Possible defect stabilization due to simultaneous deuterium exposure during annealing in self-ion damaged W

M.J. Simmonds, A. Založnik, T. Schwarz-Selinger [1], M.I. Patino,
M.J. Baldwin, R.P. Doerner, and G.R. Tynan

Center for Energy Research, UC San Diego, 9500 Gilman Dr., La Jolla, CA 92093-0417, USA
[1] Max-Planck-Institut für Plasmaphysik, Boltzmannstrasse 2, D-85748 Garching, Germany

Motivation

- Synergistic effects are known to affect defect creation/recovery strong effect on hydrogen isotope retention
- There is a strong need for simultaneous experiments (hydrogen isotope exposure during defect creation/annealing)
- These synergistic effects need to be included in the models in order to accurately predict hydrogen isotope retention and permeation

Studying annealing of heavy-ion damaged W

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Vacuum annealing (**W-A-D**)

W self-damaging → **A**nnealing (vacuum) → **D** plasma exposure → NRA & TDS

Effect of D filled defects on annealing

Vacuum annealing (**W-A-D**)

W self-damaging → **A**nnealing (vacuum) → **D** plasma exposure → NRA & TDS

Vacuum annealing after plasma exposure (**W-D-A-D**)

W self-damaging → **D** plasma exposure → **A**nnealing (vacuum) → **D** plasma exposure → NRA & TDS

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D presence during annealing clearly different

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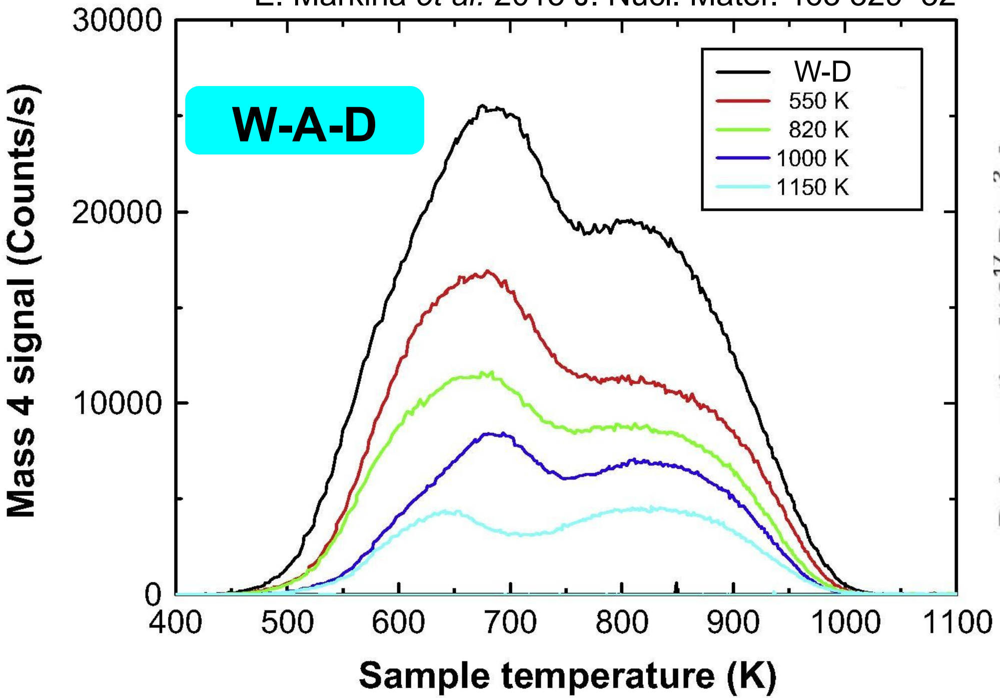

- monotonic decrease in **LT peak**
- monotonic decrease in **HT peak**

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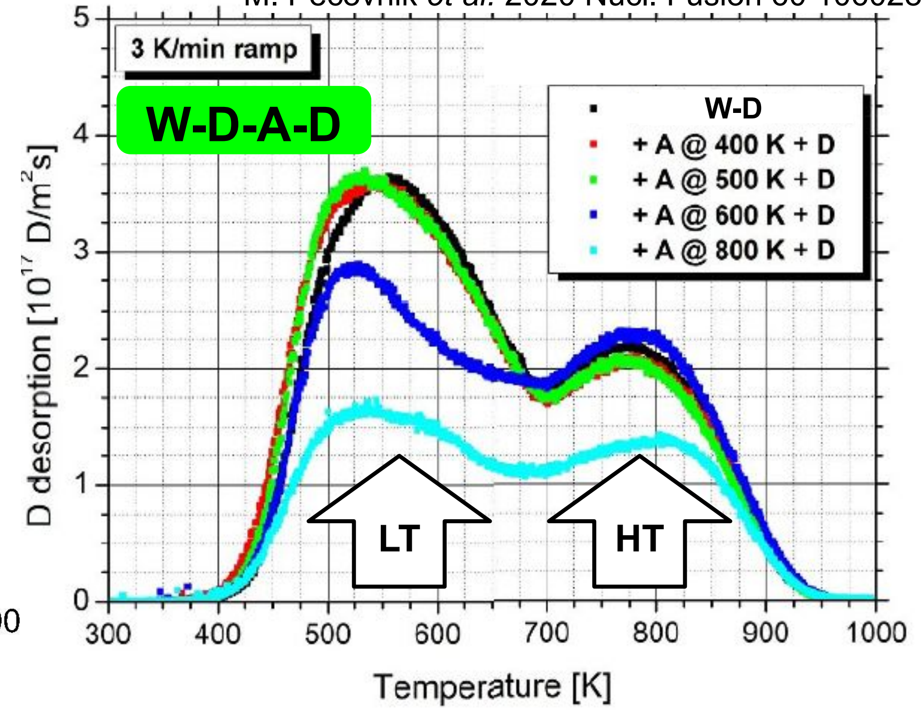


- large change in **LT peak** at **600 K** anneal
- little change in **HT peak** until **800 K** anneal

Next step: study simultaneous annealing+plasma

Vacuum annealing (**W-A-D**)

W self-damaging → **A**nnealing (vacuum) → **D** plasma exposure → NRA & TDS

Vacuum annealing after plasma exposure (**W-D-A-D**)

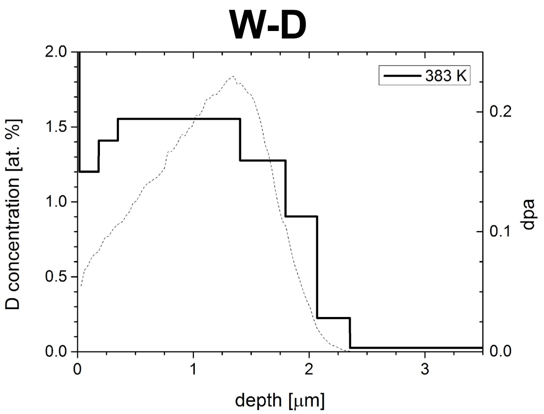
W self-damaging → **D** plasma exposure → **A**nnealing (vacuum) → **D** plasma exposure → NRA & TDS

Annealing during plasma exposure (**W-D-AD-D**)

W self-damaging → **D** plasma exposure → **A**nnealing + **D** plasma → **D** plasma exposure → NRA & TDS

W-D (no annealing fiducial)

- strong D retention in damaged zone (< 2.2 µm)

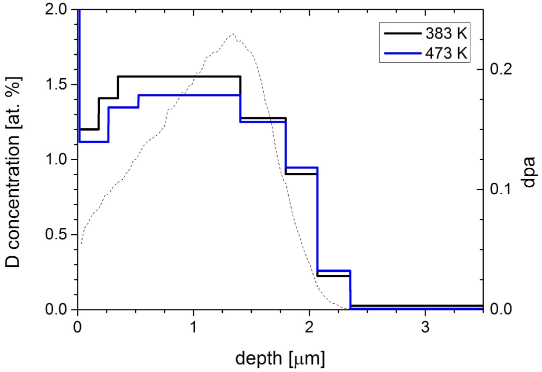


Retention diverges for 473 K anneal

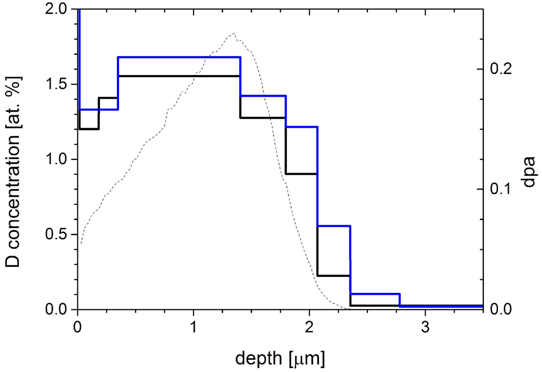
- strong D retention in damaged zone (< 2.2 μm)
- D retention change after vacuum anneal or plasma anneal

A (K)	W-A-D	W-D-AD-D
473	small decrease	small increase

W-A-D



W-D-AD-D

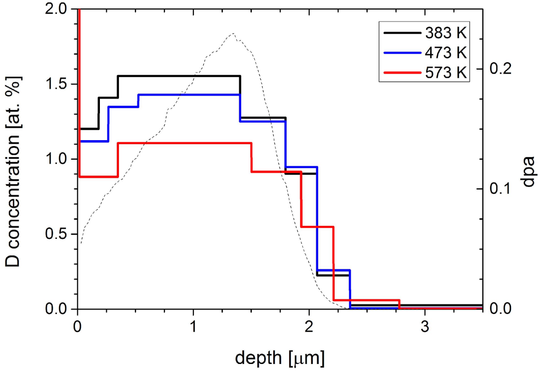


Retention continues to diverge at 573 K

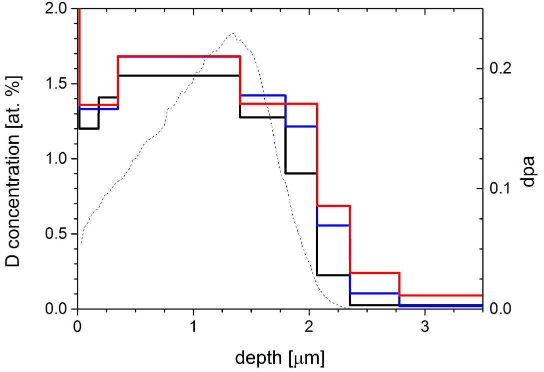
- strong D retention in damaged zone (< 2.2 μm)
- D retention change after vacuum anneal or plasma anneal

A (K)	W-A-D	W-D-AD-D
473	small decrease	small increase
573	further decrease	slight increase

W-A-D



W-D-AD-D



Retention ~constant up to 673 K plasma anneal

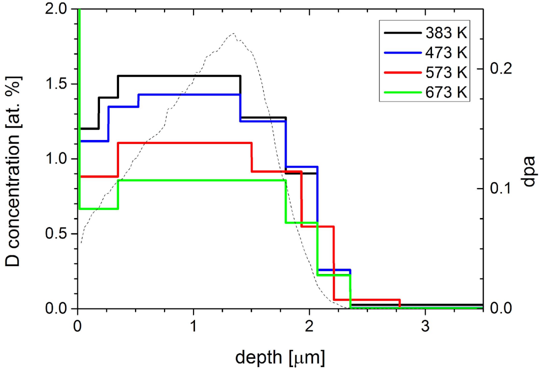
PISCES

- strong D retention in damaged zone (< 2.2 μm)
- D retention change after vacuum anneal or plasma anneal

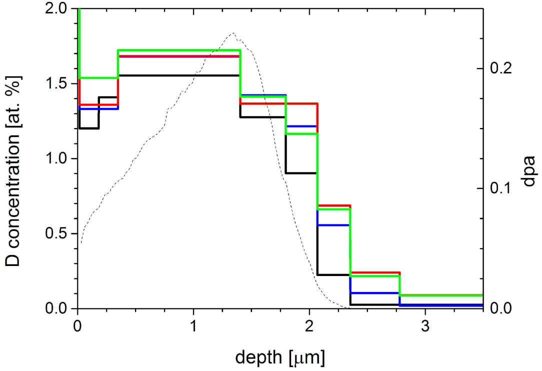
A (K)	W-A-D	W-D-AD-D
473	small decrease	small increase
573	further decrease	slight increase
673	further decrease	almost no change

- **NRA shows nearly constant D retention in damage zone up to 673 K for plasma anneal □ defect stabilization**

W-A-D



W-D-AD-D



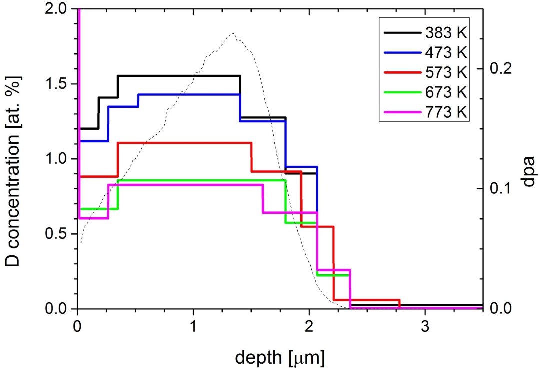
Significant D depopulation and recovery at 773 K

- strong D retention in damaged zone (< 2.2 μm)
- D retention change after vacuum anneal or plasma anneal

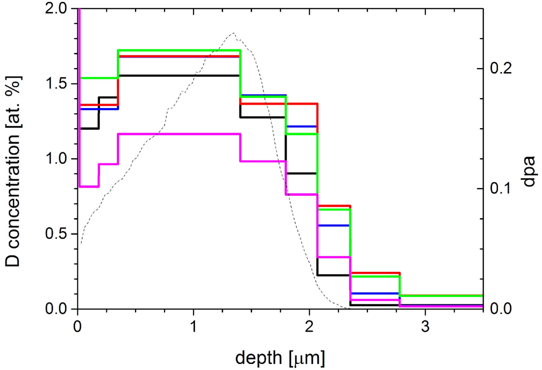
A (K)	W-A-D	W-D-AD-D
473	small decrease	small increase
573	further decrease	slight increase
673	further decrease	almost no change
773	little change	significant decrease

- **NRA shows nearly constant D retention in damage zone up to 673 K for plasma anneal □ defect stabilization**

W-A-D



W-D-AD-D



W-D (fiducial for comparison)

- two desorption peaks
  - low-temperature (LT) □ mono-vacancies/dislocations?
  - high-temperature (HT) □ vacancy clusters?

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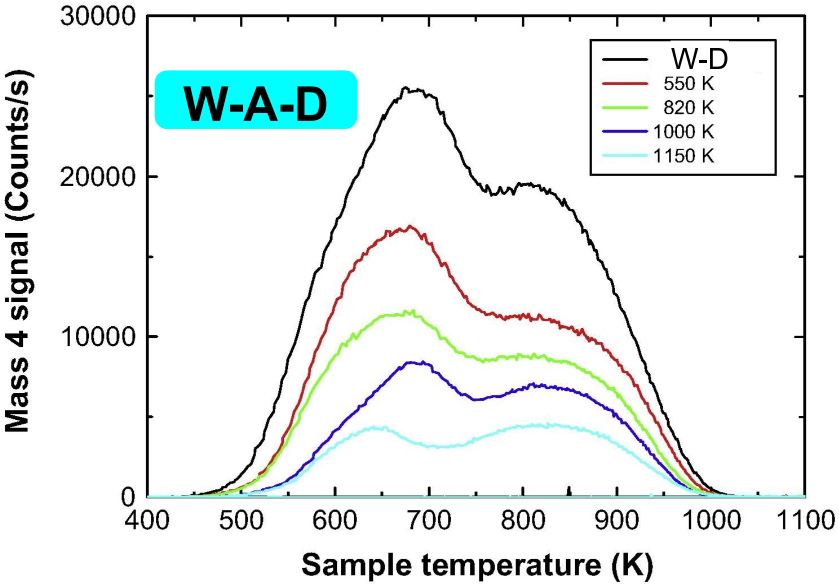
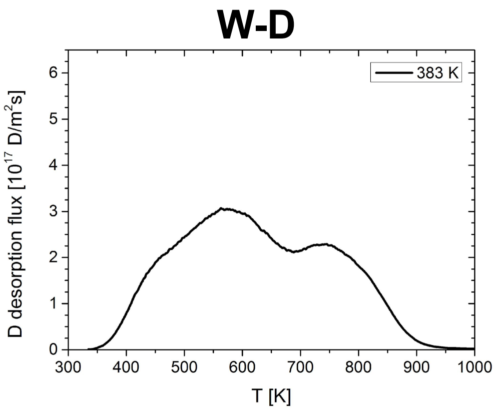




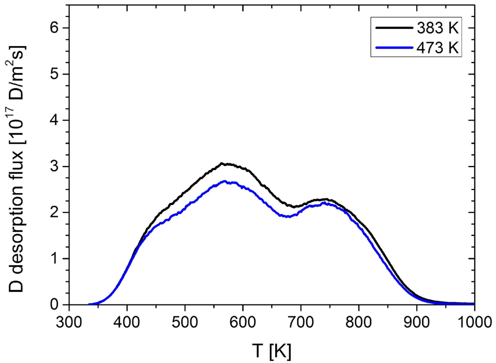
Primarily LT grows (beyond damage zone)

- two desorption peaks
  - low-temperature (LT) □ mono-vacancies/dislocations?
  - high-temperature (HT) □ vacancy clusters?

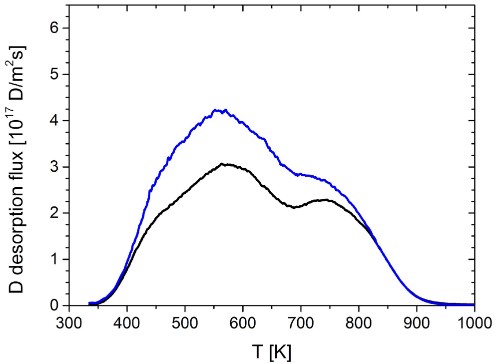
A (K)	W-A-D	W-D-AD-D
473	small decrease of LT no change of HT	strong increase of LT small increase of HT

- **NRA shows nearly constant D retention in damage zone up to 673 K for plasma anneal □ defect stabilization**

W-A-D



W-D-AD-D



LT & HT grow (beyond damage zone)

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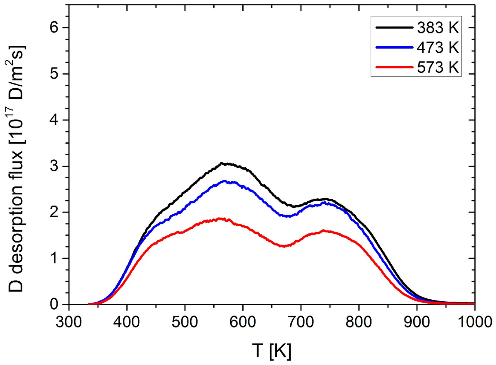
- two desorption peaks
  - low-temperature (LT) □ mono-vacancies/dislocations?
  - high-temperature (HT) □ vacancy clusters?

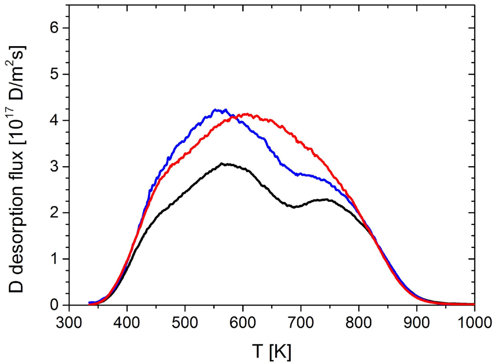
A (K)	W-A-D	W-D-AD-D
473	small decrease of LT no change of HT	strong increase of LT small increase of HT
573	strong decrease of both LT & HT	shape changed LT & HT not resolved

- **NRA shows nearly constant D retention in damage zone up to 673 K for plasma anneal □ defect stabilization**

W-A-D



W-D-AD-D



Significant growth of HT (beyond damage zone)

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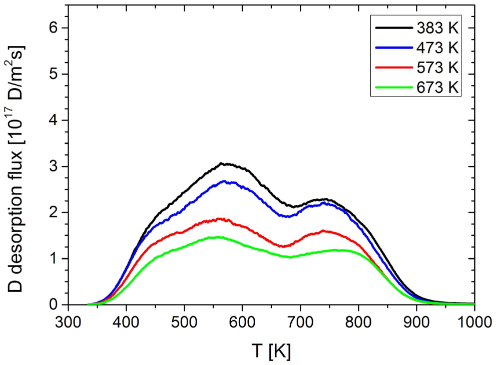
- two desorption peaks
  - low-temperature (LT) □ mono-vacancies/dislocations?
  - high-temperature (HT) □ vacancy clusters?

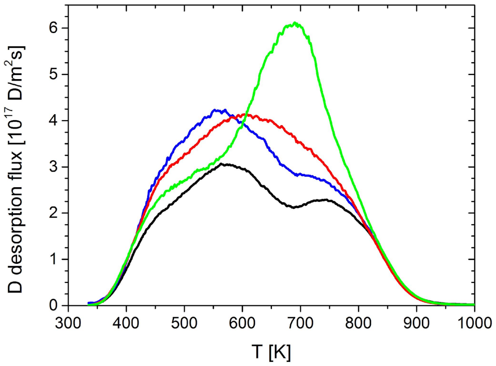
A (K)	W-A-D	W-D-AD-D
473	small decrease of LT no change of HT	strong increase of LT small increase of HT
573	strong decrease of both LT & HT	shape changed LT & HT not resolved
673	further decrease of both LT & HT	decrease of LT strong increase of HT

- **NRA shows nearly constant D retention in damage zone up to 673 K for plasma anneal □ defect stabilization**

W-A-D



W-D-AD-D



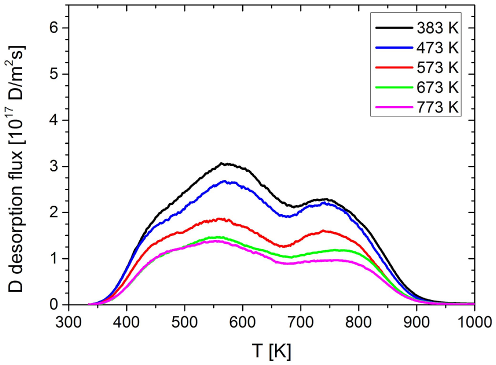
Significant shift to higher T for HT

- two desorption peaks
  - low-temperature (LT) □ mono-vacancies/dislocations?
  - high-temperature (HT) □ vacancy clusters?

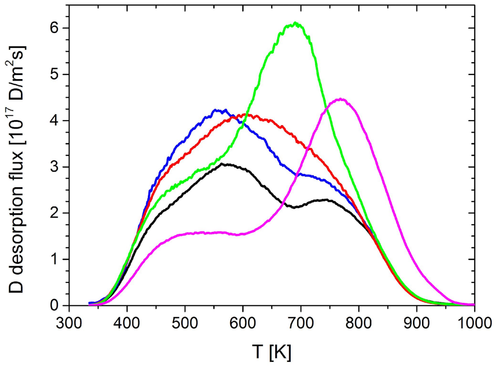
A (K)	W-A-D	W-D-AD-D
473	small decrease of LT no change of HT	strong increase of LT small increase of HT
573	strong decrease of both LT & HT	shape changed LT & HT not resolved
673	further decrease of both LT & HT	decrease of LT strong increase of HT
773	little change of both LT & HT	strong decrease of LT HT shift and still high

- **NRA shows nearly constant D retention in damage zone up to 673 K for plasma anneal □ defect stabilization**

W-A-D

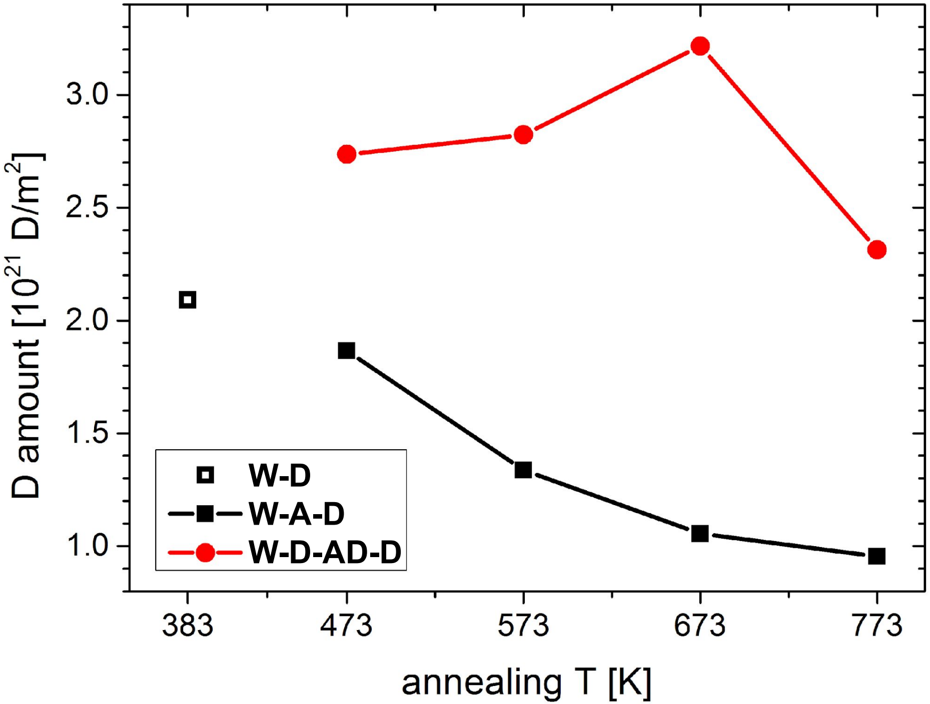


W-D-AD-D



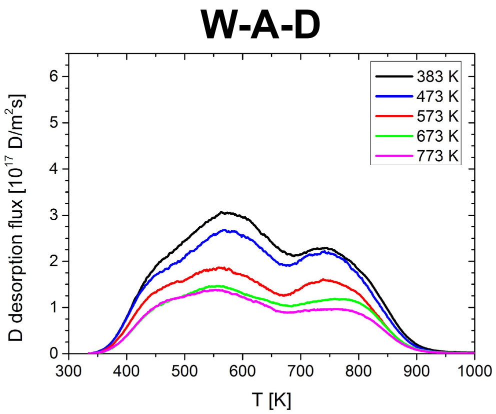
Total D Retention (TDS)

- Initial increase probably due to higher D fluence in the case of W-D-AD-D
- Clearly very different behavior of defect recovery when D is present
- Modeling can give some insight into defect stabilization in the presence of D



Vacuum anneal well fit by 3 traps

- typically 3 trap types used to model TDS spectra
  - LT peak, HT peak, & HT tail



Plasma anneal needs additional trap

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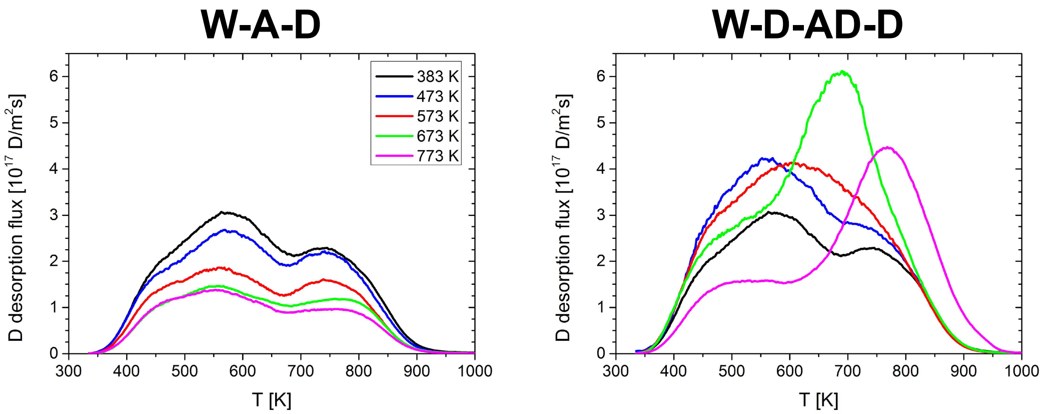
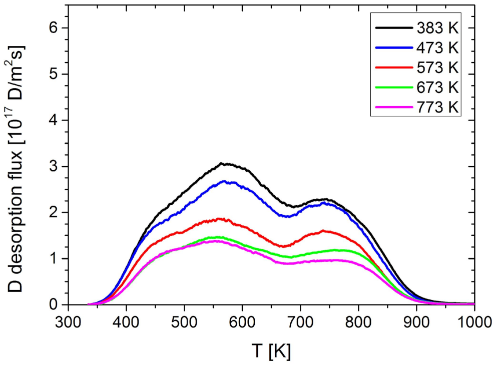
- typically 3 trap types used to model TDS spectra
  - LT peak, HT peak, & HT tail
- this work revealed the existence of the 4th trap type (small vacancy clusters?)



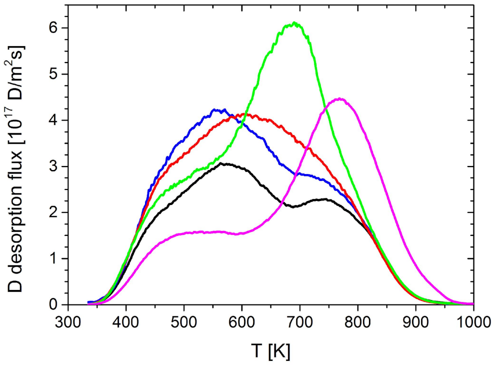
Vacuum anneal trap conc. monotonic decrease

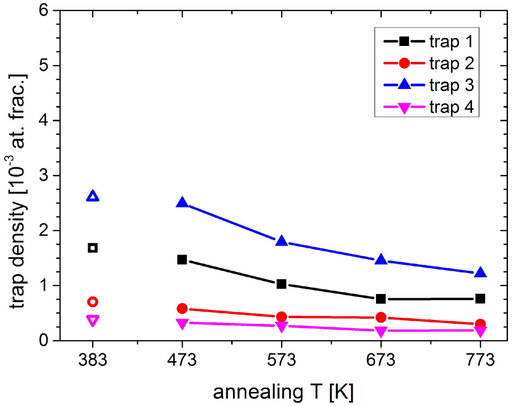
- typically 3 trap types used to model TDS spectra
    - LT peak, HT peak, & HT tail
- this work revealed the existence of the 4th trap type (small vacancy clusters?)
- W-A-D monotonically decreasing trap densities

W-A-D



W-D-AD-D





Plasma anneal exhibits complex trap evolution

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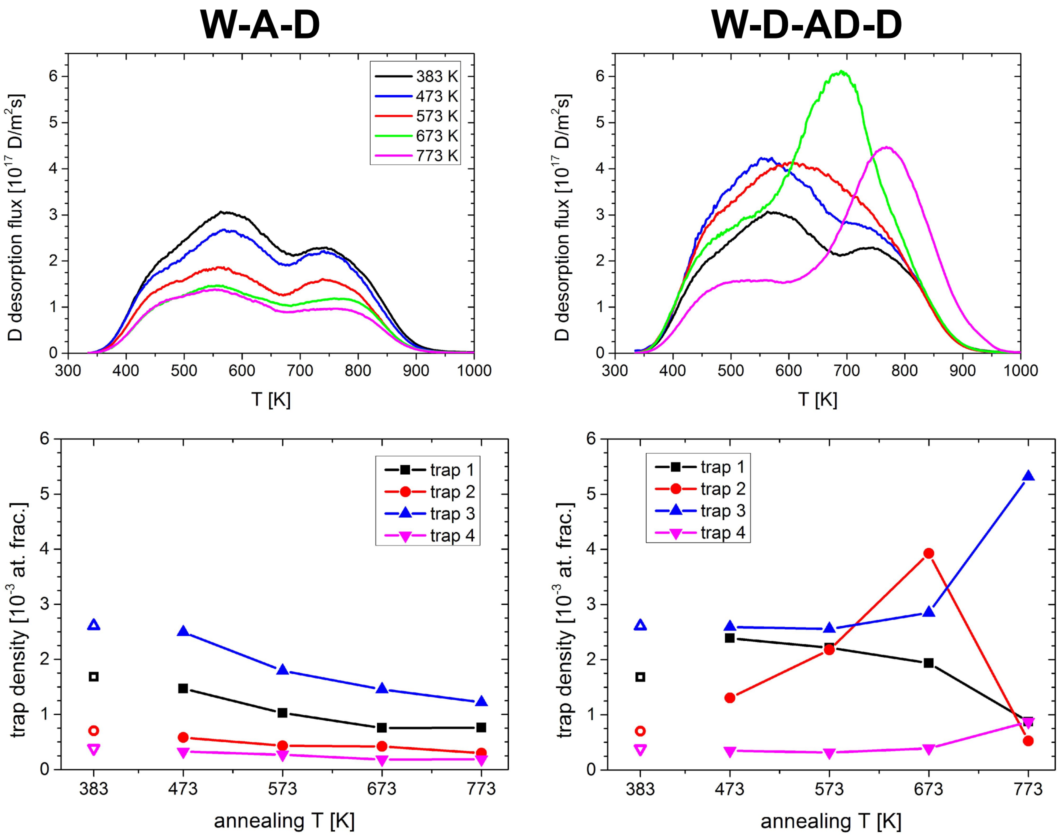
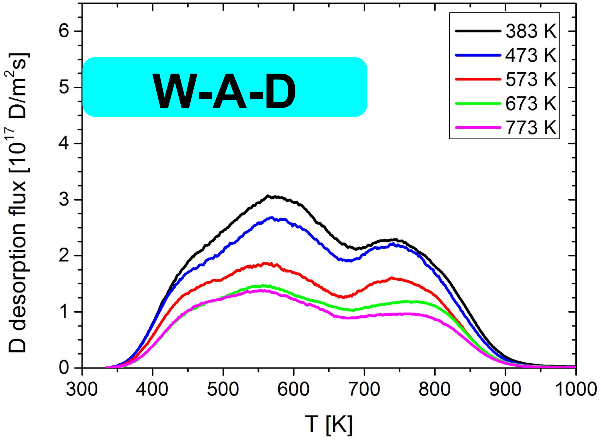
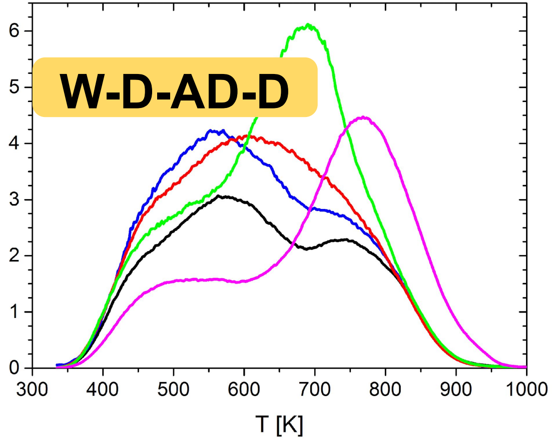
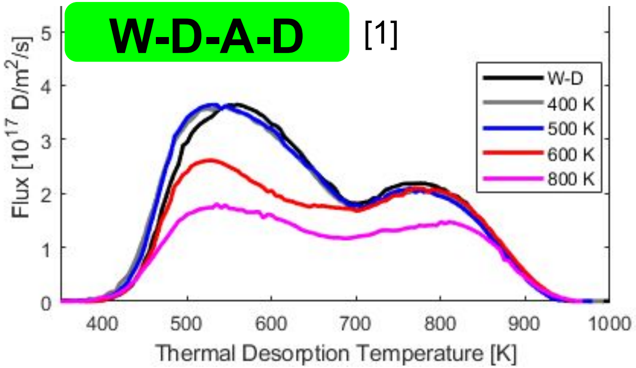
- typically 3 trap types used to model TDS spectra
  - LT peak, HT peak, & HT tail
- this work revealed the existence of the 4th trap type (small vacancy clusters?)
- W-A-D monotonically decreasing trap densities
- W-D-AD-D complex evolution of trap densities

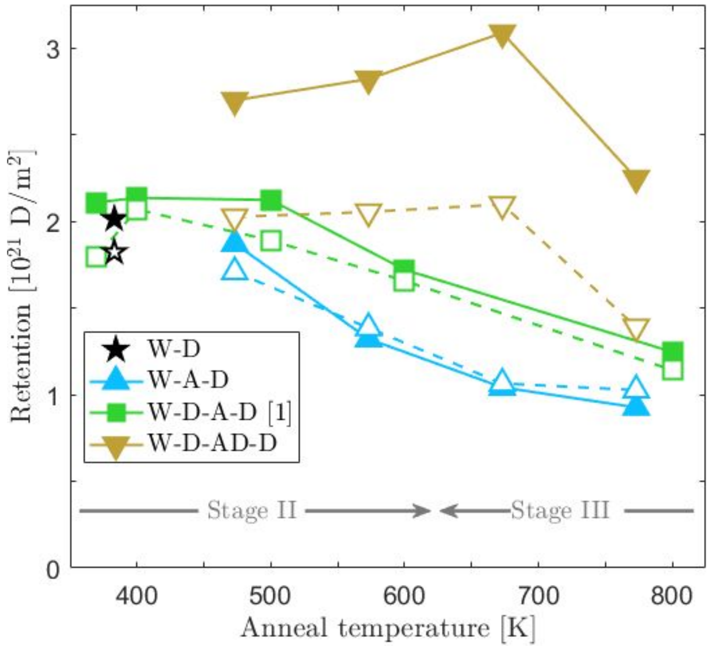


Total D retention (including W-D-A-D)









- open = NRA (damage zone)
- filled = TDS

[1] M. Pečovnik *et al.* 2020 Nucl. Fusion 60 106028

D presence during annealing clearly different

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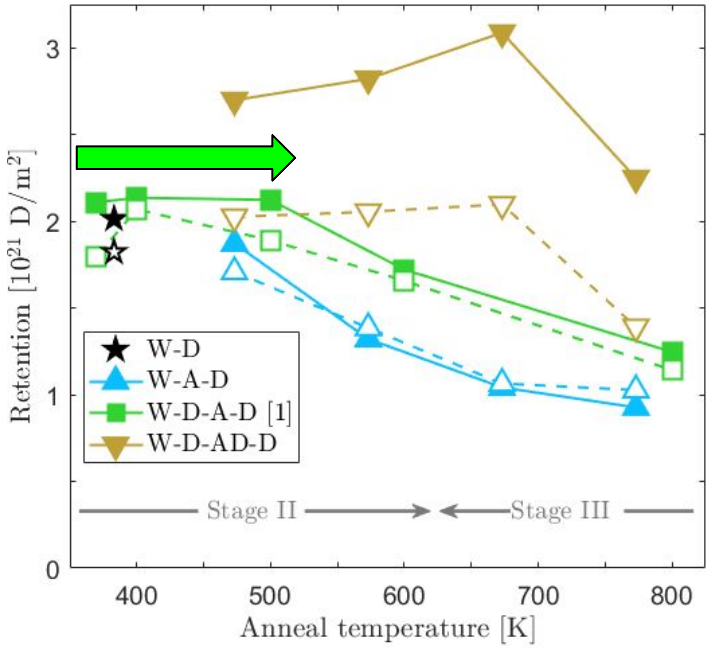
- W-A-D
  - all traps empty during anneal
- W-D-A-D
  - traps partially D filled reduced recovery
  - D continuously desorbed while held-at-temperature



- open = NRA (damage zone)
- filled = TDS

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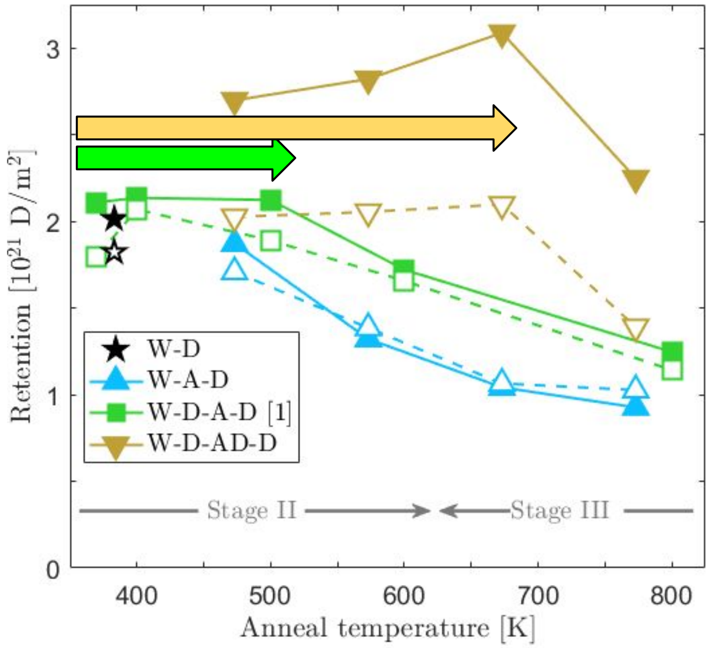
D presence during annealing clearly different

PISCES

- W-A-D
  - all traps empty during anneal
- W-D-A-D
  - traps partially D filled □ reduced recovery
  - D continuously desorbed while held-at-temperature
- W-D-AD-D
  - traps partially D filled □ reduced recovery
  - D continuously repopulated with D plasma exposure held-at-temperature
  - mobile defects annihilate at surface/GB but defects migrating further into bulk slowed/stabilized by D?



- open = NRA (damage zone)
- filled = TDS

[1] M. Pečovnik *et al.* 2020 Nucl. Fusion 60 106028

Thank you!

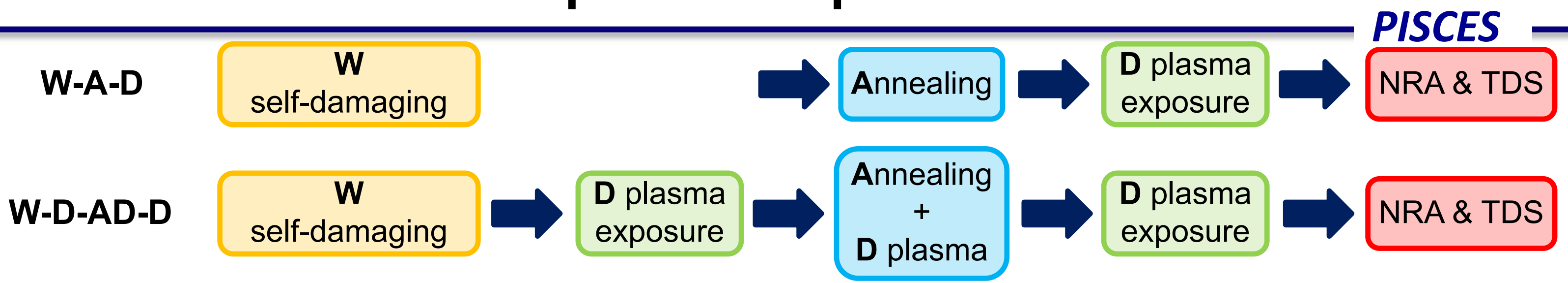
PISCES

- Annealing of W simultaneously exposed to D plasma:
  - obvious synergistic effects
  - reduced defect recovery D induced stabilization of defects
  - Further experimental details
    - M.J. Simmonds et al. 2022 Nucl. Fusion 62 036012

- Future:
  - ending Be work (Be box is gone!) and focusing on synergistic effects in W
  - finalizing plans for heavy ion accelerator (NEC) installation/coupling to PISCES-RF
  - improving modeling capabilities, including synergistic effects in the codes

Experimental Details

Sample Prep



- PCW samples:
  - 1.5 mm thick and 6 mm dia
  - polished and recrystallized

Heavy-ion induced defects

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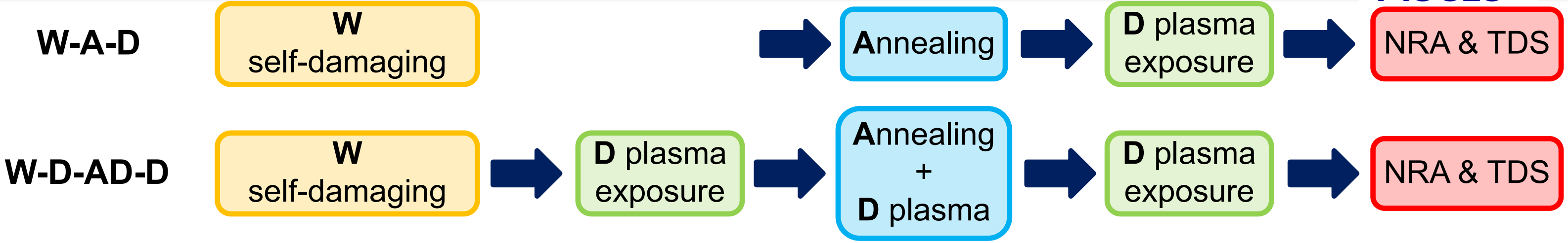

- PCW samples:
  - 1.5 mm thick and 6 mm dia
  - polished and recrystallized
- W self-damaging:
  - 20.3 MeV W^{6+} ions at 295 K
  - 7.87 x 10^{17} ions/m^2 □ 0.23 dpa

D decoration of defects

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W-A-D: W self-damaging → Annealing → D plasma exposure → NRA & TDS

W-D-AD-D: W self-damaging → D plasma exposure → Annealing + D plasma → D plasma exposure → NRA & TDS

- PCW samples:
  - 1.5 mm thick and 6 mm dia
  - polished and recrystallized
- W self-damaging:
  - 20.3 MeV W^{6+} ions at 295 K
  - 7.87 x 10^{17} ions/m^2 □ 0.23 dpa

- D plasma exposure:
  - temperature 383 K
  - flux 1.1 x 10^{21} D/m^2s
  - impact energy ~ 67 eV
  - fluence 2 x 10^{25} D/m^2 (5 h)
    (1 x 10^{25} D/m^2 before annealing)

Annealing with or without D plasma

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W-A-D: **W** self-damaging → **Annealing** → **D** plasma exposure → NRA & TDS

W-D-AD-D: **W** self-damaging → **D** plasma exposure → **Annealing** + **D** plasma → **D** plasma exposure → NRA & TDS

- PCW samples:
  - 1.5 mm thick and 6 mm dia
  - polished and recrystallized
- W self-damaging:
  - 20.3 MeV W^{6+} ions at 295 K
  - 7.87 x 10^{17} ions/m^2 □ 0.23 dpa

- D plasma exposure:
  - temperature 383 K
  - flux 1.1 x 10^{21} D/m^2s
  - impact energy ~ 67 eV
  - fluence 2 x 10^{25} D/m^2 (5 h)
    (1 x 10^{25} D/m^2 before annealing)
- Annealing:
  - 473 K, 573 K, 673 K, and 773 K for 1 h
  - D fluence 4 x 10^{24} D/m^2

Quantification of D retention

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W-A-D: **W** self-damaging → **Annealing** → **D** plasma exposure → NRA & TDS

W-D-AD-D: **W** self-damaging → **D** plasma exposure → **Annealing + D** plasma → **D** plasma exposure → NRA & TDS

- PCW samples:
  - 1.5 mm thick and 6 mm dia
  - polished and recrystallized
- W self-damaging:
  - 20.3 MeV W^{6+} ions at 295 K
  - 7.87 x 10^{17} ions/m^2 □ 0.23 dpa
- NRA & TDS:
  - ^{3}He ions with 0.5, 0.69, 0.8, 1.2, 1.8, 2.4, 3.2, and 4.5 MeV □ protons and alphas
  - TDS at 0.05 K/s

- D plasma exposure:
  - temperature 383 K
  - flux 1.1 x 10^{21} D/m^2s
  - impact energy ~ 67 eV
  - fluence 2 x 10^{25} D/m^2 (5 h) (1 x 10^{25} D/m^2 before annealing)
- Annealing:
  - 473 K, 573 K, 673 K, and 773 K for 1 h
  - D fluence 4 x 10^{24} D/m^2