Research on radiation damage of fusion relevant materials carried out at Warsaw University of Technology

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Outline

- Part I: Transmission electron microscopy investigations of self-damaged tungsten targets
- Part II: Miniaturized sample testing at Faculty of Materials Science / WUT (**Z. Pakieła** et al.)

EUROfusion Task under: PFC.SP3.1 - Role of neutron damage on retention mechanism and strength in W / Be

Partners: Institute of Plasma Physics, Garching, Germany
Jožef Stefan Institute, Ljubljana, Slovenia

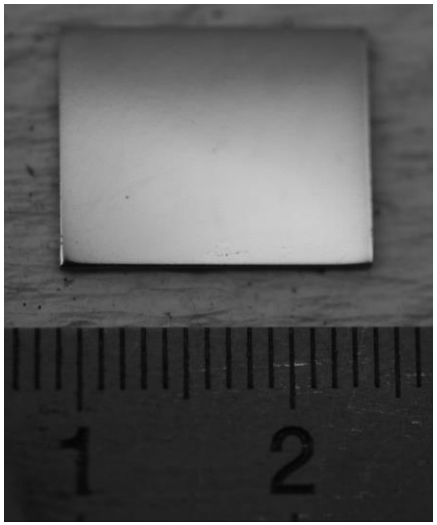
Goal: Evaluate defects morphology and distribution as well as dislocation density in the damaged W targets

Samples 2015

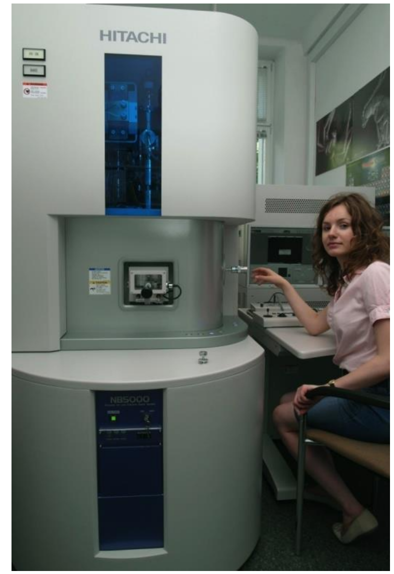
Sample number	Implantation conditions
1. A0780A	**20 MeV W ions, 0.5 dpa**, at 300K, **no pre-annealing**, exposed to D atoms at 450K
2. A0781A	**0.5 dpa**, at 300K, annealed at **600K** for 1h, exposed to D atoms at 450K
3. A0782A	**0.5 dpa**, at 300K, annealed at **800K** for 1h, exposed to D atoms at 450K
4. A0783A	**0.5 dpa**, at 300K, annealed at **1000K** for 1h, exposed to D atoms at 450K
5. A0785A	**0.5 dpa**, at 300K, annealed at **1130K**, exposed to D atoms at 450K

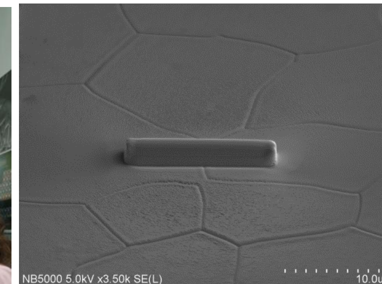
TEM on self-damaged W. Defect annealing.

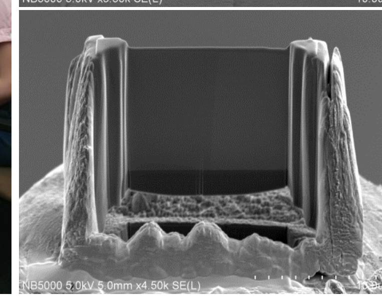
0. Self-ion damaged W plate from IPP
   W^{6+} ions, 3 MV



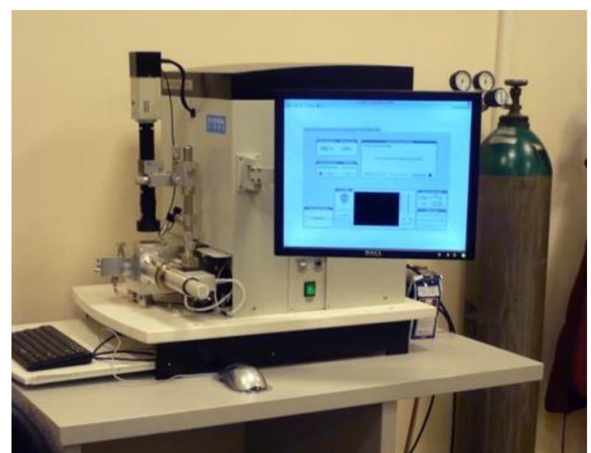
1. FIB milling – thickness 200-250 nm
   Ga ions, 40 kV, HITACH NB5000



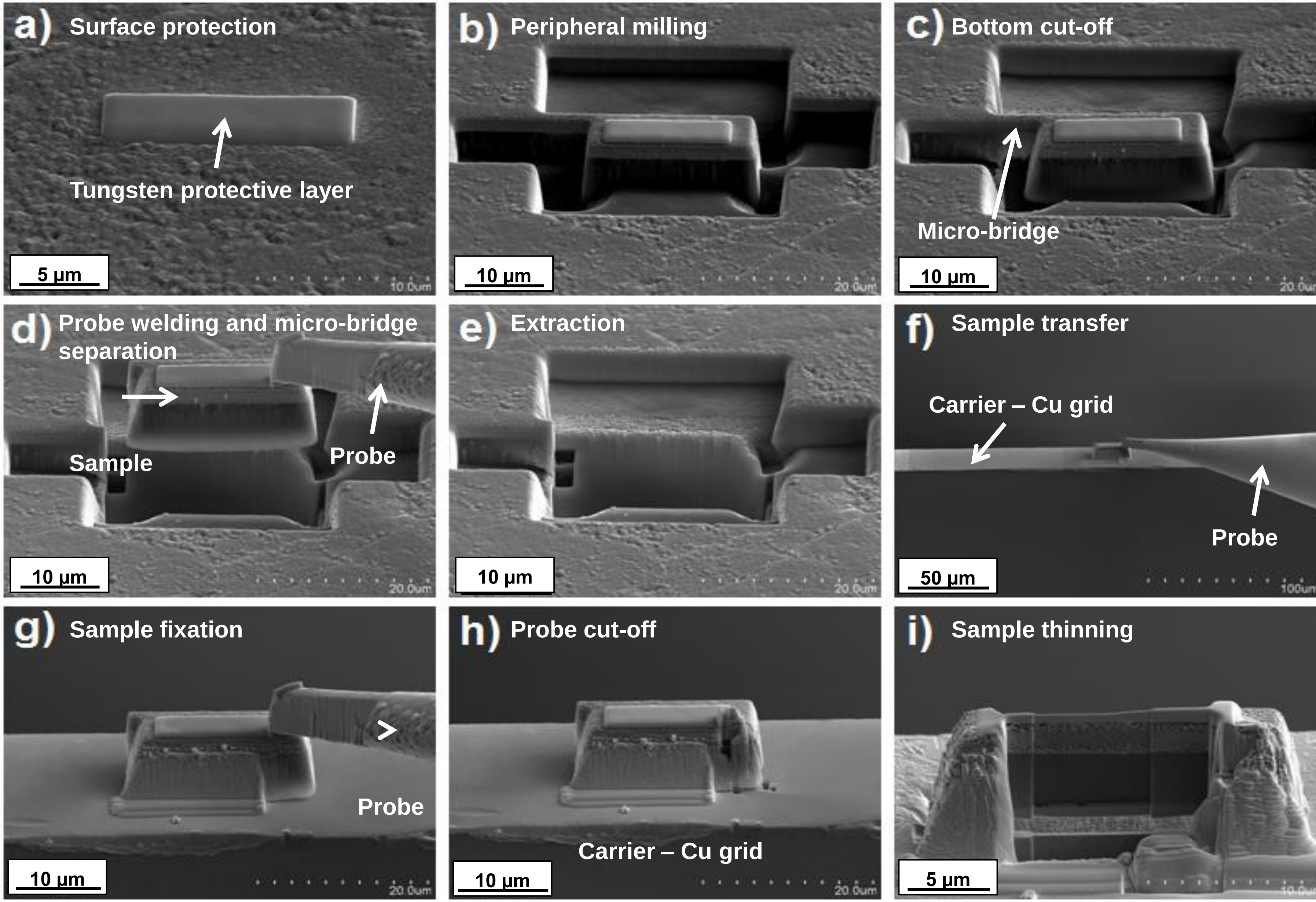




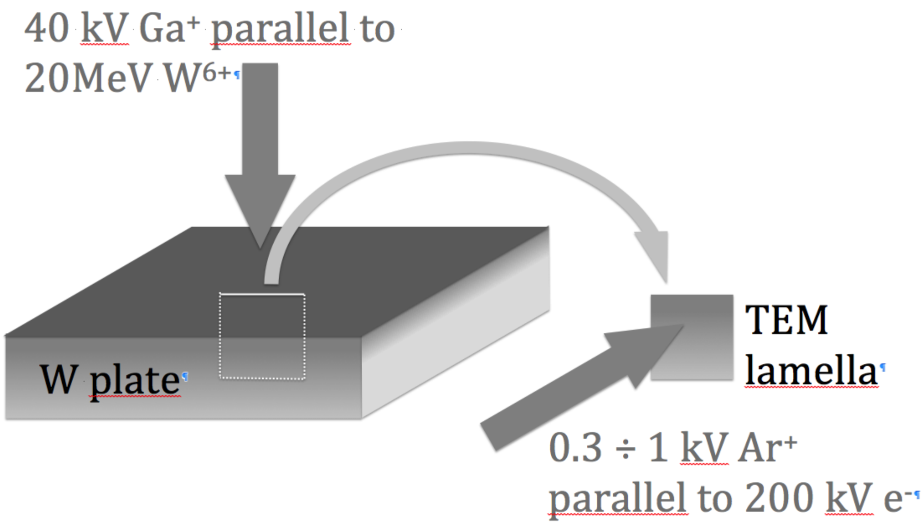
2. Thinning (gentle milling) –
   Ar ions, 1kV to 500 V

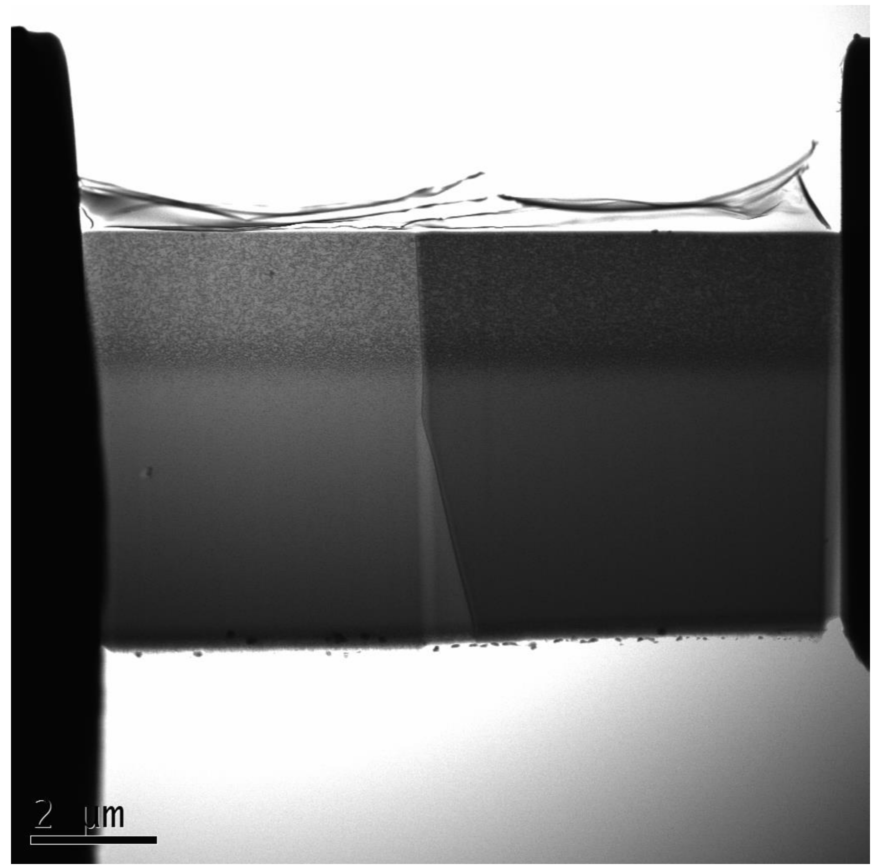


TEM on self-damaged W. Defect annealing.



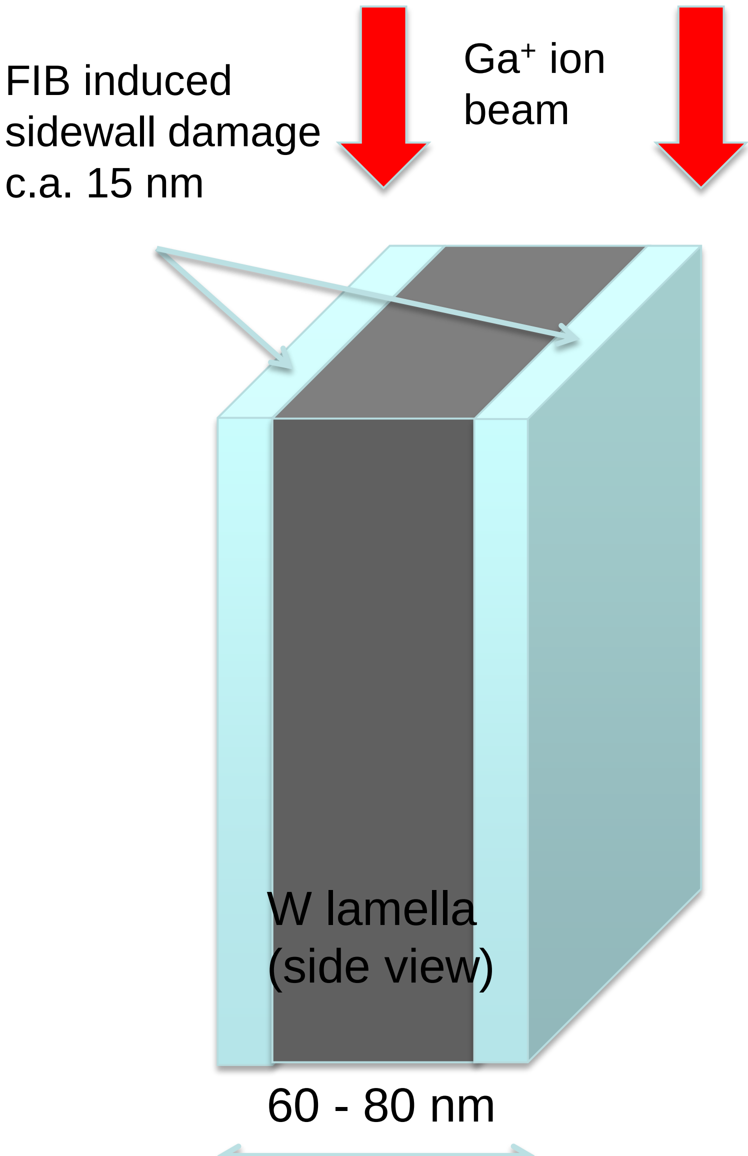
TEM on self-damaged W. Defect annealing.





W lamella

TEM on self-damaged W. Defect annealing.

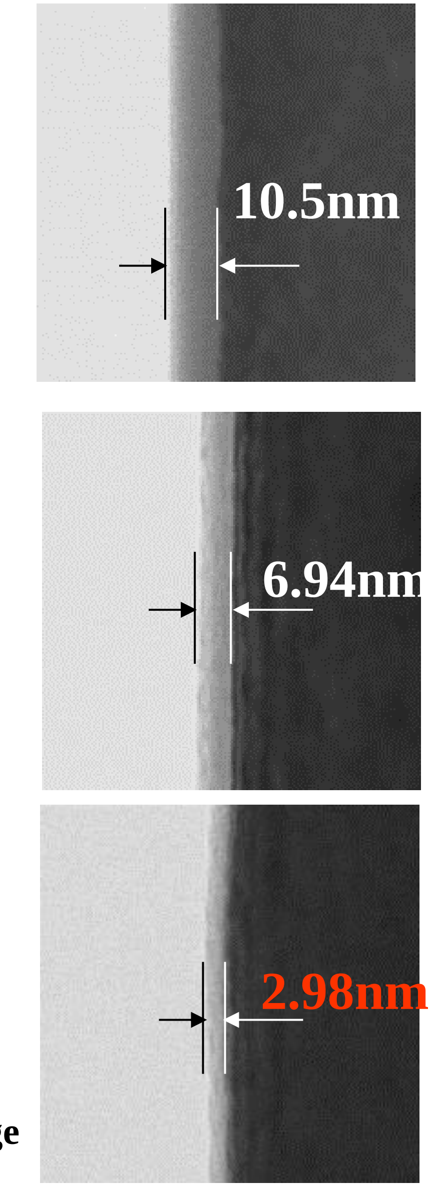


Damage layer thickness as a function of final polishing kV.

10kV

5kV

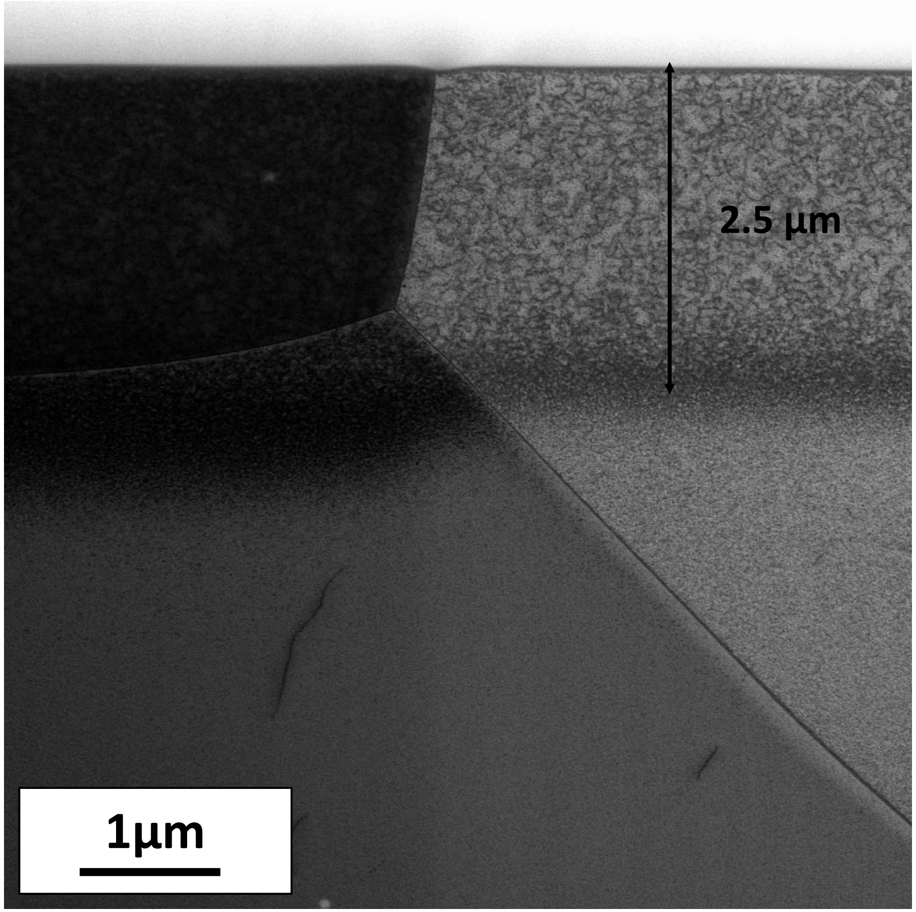
2kV



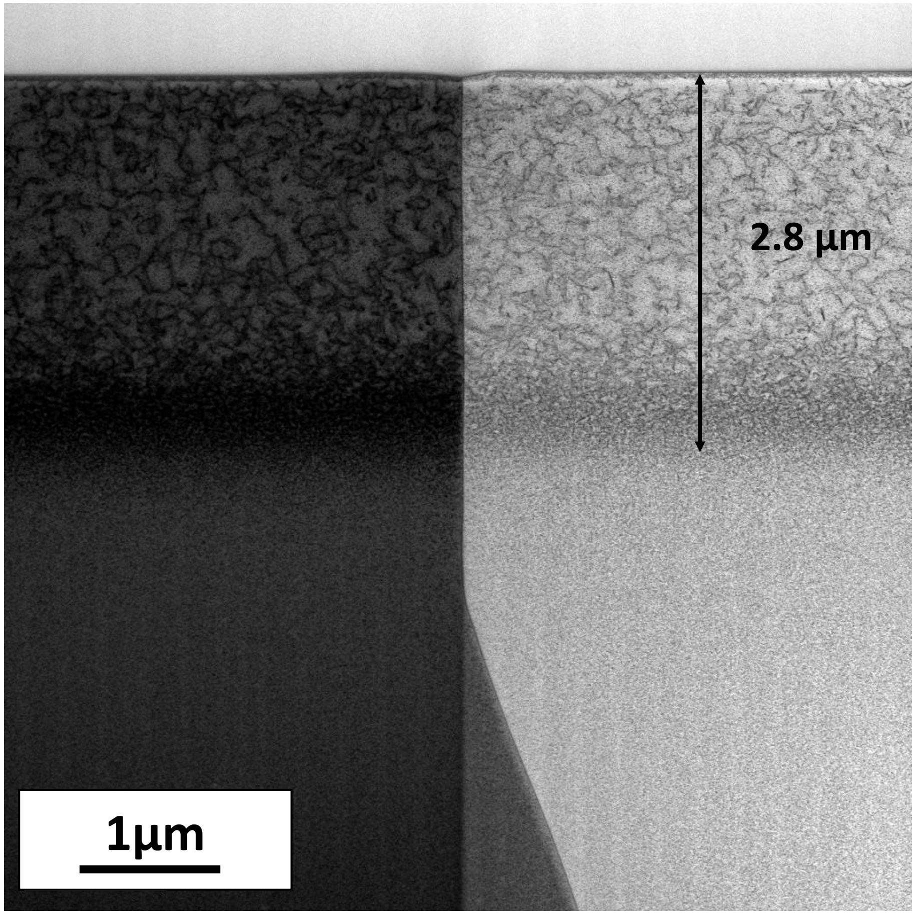
HITACHI Hi-Technologies
Specimen: Si single crystal
Remarks: 300kV TEM image

TEM on self-damaged W. Defect annealing.

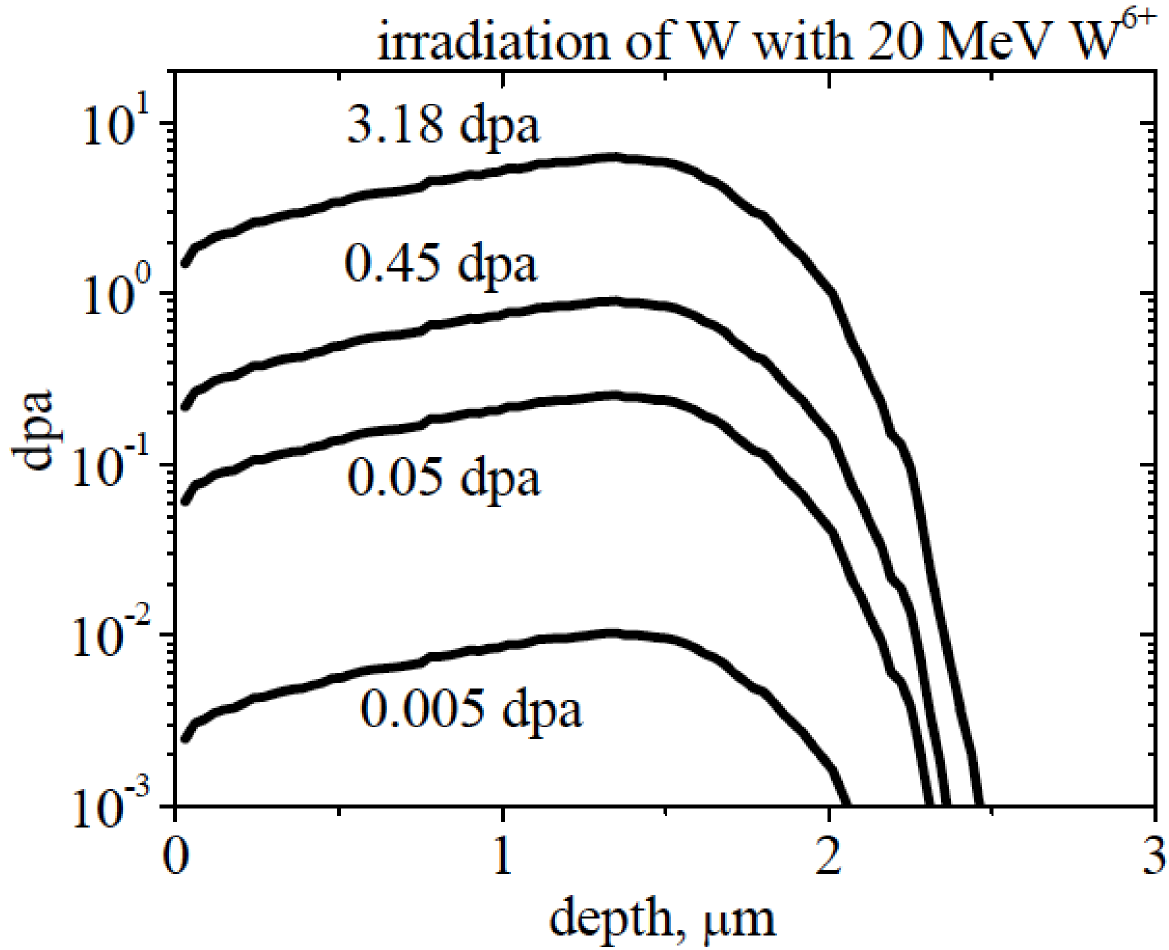
3.7*10^{18} W/m^2 (1.17 dpa)



1*10^{19} W/m^2 (3.18 dpa)



TEM on self-damaged W. Defect annealing.

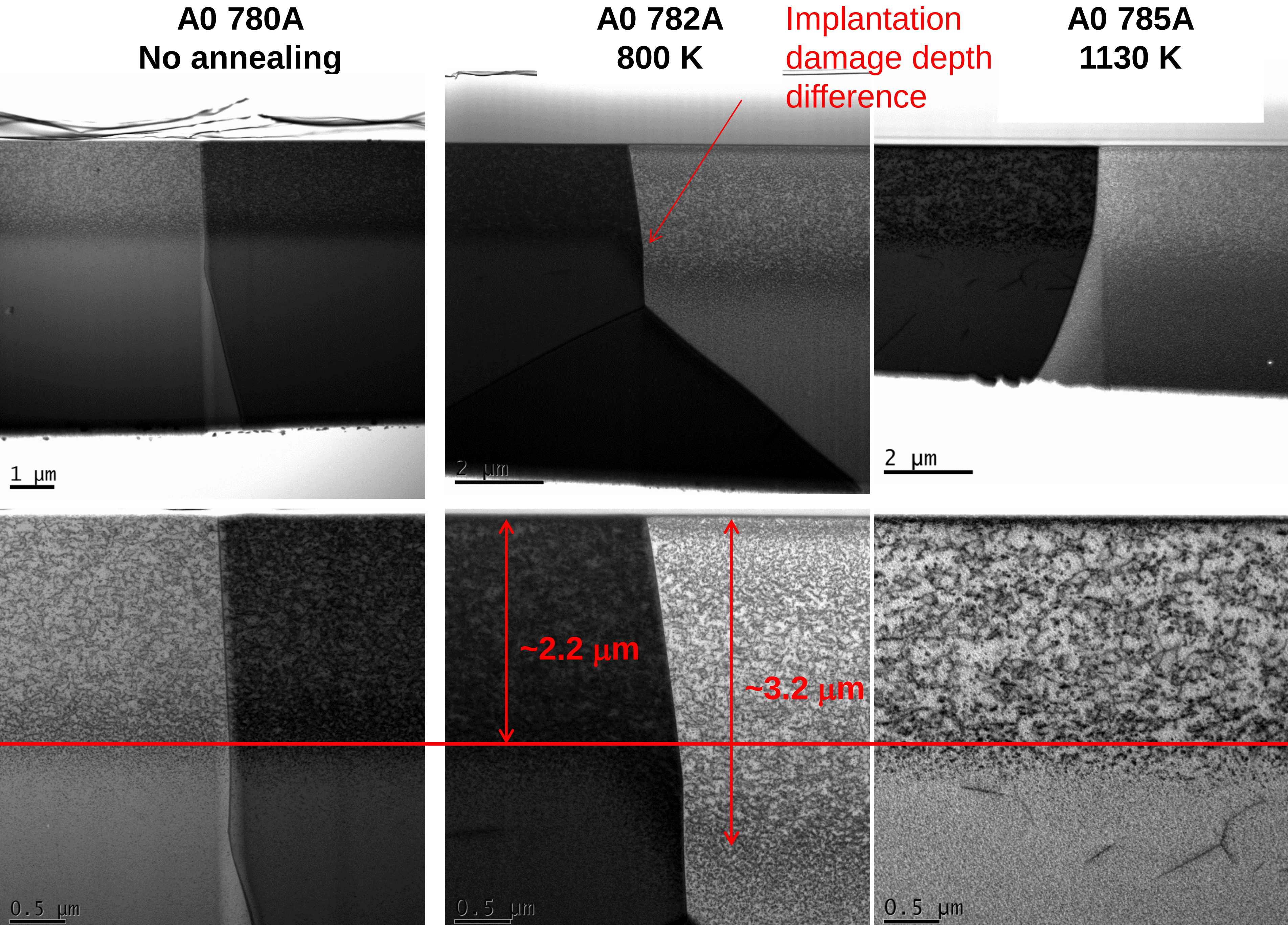


O.V. Ogorodnikova et. al., 2013, IPP

A0 780A
No annealing

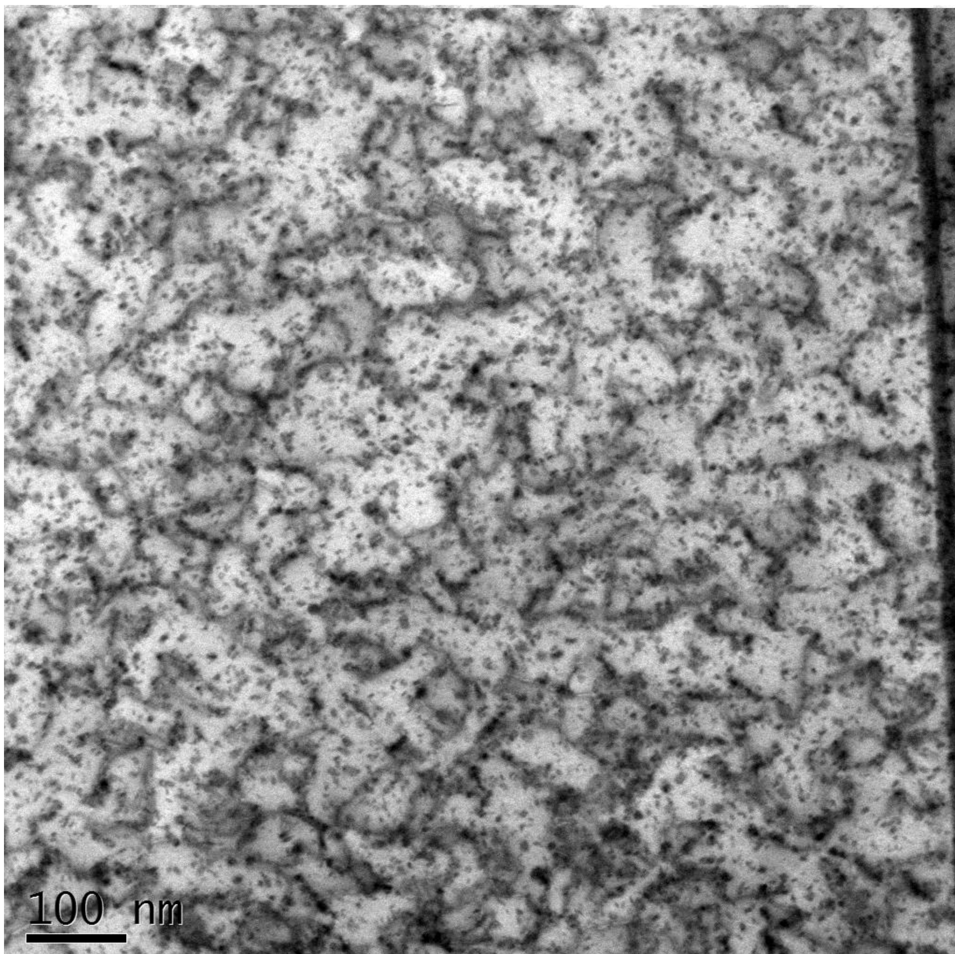
A0 782A
800 K

A0 785A
1130 K



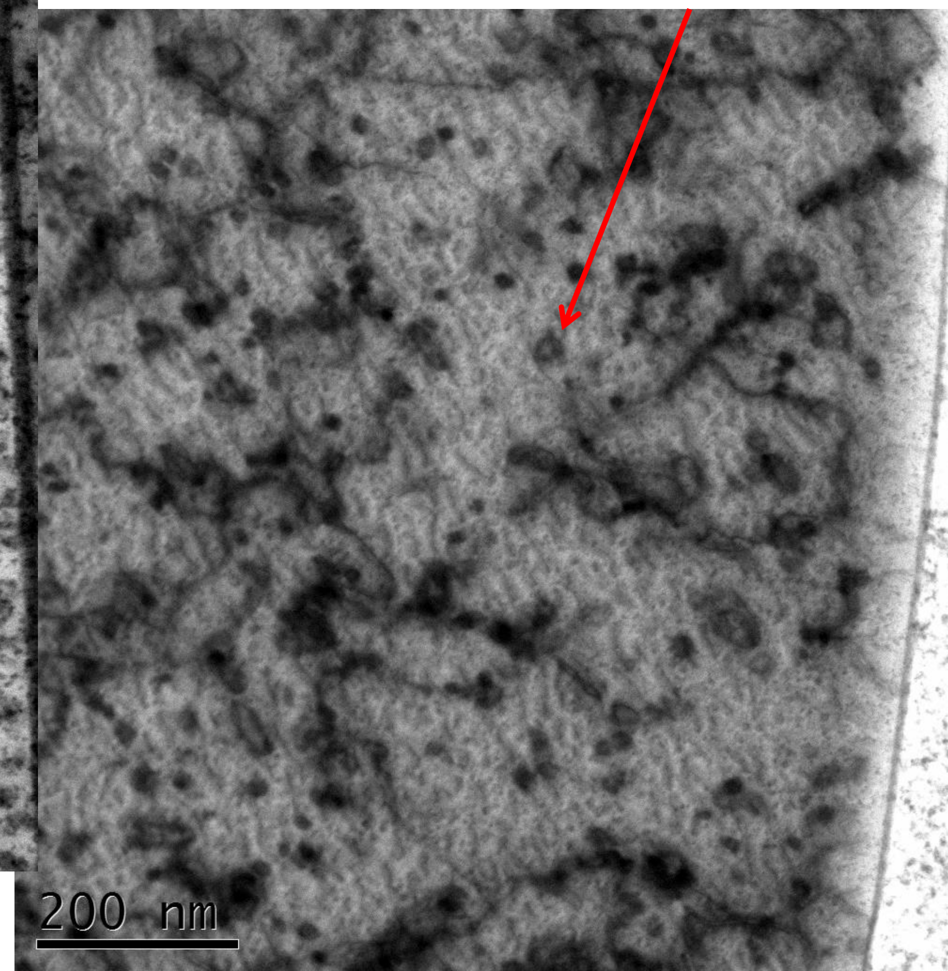
Research on radiation damage at WUT

A0 780A
no annealing

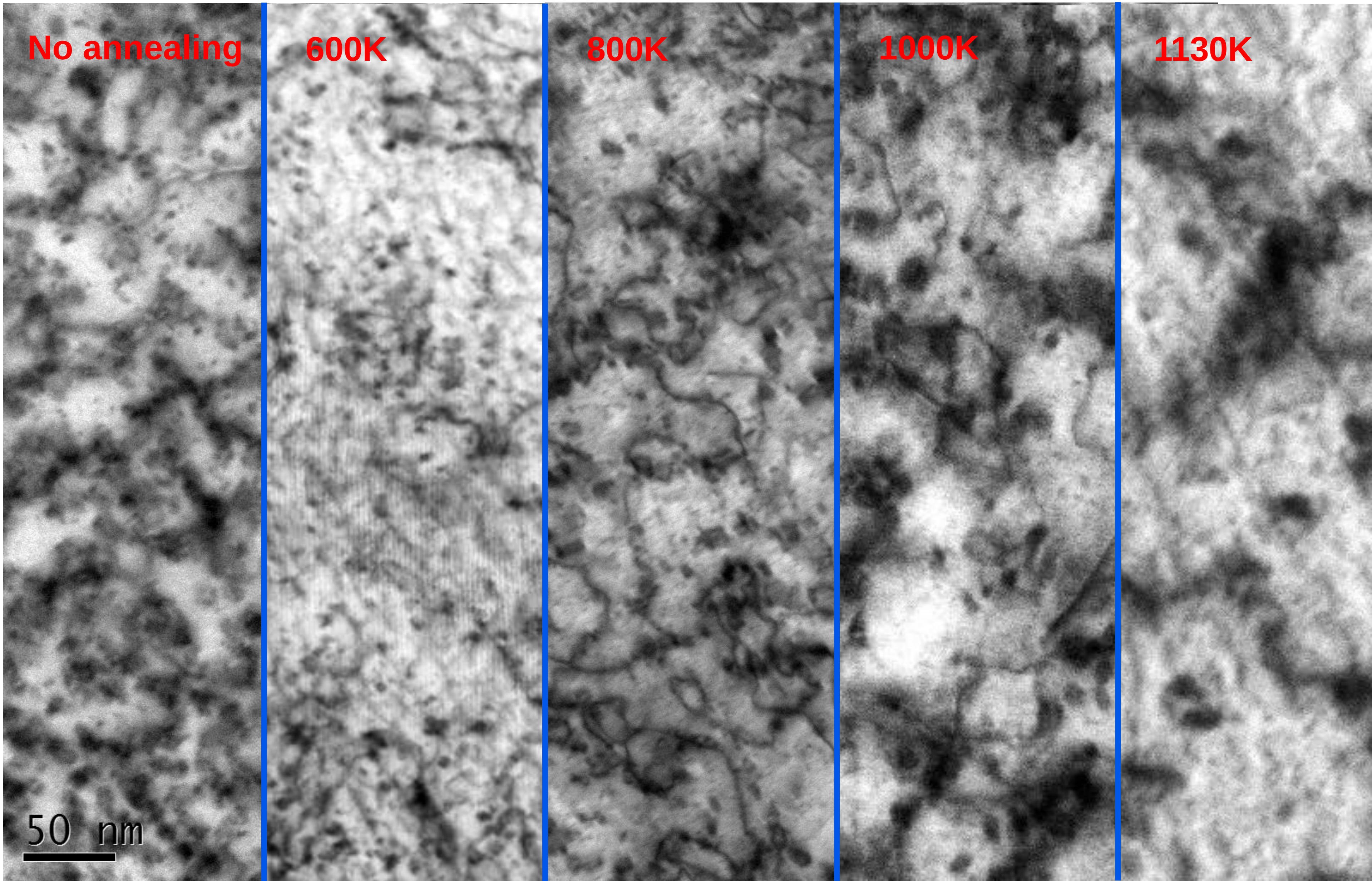


A0 785A
annealed
at 1130K

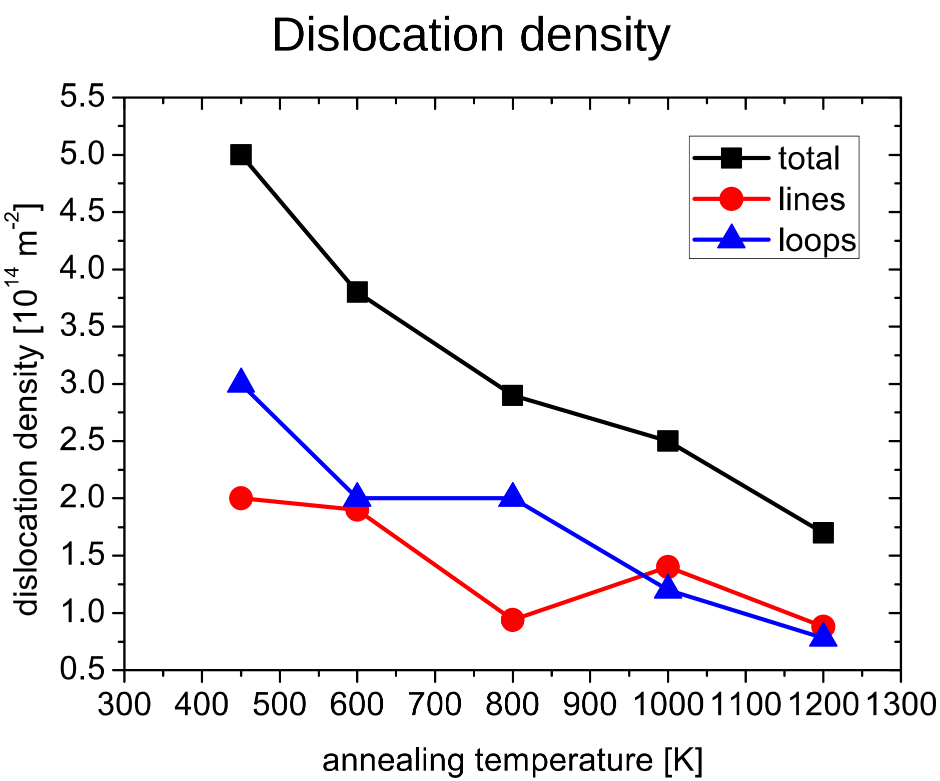
Larger dislocation loops + lower defect density

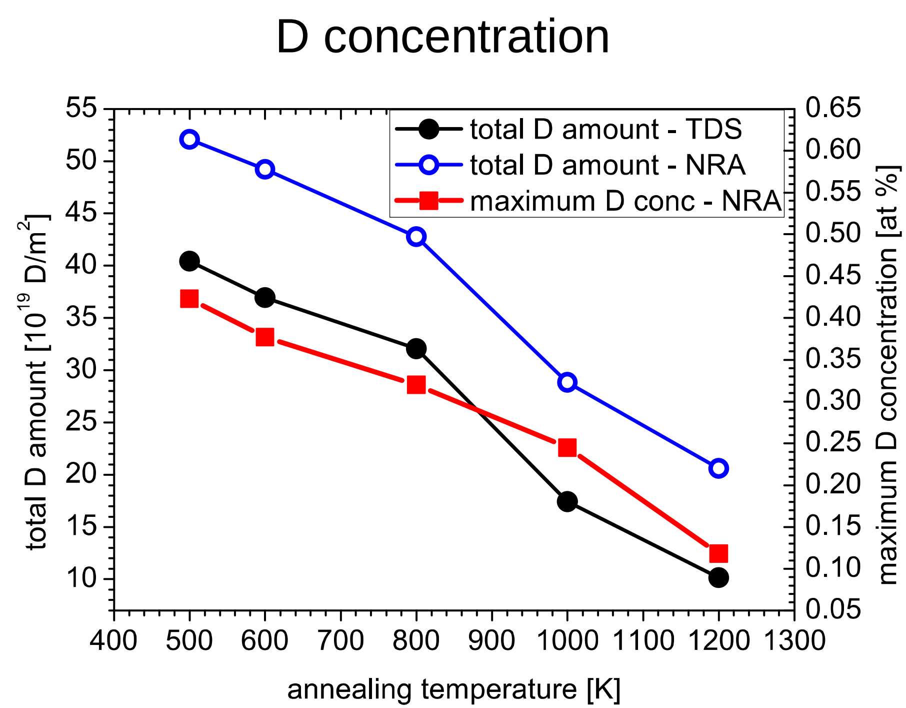


Research on radiation damage at WUT



Research on radiation damage at WUT





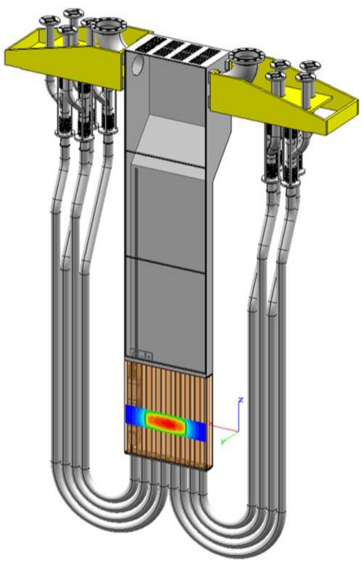
Data from JSI: A. Zaloznik, S. Markelj

“Dislocation density drops by **66 %** for the highest annealing temperature compared to the unannealed sample. This is in good agreement with the total D concentration results obtained by NRA and TDS, where also **60 %** decrease was observed.”

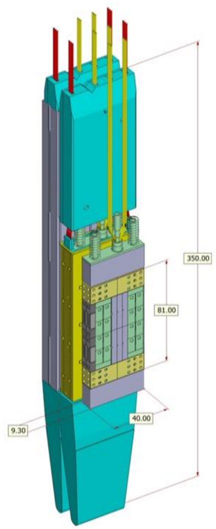
Anže Založnik, Sabina Markelj, Thomas Schwarz-Selinger, Łukasz Ciupiński, Justyna Grzonka, Primož Vavpetic, Primož Pelicon, The influence of the annealing temperature on deuterium retention in self-damaged tungsten, Phys. Scr. T167 (2016) 014031 (5pp)

Part II – Miniaturized sample testing

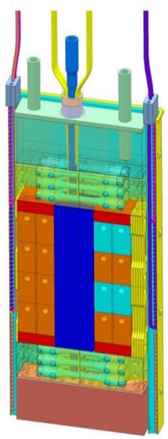
Why we need small samples in IFMIF / DONES?



HFTM Assembly



Packet of 3 Rigs



Cut-open capsule with specimens

Source: http://www.ifmif.org

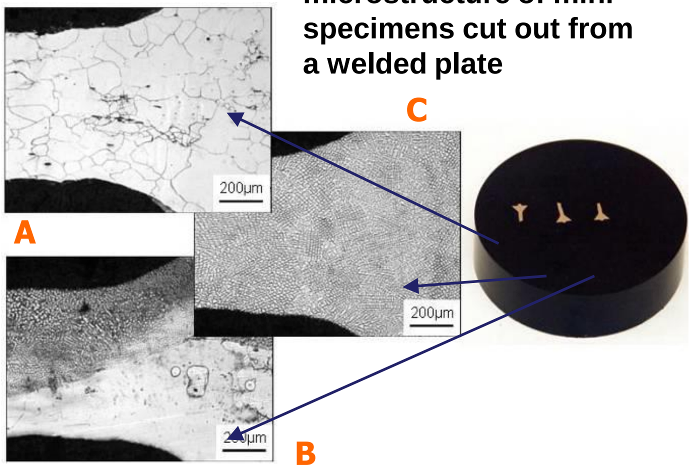
- Limited space for specimens in the irradiation capsule / limited HF volume
- For reliable engineering data for DEMO design many samples needed.
- Handling of active samples is “easier” when they are small

Is IFMIF / DONES the only reason to develop miniaturized sample testing methods?

Motivation to use miniaturized samples

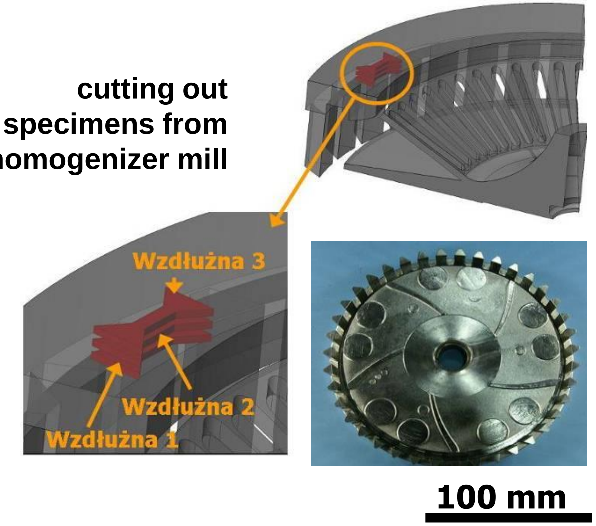
- Material / technology related: nanomaterials, weld heat affected zones, layers and coatings, gradient materials
- Design related: small cross section, complex shape (e.g. gear parts)
- Cost related: low invasiveness of the inspection vs. repair costs

microstructure of mini specimens cut out from a welded plate



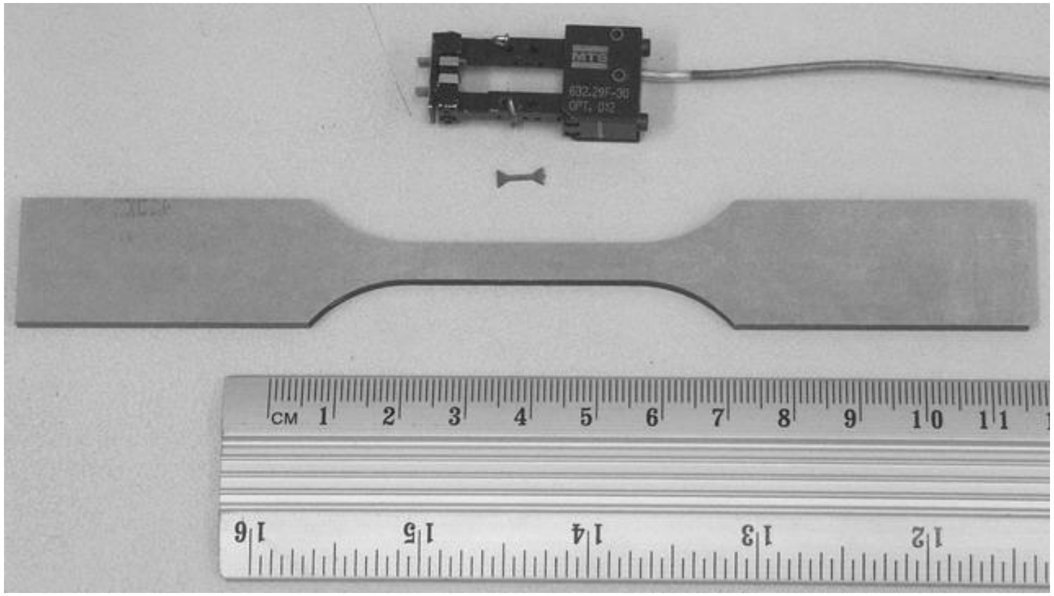
A-Plate 316L, B-HAZ+ weld, C-Weld

cutting out mini specimens from a homogenizer mill



Challenges

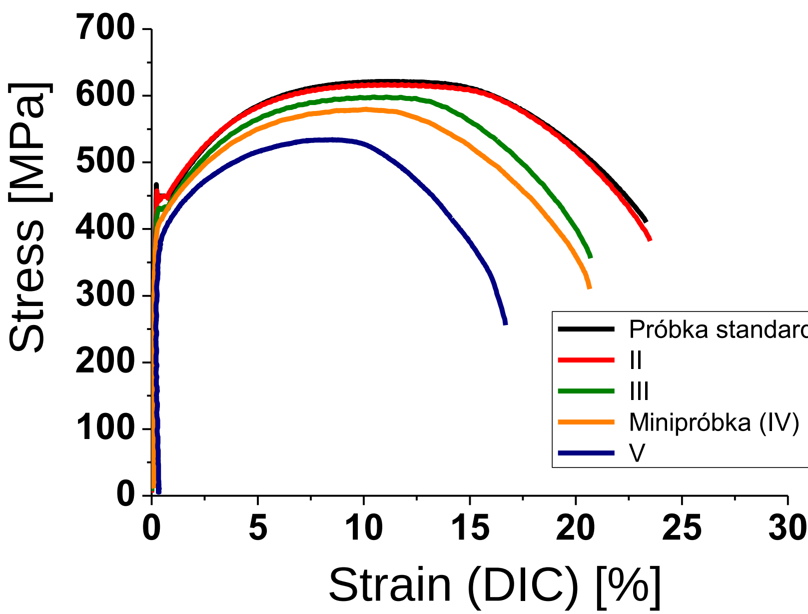
- Technical issues
  - Samples preparation
  - Gripping
  - Strain measurement
  - Alignment
  - Loading stability
  - …………..
- Size effect issues
  - Relative volume of “surface layer”
  - Microstructure elements / sample size relation
  - Sample geometry
  - ……………



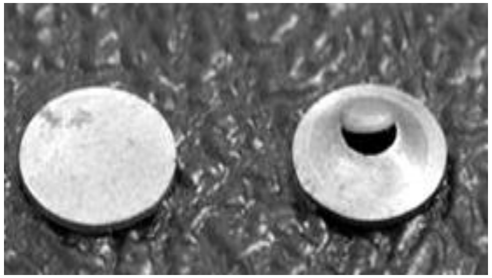
Standard and mini specimen for tensile test

Types of tests carried out at small samples at WUT

- Static Tensile Test
- Mini Disc Bend Test (MDBT)
- Fatigue Crack Growth (FCG)

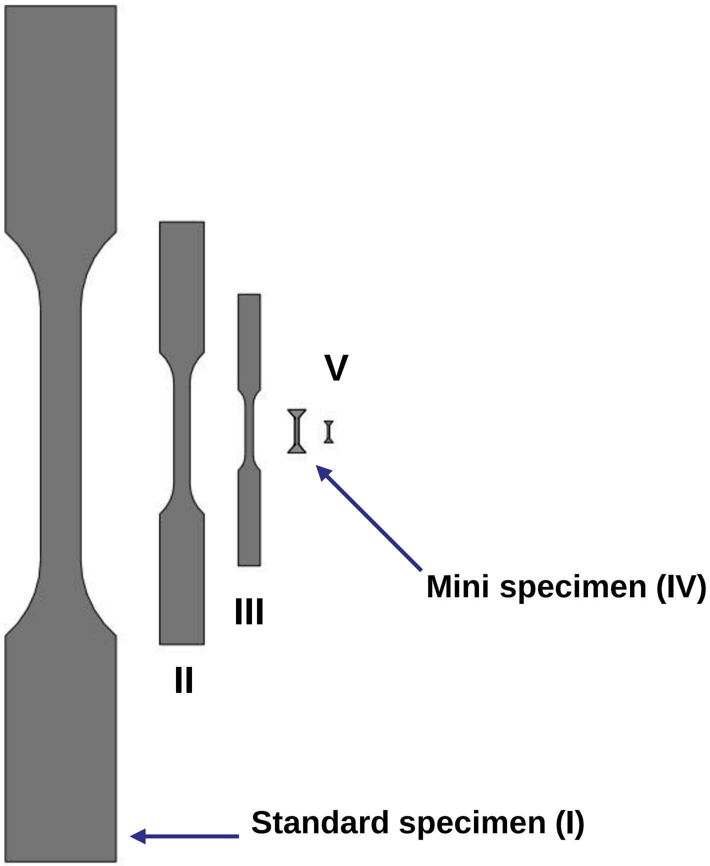


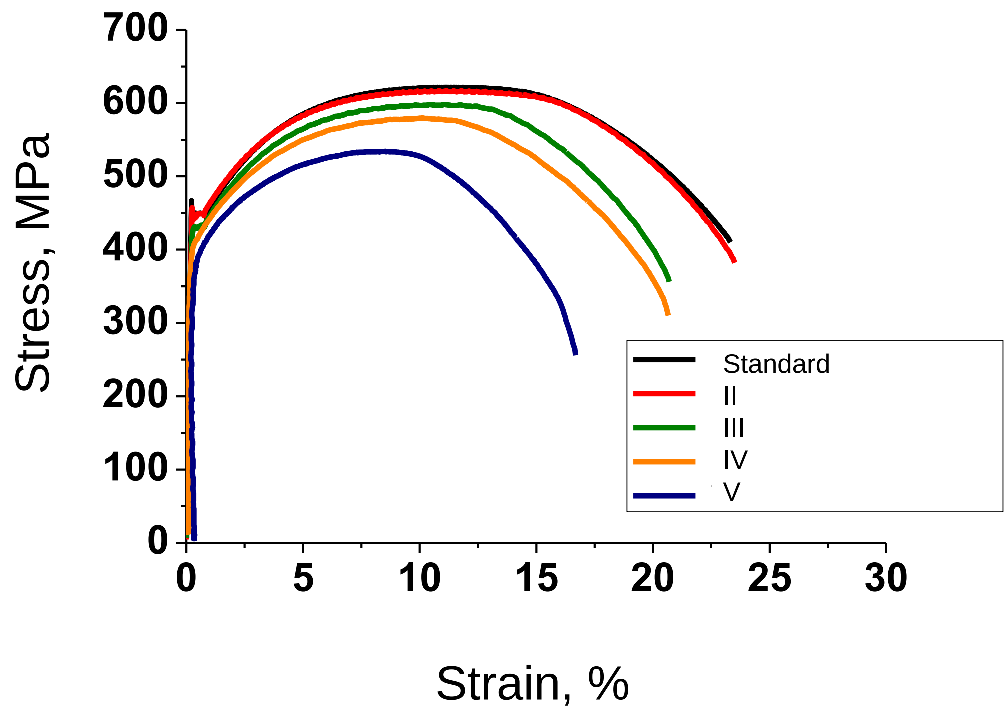
FCG test sample gripping system



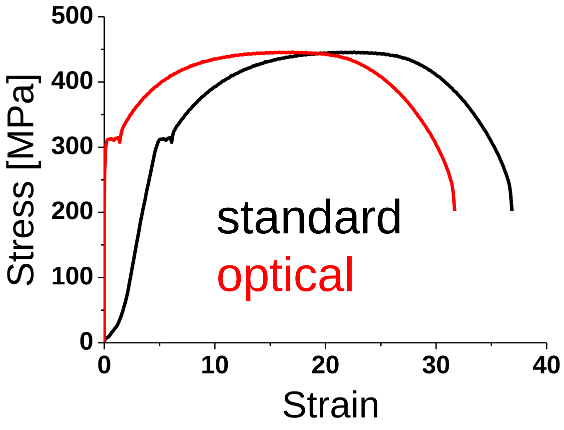
Samples before and after the MDBT

Size of specimens for static tensile tests

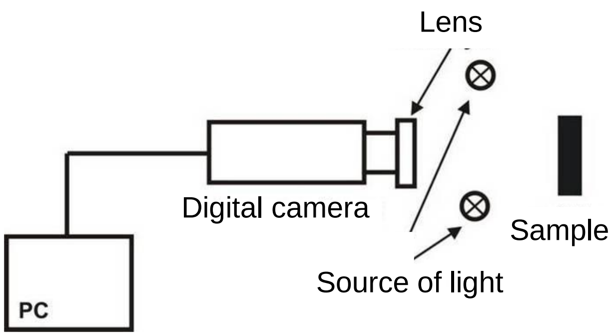




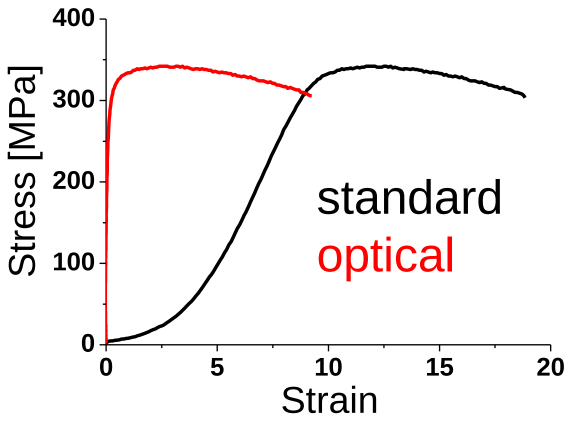
Reliable strain measurements – optical method

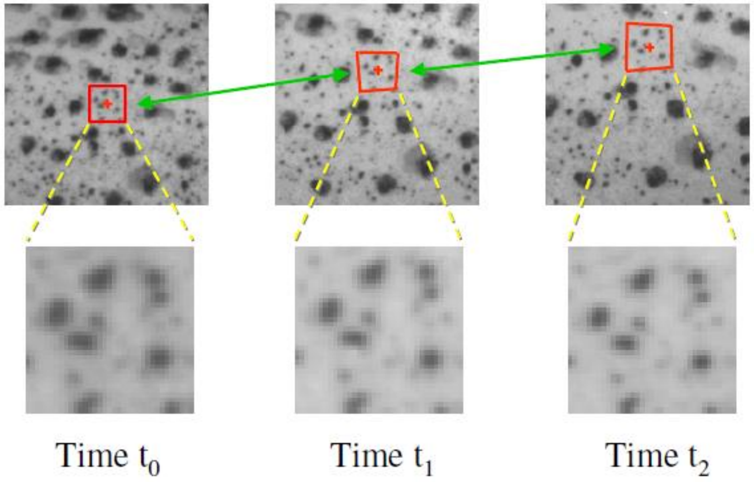


Stand for tensile tests

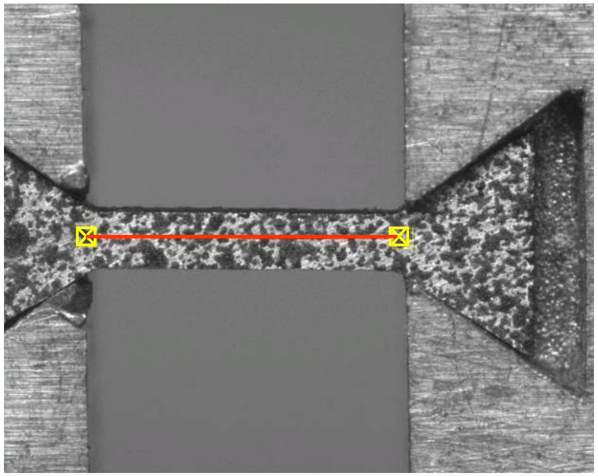


Scheme of DIC stand

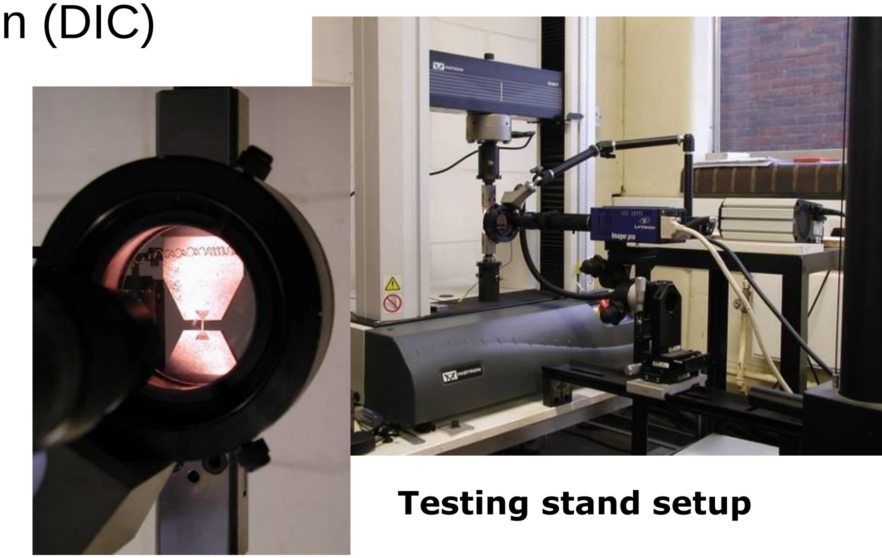




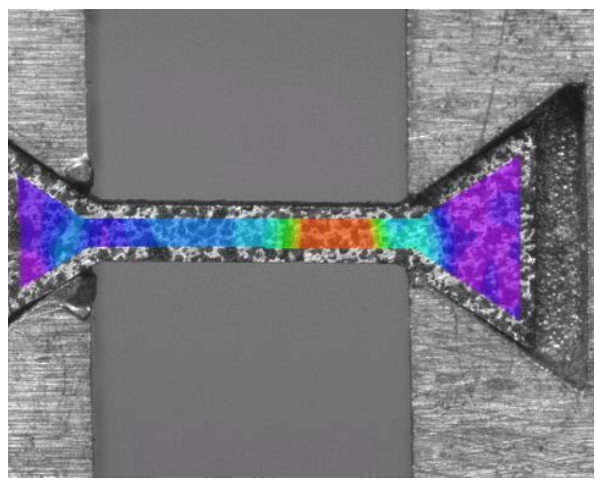
Optical method – Digital Image Correlation (DIC)



Optical extrensometer

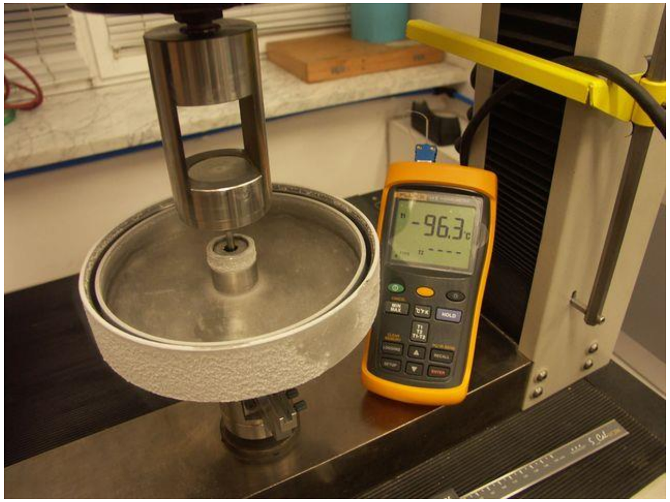


Testing stand setup

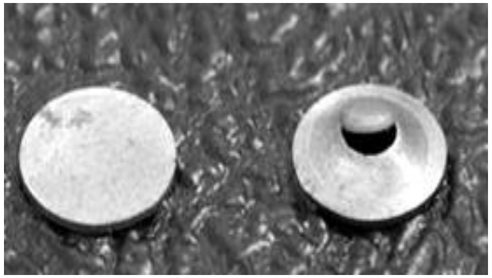


Strain field on the mini specimen surface

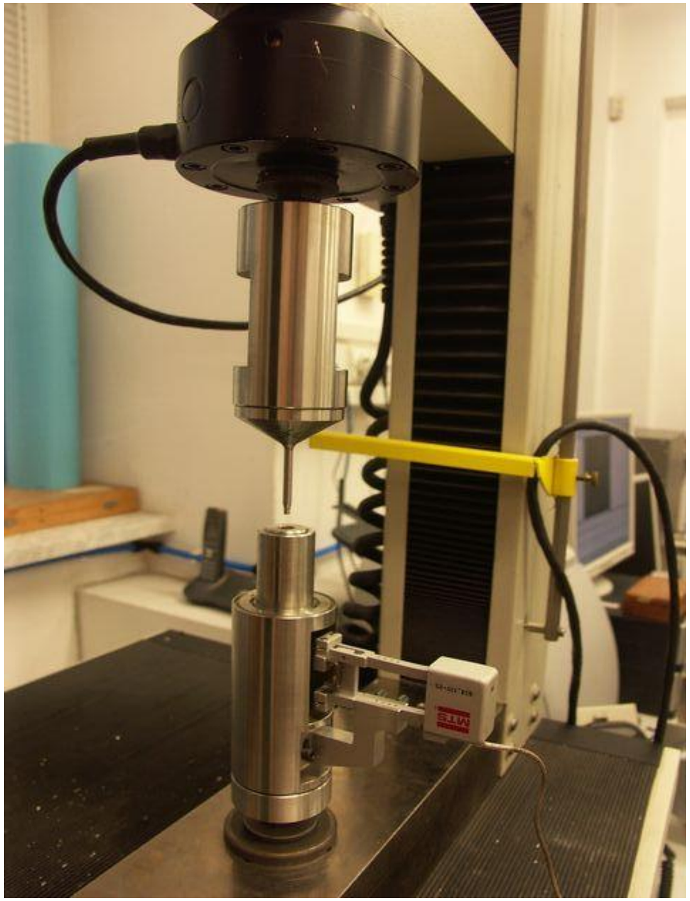
Miniaturized disc bend test



The LN container

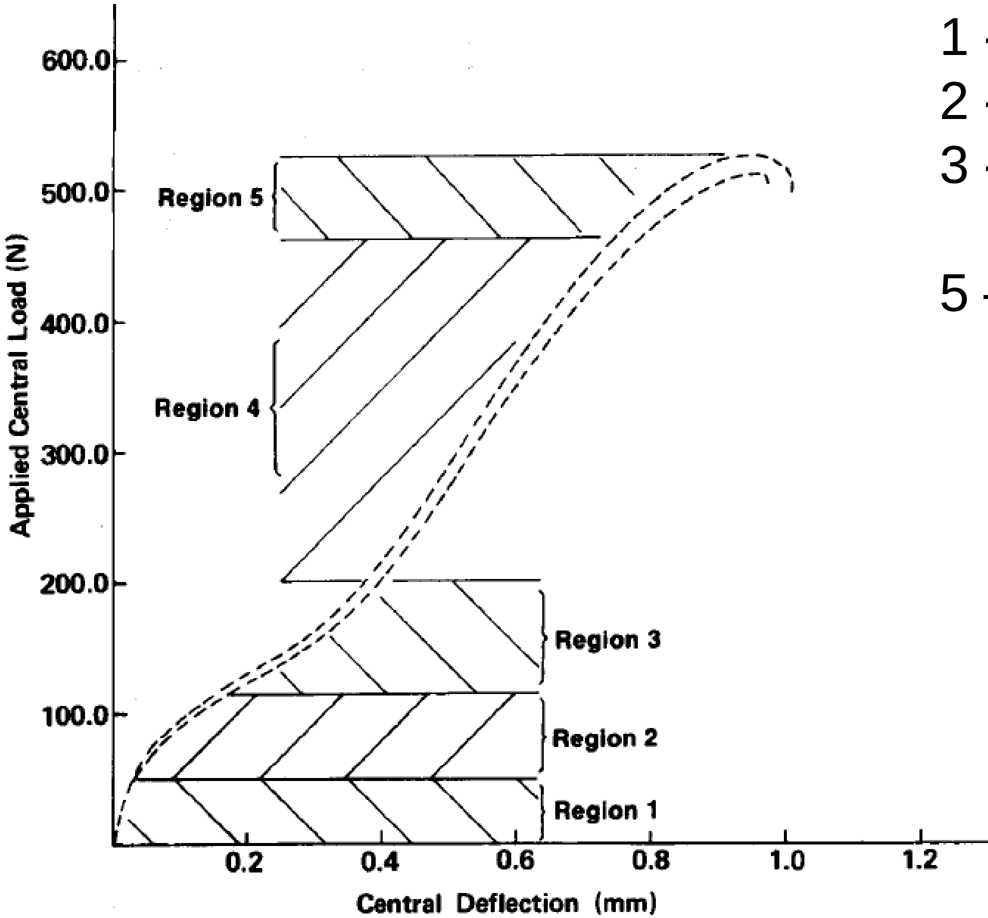


Samples before and after the MDBT

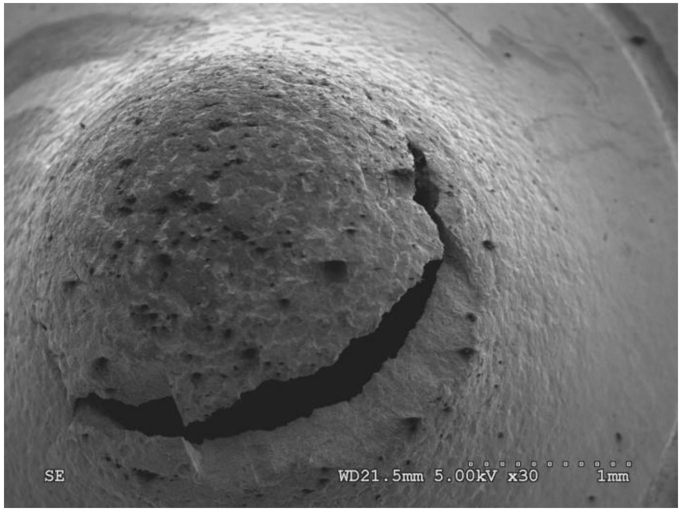


MDBT stand at WUT

The deformation stages during MDBT



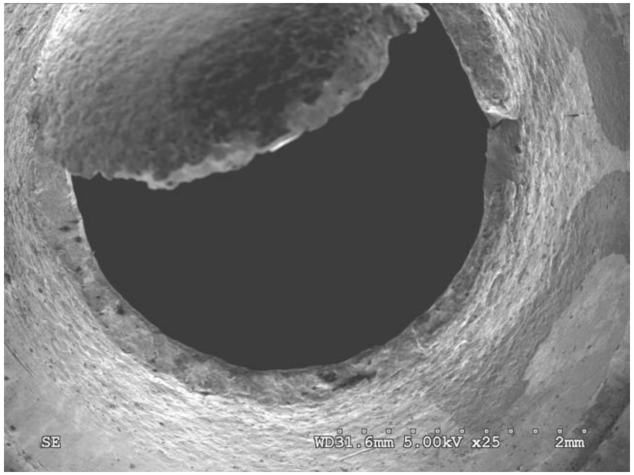
1 - elastic regime,
2 - elastoplastic regime,
3 - 4 – plastic deformation and strengthening
5 – crack initiation and propagation



M.P. Manahan „A new postirradiation mechanical behavior test - the Miniaturized Disk Bend Test", Nuclear Technlogy, Vol . 63, nov 1983

MDBT "applications"

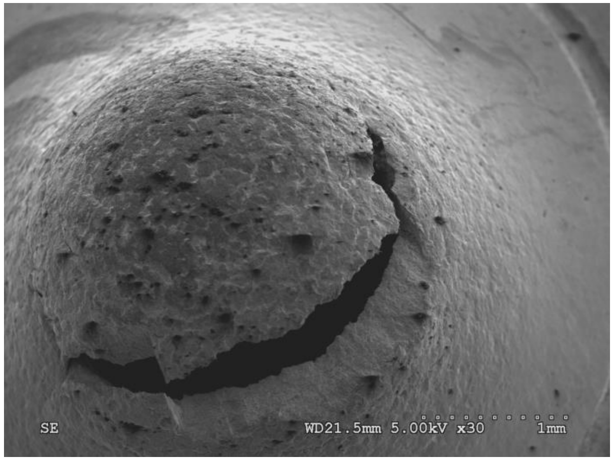
- Strength of the material – correlation with YS and UTS
- Ductile to brittle transition temperature – correlation with Charpy Impact Test
- Crack resistance – correlation with K_{IC} i J_{IC} obtained in CT test



cylinder

puncher shape

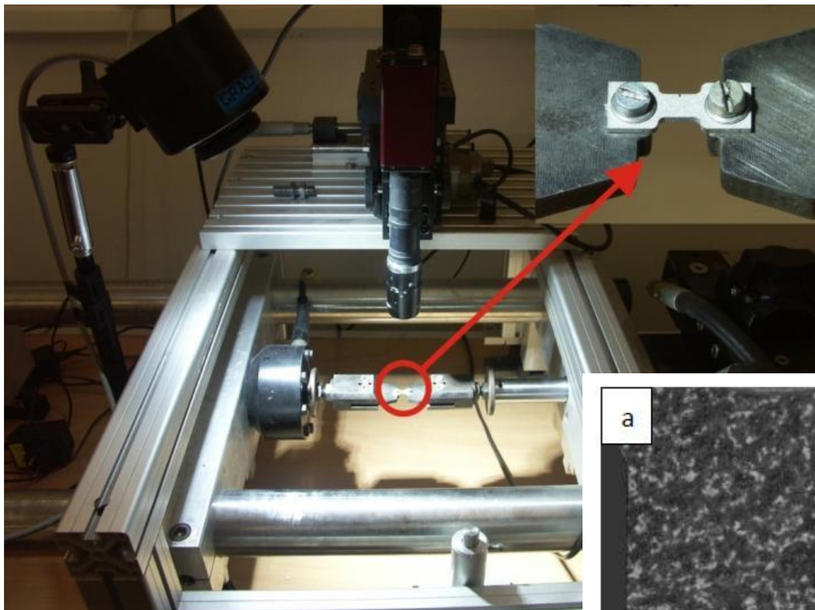
sphere

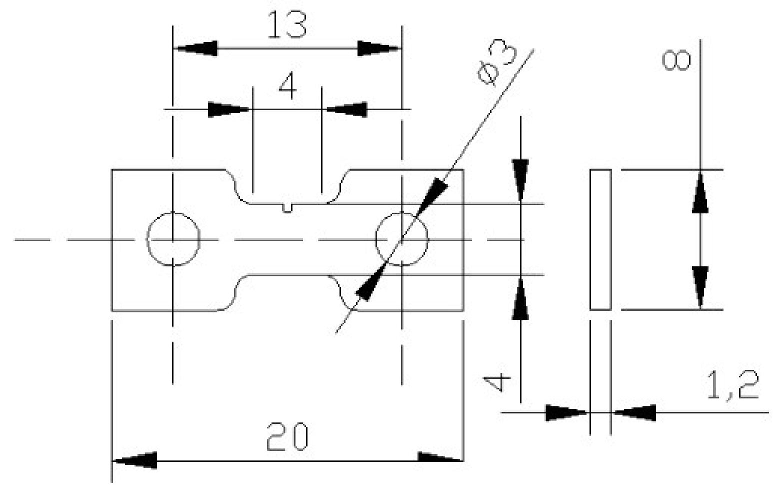


Miniaturized sample testing at WUT

Fatigue Crack Growth testing

Set-up for FCG rate (dA / dN) testing





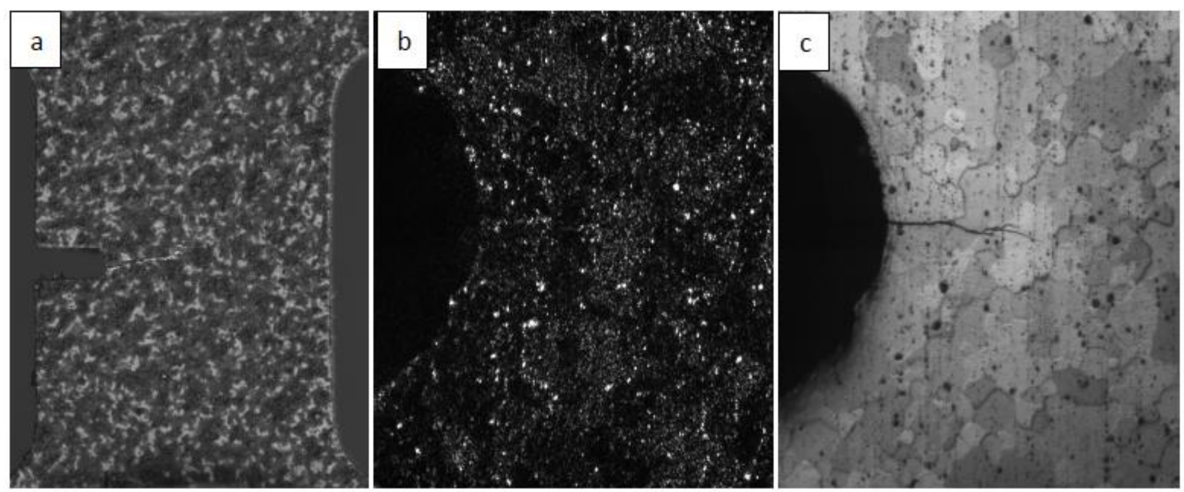


Figure 1: Sample surface pattern: a-small magnification set-up, b-large magnification set-up with laser light, c-large magnification setup with white light and polarizing filters

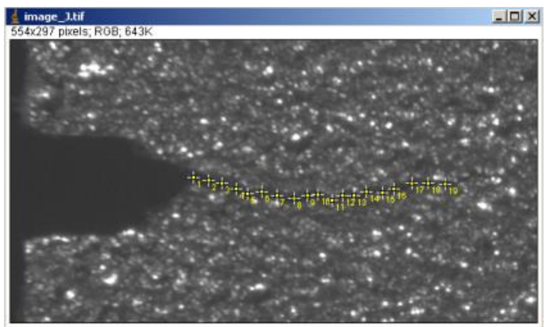
T. Brynk et al., 2010

Fatigue Crack Growth testing

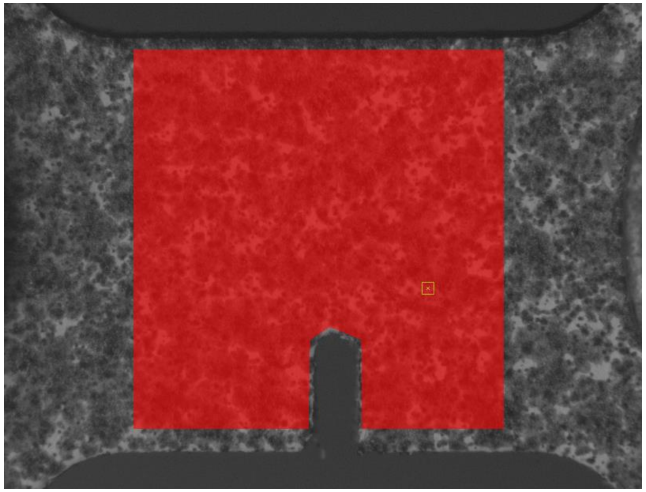
Mini-samples testing with optical crack length measurements

DIC and inverse method

Manual crack tip tracking



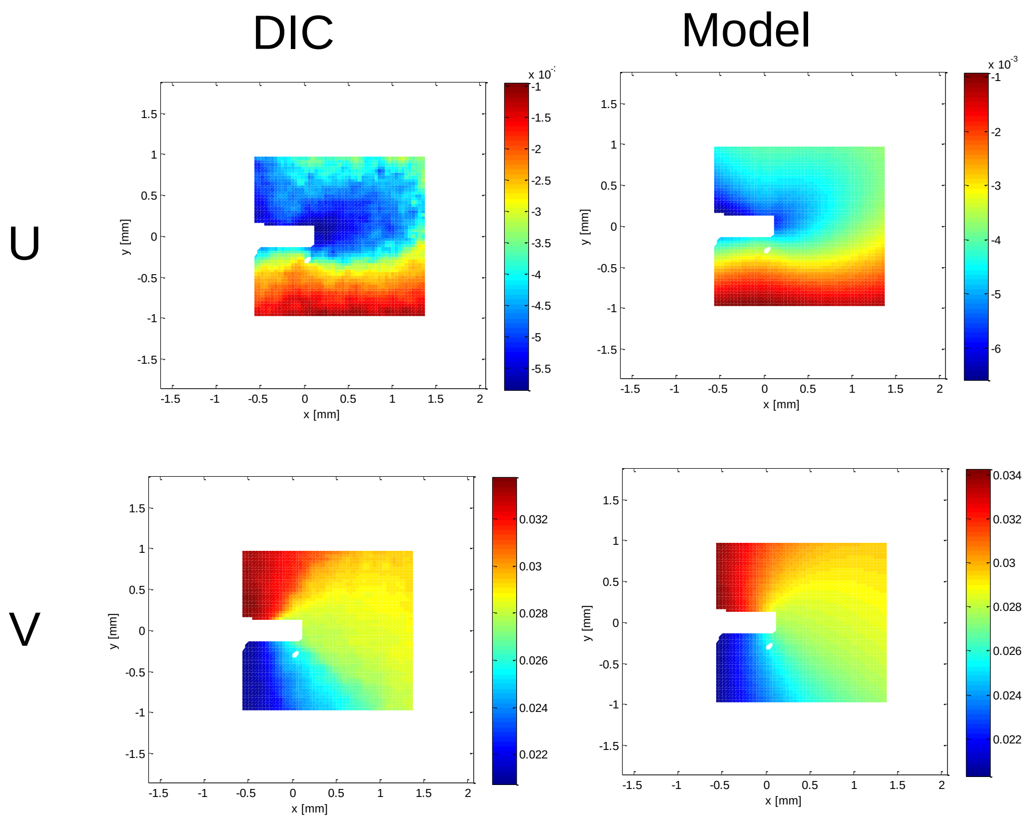
$$K_{\max} = \left(\frac{P}{Wt}\right)(\pi a)^{1/2} \cdot M_f$$



$$M_f = \left\{1{,}12 - 0{,}231\left(\frac{a}{W}\right) + 10{,}55\left(\frac{a}{W}\right)^2 - 21{,}72\left(\frac{a}{W}\right)^3 + 30{,}39\left(\frac{a}{W}\right)^4\right\}$$

a – crack length, *W* - sample width, *t* - sample thickness, *P* – maximal force

da/dN tests of ECAPed Al 5483



K_I = 14.9 MPa*m^0.5; K_{II} = 1.6 MPa*m^0.5;
x_0 =0,74 mm; y_0 = 0,21 mm

Summary

- Investigations of radiation damage in tungsten have been successfully carried out at WUT since 2011 in cooperation with foreign fusion laboratories.
- The estimated dislocations densities correlate with the amount of deuterium trapped in the material.

- Miniaturized samples testing is developed at WUT for more than 10 years.
- Considerable experience and knowledge have been gathered for different type of mechanical tests.
- The tests are used in practice to evaluate degradation of properties of components being in service at industry.