Deuterium retention in tungsten damaged by mechanical work, electrons, and fast ions

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Tritium Inventory in ITER

- **ITER will investigate a burning deuterium-tritium plasma**

  ⇒ **Problem**: Radioactivity of tritium (T)

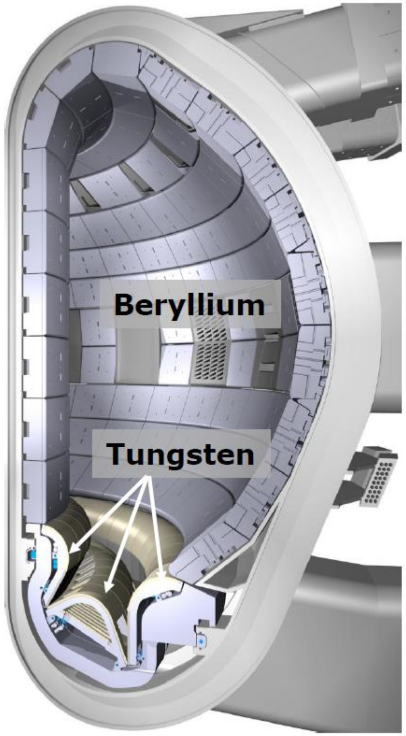
  - T-inventory in ITER is restricted to 700 g due to safety reasons
  - T is a precious resource ⇒ should not be „stored“ in the wall

- **ITER will use Be and W**

  as plasma-facing materials

- **W is a candidate material for DEMO**

⇒ Need to understand T retention mechanisms in W



Predicted edge plasma conditions:

- Wall: 800 m^2, **~10^{21} /m^2s**

  surface temperature: **~450 K**

- Divertor: <10 m^2, **~10^{24} /m^2s**

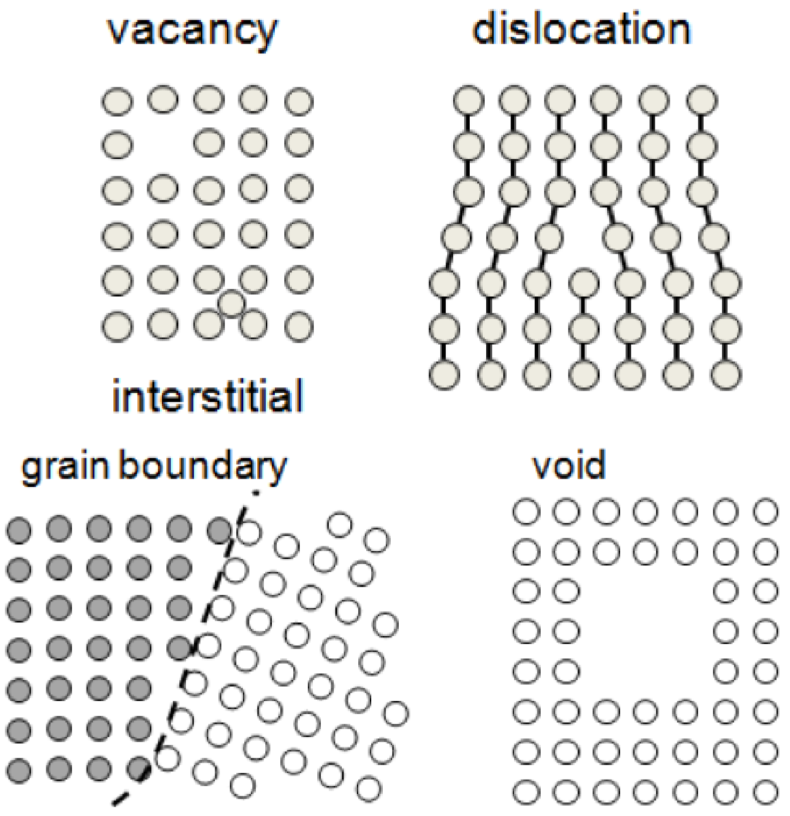
  surface temperature: **~1000 K**

Tritium retention in tungsten

H solubility in W is extremely low ⇒ presence of lattice defects determines H isotope retention in W

- Intrinsic defects:
  Dislocations, impurities,
  grain boundaries, voids
- Ion- and neutron-induced defects:
  Vacancies, vacancy clusters,
  dislocation loops,…

Features of H trapping by each type of defect are still unclear!



⇒ Dedicated experiments studying H interaction with different types of defects in W are required

Intrinsic defect density in W

- As received: 10^{-4} – 10^{-3} M. Balden et al. JNM 452 (2014) 248
- Recrystallized: $2x10^{-5}$ – 10^{-4} M. Balden et al. JNM 452 (2014) 248
- Single crystal: $<5x10^{-6}$

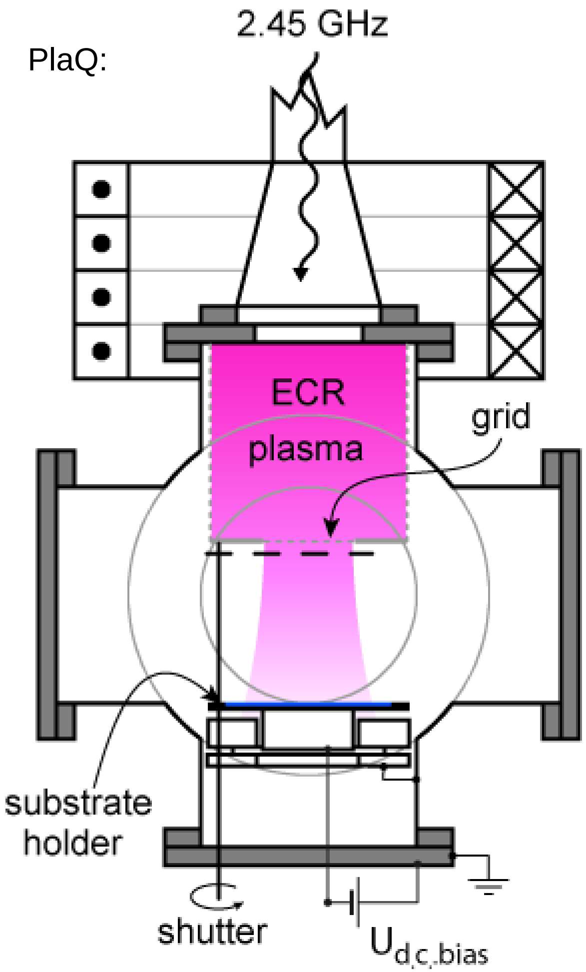
- Depending on material grade and pre-treatment
- Scatter from sample to sample even from the same batch

⇒ **Experiments at low dpa levels require recrystallized W samples or single crystals!**

Defect decoration by gentle plasma loading

<u>Deuterium decoration of defects:</u>

- Low-temperature ECR plasma:
  - Energy: „<5eV/D" (floating targets)
  - Ion flux: 5.6 $\times 10^{19}$ D/(m^2s)
    (97% as D_3^+, 2% as D_2^+, 1% as D^+)
- Homogenous plasma: Five samples at once
- "Gentle" loading:
  - No additional defect creation
  - No blistering



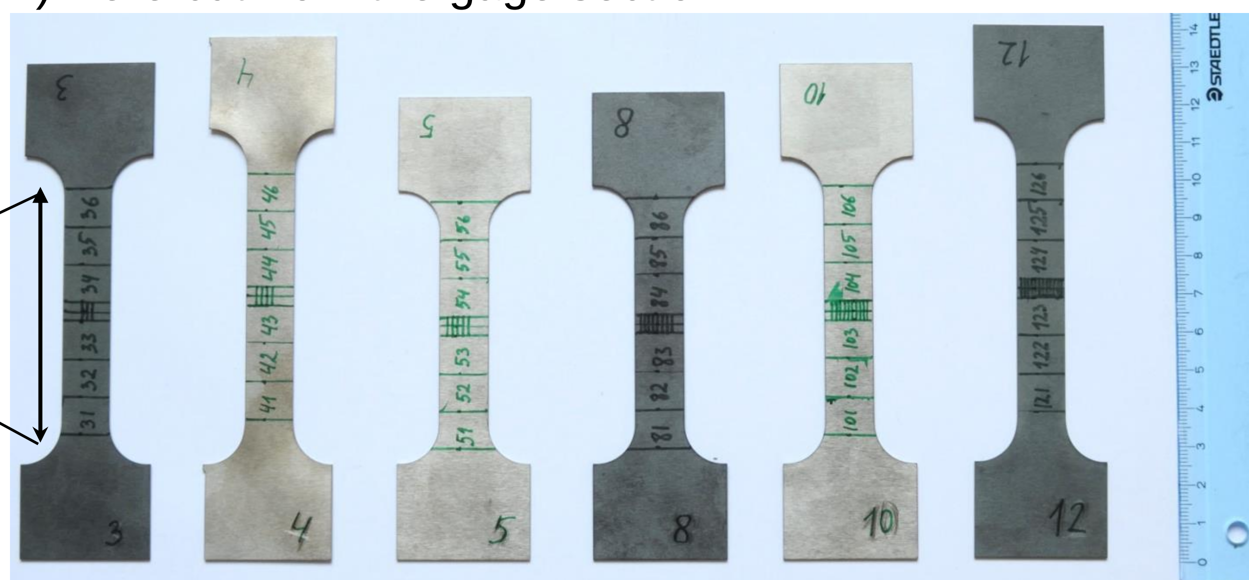
A. Manhard et al., Plasma Sources Sci. Technol. ***20*** *(2011) 015010*

Dislocation-dominated samples

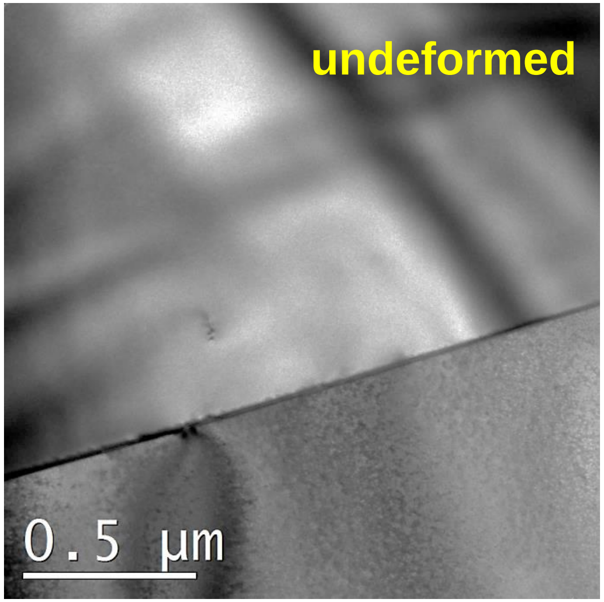
Idea: Introduce dislocations by tensile deformation at temperatures above the ductile-to-brittle transition temperature (DBTT)

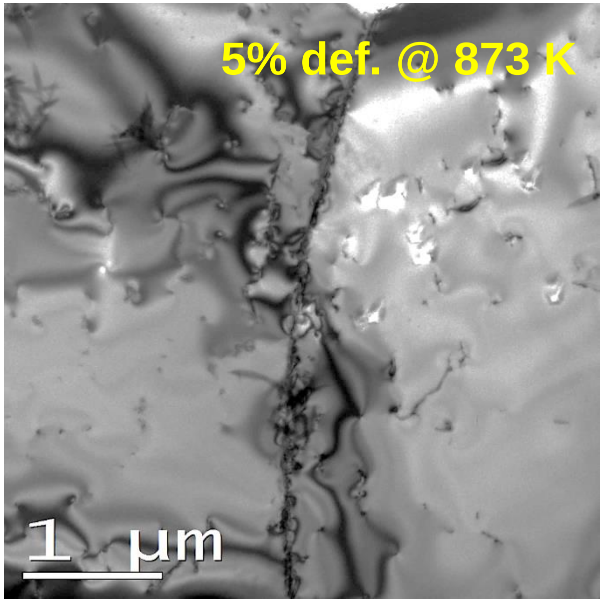
- Tensile specimens of hot-rolled W with 99.97 wt.% purity
- Recrystallized at 1873 K for 1 hour in vacuum
- Tensile deformations at temperatures of 573 K and 873 K to strains from 3% to 39%
- Samples (10×10 mm²) were cut from the gage section

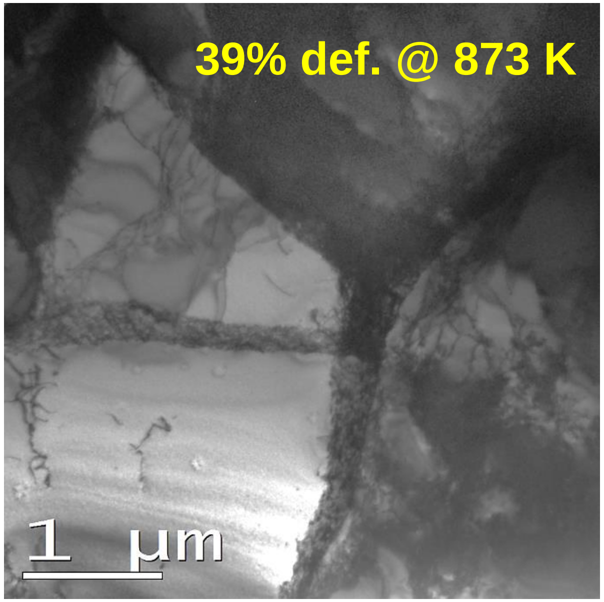
gage section

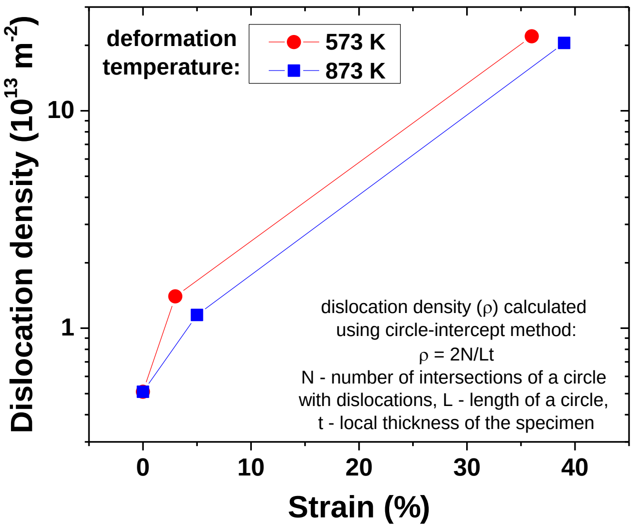


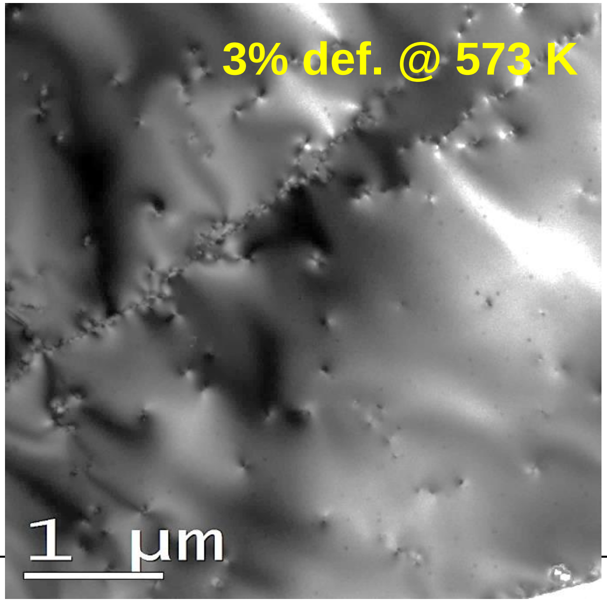
Dislocation-dominated samples: microstructure

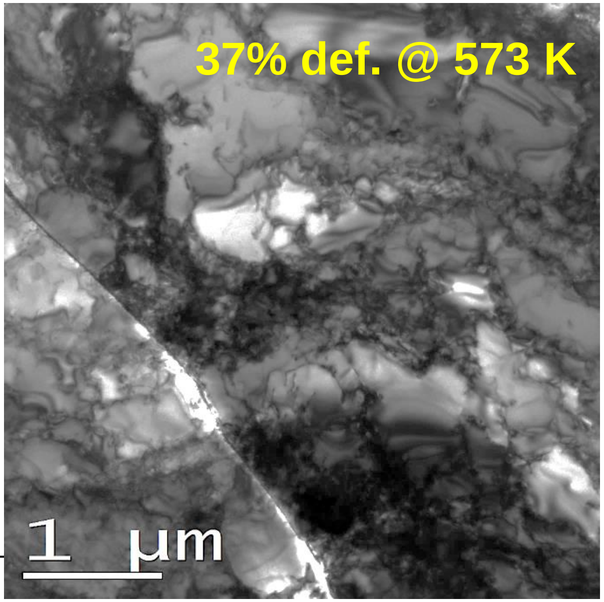






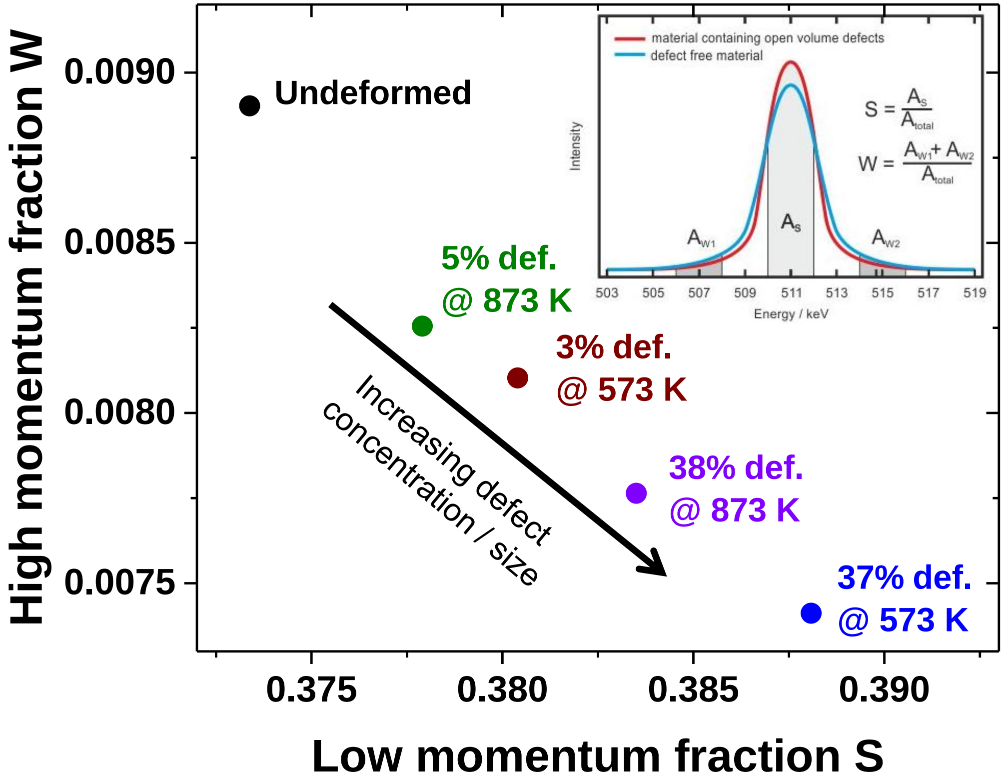




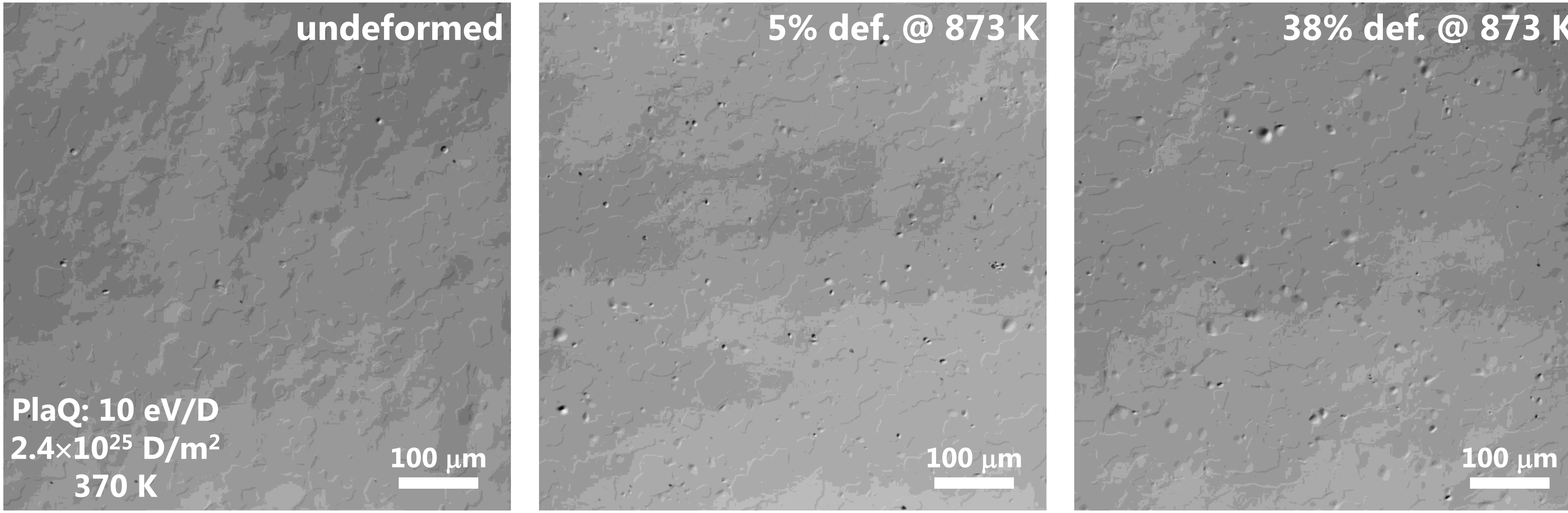


Dislocation-dominated samples: defect density

- Positron annihilation Doppler broadening spectroscopy (DBS) shows almost linear correlation of W vs. S with the amount of plastic deformation
  $\Rightarrow$ the nature of positron-trapping defects does not change with increasing deformation
  $\Rightarrow$ higher deformation levels lead to higher concentration of positron-trapping defects

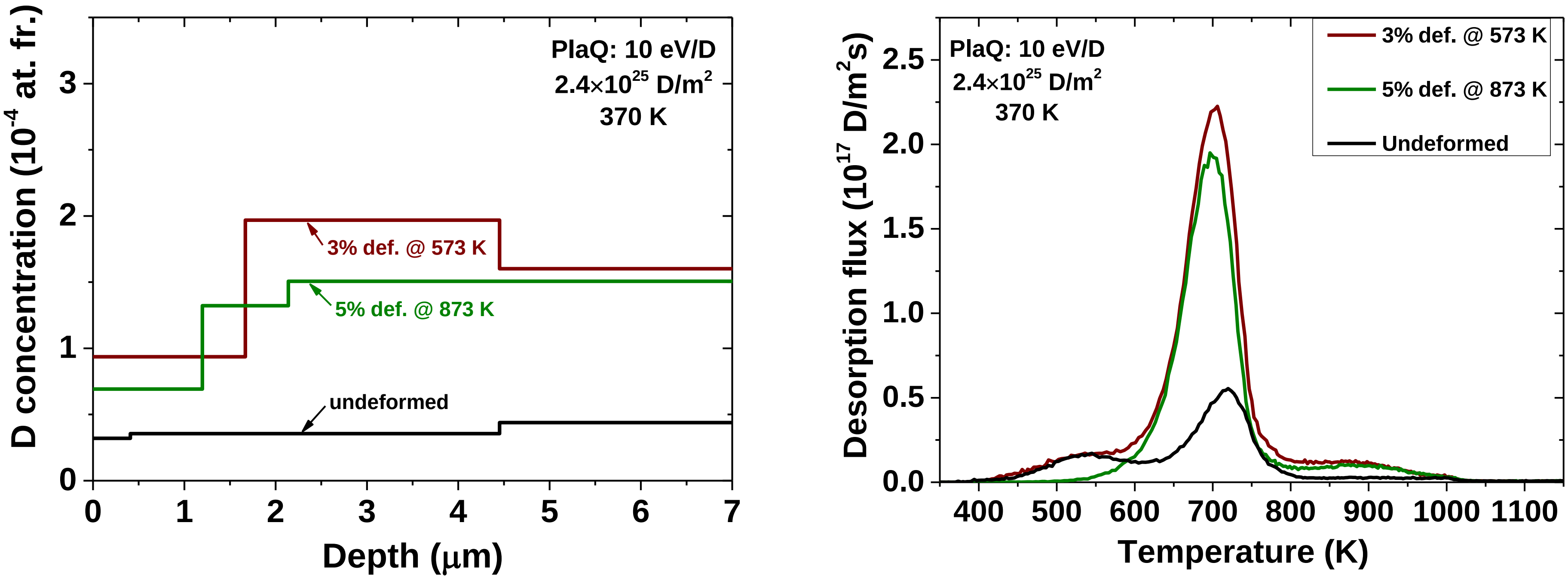


Dislocation-dominated samples: D retention



- Increasing deformation level leads to increase in number density of blister-like structures

Dislocation-dominated samples: D retention



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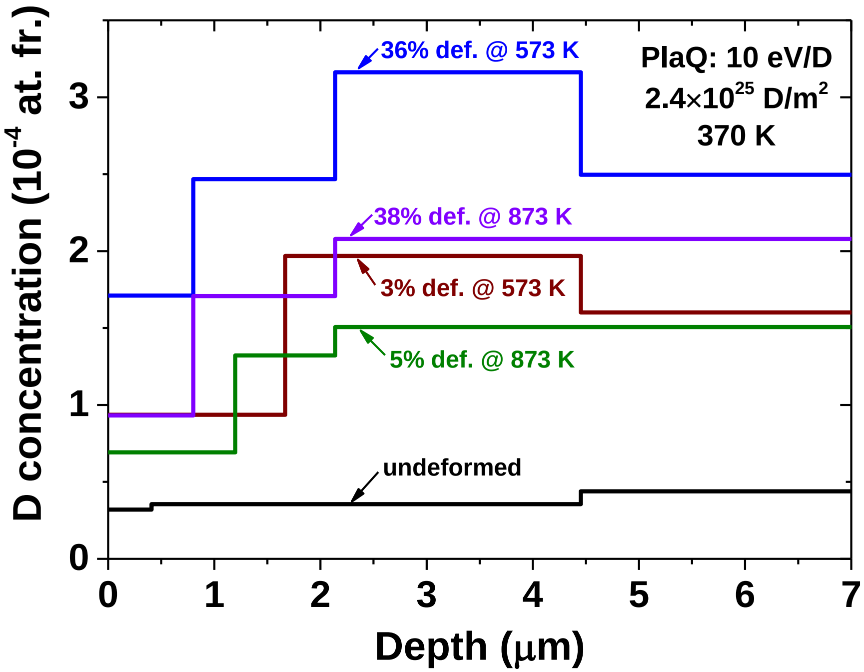
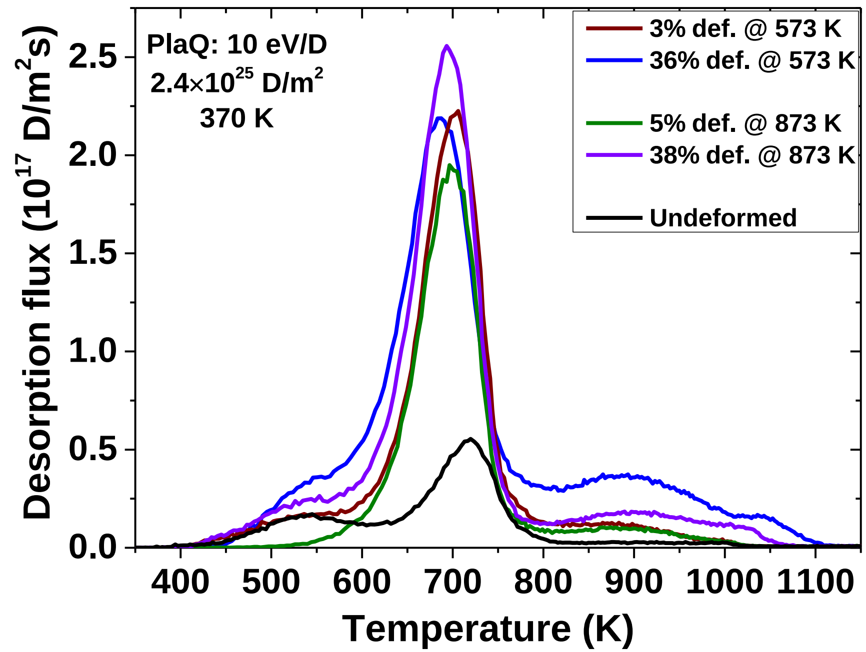
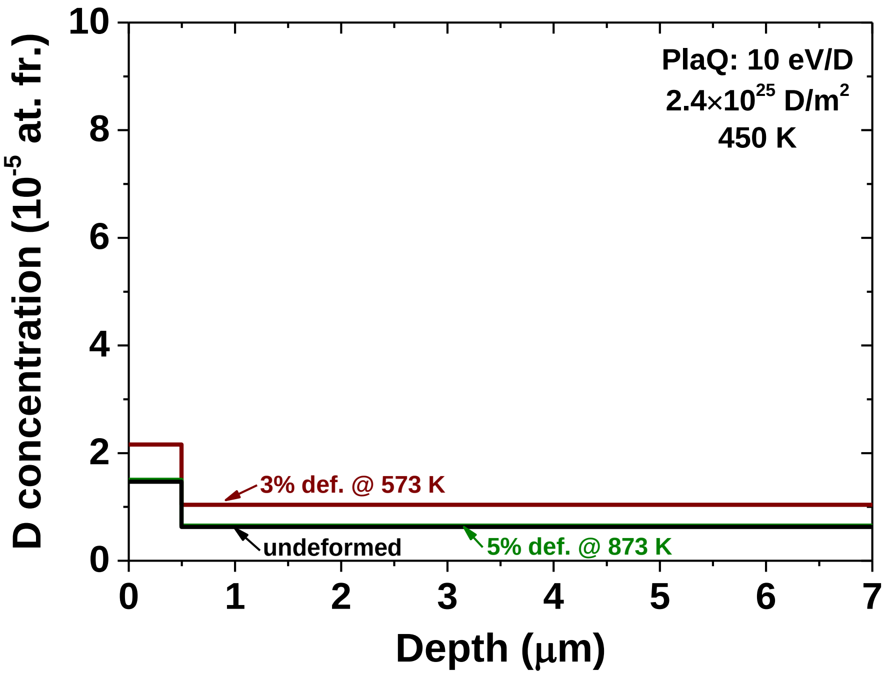
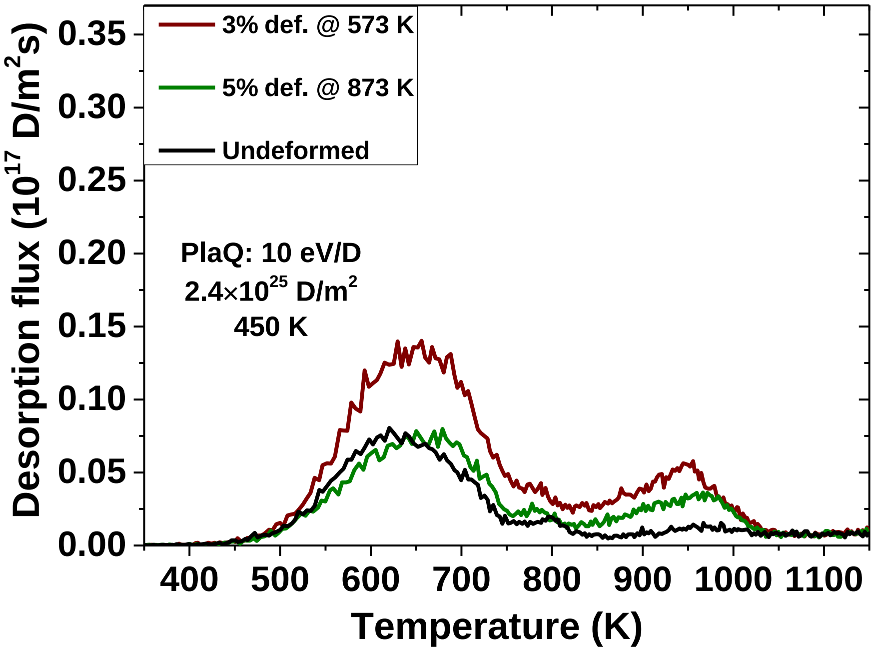




- Increasing deformation level leads to increase in D concentration
- TDS spectra have a complicated multi-peak structure
- D depth profiles and TDS spectra seem to be affected by the presence of blisters

Dislocation-dominated samples: D retention





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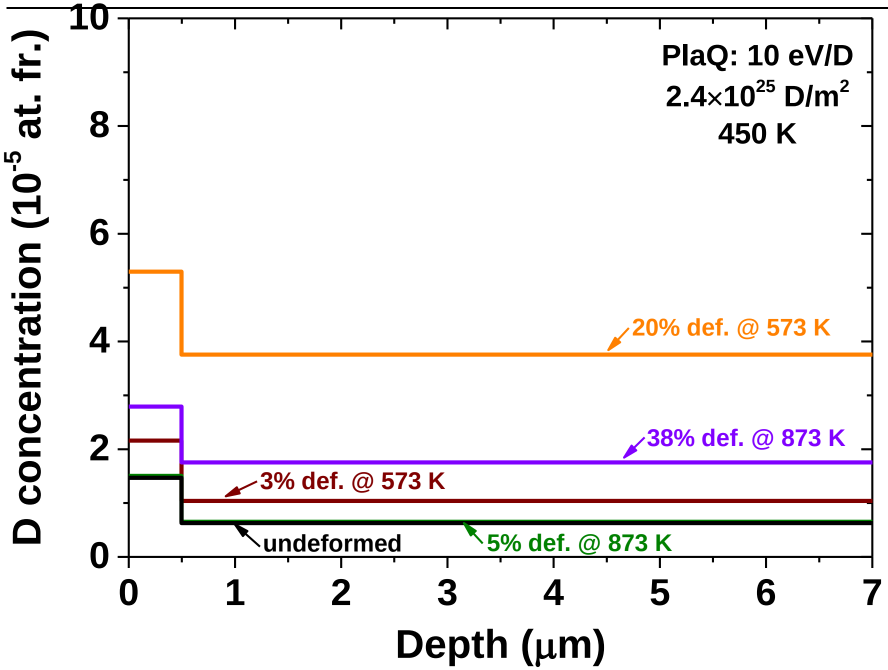
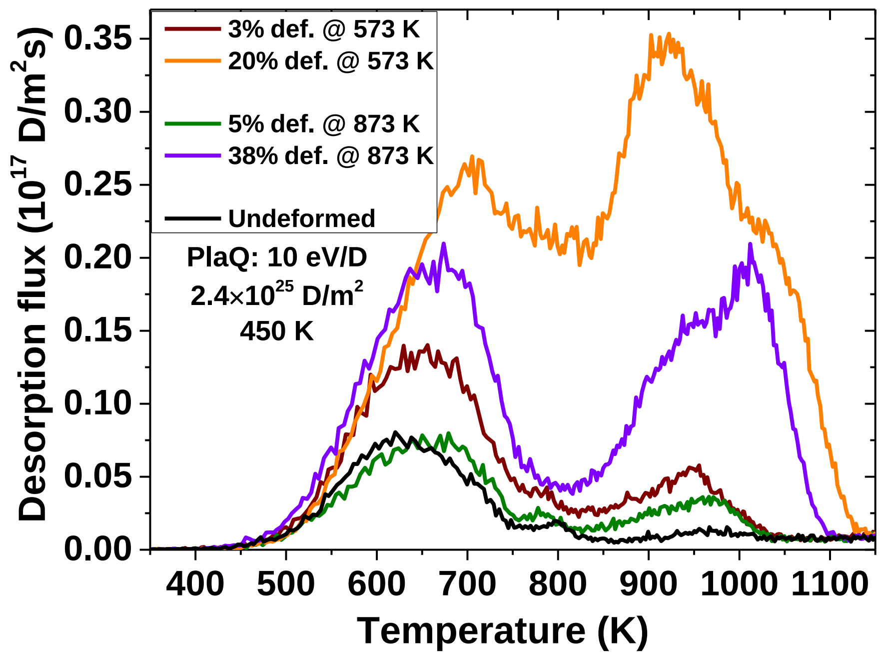




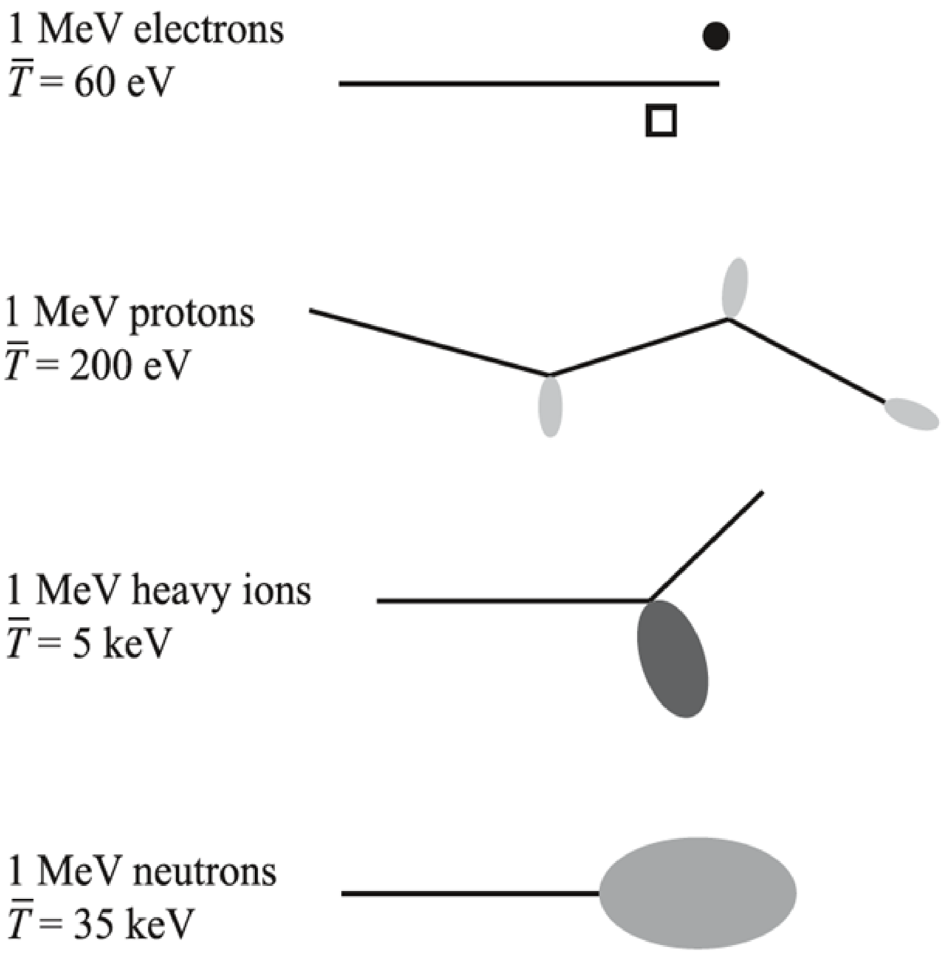
- Plasma exposure at 450 K does not lead to formation of blisters
- TDS spectra have high-temperature peaks, amplitudes increase with increasing deformation level

⇒ presence of trapping sites with high H binding energies (similar to vacancies and vacancy clusters)

⇒ Formation during deformation or during plasma exposure (e.g. nucleation of bubbles on dislocations)?

Creation of radiation damage

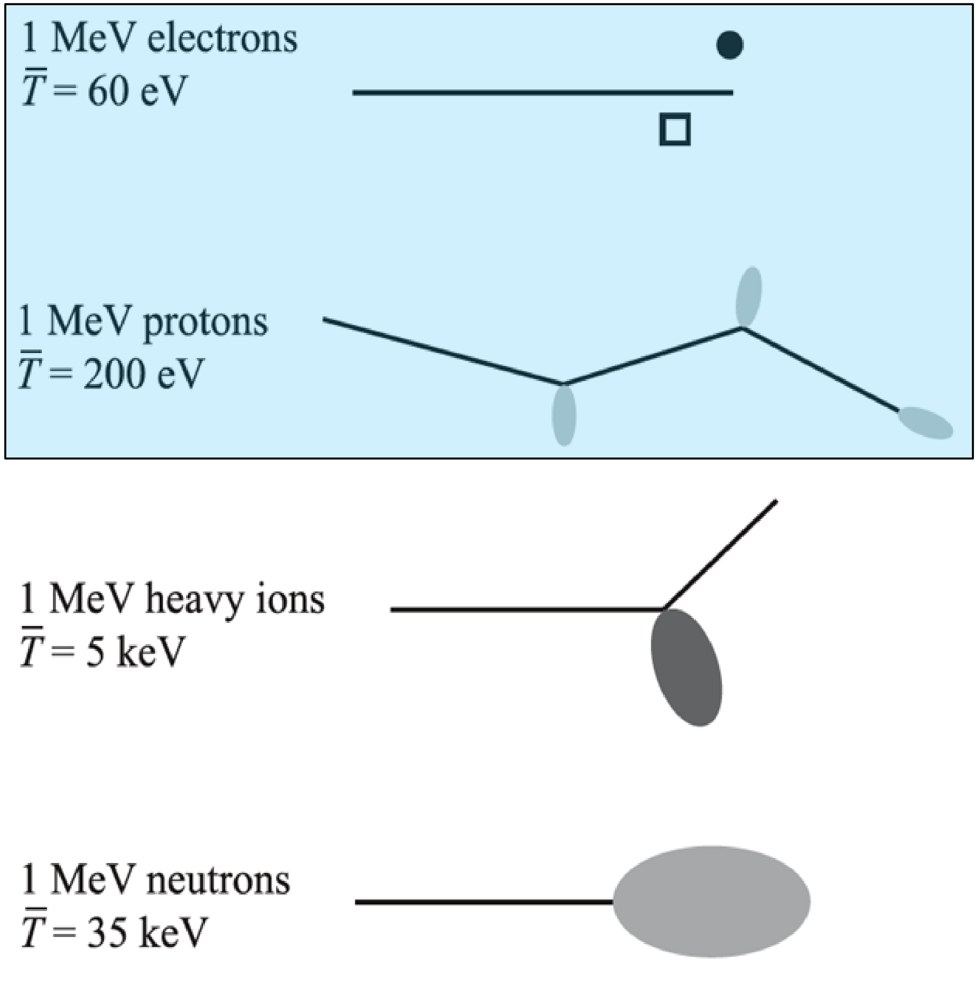
- Simplest radiation defects - Frenkel pairs



G.S. Was, Fundamentals of Radiation Materials Science, Springer Berlin Heidelberg, 2007.

Creation of radiation damage

- Simplest radiation defects - Frenkel pairs
- Can be created by irradiation with electrons and light ions (p)



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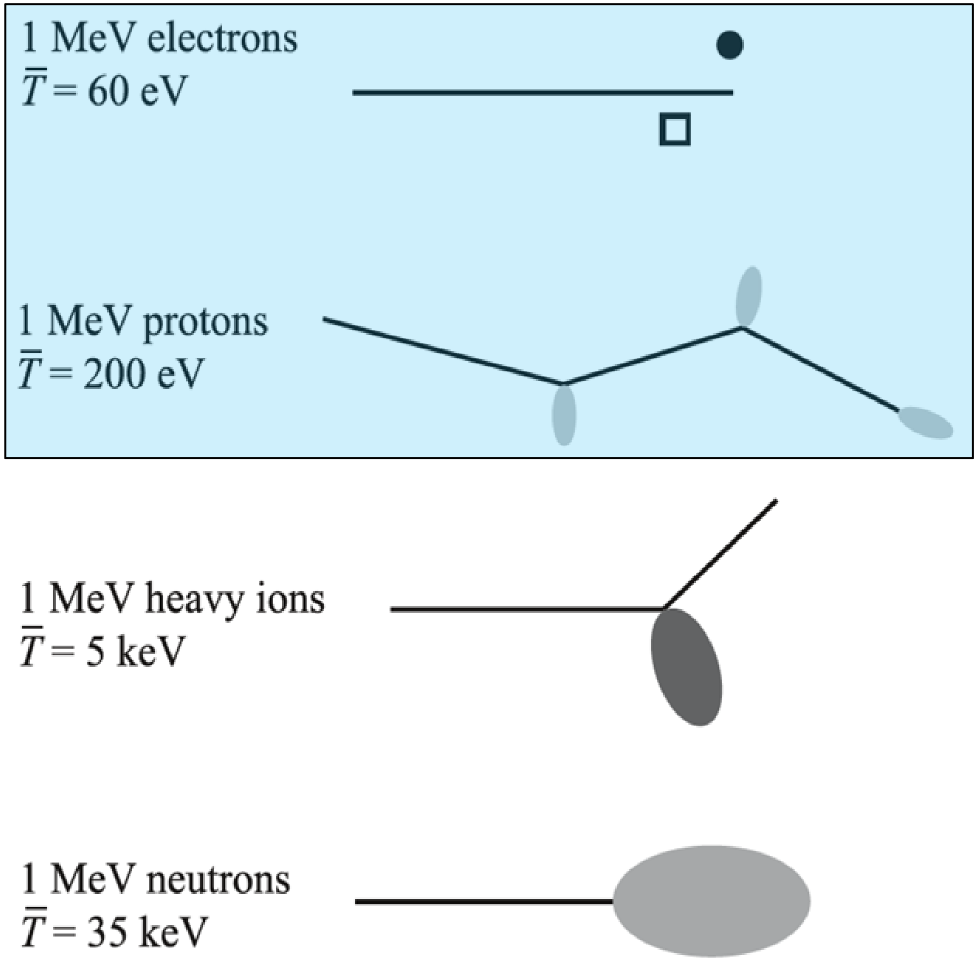

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Creation of radiation damage

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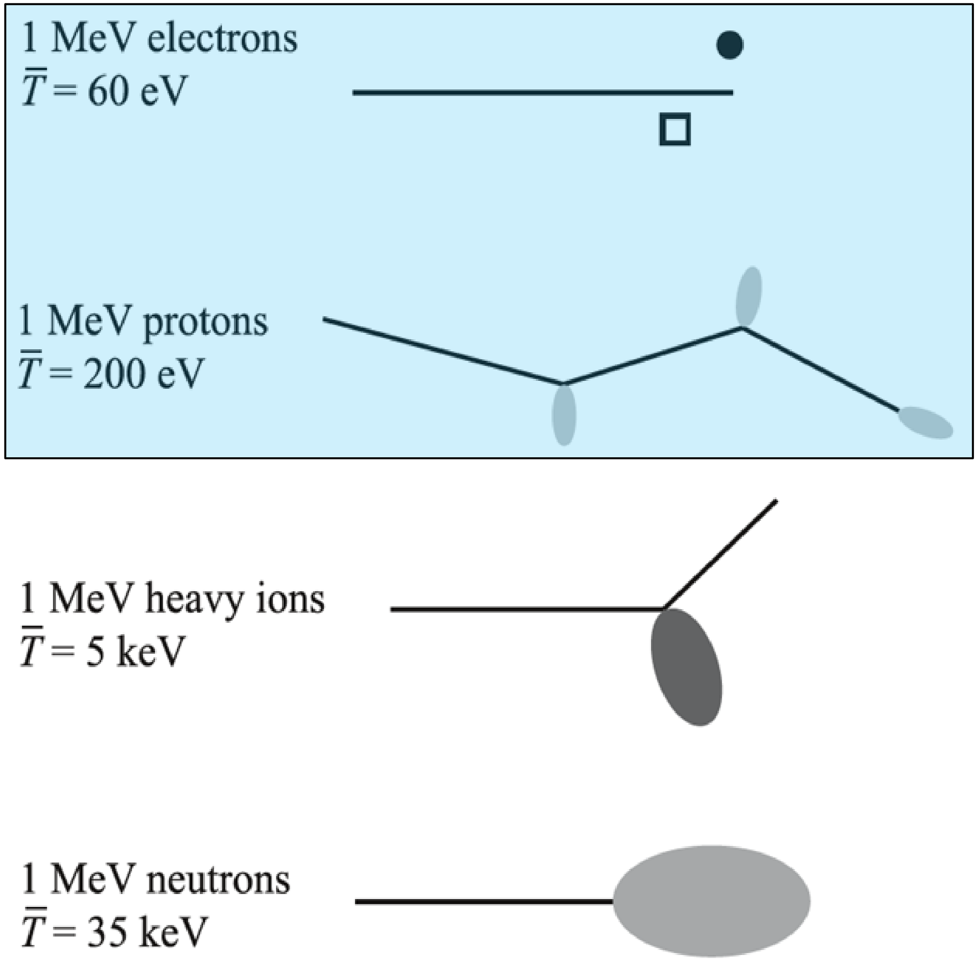
  - ✓ Low irradiation temperatures (< 550 K for W) → avoid vacancy mobility [1,2]



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[1] H. Eleveld, A. van Veen, J. Nucl. Mater. 212 (1994) 1421.
[2] A. Debelle, M.F. Barthe, T. Sauvage, J. Nucl. Mater. 376 (2008) 216

Creation of radiation damage

- Simplest radiation defects - Frenkel pairs
- Can be created by irradiation with electrons and light ions (p)
  - ✓ Low damage levels
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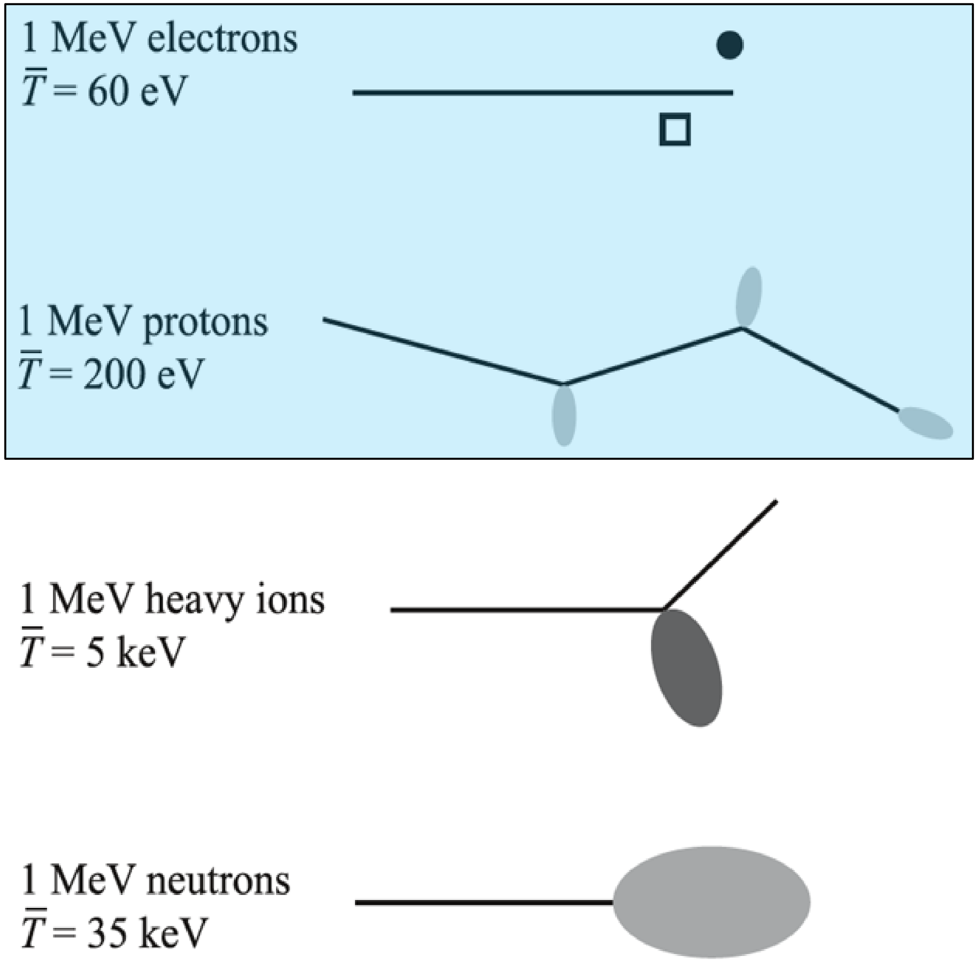
⇒ Avoid formation of secondary defects (vacancy clusters, pores,..), which is typical at high damage levels



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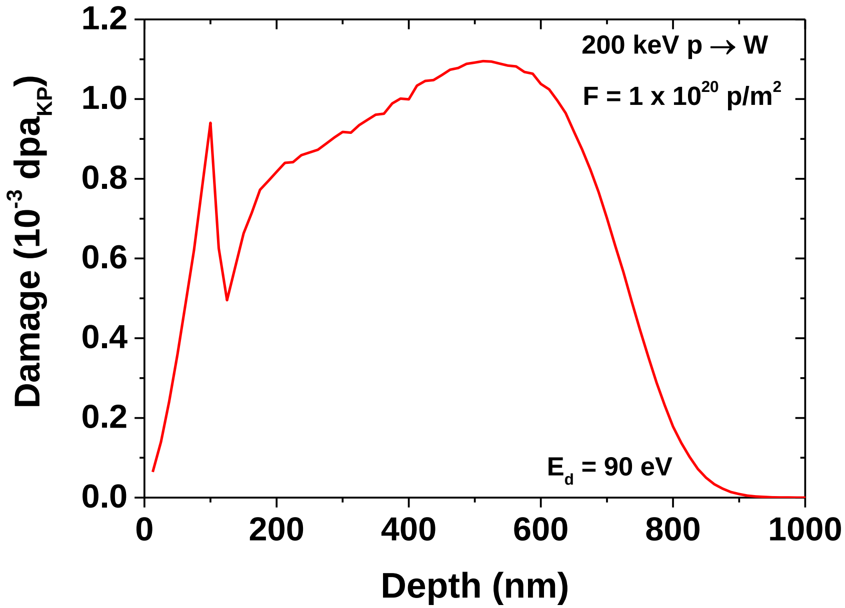
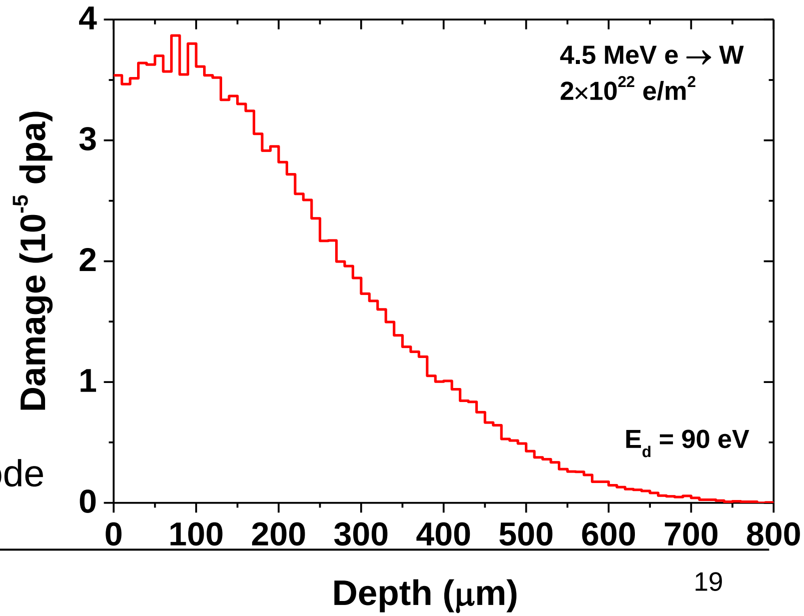
Vacancy-dominated samples

Idea: Introduce mainly single vacancies by damaging with protons or electrons

- Samples: High-purity W (100) single crystals
- Damaged by 200 keV protons and 4.5 MeV electrons to low damage levels (below 10^{-3} dpa_{KP}) at 295 K
- Post-irradiation annealing of samples in vacuum for 15 min at temperatures in the range of 550-1800 K
  $\rightarrow$ study the annealing and clustering behavior of single vacancies

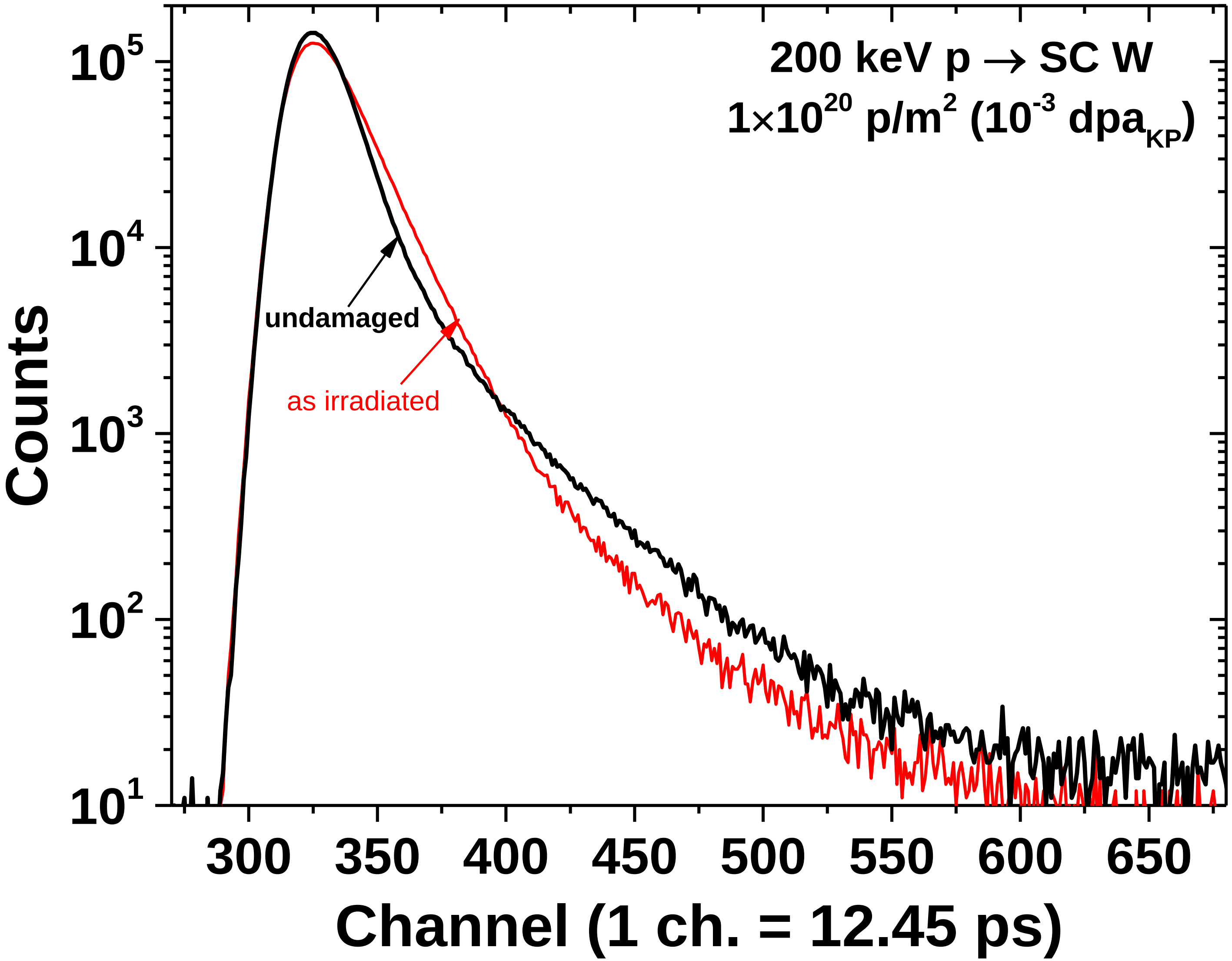




Damage calculated using the Monte-Carlo ElectronDamage code

L. Messina, Master Thesis; Politecnico di Torino, Kungliga Tekniska Högskolan, 2010

PALS measurements



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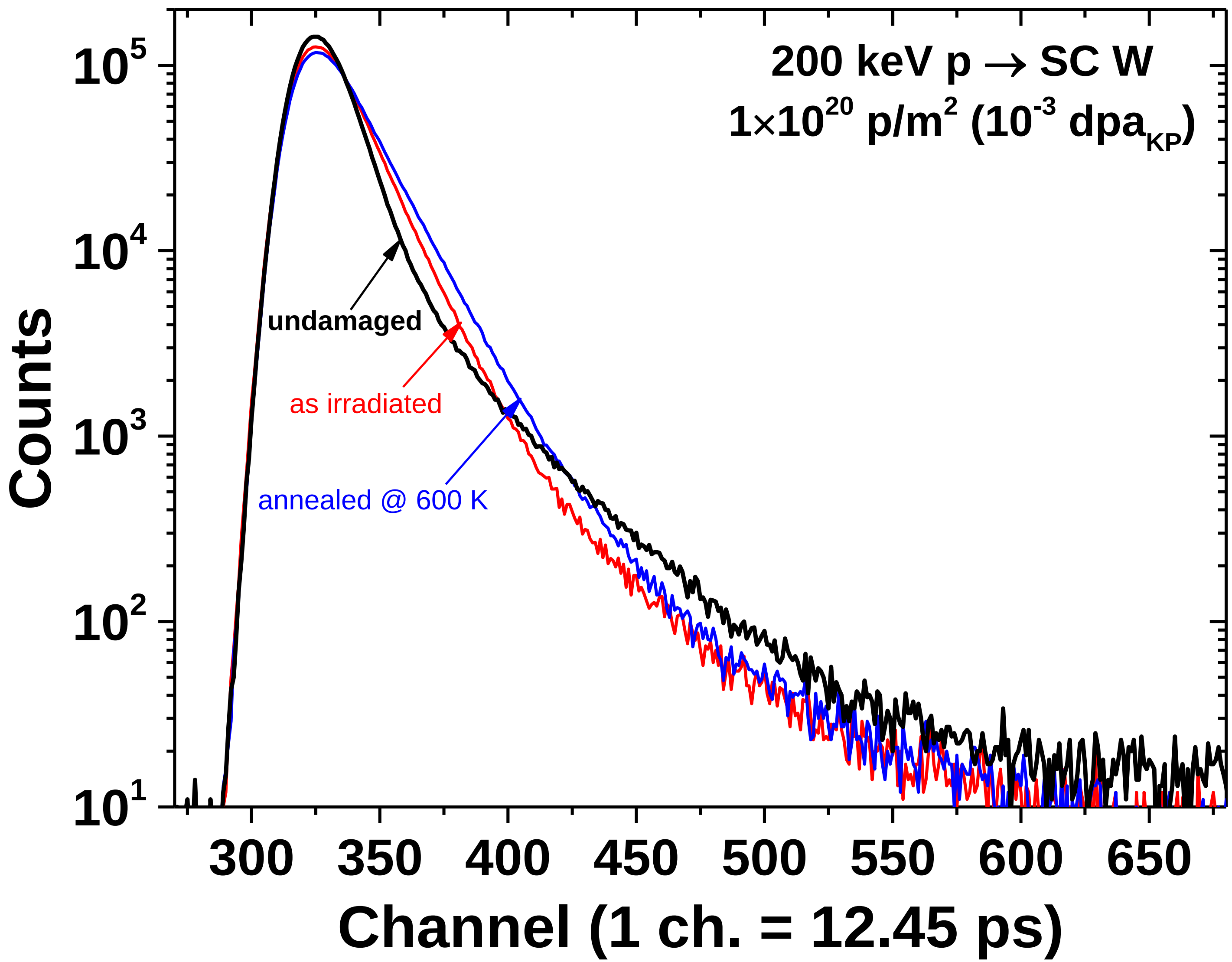

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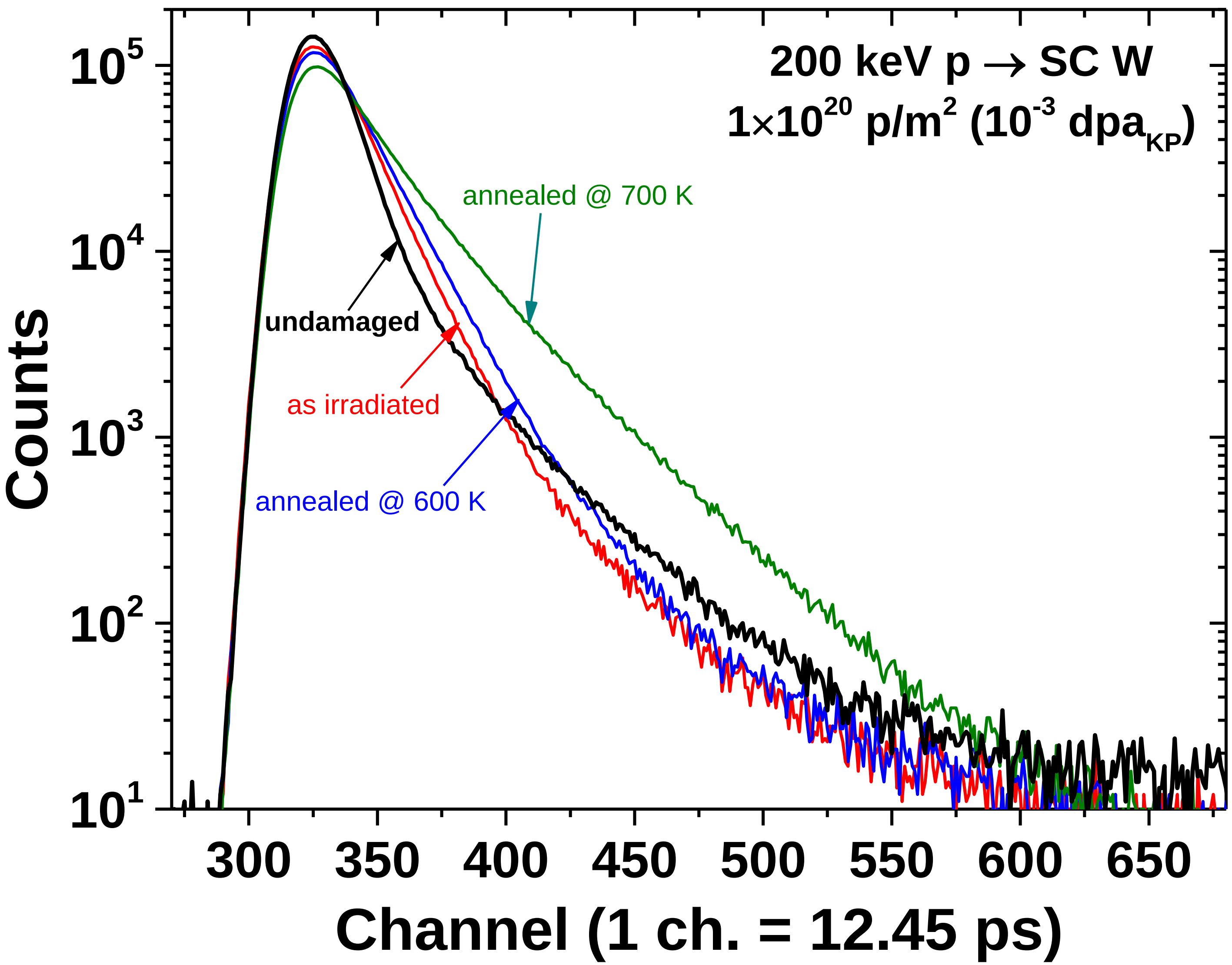

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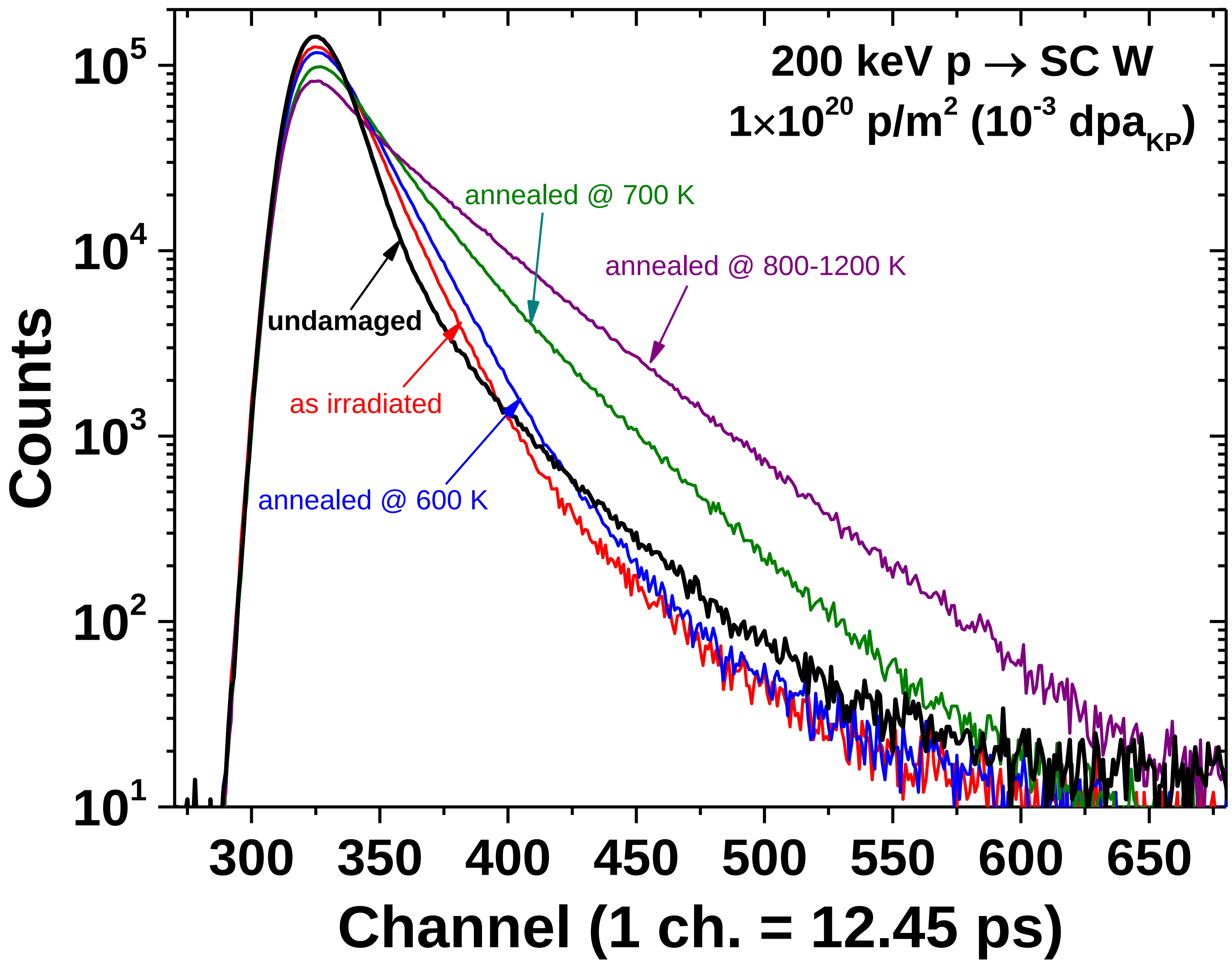

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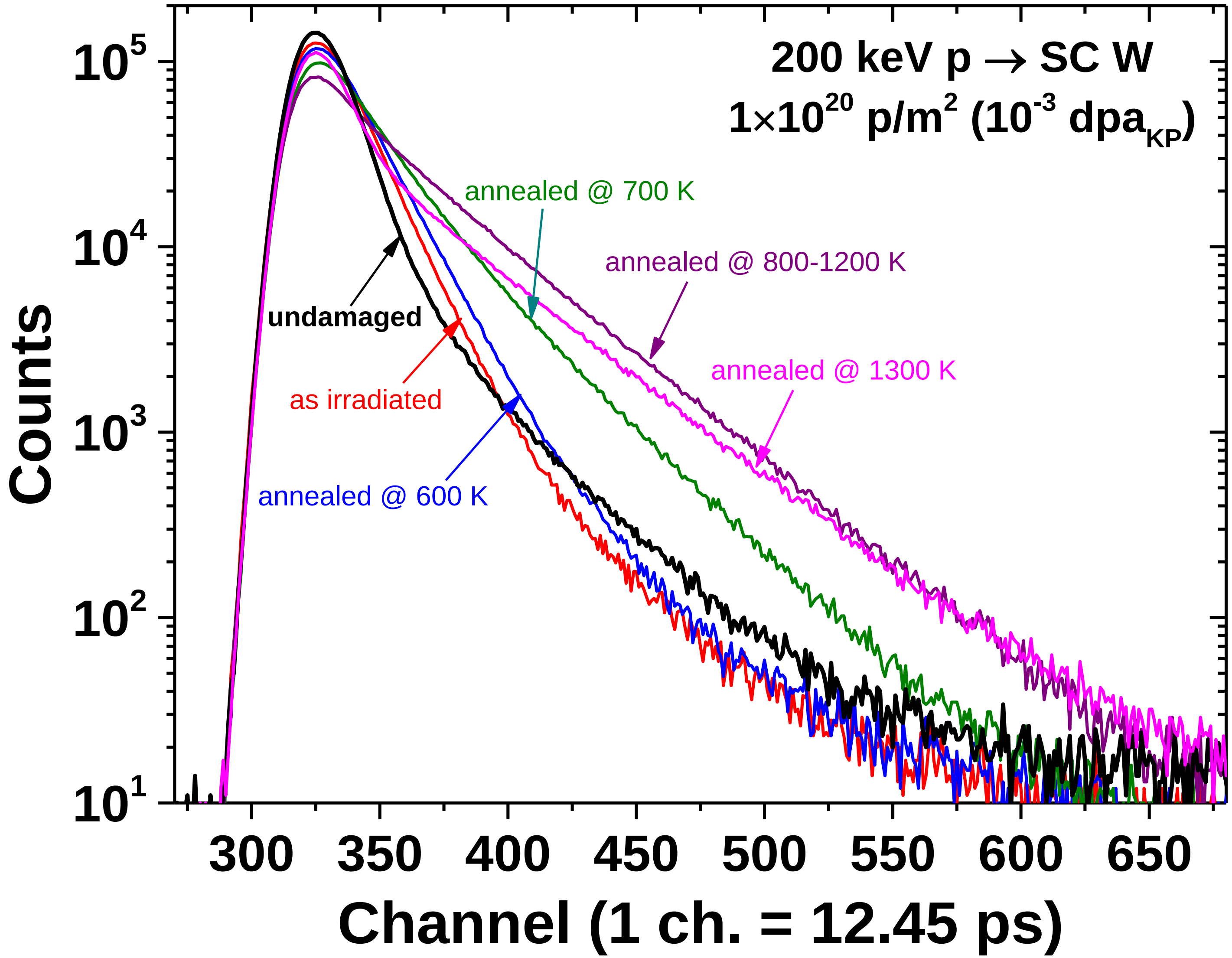

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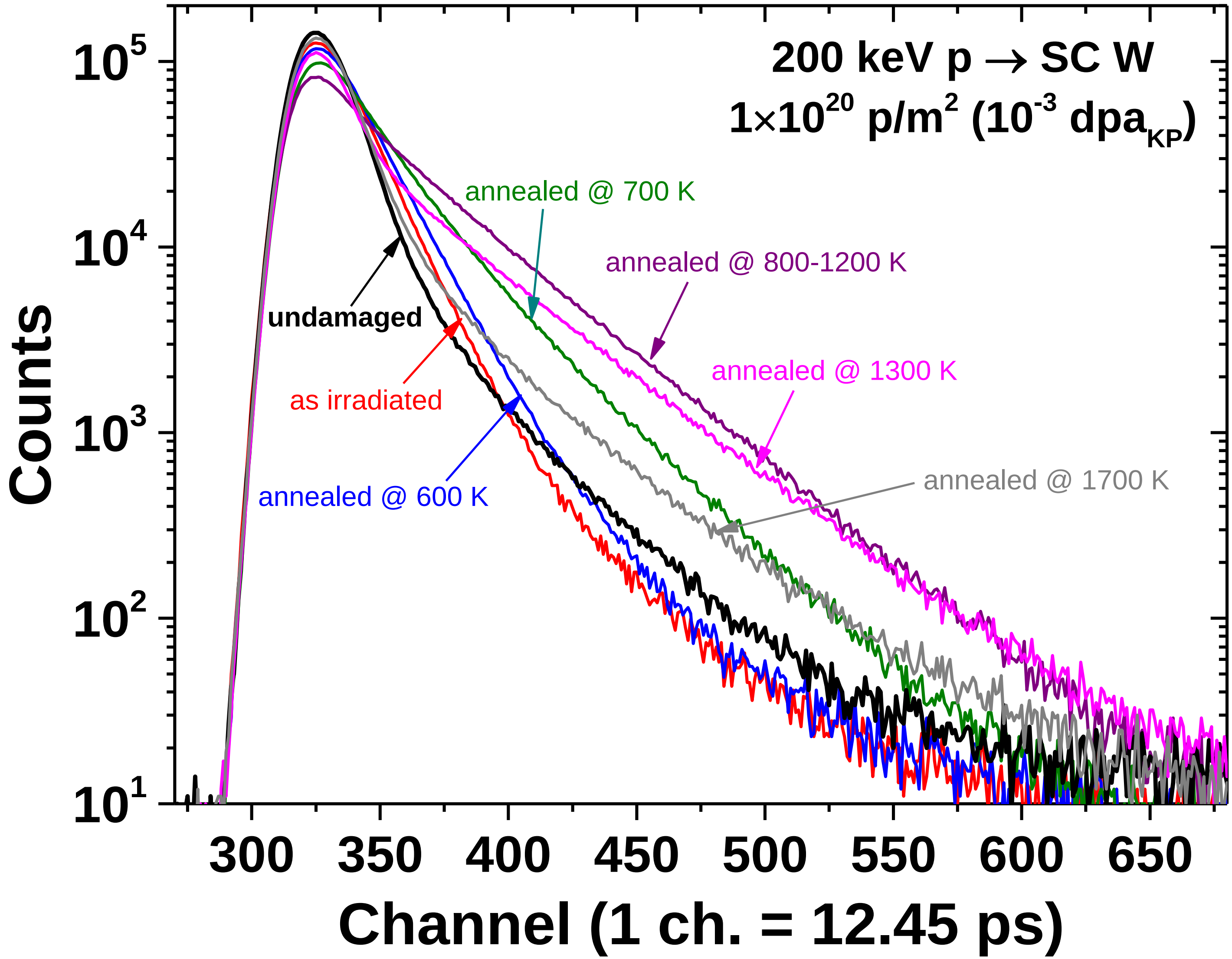

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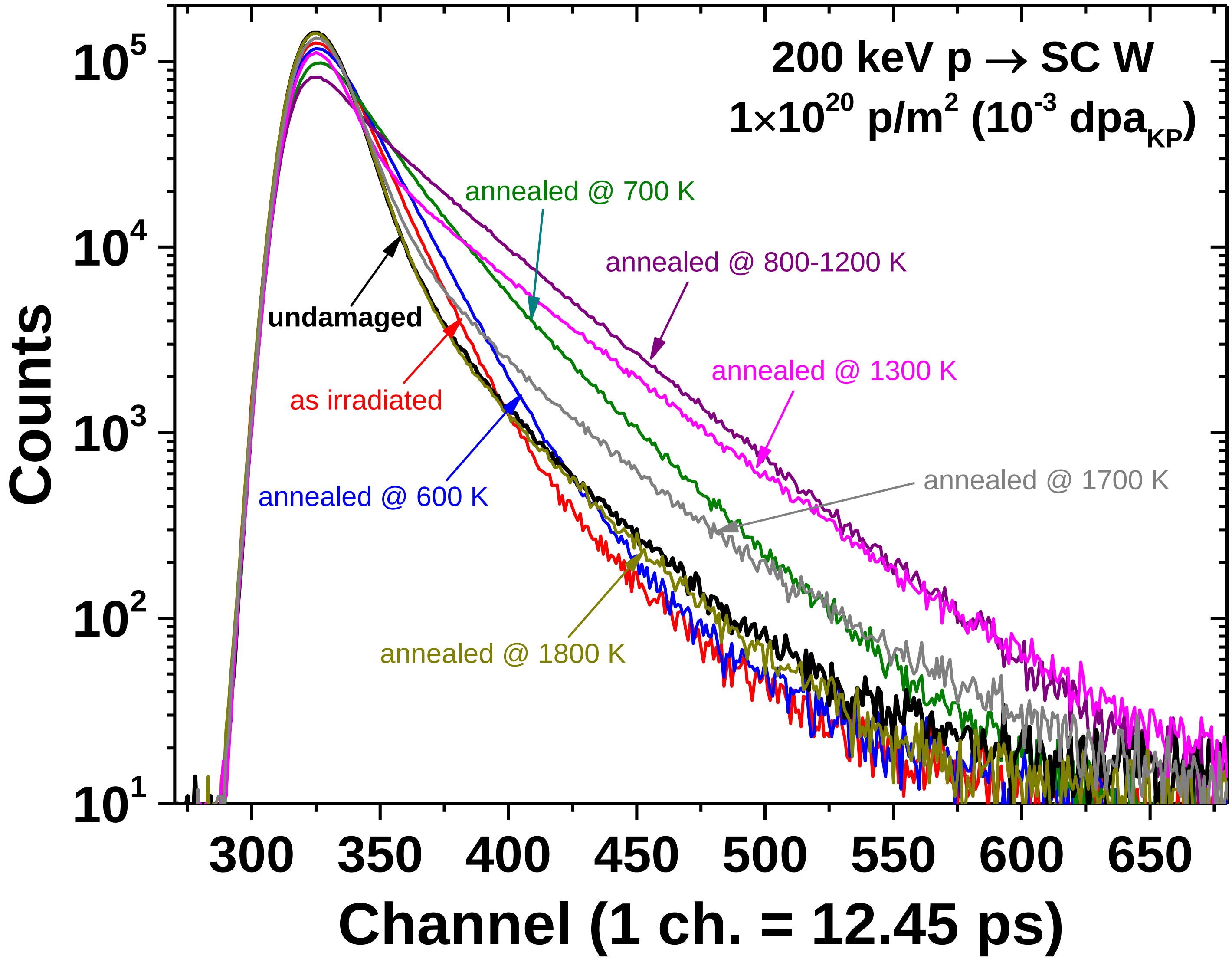
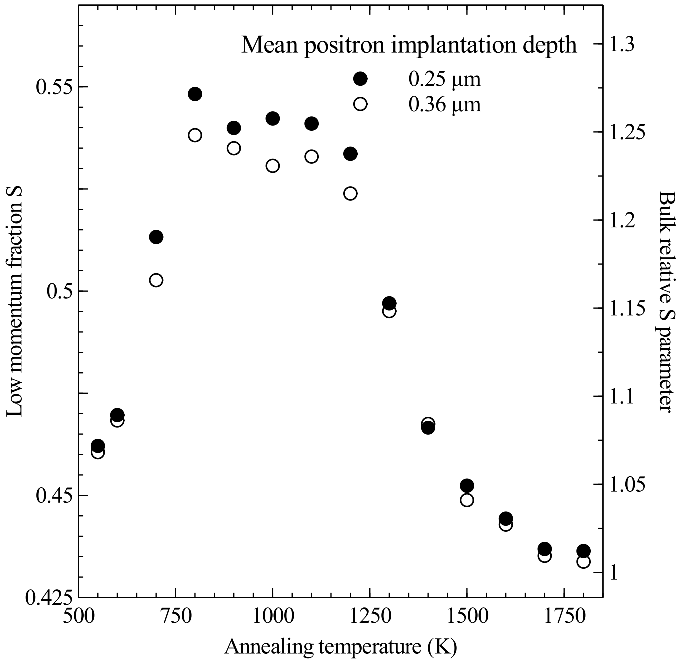
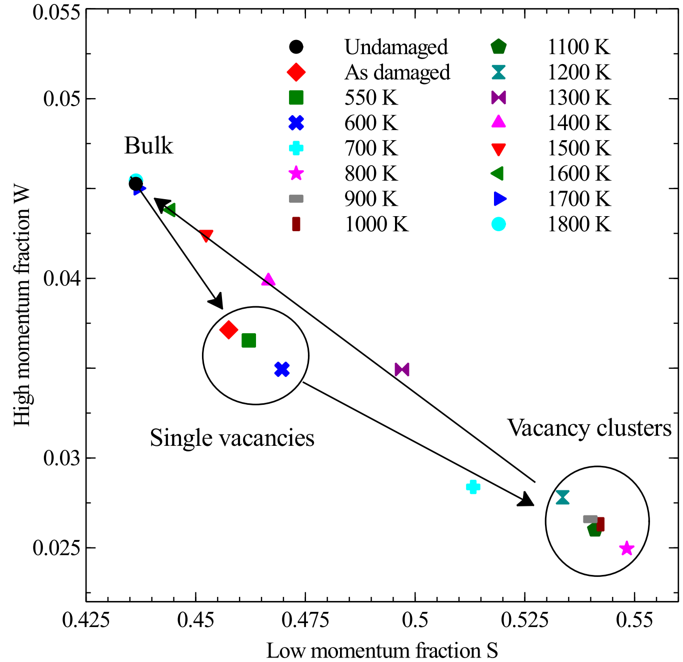


Vacancy-dominated samples: Annealing of vacancies



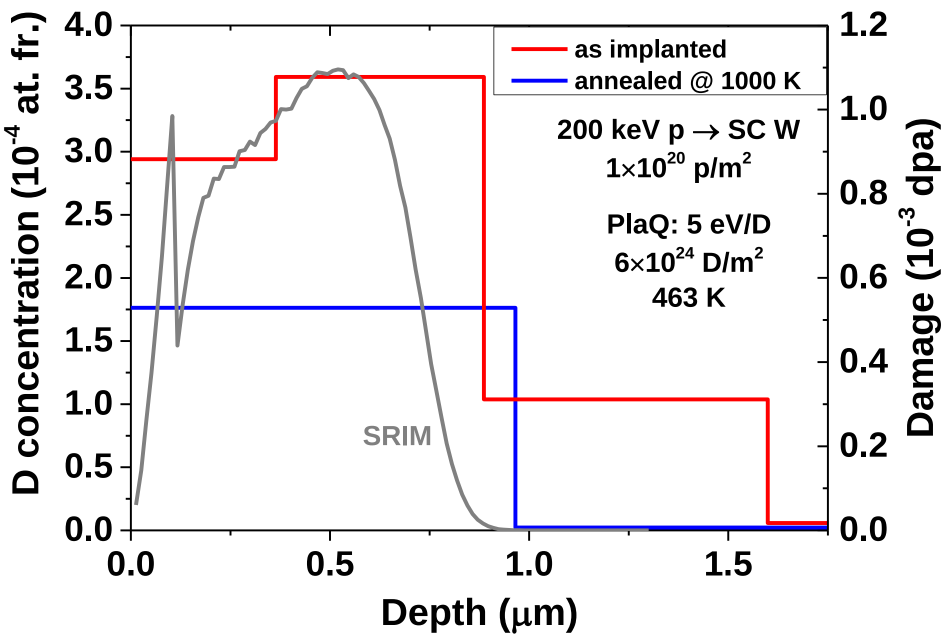


- Post-irradiation annealing at T > 600 K leads to sharp increase of S-parameter $\Rightarrow$ vacancies become mobile and start to agglomerate in clusters
- Same S-parameter after annealing between 800 K $\leq$ T $\leq$ 1200 K $\Rightarrow$ stable configuration of vacancy clusters
- Reduction of S-parameter after annealing at T $\geq$ 1300 K $\Rightarrow$ partial annealing of vacancy clusters
- Complete recovery of the defects after annealing at 1800 K

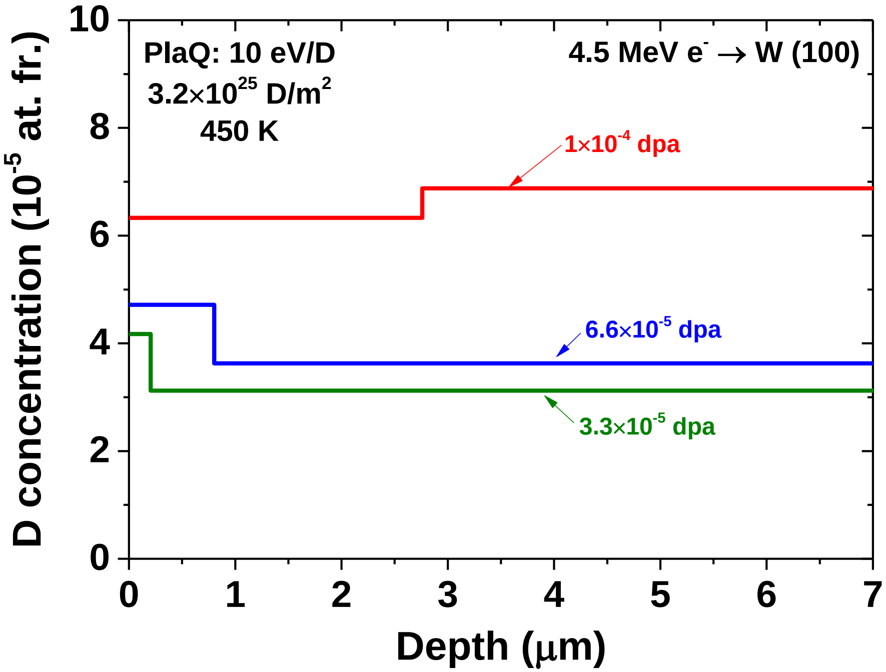
H interaction with vacancies and vacancy clusters in W

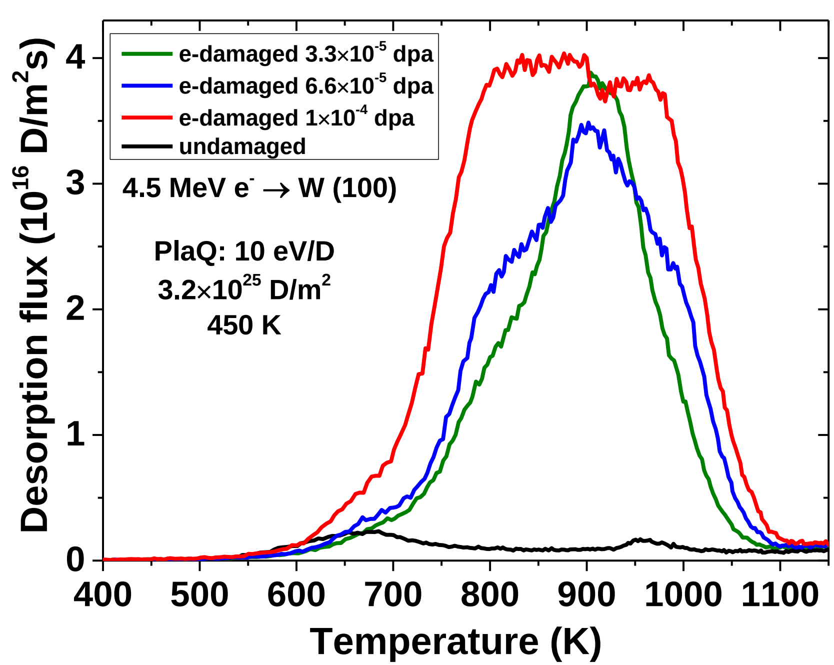
- Use of W single crystals (extremely low amount of intrinsic defects)
- No D is detectable in non-damaged single crystals

  ⇒ Minimal defect creation by plasma exposure
- Depth distributions of damage is in agreement with SRIM
- A good system for studying H interaction with vacancies/vacancy clusters



MeV electron-irradiated samples: D retention





- D concentration in undamaged sample ~5×10^{-6} at.fr.
- Increase of D bulk concentration with dpa level
- The peak position on TDS spectra is located at unexpectedly high temperatures (900 K) and broadens with increasing fluence

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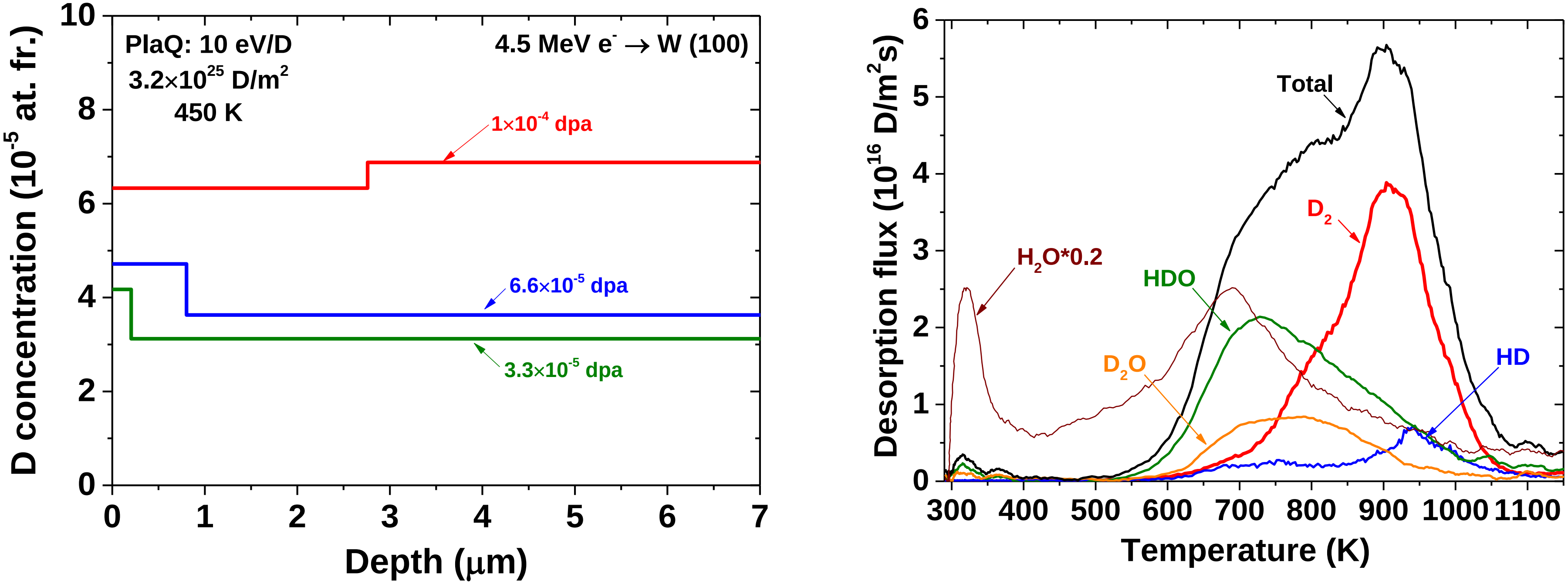


- D concentration in undamaged sample ~5×10^{-6} at.fr.
- Increase of D bulk concentration with dpa level
- The peak position on TDS spectra is located at unexpectedly high temperatures (900 K) and broadens with increasing fluence
- Strong influence of heavy water (HDO, D_2O) due to low amounts of trapped D

Damaging by different ion species

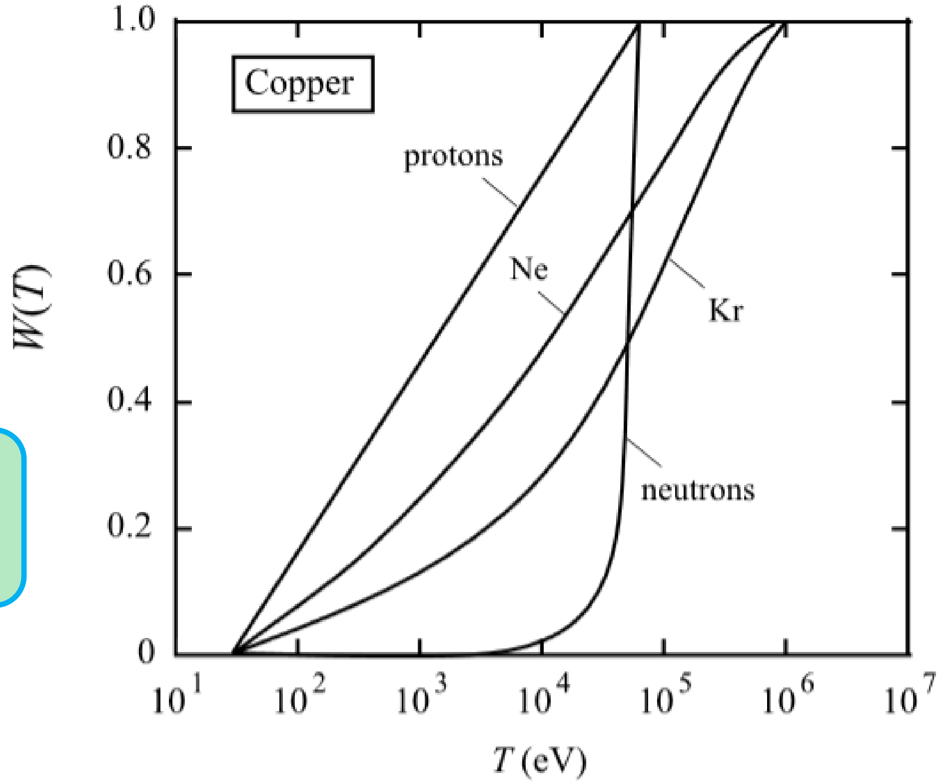
Neutrons:

narrow recoil energy spectra

Ions:

broad recoil energy spectra

Different weighted PKA spectra

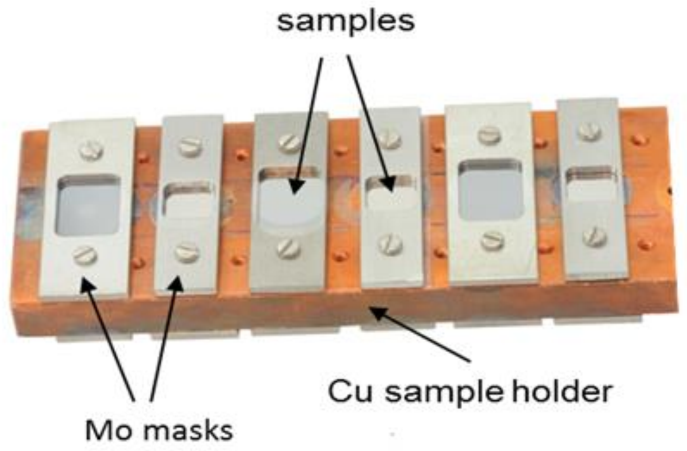


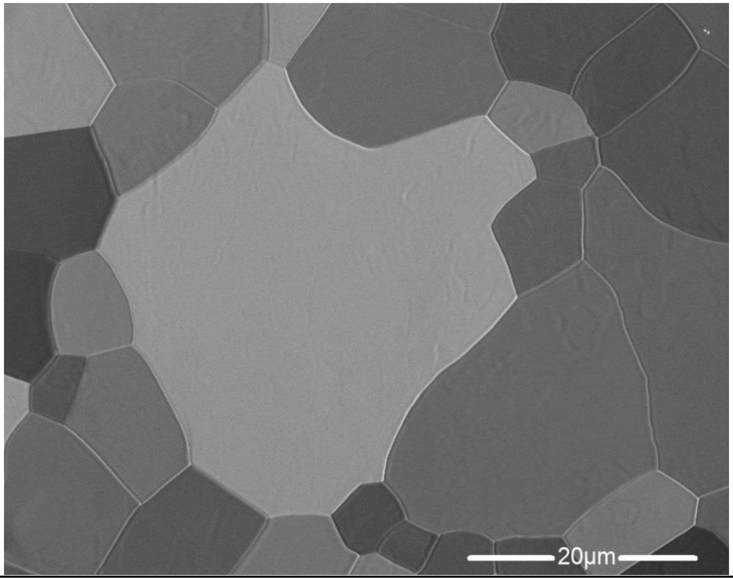
Weighted recoil spectra W(T) vers recoil energy T for 1 MeV particles in copper from Gary S.Was: „Fundamentals of Radiation Materials Science“

- **Heavier ions are closer to neutrons**
- **than light ions**

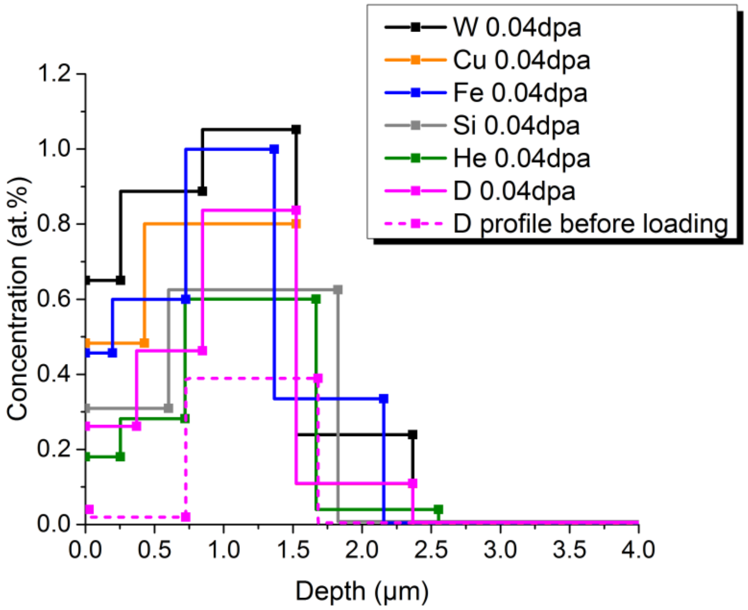
Damaging by different ion species (2)

- 3 MV IPP tandem beam line “TOF”
- Quadrupole triplet for beam focusing
- x- and y- deflection plates for beam scanning/spreading
- Faraday corner cups for flux measurement
- **Tungsten was damaged with different ion species (D, He, Si, Fe, Cu, W) at energies between 0.3 and 20 MeV to 0.04 dpa_{KP} and 0.5 dpa_{KP} in the damage peak maximum**
- **Energies were chosen to get similar damage**
- **range < 2µm**



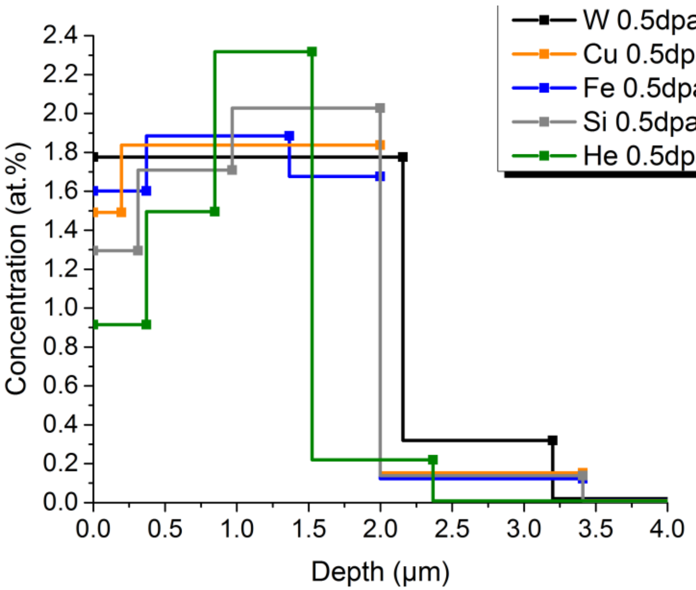


Damaging by different ion species: D depth profiles



Small dpa level (0.04 dpa)

- Comparable D concentrations for all heavy ions
- Somewhat smaller D concentrations for light ions



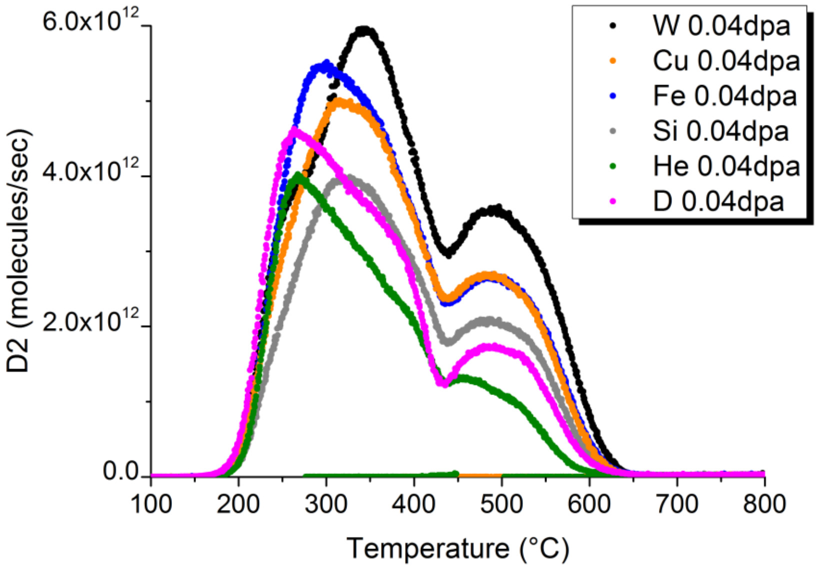
High dpa level (0.5 dpa)

- Identical for all heavy ions
- Somewhat different behaviour for He

Damaging by different ion species: TDS

Small dpa level (0.04 dpa)

- Comparable TDS spectra for all heavy ions
- Systematic mass dependence for lighter ions



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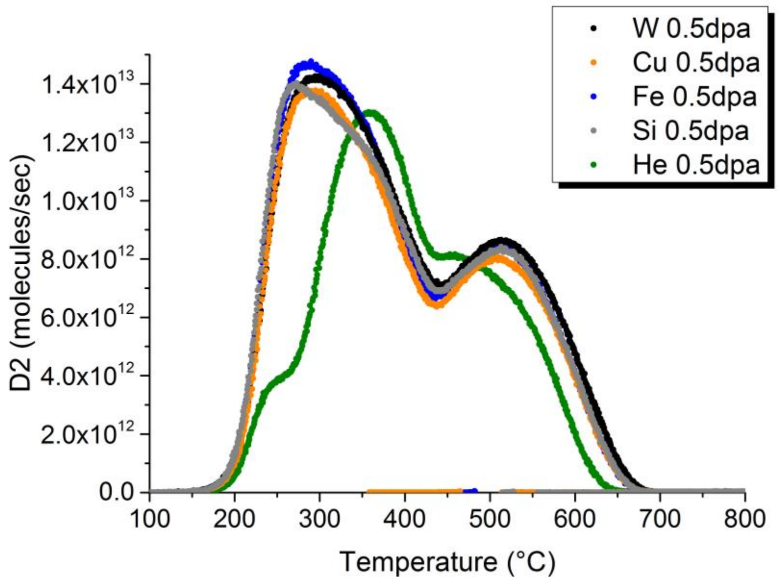
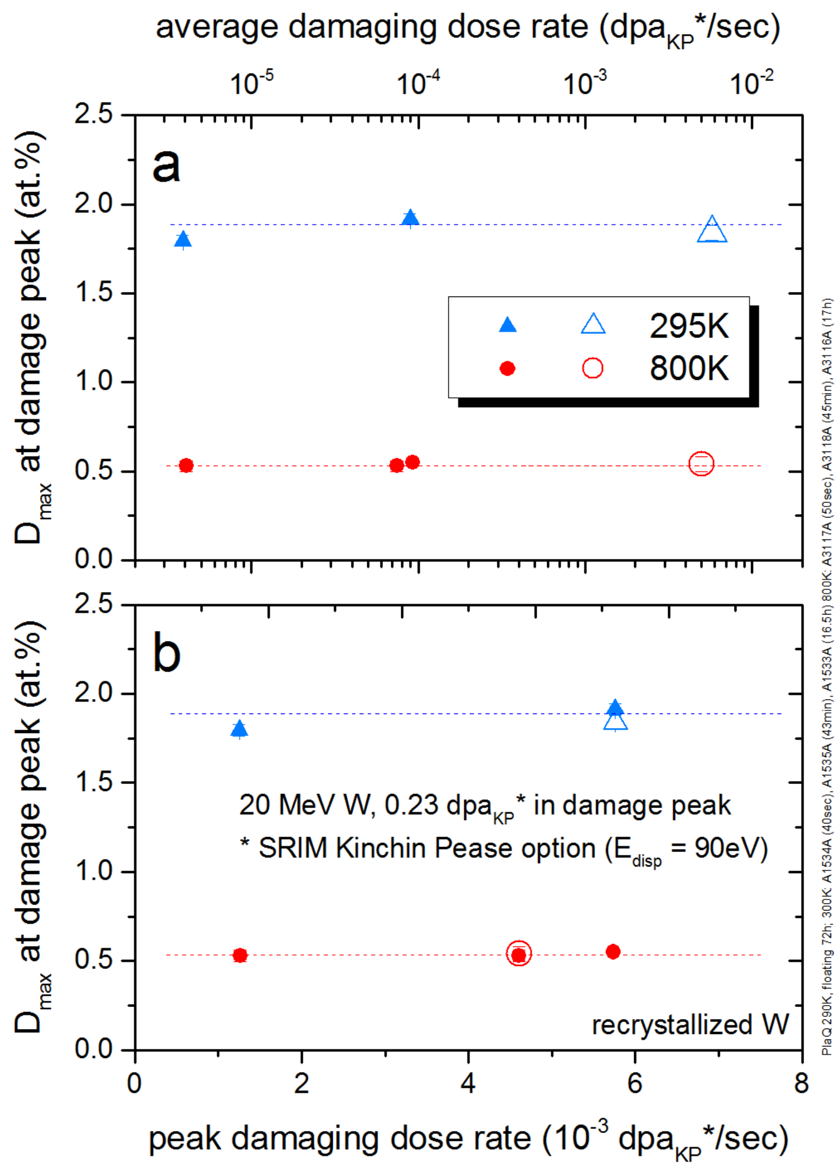


Damage rate dependence

0.23 dpa level

- No damage rate dependence



Conclusions

Lack of reliable data on H interaction with different types of defects in W

⇒ Samples with defined specific defects for fundamental understanding

- **Dislocations by mechanical work at elevated temperatures**
  - Dislocations have only small influence on D retention at 450 K
  - Mechanical damage faciliates formation of blisters

- **Single vacancies by MeV electron beam or 200 keV proton irradiation**
  - Vacancy clusters > 600 K; Complete defect annealing > 1700 K
  - Heavy water at low dpa levels

Samples damaged by different MeV ions

- Comparable results for all heavy ions (especially at high dpa)
- No damage rate dependence