O-9

D retention in bulk Be and D co-deposited in Be layers studied by 3 different thermal desorption techniques and their modelling by CRDS

19th June 2019 I Miro Zlobinski*, ***D. Matveev***, M. Eichler, T. Dittmar, G. De Temmerman, C. Porosnicu, B. Unterberg, G. Sergienko, S. Brezinsek, D. Nicolai, A. Terra, M. Rasinski, B. Spilker, M. Freisinger, S. Möller, Ch. Linsmeier, C. P. Lungu, P. Dinca

Jülich, Germany

St Paul Lez Durance Cedex, France

Bucharest, Romania

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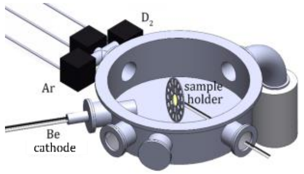
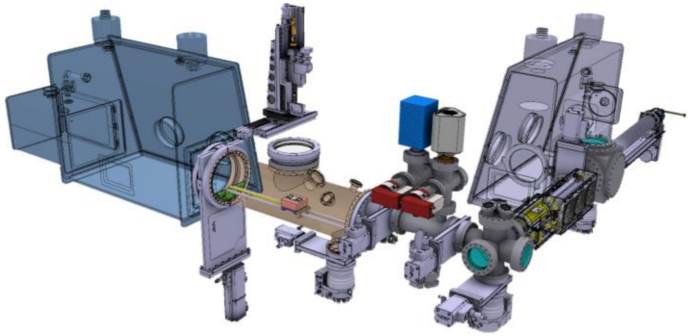
Outline

- ***Be/D layer*** production on W (HiPIMS)

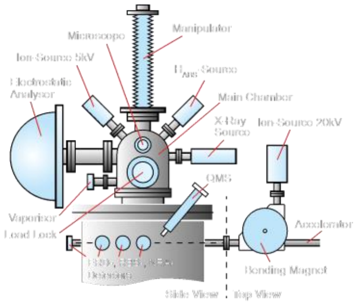


INFLPR

- new Analysis device (FREDIS)
- LID (Laser-Induced Desorption) in FREDIS
- TDS in FREDIS
- Modelling

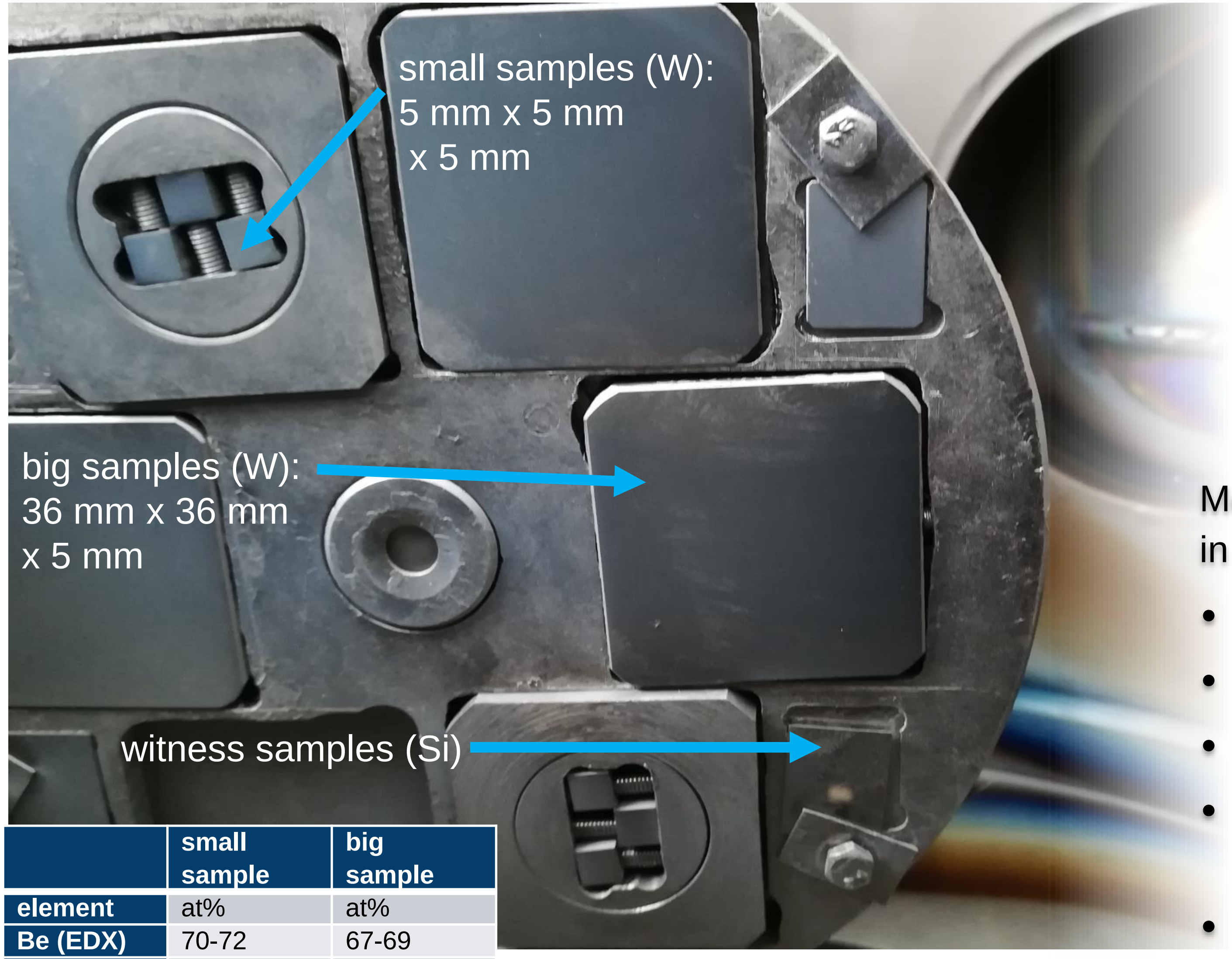


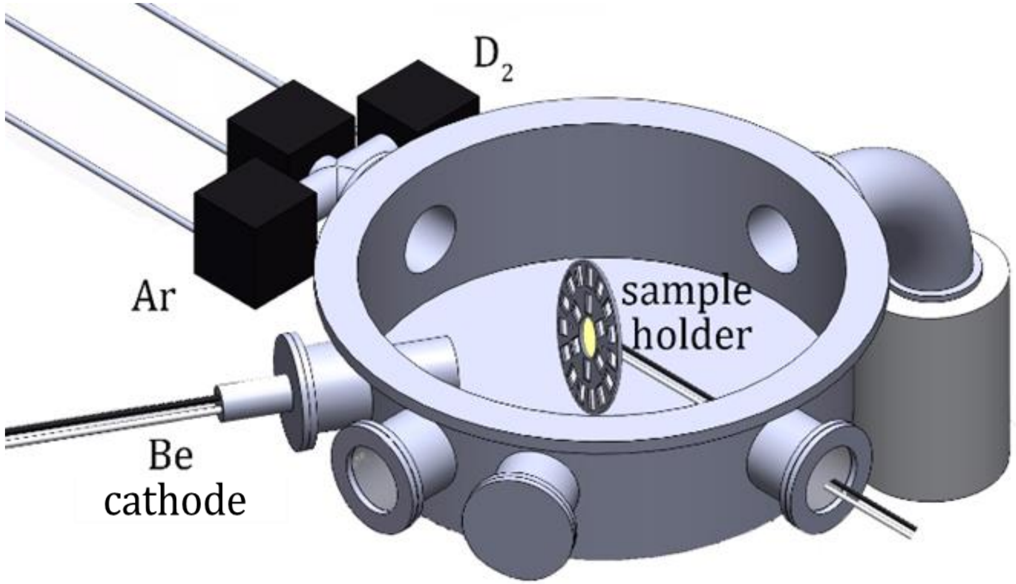
- ***Bulk Be:*** D implantation & TDS (ARTOSS)
- Modelling
- Summary & Outlook



- Good news: GO for new Hot Cells in Jülich

Be Layer Deposition in Bucharest





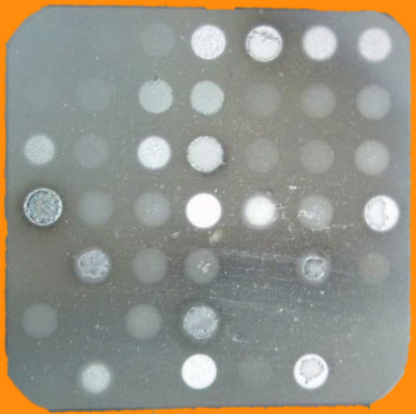
	small sample	big sample
element	at%	at%
Be (EDX)	70-72	67-69
D (NRA)	25-27	28-30
H (LID)	1.1	1.1
O (EDX)	1.0	1.9
C (EDX)	1.8	0.6
N (EDX)	0.4	0.4

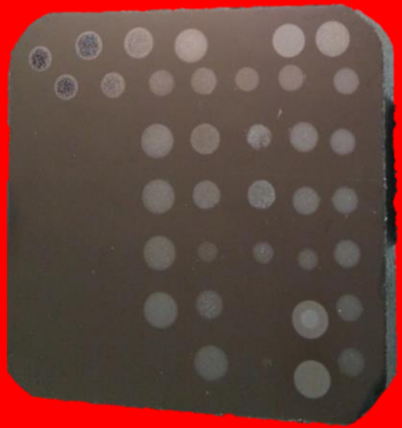
Method: High Power Impulse Magnetron Sputtering (HiPIMS) in D atmosphere by INFLPR

- pulsed magnetron plasma: several MW/m^2 during 3 µs
- Be layer thickness: **1 µm** and **10 µm**, new: **20 µm**
- D content: **1-30 at%** (measured by NRA)
- substrate: 5 mm polished W (IGP) by Plansee with grains elongated perpendicular to the surface
- <3E-6 hPa base pressure, 2E-2 hPa after gas inlet
- sample temperature ≈ 340 K, RT

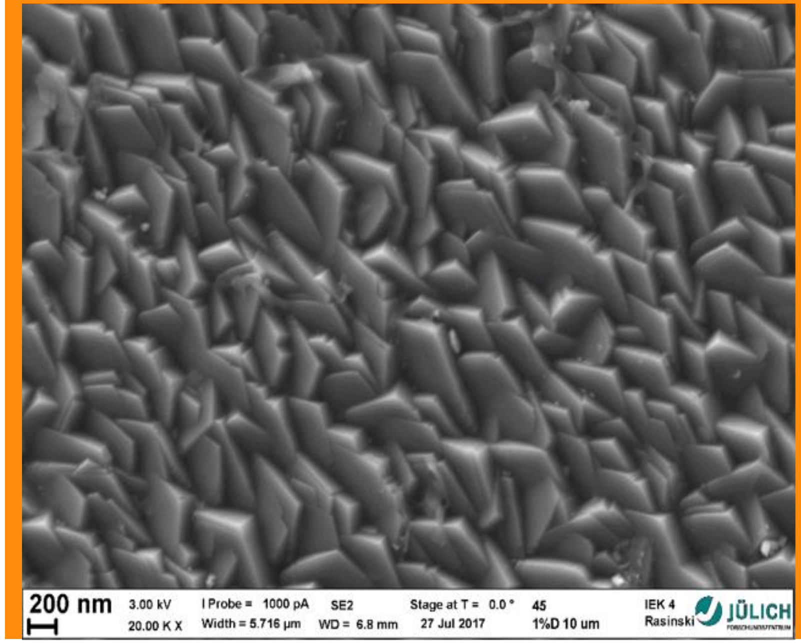
Microstructure of co-deposited Be/D layer

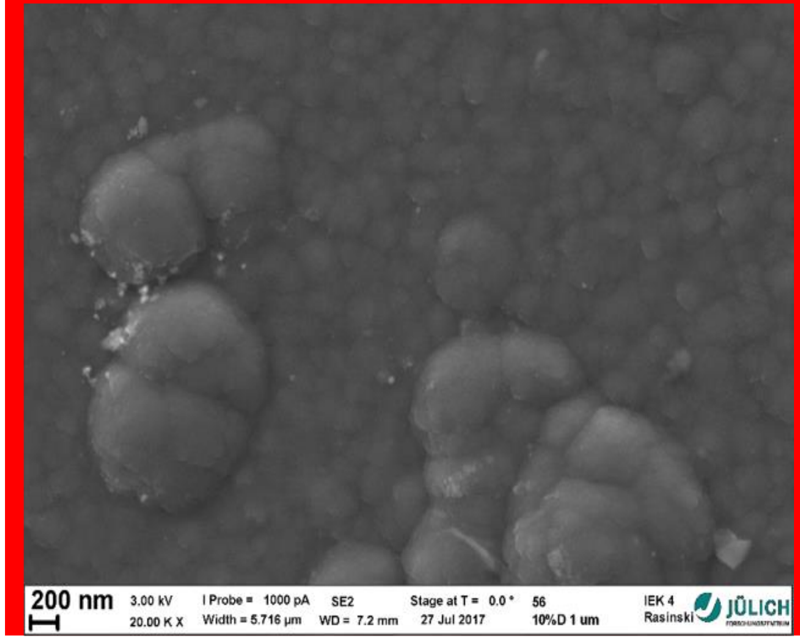
HiPIMS layers

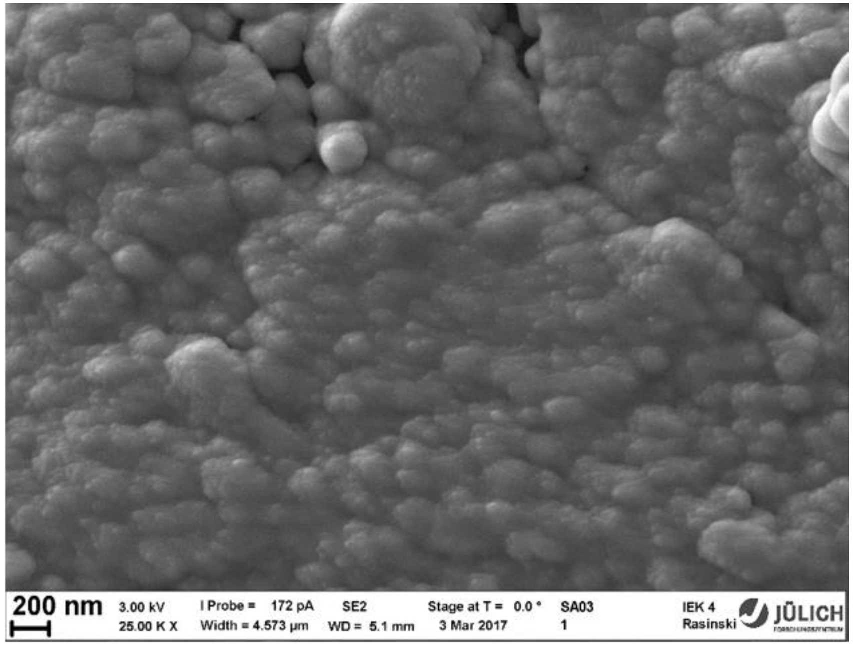




tokamak layer







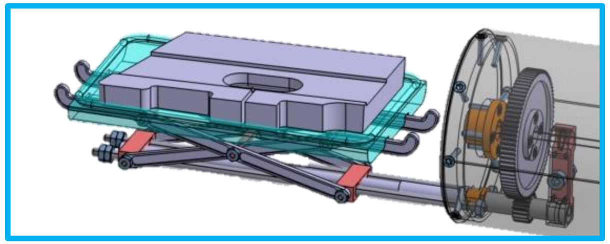
1.2-1.8 at% D
10 µm

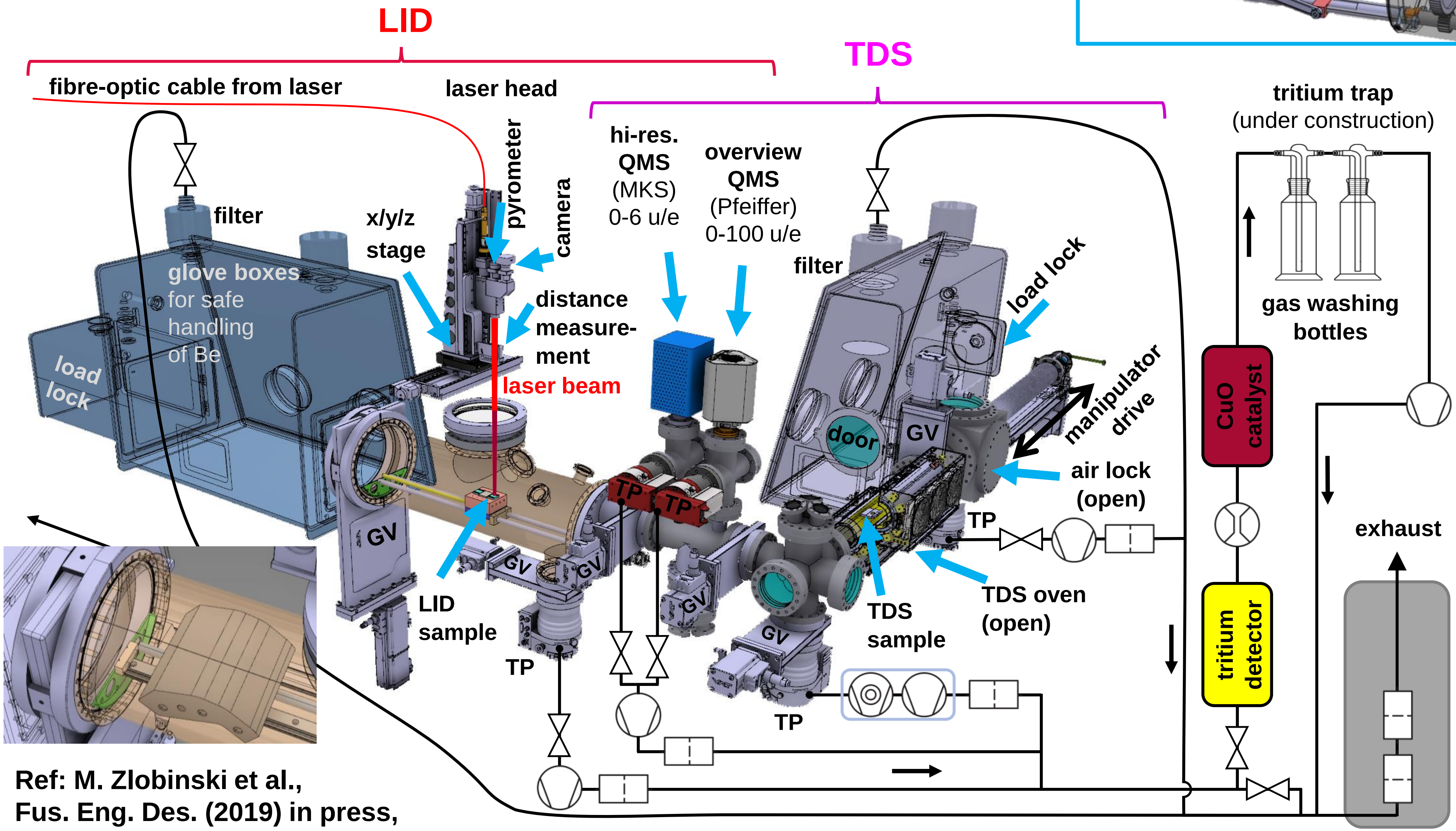
27-30 at% D
1 µm

layer from JET

Analysis Device in Jülich: FREDIS

(Fuel Retention Diagnostic Setup)



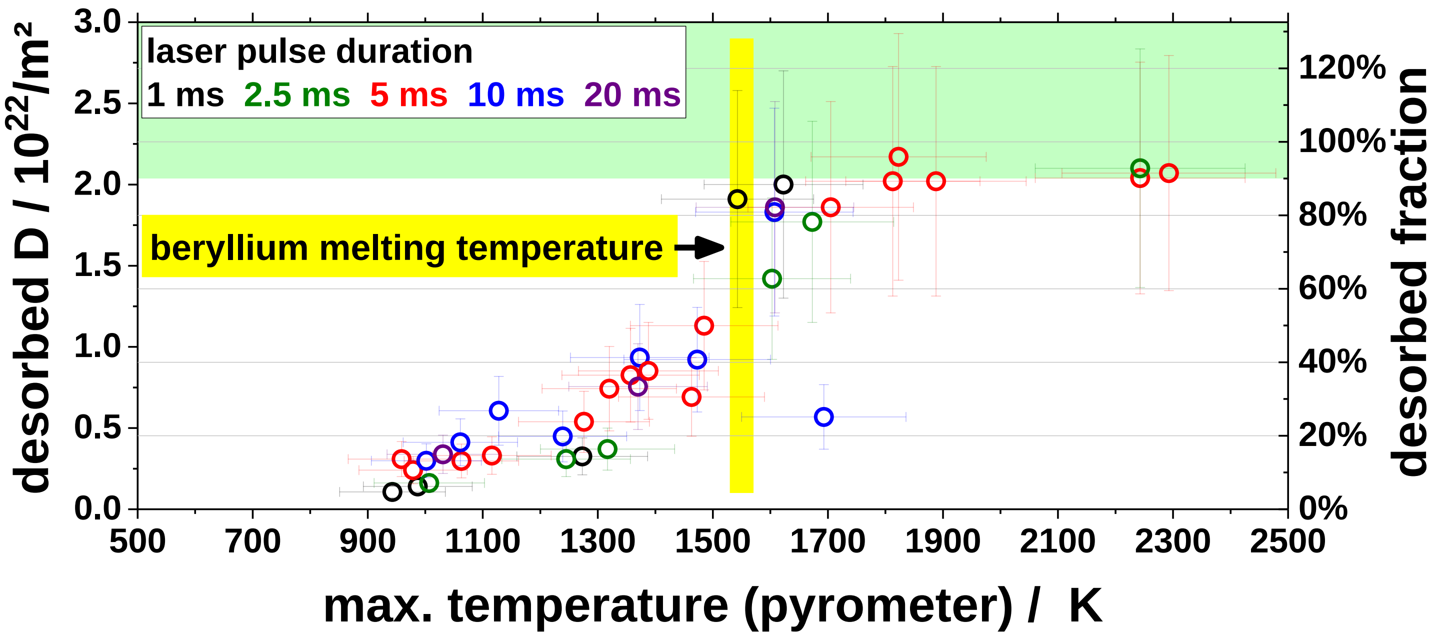


FREDIS features:
beryllium compatible
soon: tritium compa-tible

located in radiation controlled area

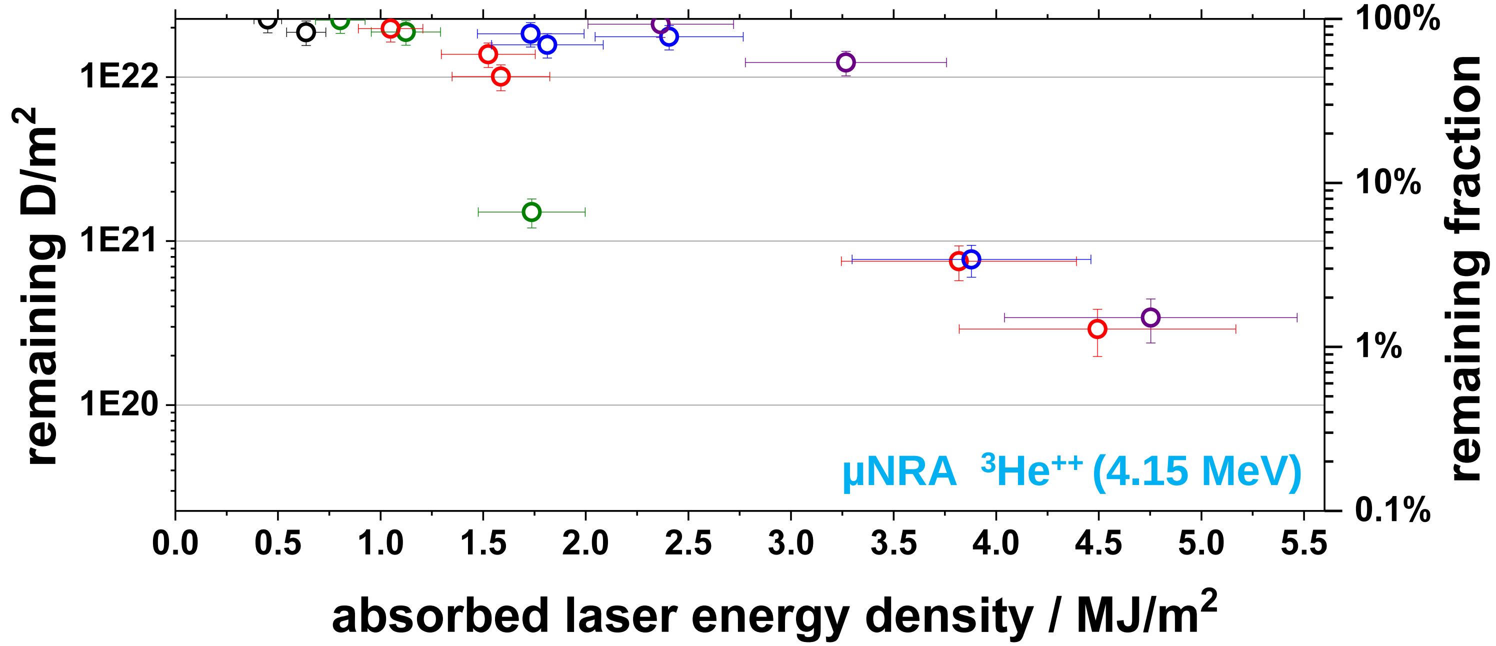
Ref: M. Zlobinski et al.,
Fus. Eng. Des. (2019) in press,
doi:10.1016/j.fusengdes.2019.02.035

LID desorption efficiency on 10 µm Be layer with 1.6 at% D



below Be melting:
up to **50% D desorption**
with a single laser pulse

with Be melting:
>99% D desorption
with a single laser pulse

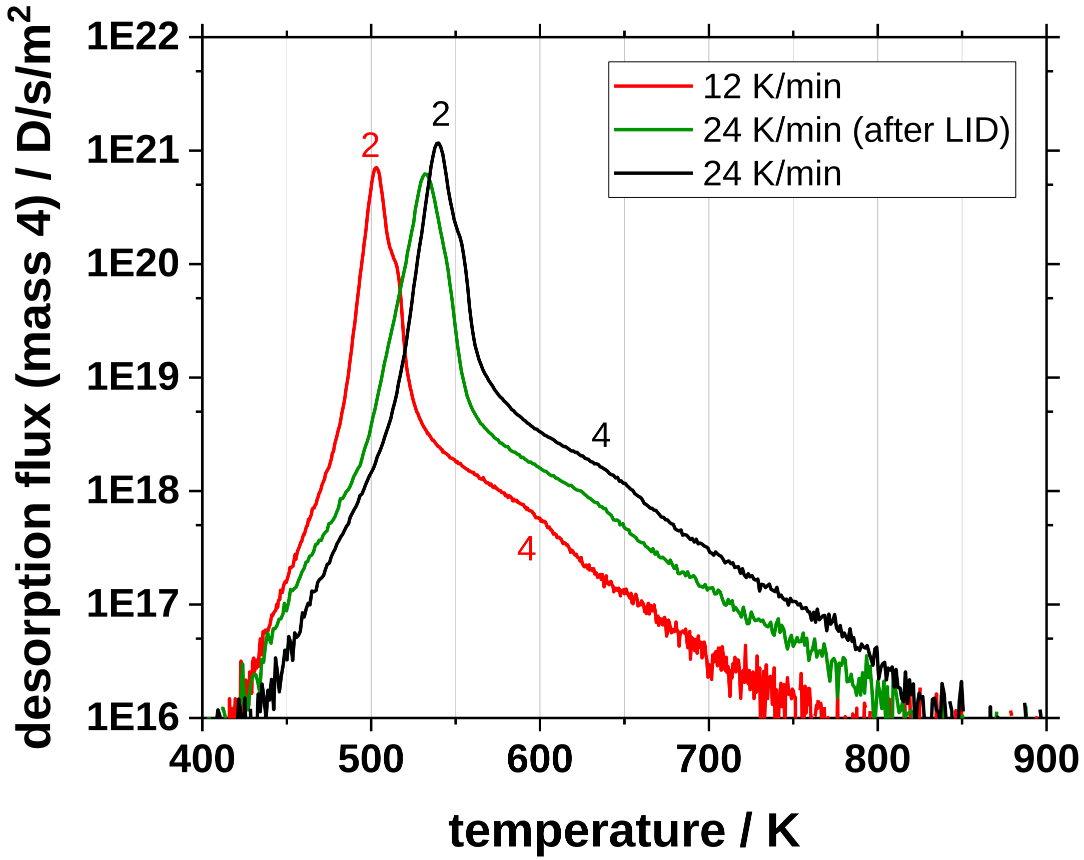


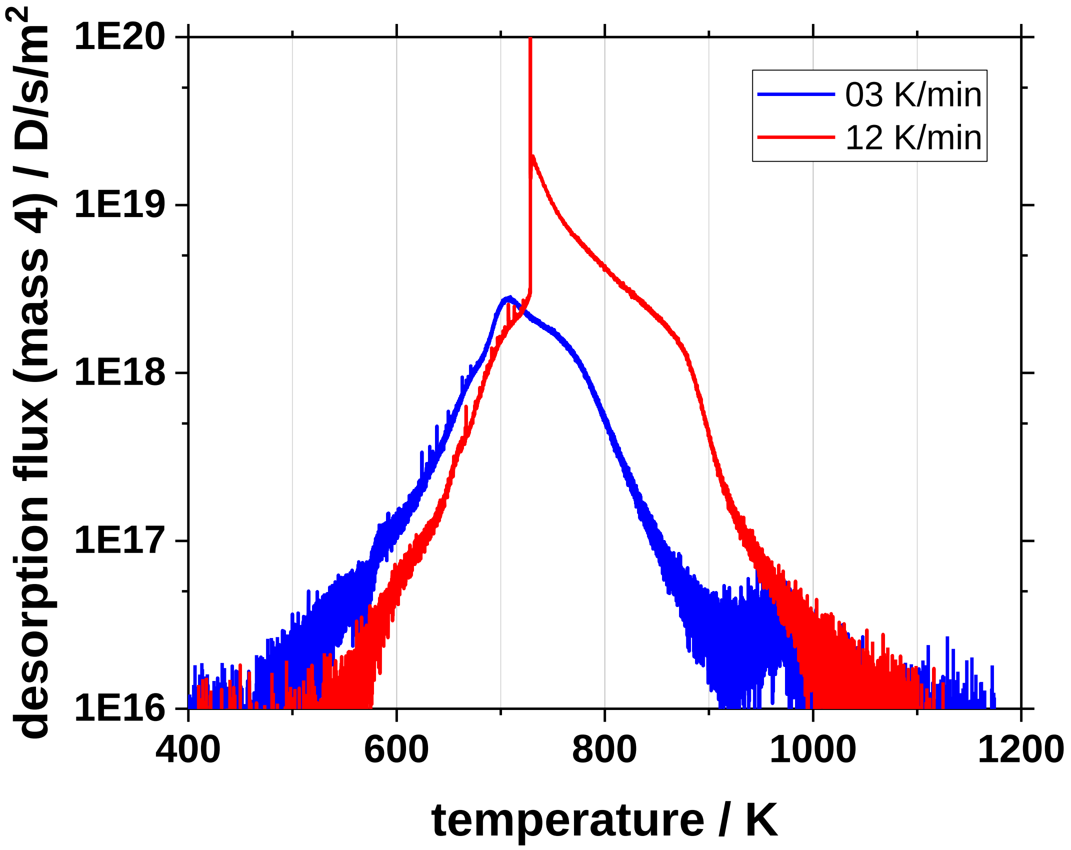
NRA in laser spot centre
shows that up to 99%
of D is desorbed

for 1 µm Be layer with 30 at% D:

>99% D desorbed
by one laser pulse
without melting

TDS spectra: 1 µm, 25-30 at.% D vs 10 µm, 1 at.% D

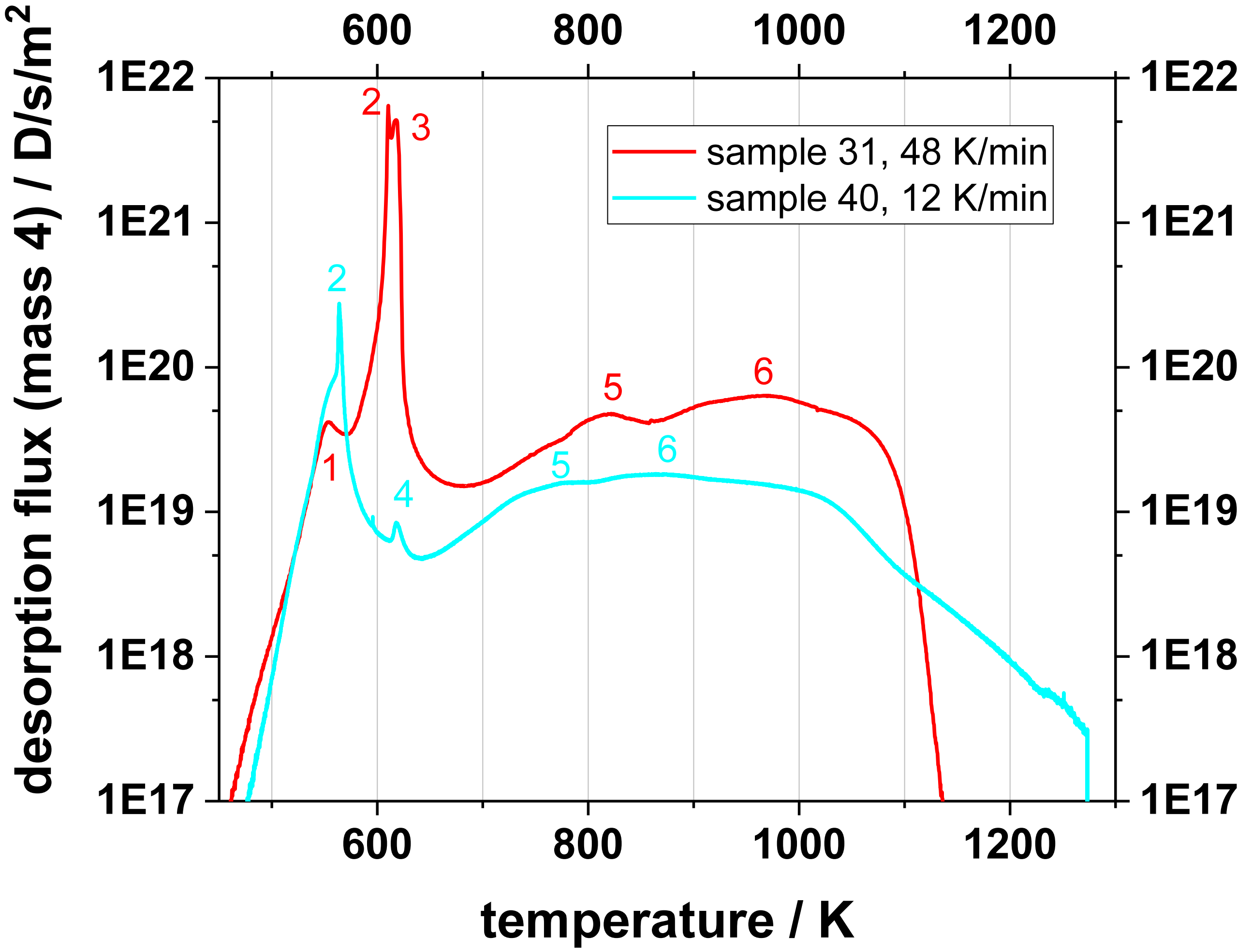




high temperature TDS peak could be the reason for the lower LID efficiency below melting

ultra-sharp TDS jump: instantaneous (within 0.4 s), by factor ~30
only in thick layers (fast heating of 10 µm, all 20 µm)
reason: layer detachment ???

TDS spectra: at least 6 TDS peaks observed



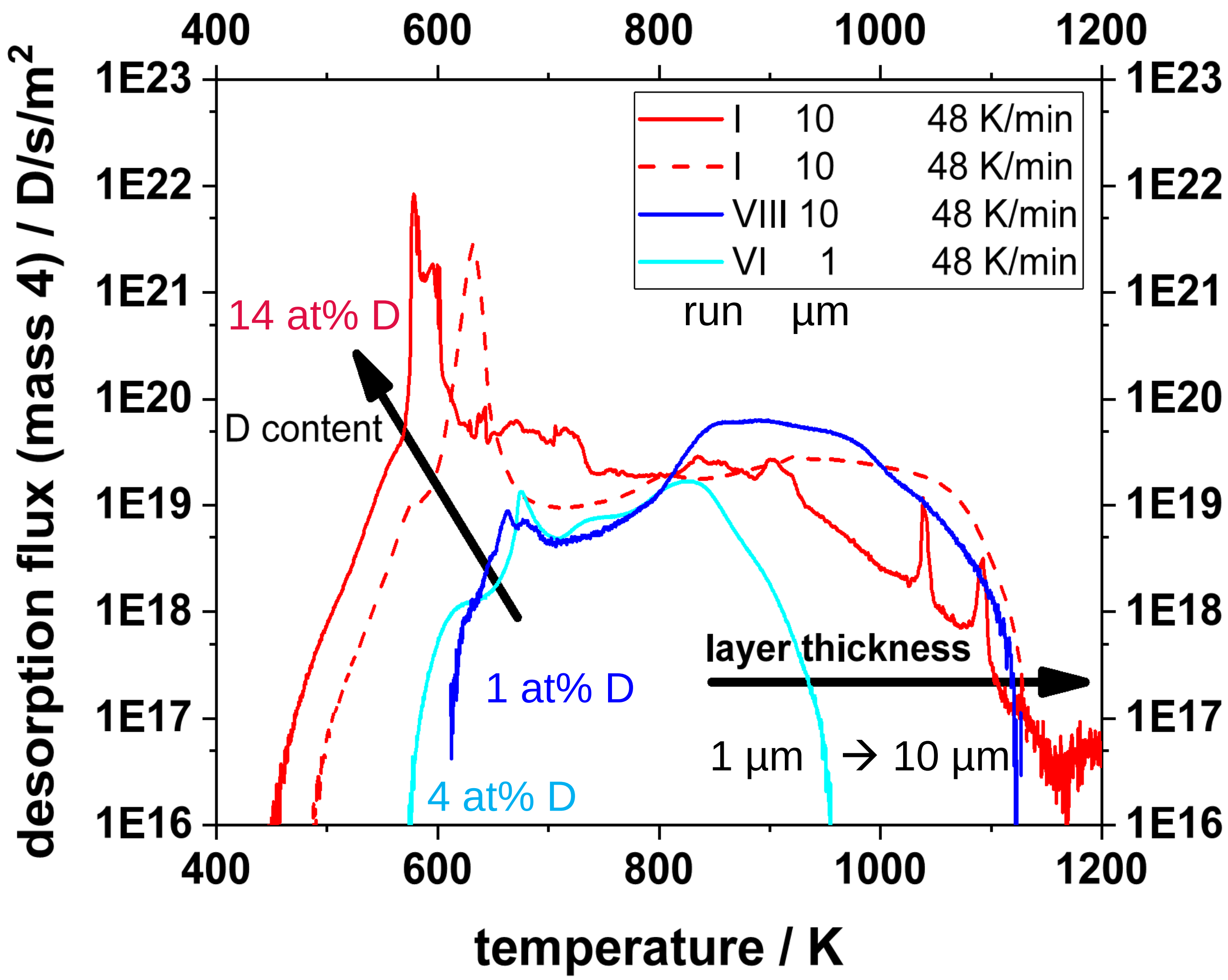
Be/D layer on W

narrow Low Temperature (LT) peak: consists of 2-3 peaks

small peak 4

broad High Temperature (HT) peaks: often several peaks

TDS spectra: general observations



Be/D layer on W

Coupled Reaction-Diffusion Systems (CRDS)

The code is similar to TMAP-7, TESSIM, MIMPS and others diffusion-trapping codes

Basic equations:

$$\partial_t c_i(x,t) = \underbrace{D_i(T(t))\partial^2_{xx} c_i(x,t)}_{\text{diffusion}}$$

$$+ \underbrace{R_i(\{c\},T(t))}_{\text{reactions (trapping,etc)}} + \underbrace{S_i(x,t)}_{\text{sources}}$$

$$\frac{d\sigma}{dt} = j_{bs} - j_{des} \quad \textit{surface coverage}$$

$$\partial_x c_H\Big|_{x=0} = \frac{j_{bs}}{D(T(t))} \quad \textit{bounary condition}$$

$$j_{bs} = \gamma\left(1 - \frac{\sigma}{\sigma_{max}}\right) c_H(0,t) \quad \textit{bulk}\rightarrow\textit{surf}$$

$$j_{des} = 2\kappa\sigma^2 \quad \textit{desorption from surface}$$

Modes of operation:

(1) Equivalent to TMAP-7 with either immediate desorption ($c_{|x=0} = 0$) or with molecular desorption flux

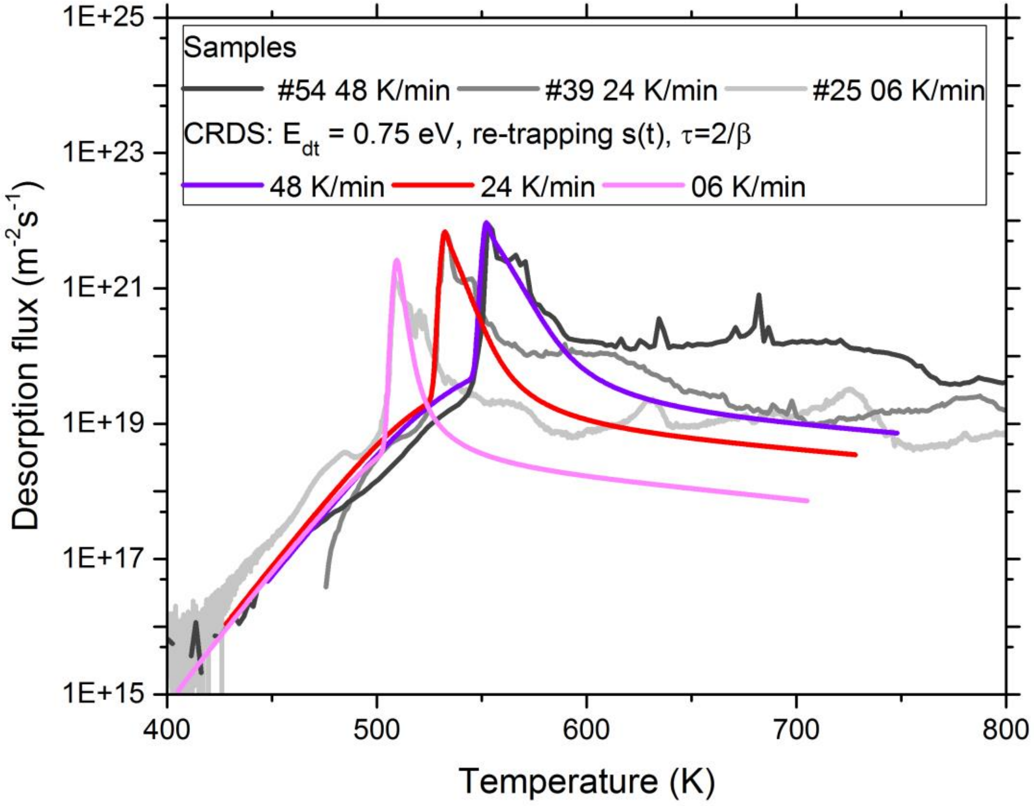
$$\Gamma = \kappa\, {c_{|x=0}}^2$$

(2) Accounting for actual surface coverage with saturation effects: $0 \le \sigma \le \sigma_{max}$

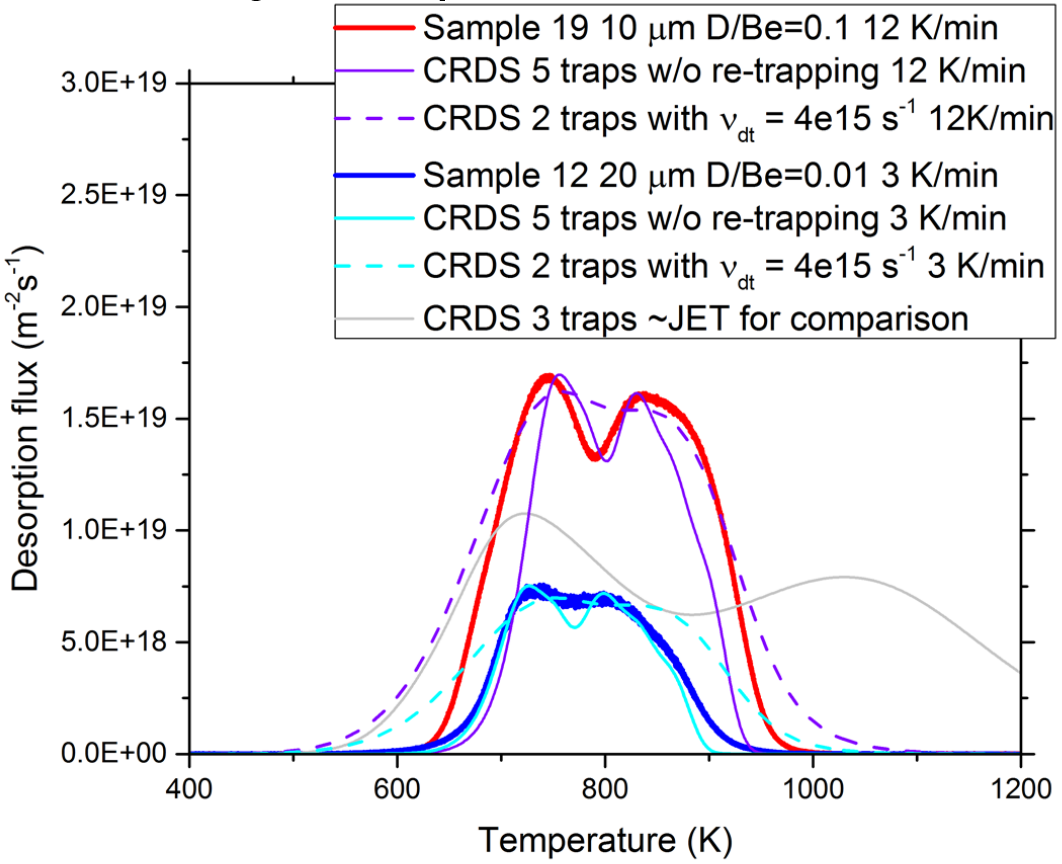
Further features:
- multiple-occupancy of traps
- trap mobility

Modelling of D release from Be/D co-deposited layers

Sharp low temperature release



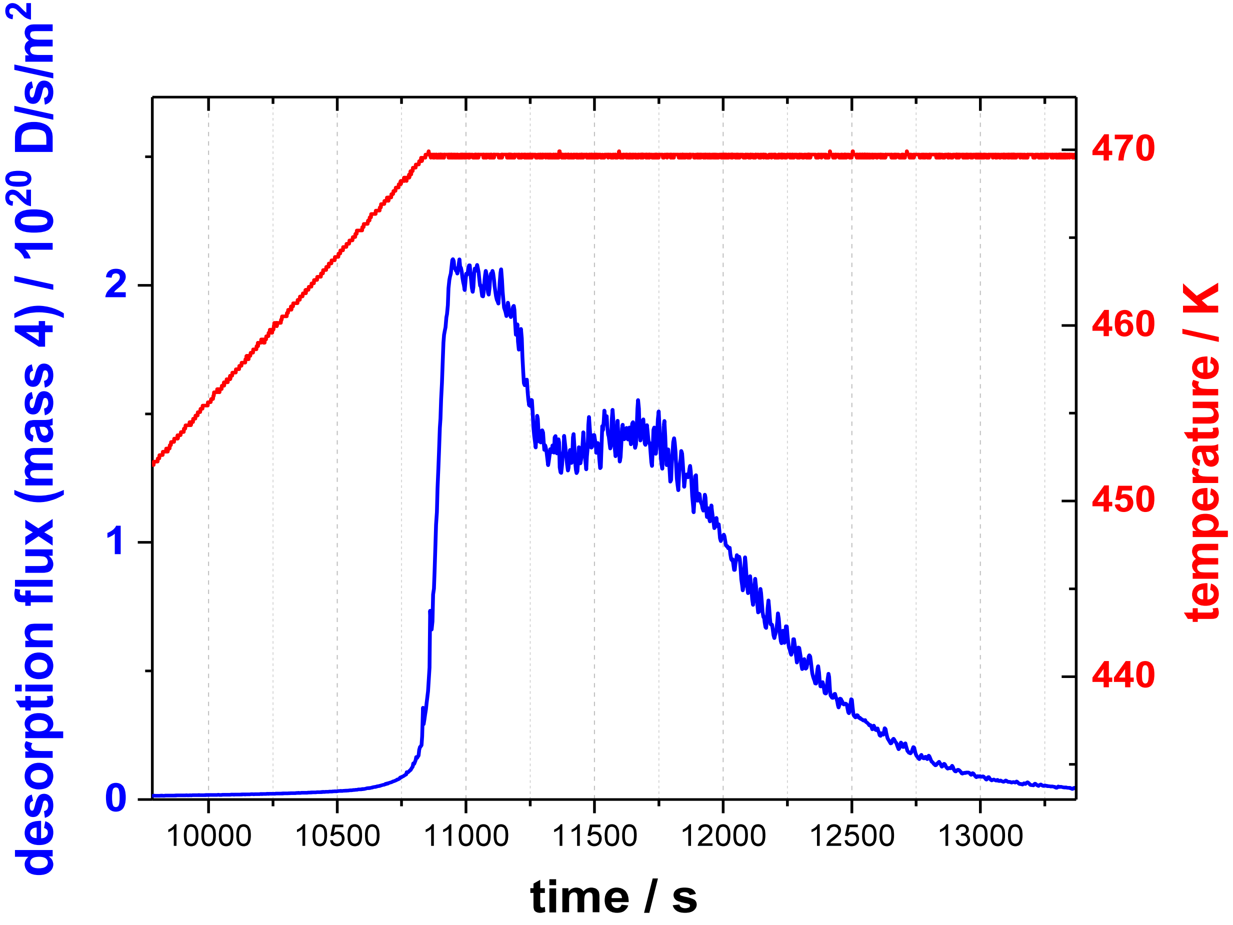
High temperature release

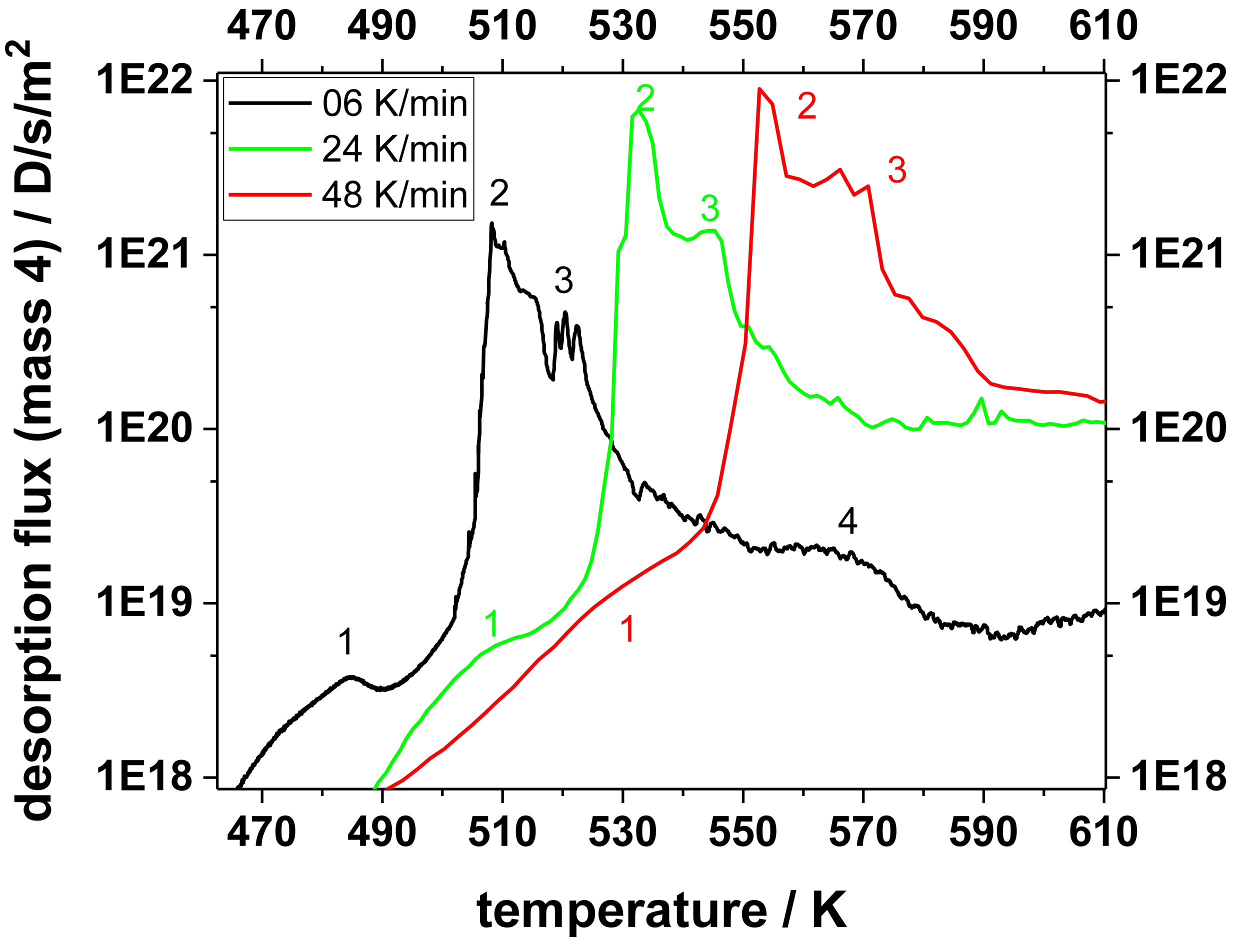


$s(t)=(1-\text{erf}[(t-t_0)/\tau])/2$ switch off re-trapping (e.g. trap annealing)

collective de-trapping, e.g. release from gas bubbles by their opening or percolation through a porous network at threshold temperature t_0

Ramp & Hold experiment: nature of low-temperature peaks





release is not governed by de-trapping;

permanent supply of hydrogen [Baldwin et. al: “hydrides”]

alternatively: strongly surface limited release

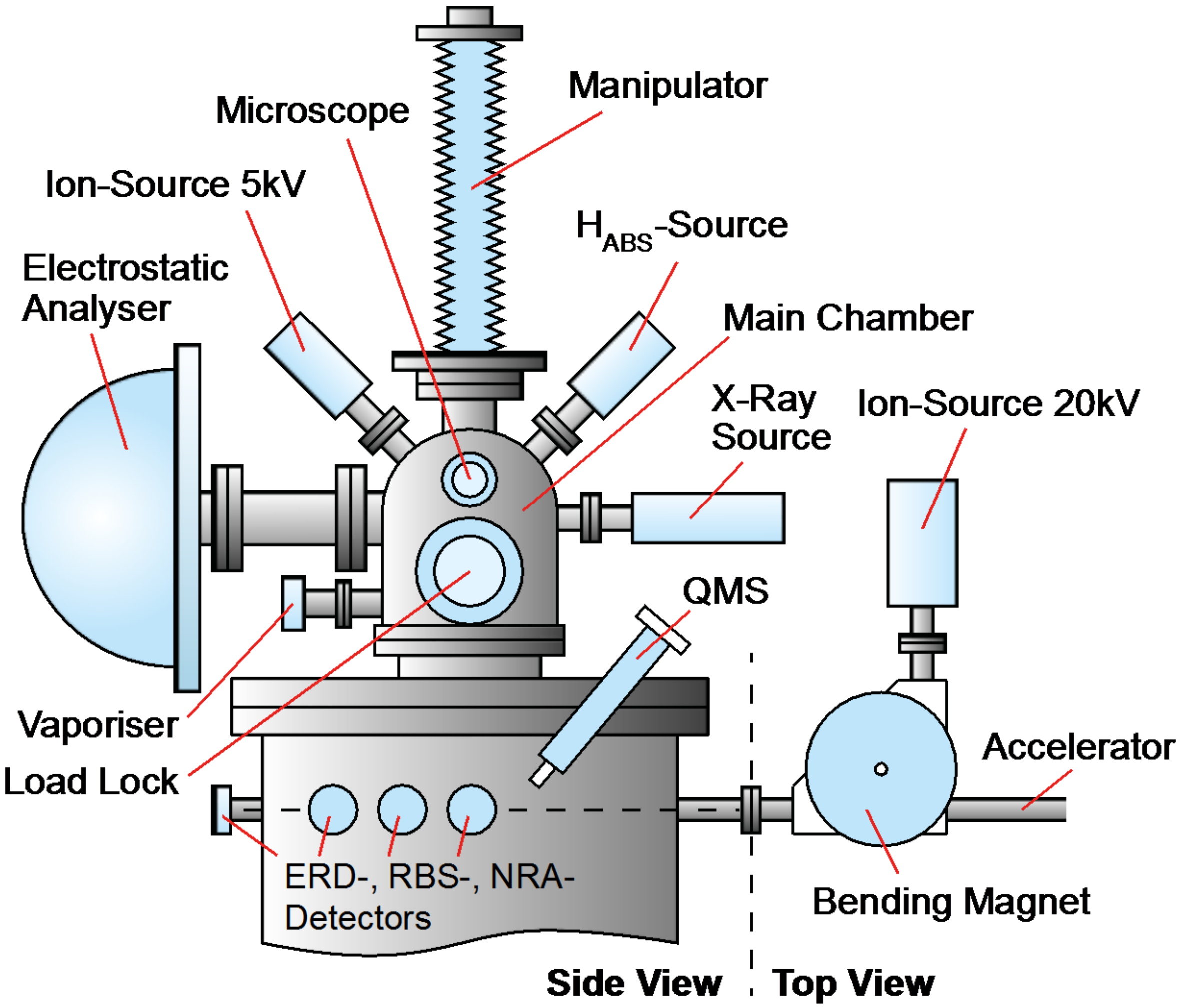
Material Change

- INFLPR/FREDIS: sputtered Be layers in D gas atmosphere → co-deposited D layers on W

Material change: Still Be but …

- ARTOSS: Bulk Be (Be SC, Be polycrystal) with implanted D

ARTOSS (name could mean… "All Relevant Techniques Of Surface Science")



- Mass & energy separated ion source 0.1-20 kV
- Sputter cleaning ion source, 1-5 kV
- Thermal atomic H source
- Electron beam evaporator
- Accelerator beam: **NRA**, **RBS**, …
- **QMS** mass spectrometer for **TPD**, **TDS**
    - **TDS heating by e⁻ beam**
- **XPS**: X-ray source and electrostatic analyser
- Base pressure < 5 · 10^{11} mbar
- Beryllium compatible

[T. Dittmar, M. Eichler]

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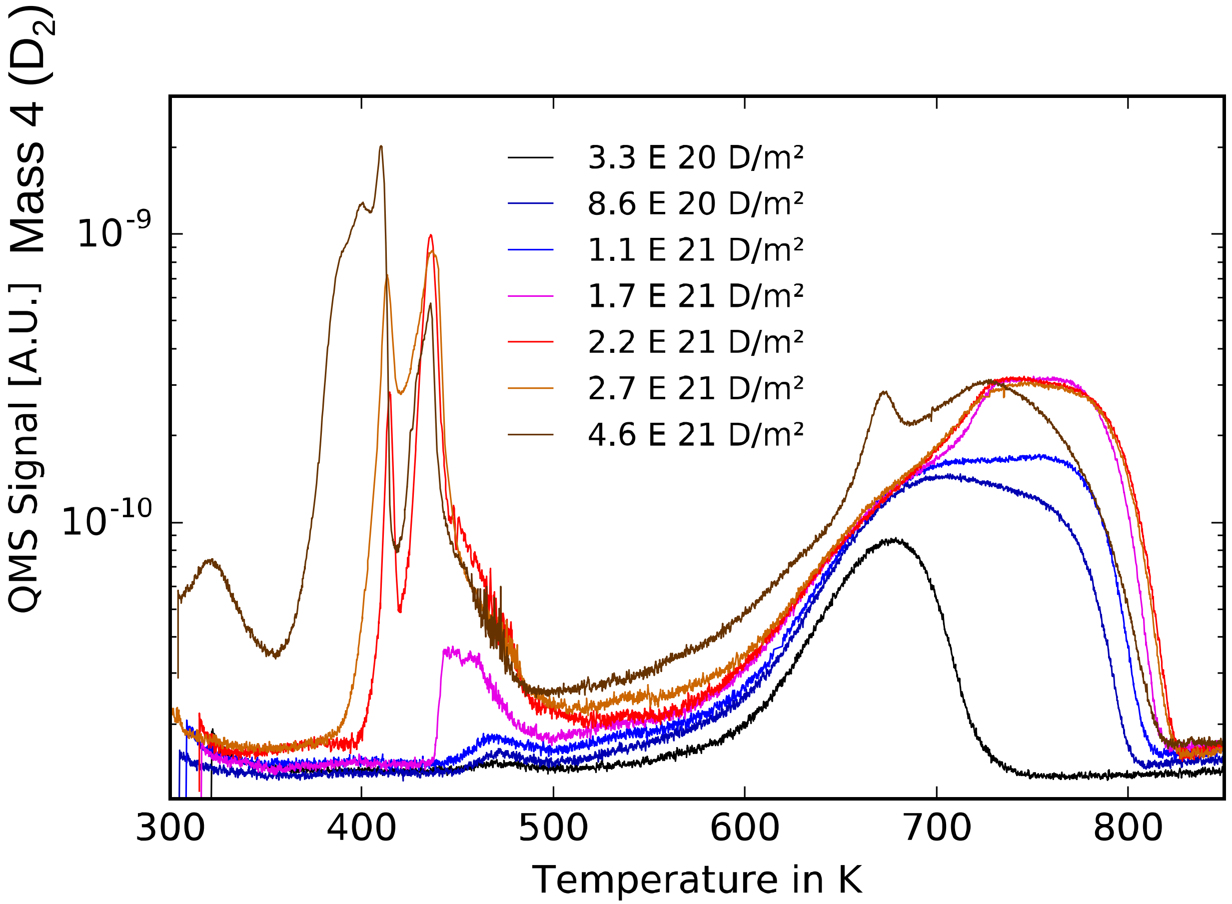
Beryllium single crystal fluence scan

implantation: D_3^+ (1 keV/D), 1E18 /m^2/s

TDS heating rate: 0.01 K/s



- High-temperature peak shifting
  - Multitrapping in single vacancies?
  - Depth effect?
- Low-temperature peak splitting
  - LT-peak consists of min. 2 sharp peaks
  - Cannot be explained with Arrhenius release
- Pre-LT-Peak
  - Release of solute Deuterium

Ref: M. Eichler,
Nuc. Mat. En. 19 (2019) 440–444,
doi:/10.1016/j.nme.2019.03.018

[T. Dittmar, M. Eichler]

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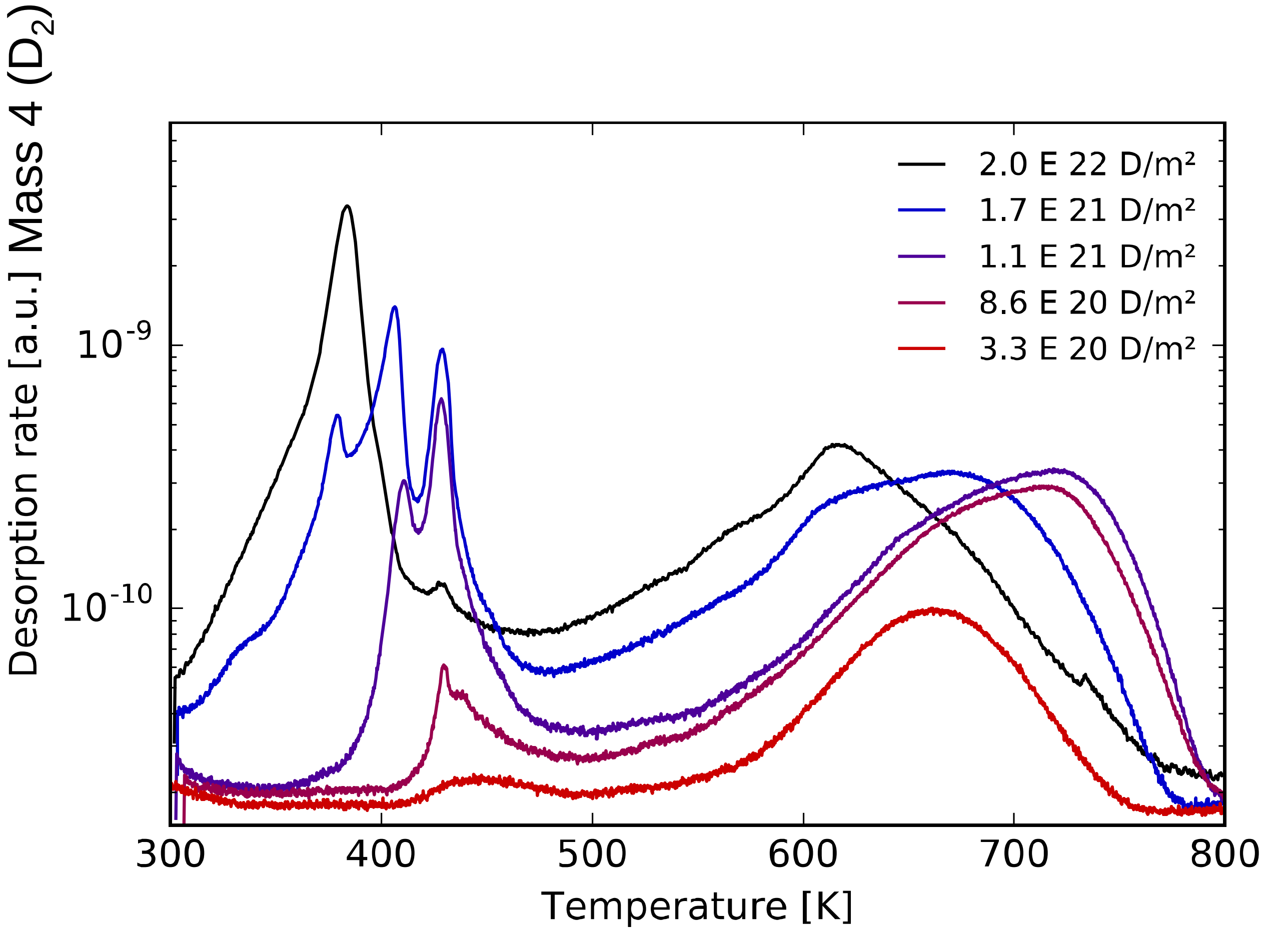

- High-temperature peak shifting
  - Similar behaviour as single crystal
- Low-temperature peak splitting
  - LT-peak consists of min. 3 sharp peaks (0.43 eV, 0.67 eV and 0.82 eV)
  - Forms single dominant peak at high fluence
- Pre-LT-Peak
  - Visible as „shoulder“

Compare [Baldwin et. al: “only 1 LT peak”]

[T. Dittmar, M. Eichler]

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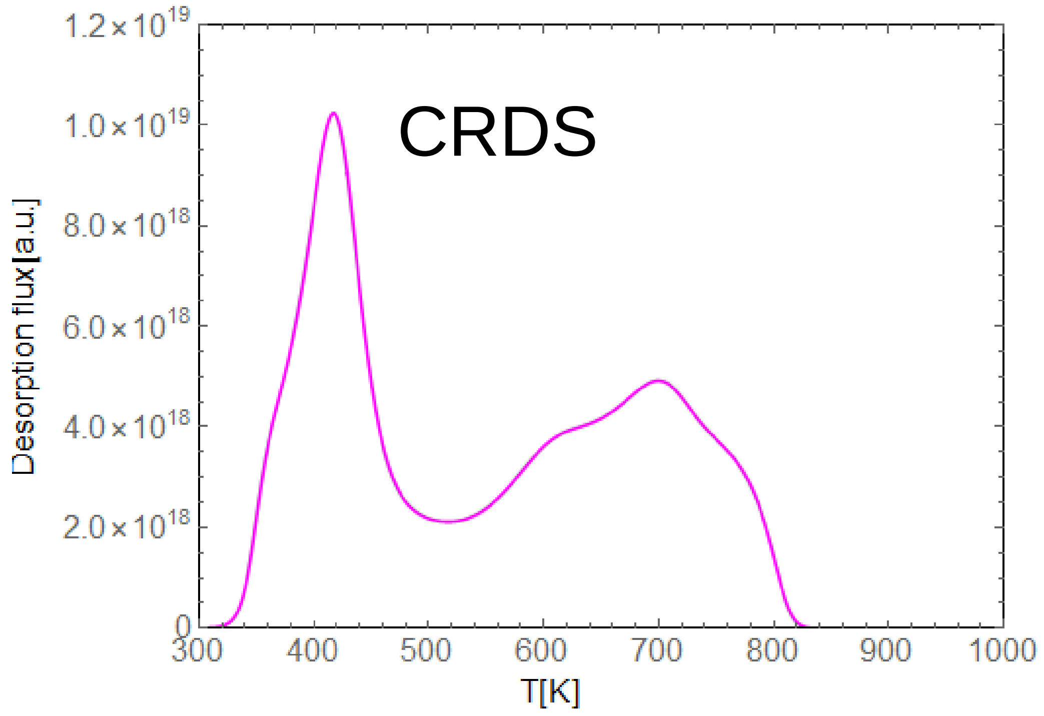
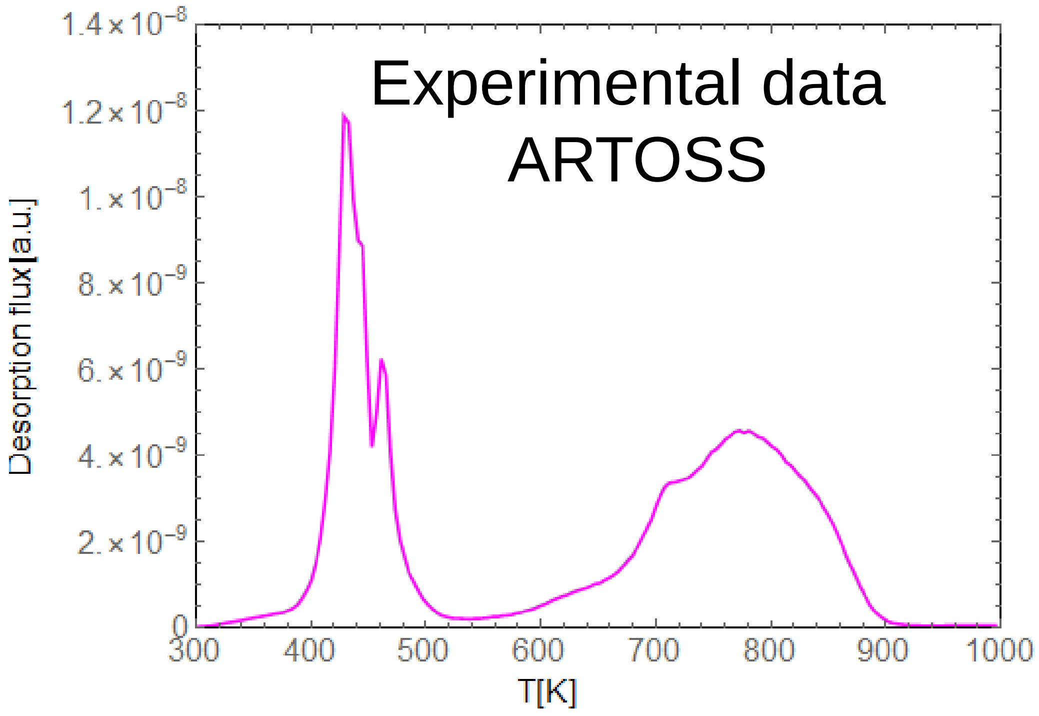
Modelling of D retention in crystalline Be

Modelling of new ARTOSS data (ongoing)
Defect evolution during implantation is taking into account directly in simulations
Slow down of defect creation (net) and defect saturation are reproduced





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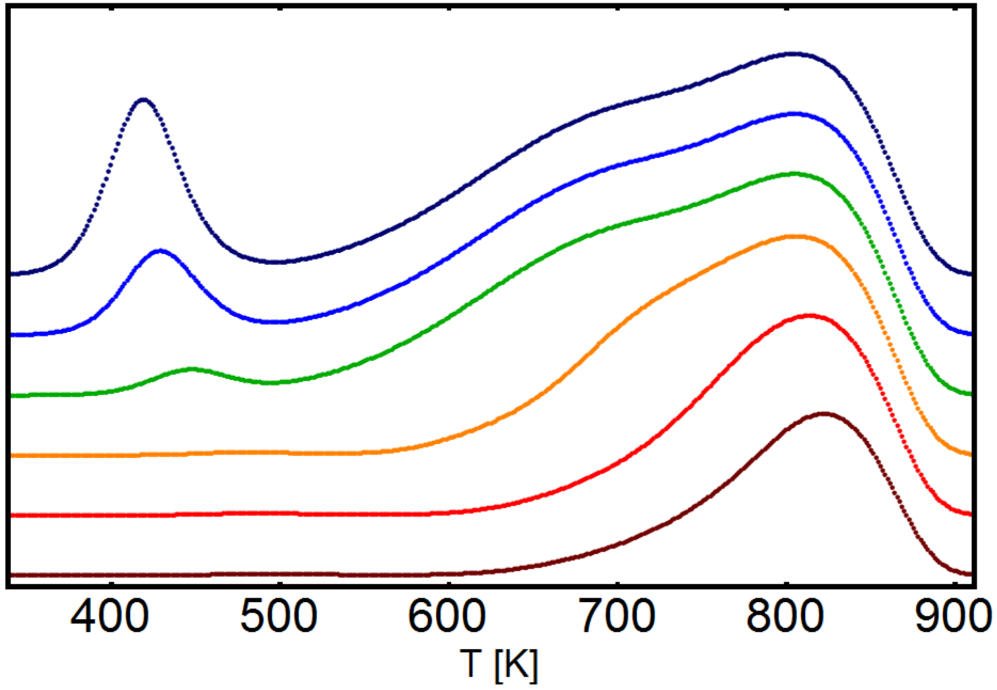
[D. Matveev]

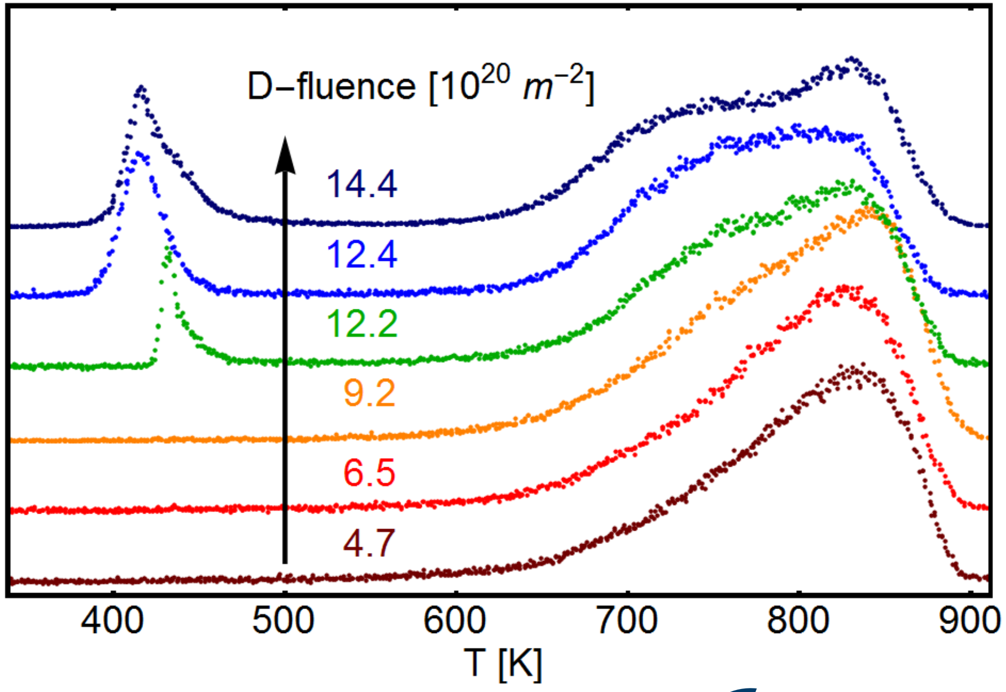
Modelling of D retention in crystalline Be

Qualitative agreement
- high temperature peak shift (effect of re-trapping) and shoulder (multiple-trapping)
- low temperature peak threshold (surface occupation after bulk saturation)

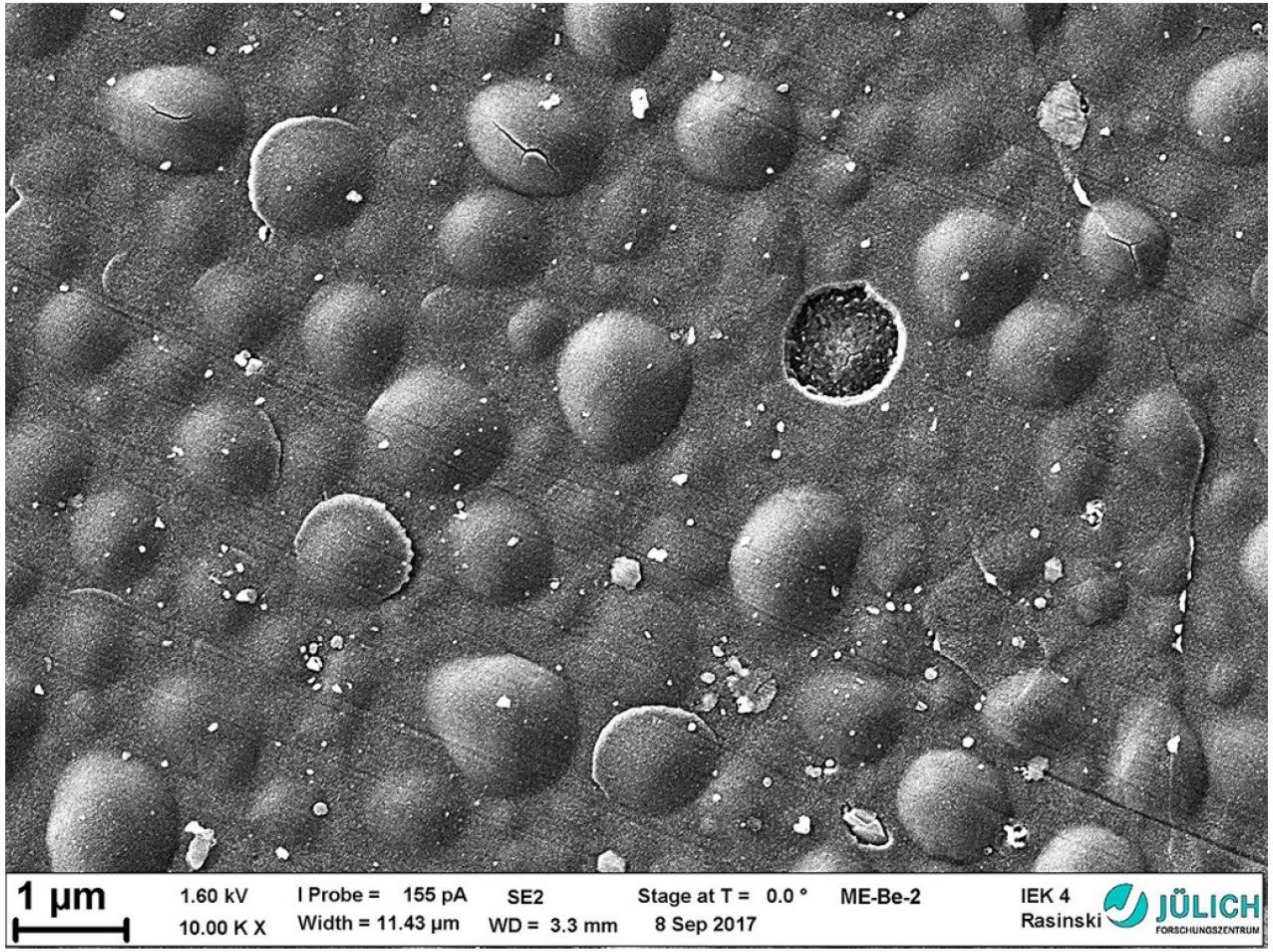
CRDS



Experimental data: M. Reinelt, NJP 2009



Reason for LT peak splitting in ARTOSS



Hypothesis:

different adsorption surfaces for D

e.g. blisters

Be polycrystal after implantation of D_3^+ at 2 keV/D. Blisters are formed on the surface, which are partially cracked open on the top while others are peeled off or in the process of flaking

[T. Dittmar, M. Eichler]

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Summary

LID (Laser-Induced Desorption within ms):

- for 1 µm HiPIMS Be/D layer with 30 at% D: **complete D desorption** (i.e. >99%) possible by a single laser pulse without melting
- for 10 µm HiPIMS Be/D layer with 1.6 at% D: up to **50% desorption** until melting, **nearly complete D desorption** (ca. 99%) possible with melting

key for understanding probably in TDS spectra

TDS (slow Thermal Desorption Spectrometry):

- Be: 1st time observation of up to **3 LT subpeaks**
- LT peak contribution increases with D concentration/implantation flux
- LT peak **very narrow** → cannot be simulated by standard diffusion-trapping model
  need to introduce: collective de-trapping, switch off re-trapping, …

- HT peak (multipeak) widens with D layer thickness/D depth
- qualitatively similar TDS spectra for bulk Be (single crystal, polycrystal) and sputtered Be layers
- ultra-sharp peak → **???**

Outlook

- Is the laser desorption temperature determined or diffusion dominated?
  $\rightarrow$ analysis of increasing heating by LID on same position

- **modelling** of TDS and LID ongoing to transfer the results
  - dominant effects
  - sensitivity of desorption on binding energy, diffusion activation energy, …

- new TDS setup: **i-TDS** (inductive TDS): ca. 2000 K in ca. 1 min; between LID and classical TDS

- LID and TDS of our 1st **JET samples** with thick Be co-deposited layers in FREDIS
  - tokamak layers behaviour compared to HiPIMS layers (are they reactor relevant?)
  - differences between T and D desorption?

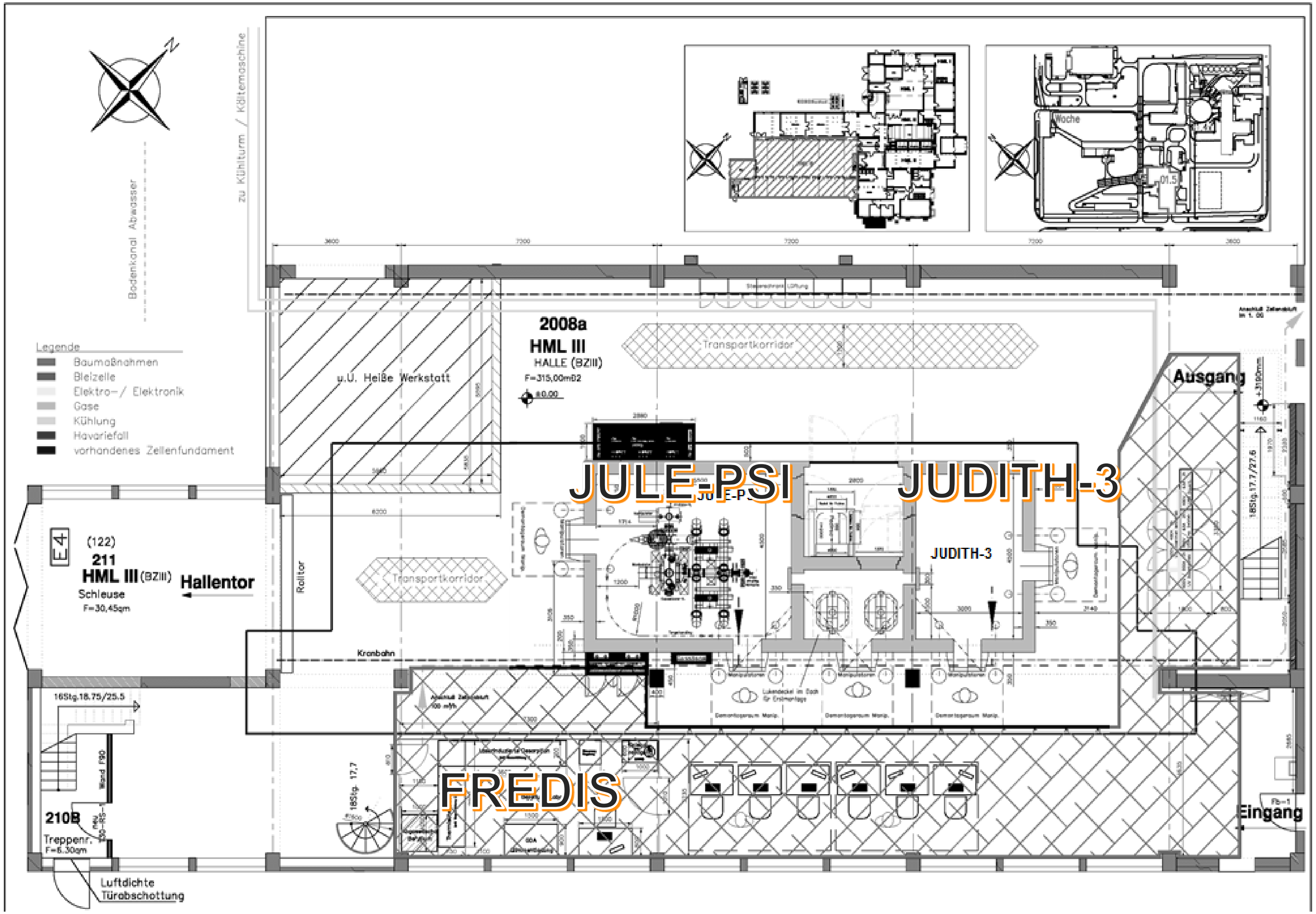
- future application and modelling of LID in **JET** and **ITER**

Jülich-HML (High temperature Material Laboratory)

few weeks ago:
funding granted for building of new hot cells

for:
- linear plasma device:
  JULE-PSI
- high heat flux test device:
  JUDITH-3
- analysis labs, …

to handle activated material and beryllium



Thanks for your attention