Max-Planck-Institut für Plasmaphysik

The influence of radiation damage on D retention in W: IPP activities

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Content:

- Introduction and IPP activities
- Review of recent work and IPP expertise
- D keV implantation
- Damage rate dependence
- TEM / PALS
- Summary

EUROfusion

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

Issues

H retention in damaged W

- Additional damaging (e.g. neutron damage or high energy ion implantation) strongly enhances H retention
- n damage simulated in lab experiments by self ion implantation (20 MeV W, no chemical effects)

H retention in damaged W

- Dependence on type of ion, damage rate, damage temperature
- Nature of trap sites (vacancies, dislocation loops, nano-voids, voids, …) and analysis of damage

IPP activities

- E-beam damaging (collaboration with Yury Gasparyan, "MEPhI" Moscow)
- Mechanically damaged samples (collaboration with D. Terentyev, MOL)
- Characterisation of damage: TEM and PAS/PALS
  - TEM: L. Ciupinsky WUT
  - PAS/PALS: TU Munich (PLEBS/NEPOMUC)
  - PAS/PALS: I. Uydenhowen (MOL)
- Damage during implantation: J. Bauer
- Simulation of damage: SRIM (is not consitent)

E-beam damaged W (MEPhI, Moscow)

E beam damage produces vacancies only (?)

- E-beam damaging carried out in Moscow
- Characterization of damage (MEPhI)
- “gentle” loading in PlaQ (decoration of defects)
- D depth profiles by NRA
- TDS (MEPhI and/or IPP)
- ….

IPP expertise

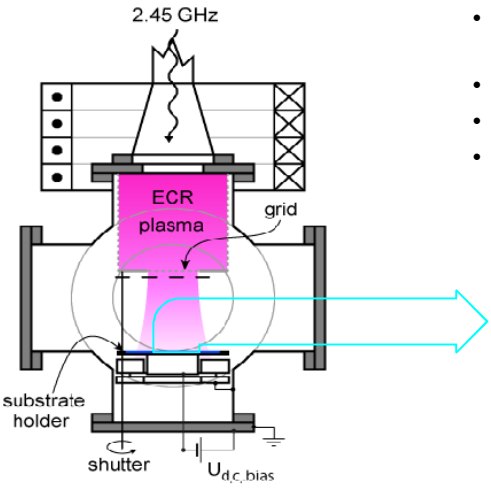
- Ion beam damaging (predominantly we use 20 MeV W self-implantation, but “any” ion species can be used)
- “gentle” D loading using quantified plasma source (PlaQ) (decoration of defects)
- Ion beam implantation (HSQ) (high current, mass-separated)
- Ion beam implantation and in-situ ion beam diagnostics (dual-beam device)
- D depth profiles and total amounts (NRA and TDS)
- Surface morphology by SEM (combined with FIB cross sectioning)
- Deposition of W thin films
- Sample preparation / treatment (annealing up to 2150 K)

“gentle” D loading

using quantified plasma source

(PlaQ)

PlaQ (versatile plasma implantation source)

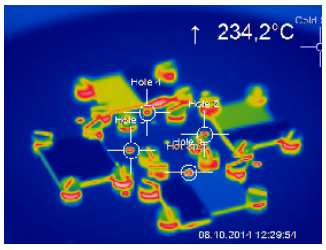


Sketch of plasma source PlaQ

❑ **D implantation: PlaQ[1]**

- 1.0 Pa: D_3^+(94%) + D_2^+(3%) + D^+(3%)
- Flux: 9.9×10^{19} $D\cdot m^{-2}\cdot s^{-1}$ (at 200 V)
  1.07×10^{20} $D\cdot m^{-2}\cdot s^{-1}$ (at 600 V)
- Fluence: 1×10^{23} - 6×10^{24} $D\cdot m^{-2}$
- Ion energy: 10 to 600 eV
- Temperature: 230 to 800 K
  (2 substrate holders for low and high T operation)

*[1] A Manhard, et al. 2011 Plasma Sources Sci. T. **20** 015010*



IR camera view of samples mounted in PlaQ

What have we learned in the near past?

- Saturation effects

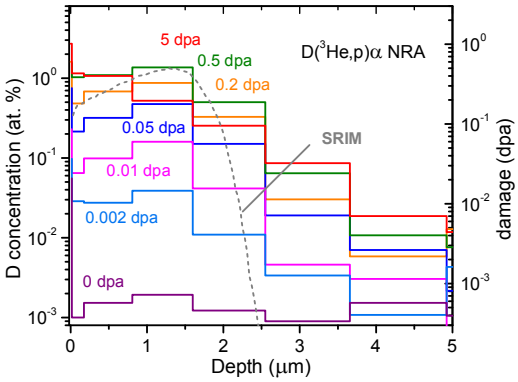
- Annealing of damage

D retention in self-damaged W: saturation effects

Motivation
- Predicting D retention in n-damaged W
- self-damaged W as proxy (20 MeV W^{6+})

Experiment
- D decoration of defects
  - < 5eV/D (no additional damage)
  - 450 K (no damage annealing)
- Analysis by
  - NRA (depth profile < 8 µm)
  - TPD (total amount)

Results :
- D follows predicted damage profile
- D retention
  - increases linearly at low dpa
  - saturates around 1 dpa



D depth profiles in tungsten self-damaged to different peak damage levels (dpa from SRIM, fc)

T. Schwarz-Selinger et al., unpublished results

D retention in self-damaged W: saturation effects

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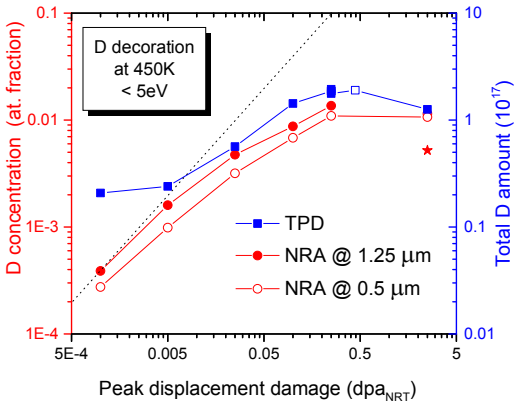
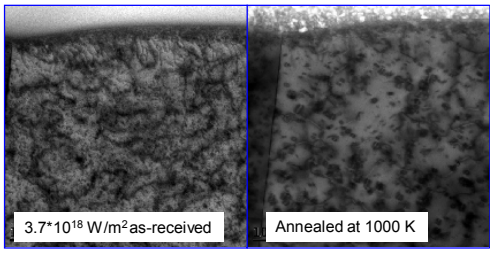


D retention in self-damaged tungsten for different peak damage levels

T. Schwarz-Selinger et al., unpublished results

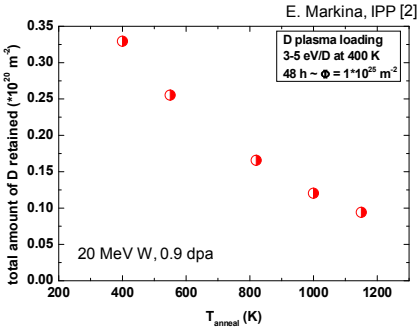
Self-damaged tungsten: Annealing of defects

- Annealing of defects already at low temperatures
- 30% remaining H-decorable damage after annealing at 1200 K
- Residual damage still visible in TEM
  after annealing at 1000 K
  ⇒ dislocation loops, long dislocations



J. Grzonka et al. [1]

E. Markina, IPP [2]



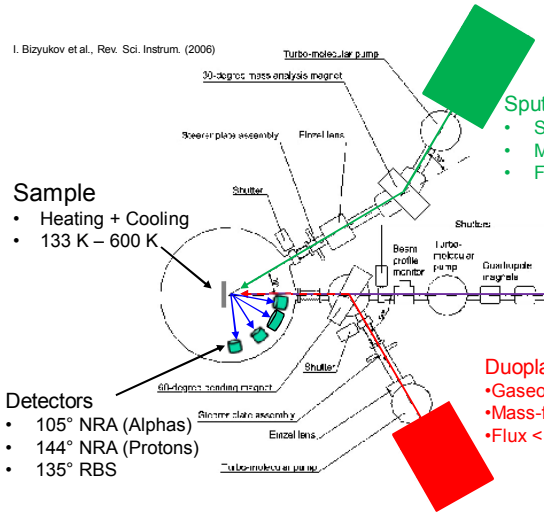
*[1] J. Grzonka et al., Nucl. Instrum. Meth. B **340**, 27–33 (2014)*
*[2] E. Markina et al., J. Nucl. Mater. **463**, 329–332 (2015)*

D keV implantation:

D induced damage

PhD thesis project of Johannes Bauer

Dual Beam Experiment



Sputter ion source
- Solid target materials → TiD, TiH, C…
- Mass-filtered
- Flux < $2 \cdot 10^{18}$ D/m²s, 5 keV/D < E_{ion} < 12 keV/D

Sample
- Heating + Cooling
- 133 K – 600 K

3 MV Tandem Accelerator for ion beam analysis
- ^{3}He, ^{4}He, D
- NRA

Duoplasmatron
- Gaseous species D, H, He, Ar
- Mass-filtered
- Flux < $6 \cdot 10^{18}$ D/m²s, 1.5 keV/D < E_{ion} < 3.0 keV/D

Detectors
- 105° NRA (Alphas)
- 144° NRA (Protons)
- 135° RBS

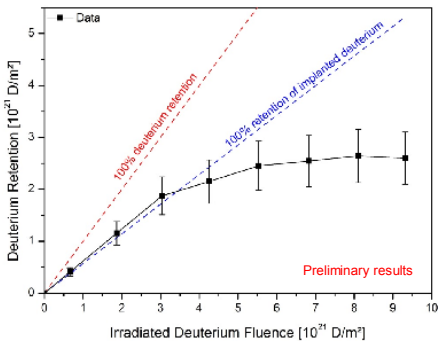
➢ In-situ: Duoplasmatron ion source + NRA, RBS
➢ In-situ + real time: Sputter ion source + NRA, RBS

D keV implantation at low temperature

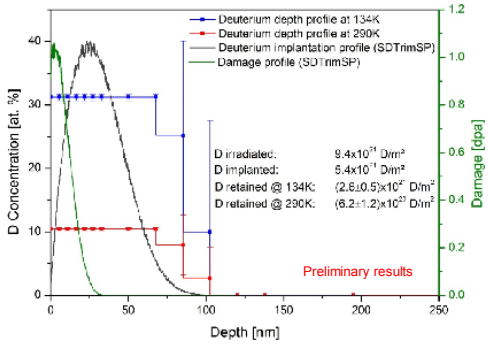
Sample: Polycrystalline W (99.97 wt.%), annealed to 2000 K for 5 min, electro polished

Implantation: E_{ion} = 3.0 keV/D, α = 0°, **T_{sample} = 134 K**, Flux = 6.8 x 10^{17} D/m²s, fluence up to 9.4 x 10^{21} D/m²

D fluence series



Depth profiles



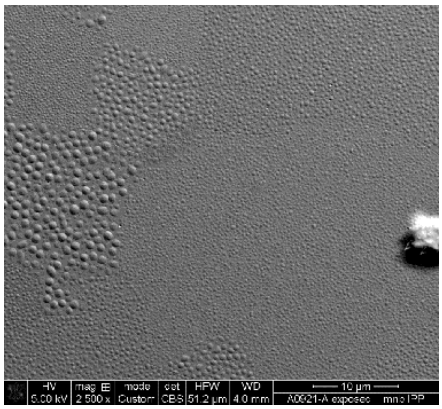
➢ ~ 100% retention for low fluence
➢ Saturation of retention for higher fluence

➢ ~ 30% D concentration @ 134K
➢ ~ 10% D concentration after warm up 290K
➢ D retained in implantation zone
➢ ~ 1 dpa damage induced during implantation

J. Bauer et al., unpublished results

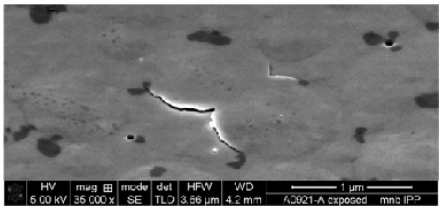
Surface morphology

Vertical SEM image



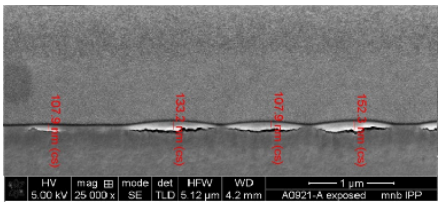
- Strong blistering observed
- Blisters occur within grains
- Mainly circular shape
- Blister size & density depends on grain orientation

Tilted SEM image



- Burst blisters observed

Vertical FIB cut + tilted SEM image



- Blister located within or just behind implantation zone

J. Bauer et al., unpublished results

Damage rate dependence of trap formation in W by MeV self-implantation

T. Schwarz-Selinger

Experimental strategy

3 MV tandem accelerator at IPP:

- Implantation with 20MeV W^{6+} until saturation (= 0.25 dpa_{NRT})
- At room temperature
- Homogenous implantation profile by x-y-scanner
- Accurate flux measurement by four corner cups

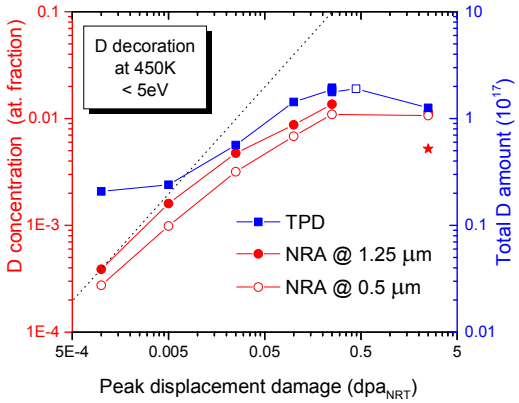
ECR Plasma PlaQ at IPP:

- Gentle plasma loading at 290K with $5\times10^{19}D/m^{-2}s^{-1}$, <5eV/D
- To minimize defect creation by D and thermal defect annealing
- 72h to saturate all existing traps

3 MV tandem accelerator at IPP:

- ^3He NRA depth profiling

Insert: D saturation in self-damaged tungsten



- D uptake in damaged zone saturates above 0.25 dpa

T. Schwarz-Selinger et al., unpublished results

Typical times after the cascade

local heating by electrons within cascade

⇒ recombination of point defects

⇒ diffusion of defects to sinks

(grain boundaries, surfaces, dislocations)

⇒ what remains are defects around ion track

⇒ **individual cascades** can be considered separated,

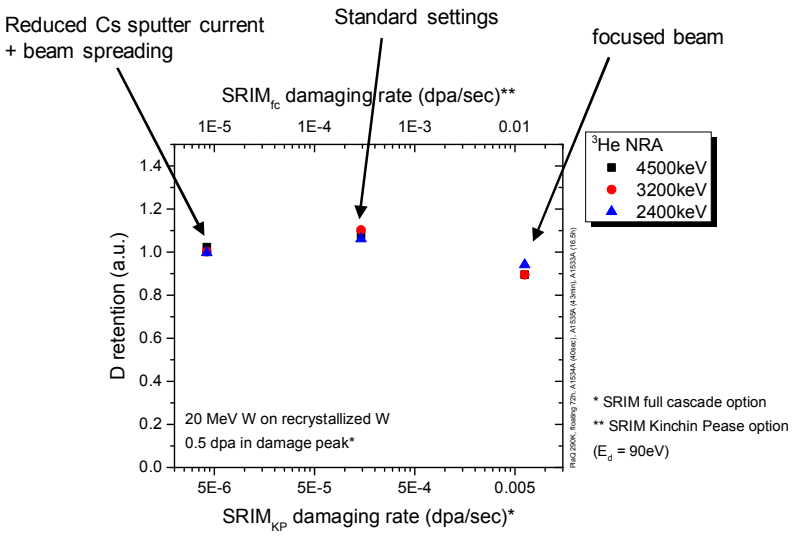
both in time and space:

time scale: 10-100ps

collision size: $(20\ nm)^3$

⇒ No rate dependence expected

Damage rate dependence



T. Schwarz-Selinger et al., unpublished results

For the future

- Are these defects representative for neutron damaged W?
  - Different PKA spectrum ⇒ effect of different ions
  - PAS/PALS study with NEPOMUC

TEM

Warsaw University of Technology (WUT)

L. Ciupinski, J. Grzonka

TEM (Warsaw Technical University = WUT)

Five tungsten samples from IPP/JSI listed in table 1 were investigated. They were chemo-mechanically polished samples self-implanted in the tandem laboratory setup TOF at room temperature with a 20 MeV W^{6+} ions, fluence 7.8×10^{17} m^{-2}, corresponding to peak damage level of 0.5 dpa (SRIM calculation – 90 eV displacement energy).

W samples were annealed to different temperatures after damaging at 300 K.

A0780A	**damaged at 300K, no pre-annealing**
A0781A	damaged at 300K and annealed at 600K for 1h
A0782A	damaged at 300K and annealed at 800K for 1h
A0783A	damaged at 300K and annealed at 1000K for 1h
A0785A	damaged at 300K and annealed at 1130K for 1h

Collaboration with Anže Založnik and Sabina Markelj, JSI Ljubljana, Slowenia

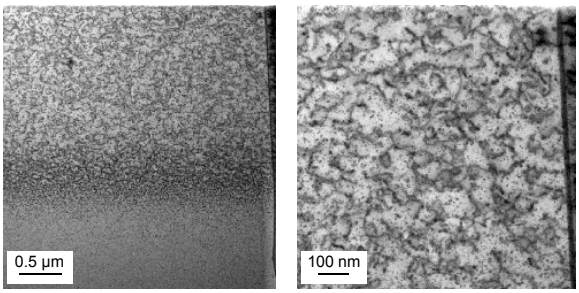
A. Zaloznik, S. Markelj, T. Schwarz-Selinger, L. Ciupinski, J. Grzonka, P. Vavpetic, P. Pelicon, presented at PFMC-15, accepted for publication in Physica Scripta

TEM annealing series

A0780A

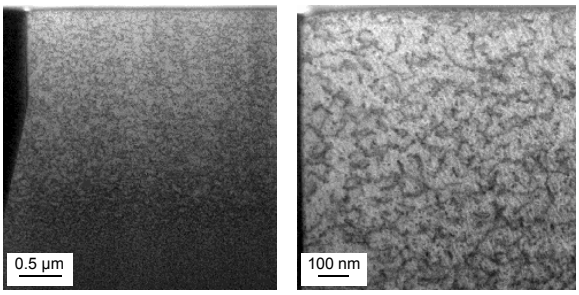
damaged at 300 K

no annealing



A0781A

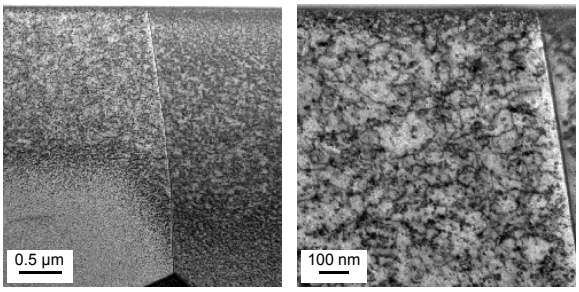
damaged at 300 K and annealed at **600 K** for 1 h



TEM annealing series

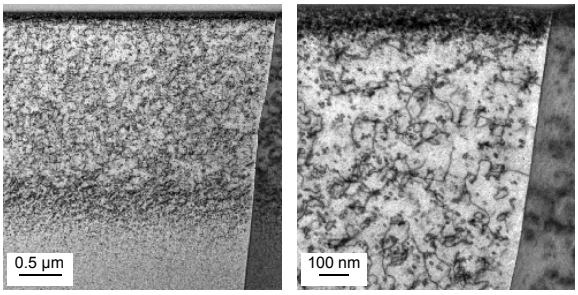
A0782A

damaged at 300 K
and
annealed at **800 K**
for 1 h



A0783A

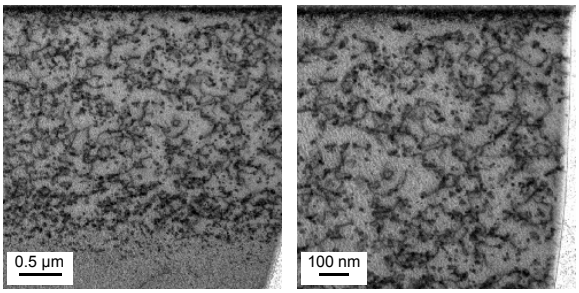
damaged at 300 K
and
annealed at **1000 K**
for 1 h



TEM annealing series

A0785A

damaged at 300 K
and
annealed at **1130 K**
for 1 h

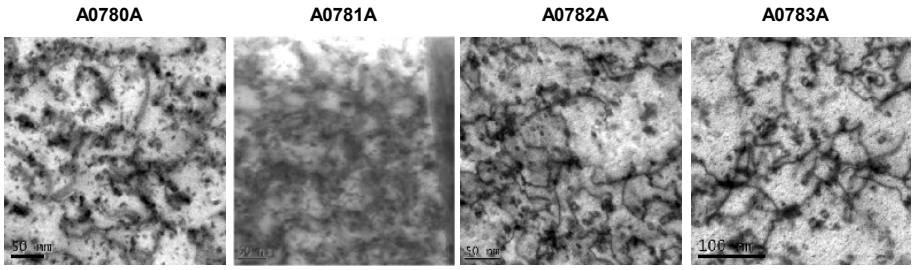


Bright field STEM images of damaged region of W samples with magnified near surface area

Collaboration with Anže Založnik and Sabina Markelj, JSI Ljubljana, Slowenia

A. Zaloznik, S. Markelj, T. Schwarz-Selinger, L. Ciupinski, J. Grzonka, P. Vavpetic, P. Pelicon, presented at PFMC-15, accepted for publication in Physica Scripta

TEM annealing series



Bright field STEM images of damaged region of W samples annealed at different temperatures showing the growth of dislocation loops

Collaboration with Anže Založnik and Sabina Markelj, JSI Ljubljana, Slowenia

A. Zaloznik, S. Markelj, T. Schwarz-Selinger, L. Ciupinski, J. Grzonka, P. Vavpetic, P. Pelicon, presented at PFMC-15, accepted for publication in Physica Scripta

TEM annealing series

Table 2. Dislocation density values of W samples calculated for near surface and intermediate regions

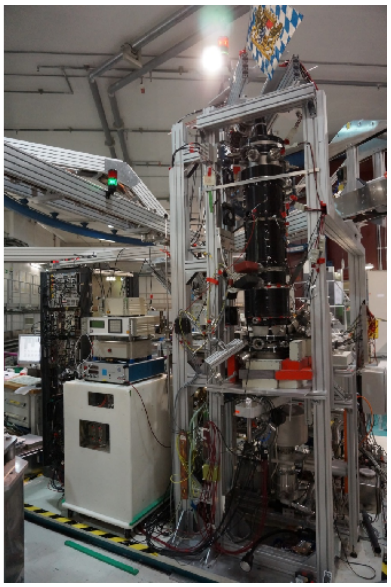
Sample	Region	Total dislocation density [m^{-2}]	Radiation-induced dislocation lines [m^{-2}]	Radiation-induced dislocation loops [m^{-2}]
A0 780A	Near surface	$4.6*10^{14}$	$1.9*10^{14}$	$2.7*10^{14}$
	Intermediate	$5.0*10^{14}$	$2.0*10^{14}$	$3.0*10^{14}$
A0 781A	Near surface	$2.3*10^{14}$	$1.2*10^{14}$	$1.1*10^{14}$
	Intermediate	$3.8*10^{14}$	$1.9*10^{14}$	$2.0*10^{14}$
A0 782A	Near surface	$2.4*10^{14}$	$9.9*10^{13}$	$1.4*10^{14}$
	Intermediate	$2.9*10^{14}$	$9.4*10^{13}$	$2.0*10^{14}$
A0 783A	Near surface	$2.5*10^{14}$	$1.6*10^{14}$	$9.9*10^{13}$
	Intermediate	$2.5*10^{14}$	$1.4*10^{14}$	$1.2*10^{14}$
A0 785A	Near surface	$1.9*10^{14}$	$1.3*10^{14}$	$6.0*10^{13}$
	Intermediate	$1.7*10^{14}$	$8.8*10^{13}$	$7.8*10^{13}$

Collaboration with Anže Založnik and Sabina Markelj, JSI Ljubljana, Slowenia

A. Zaloznik, S. Markelj, T. Schwarz-Selinger, L. Ciupinski, J. Grzonka, P. Vavpetic, P. Pelicon, presented at PFMC-15, accepted for publication in Physica Scripta

PALS (Technical University Munich = TUM)

Pulsed Low Energy
Positron System
(PLEPS)
@
Neutron Induced
Positron Source
Munich
NEPOMUC
@
MLL Garching Germany



PALS (TUM)

Recrystallized MF ref Plansee tungsten

Sample	Irradiation	Dose	Dpa_{NRT}
	None	0	
	3.5 MeV e beam	2.6×10^{18} e/cm^2	
	3.5 MeV e beam	1.3×10^{19} e/cm^2	
A1582	20MeV W^{6+}	1.6×10^{12} W/cm^2	0.005
A1583	20MeV W^{6+}	1.6×10^{14} W/cm^2	0.5

First set of positron lifetime spectra were measured
Data pending for evaluation

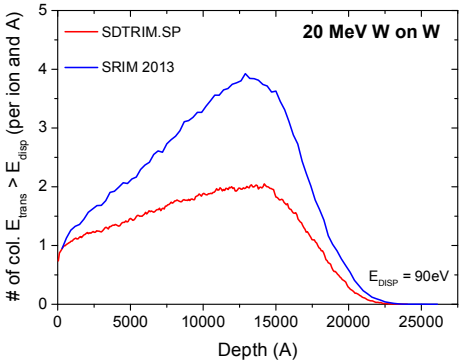
SRIM

A few comments about SRIM

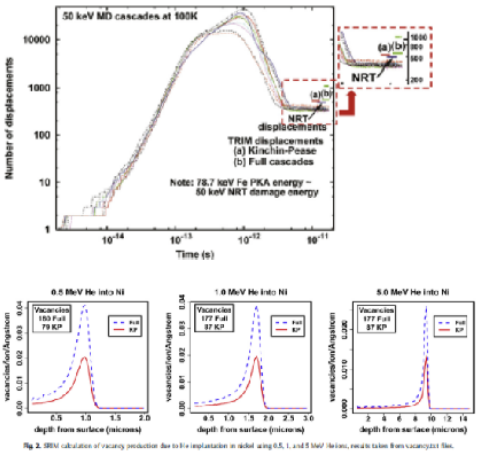
Damage profile calculation (SRIM/SDTrimSP)

Concern: SRIM versus SDTrimSP (W self irradiation)

- Two similar codes deliver different damage profiles although
- Both use Ziegler-Biersack stopping power
- ????



SRIM for calculation of damage: Results are inconsistent



In summary, if a researcher intends to use SRIM to compute dpa from an ion irradiation experiment and wishes to compare the data to the results of neutron or other ion irradiation experiments, it is important that the calculation produce values consistent with the international standard [12,17]. The following recommendations should be complied with:

(1) run SRIM using the "Quick" Kinchin and Pease option,
(2) choose the recommended displacement threshold energy from Ref. [17], which is 40 eV for iron or iron-based alloys,[1]
(3) set the lattice binding energy to zero,
(4) compute the damage energy according to Eq. (5b), and
(5) use the computed value of T_{dam} to calculate the number of displacements according to Eq. (1).

Calculating the number of displacements based on the damage energy is preferred over simply reporting the number of vacancies in the vacancy.txt file even when using the K–P option because the F–C calculations discussed above indicate there is a significant error in SRIM's internal model for calculating the number of vacancies. It is likely that the same error influences the vacancy calculation in the K–P case. Finally, in order to avoid misleading comparisons of data from different experiments, authors should fully describe how they have calculated any dpa values they report and endeavor to determine how the dpa were calculated in any previous experiments to which they compare their own data. Although the NRT model has been broadly used since the mid 1970s, some historical data was reported using the other dpa variants discussed above, and other computer codes such as E-DEP (Energy Deposition) [30] were formerly used to calculate dpa.

Stoller et al., Nucl. Instrum. Meth. B **310**, 15 (2013)

Summary

e beam damaged W:
- collaboration with MEPhI started and will be continued

D ion with keV energies produce substantial damage
- At 134 K D concentration up to 30%
- Warming to 290 K reduces D concentration to 10%
- Strong grain-orientation-dependent blistering

Damage rate dependence
- Variation of damage rate by factor of > 1000 → no significant difference
- Problem: calculation of damage by SRIM is questionable

TEM
- Ongoing collaboration with WUT (and JSI)
- Annealing changes dislocation density
- Residual damage still visible in TEM after annealing at 1100 K

PALS
- Collaboration with TU Munich
- First measurements taken, analysis is pending

EUROfusion

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.