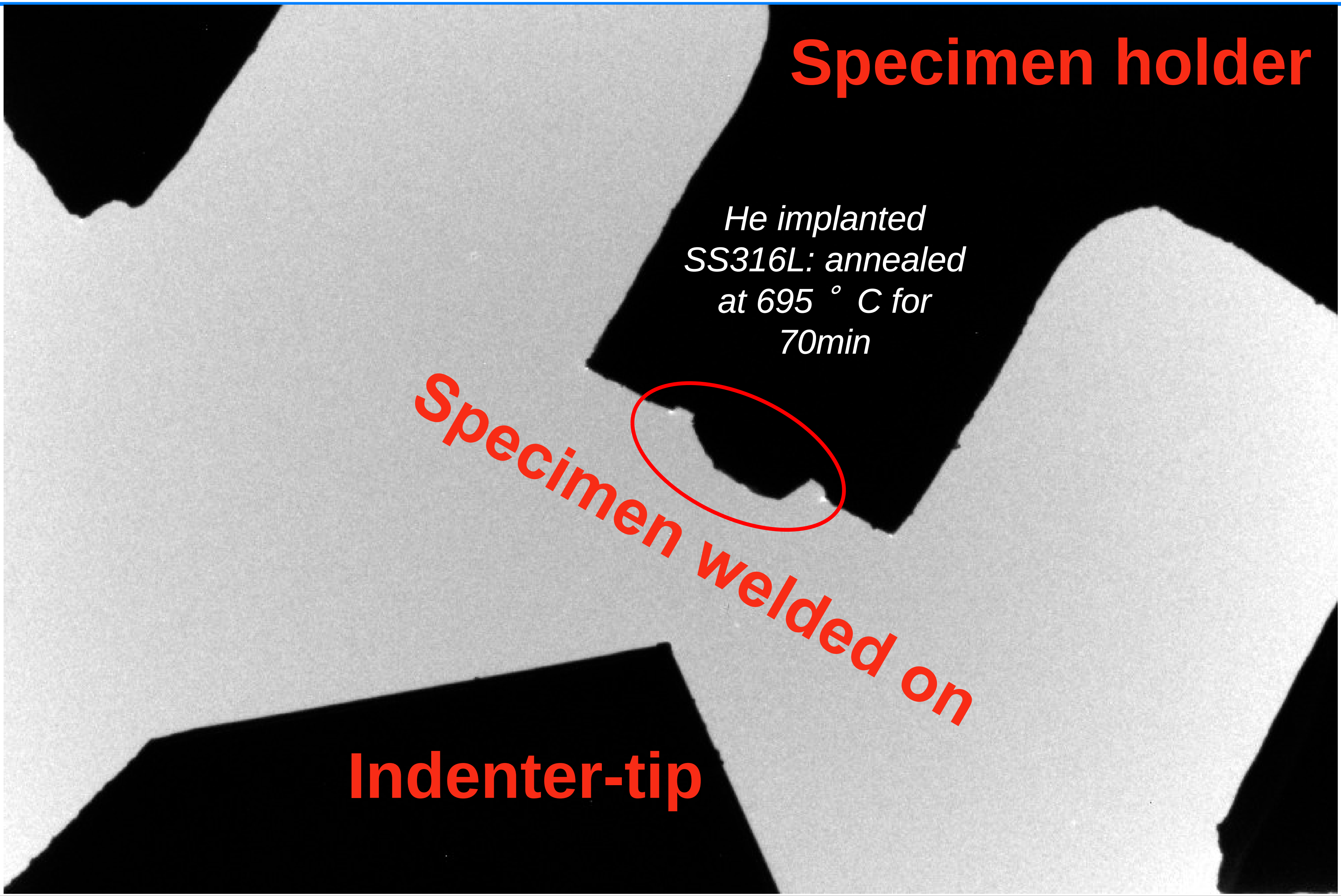
1. In-situ compression test

2. Post examination by TEM

- angle: cos Φ = barrier strength (α) of bubble
- g.b analysis: Burgers vector (**b**)
- stereo pair

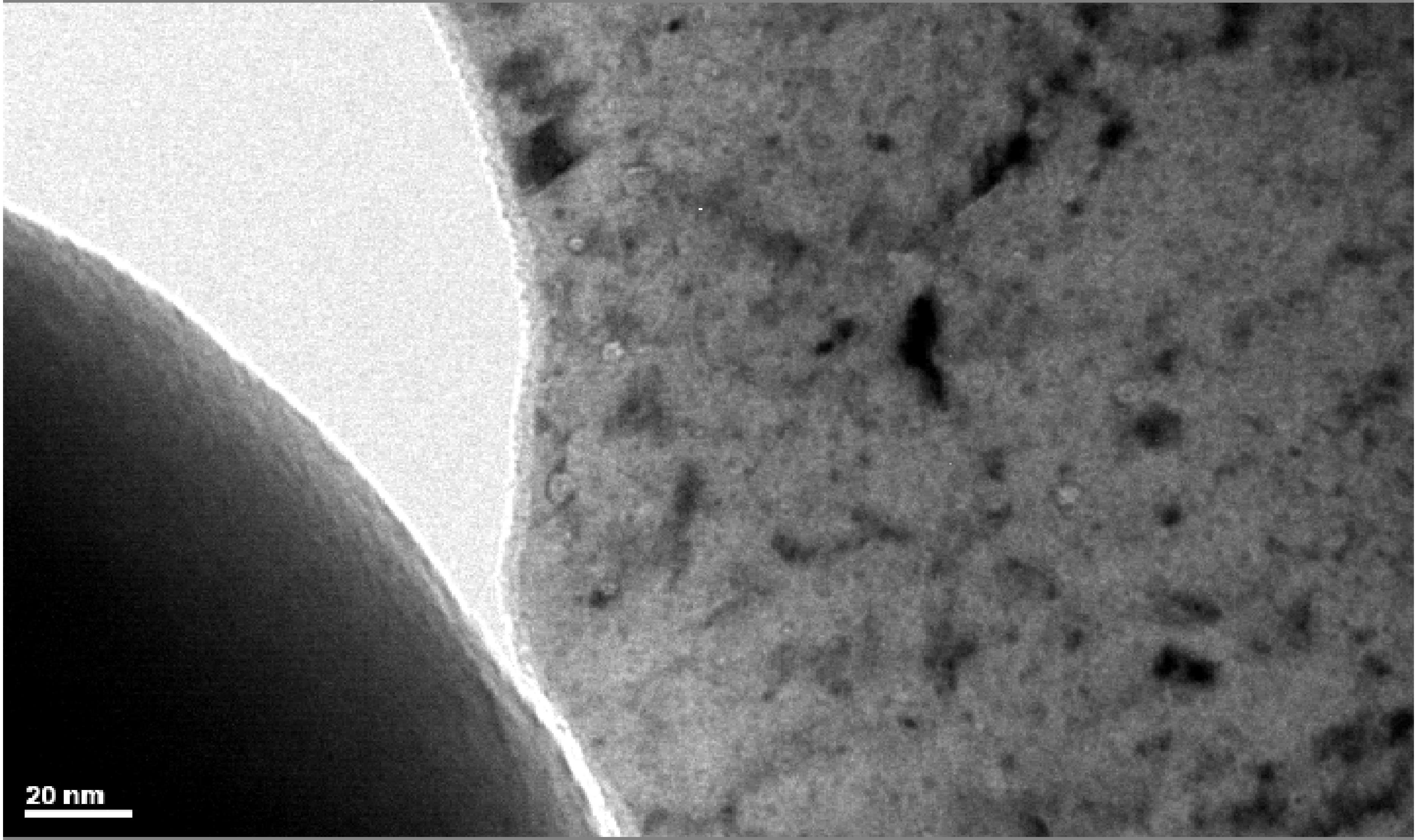


Future Prospects: In-situ experiment



Future Prospects: In-situ experiment

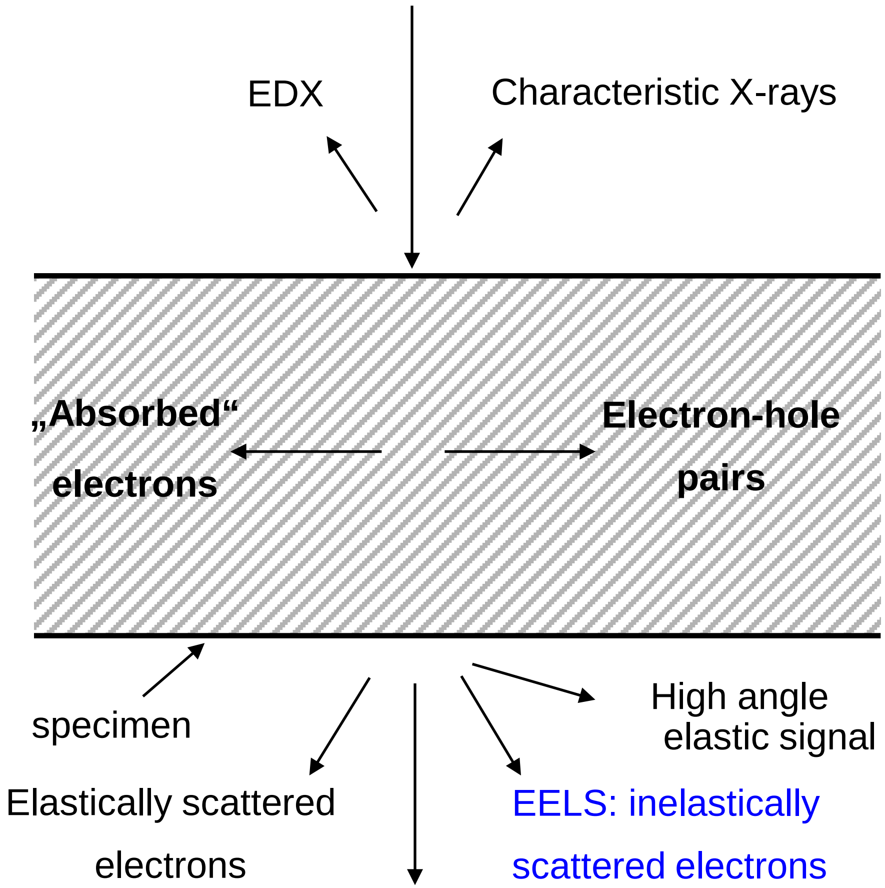
He implanted SS316L: annealed at 695 ° C for 70min



Basic Principles: Interaction of electrons with matter

Transmission Electron Microscope (TEM)

Incident beam intensity I_o



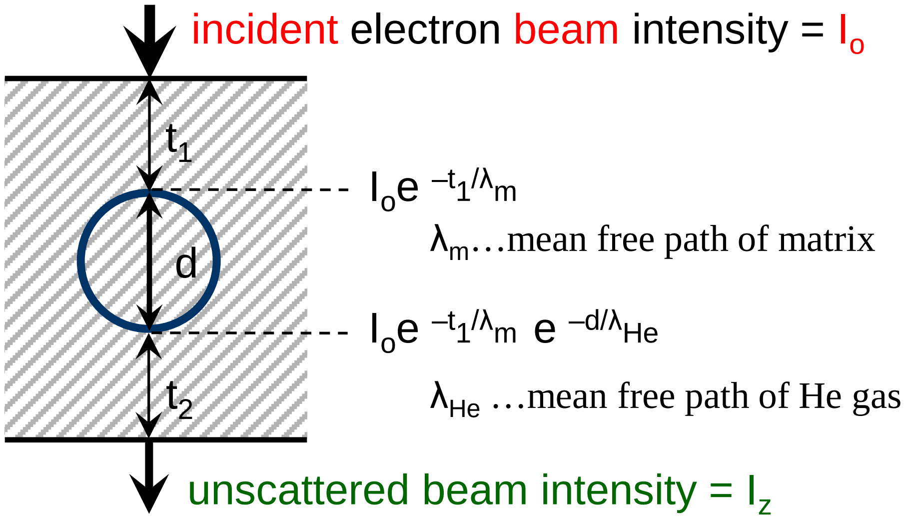
unscattered beam intensity I_z

Scanning TEM (STEM) Electron Energy Loss Spectroscopy (EELS)

Incident beam intensity I_o

reduced by

inelastic scattering events: matrix & bubble



$$I_z = I_o e^{-t_1/\lambda_m} e^{-d/\lambda_{He}} e^{-t_2/\lambda_m}$$