TDS round robin samples:

Motivation, preparation and pre-characterization

Thomas Schwarz-Selinger, Liang Gao, Wolfgang Jacob

Max-Planck-Institut für Plasmaphysik, 85748 Garching, Germany

Starting point

- Decision on a TDS round robin experiment during the meeting in Seoul 2015
- Wolfgang Jacob (IPP Garching) volunteered for sample preparation
- Heun Tae Lee (Osaka University) volunteered to coordinate the task and take care of data analysis
- Now: Talk on samples themselves:

  Motivation – preparation – pre-characterization

- Following talk by Heun Tae Lee about the round robin results

What we needed

The basic aim is to compare TDS spectra measured with different TDS set-ups in terms of:

- D amount
- desorption temperature

➢ Participating groups: 10 – 15 with two to three samples each

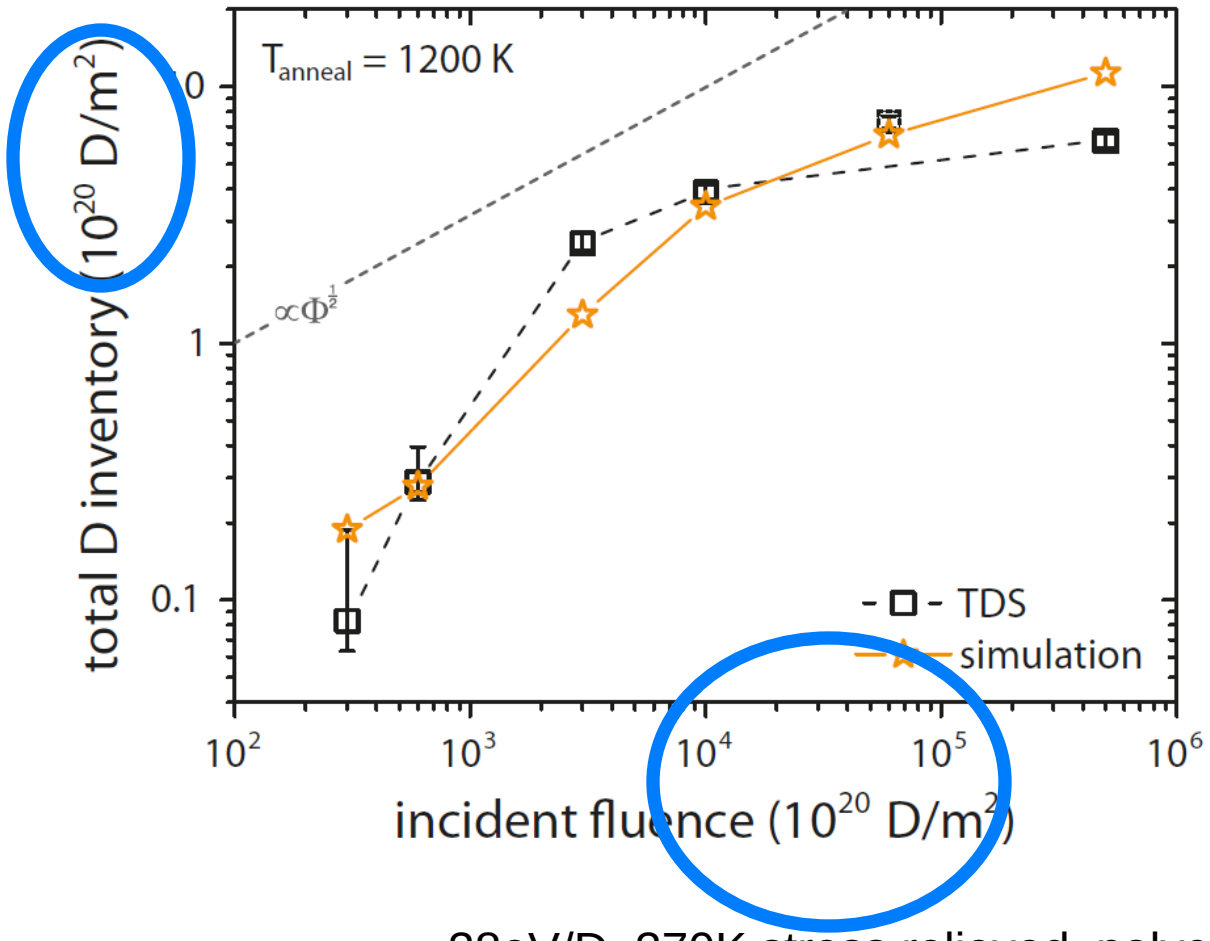
⇒ A set of about 50 samples is necessary

➢ Samples should

- have identical D content
- be stable over time (outgassing)
- be tungsten (T measurement)

What we needed

Initially it was suggested to produce reference samples by plasma loading



38eV/D, 370K stress relieved, polycrystal. Plansee W
A. Manhard, PhD Thesis, University Augsburg

D implantation for 50 samples?

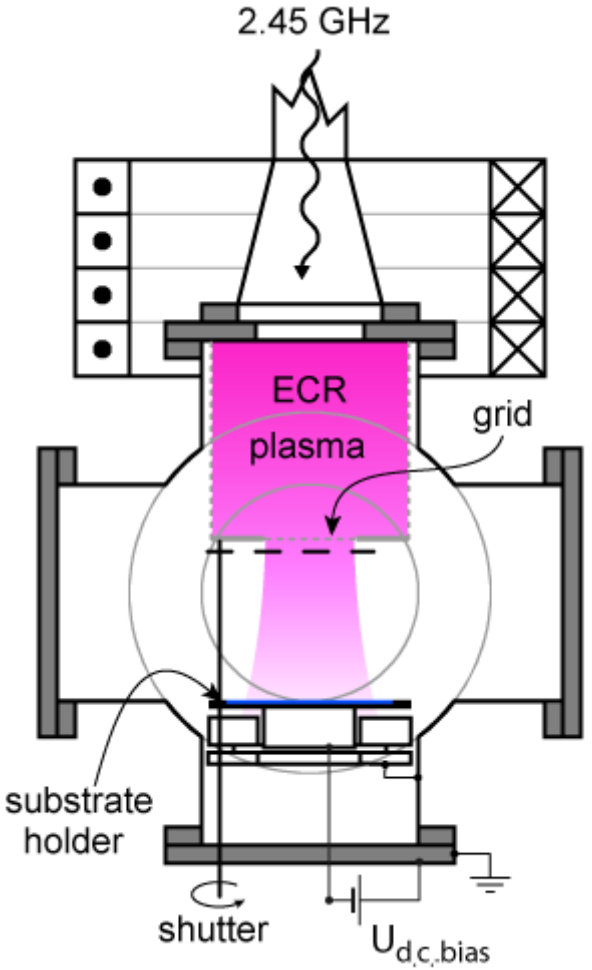
Low-temperature ECR plasma PlaQ:

- Ion flux absolutely calibrated:

  1.0×10^{20} D/(m^2s)

  (97% as D_3^+, 2% as D_2^+, 1% as D^+)
- Ion fluence: 1.0×10^{24} D/m^2
- Bias: floating up to -600V
- 200K < T < 800K
- 5 samples simultaneously

⇒ 10 exposures a ≈ 1 day + pumping



≈ one month of operation while the system is booked out 24/7

A. Manhard, Plasma Sources Science and Technology 20, 015010 (9pp) (2011).

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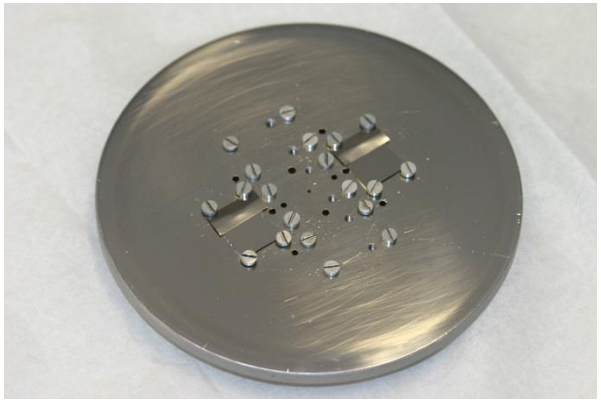

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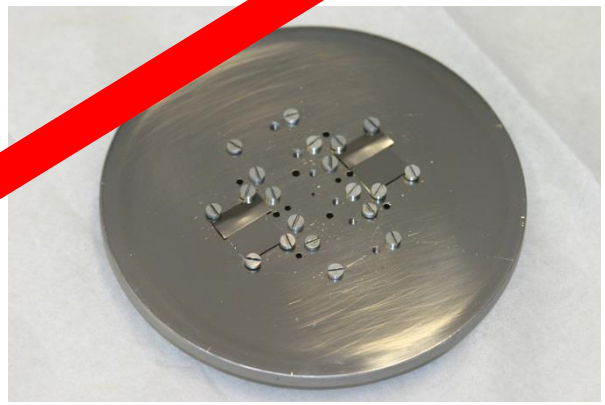

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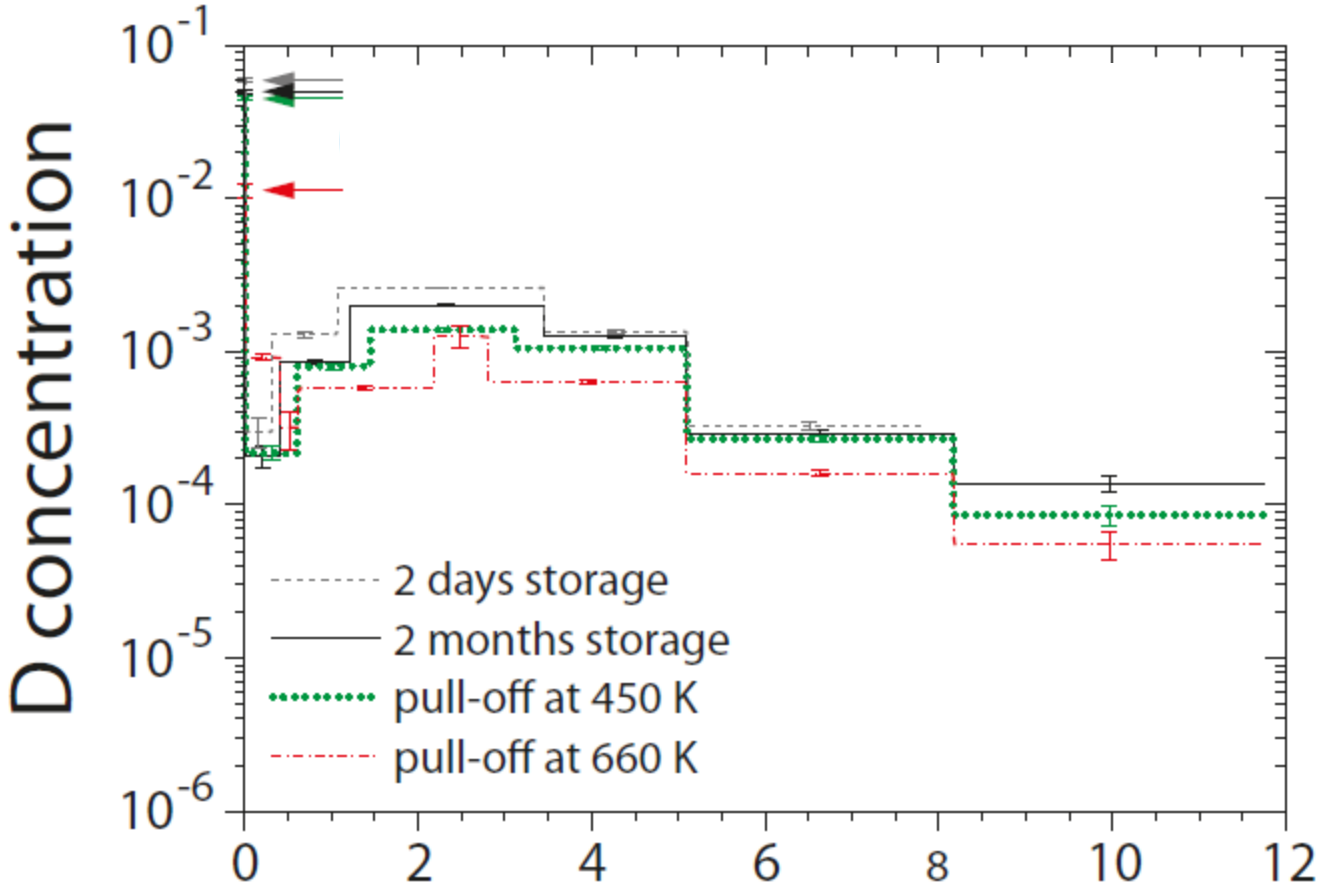
What we did not want: outgassing



38eV/D, stress relieved, polycrystal. Plansee W
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De Temmerman scaling

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Deuterium retention and release in tungsten co-deposited layers

G. De Temmerman [a,*], R.P. Doerner [b]

[a] EURATOM/UKAEA Fusion Association, Culham Science Centre, Abingdon OX14 3DB, UK
[b] Center for Energy Research, University of California at San Diego, CA-92093 La Jolla, USA

$$\frac{D}{W} = 5.7 \cdot 10^{-8} \cdot r_d^{-0.41\pm0.1} \cdot E_n^{1.88\pm0.4} \cdot exp\left(\frac{779 \pm 220}{T}\right),$$

where r_d is in units of $10^{15} at \cdot cm^{-2} \cdot s^{-1}$, E_n in eV and T in K, and with 293 K $\leqslant T \leqslant$ 600 K and 60 eV $\leqslant E_n \leqslant$ 280 eV.

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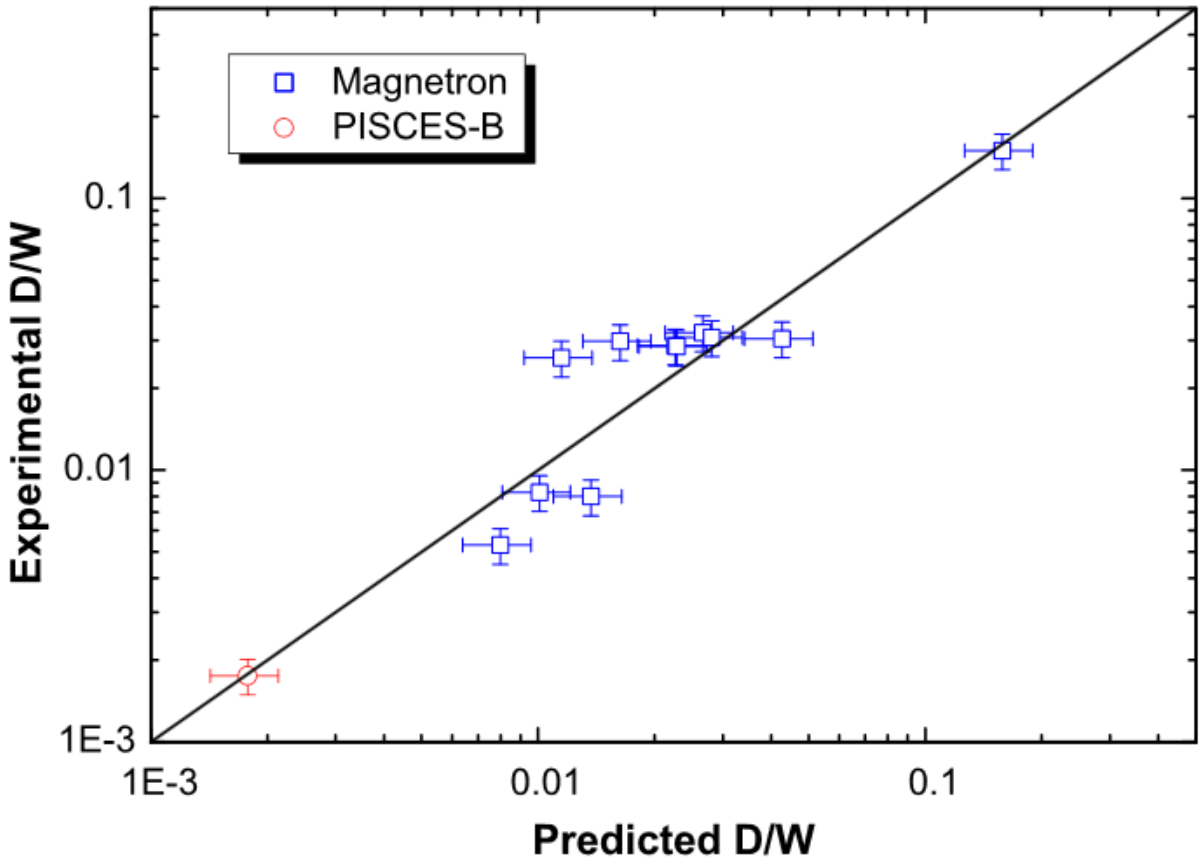

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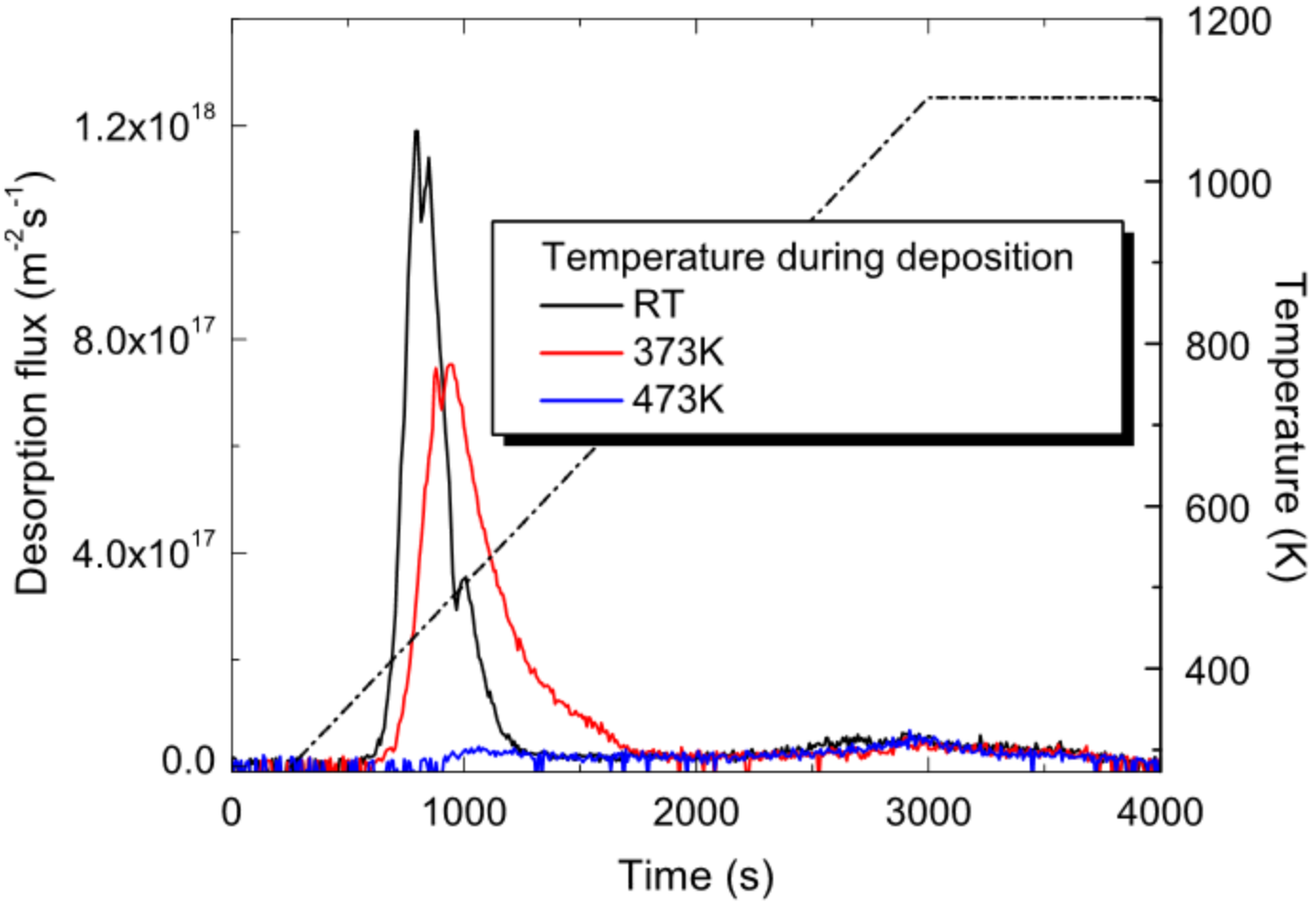


Sample preparation: substrate

- Polycrystalline, hot-rolled W, 99.97 wt.-% purity (Plansee SE Austria),
  $10 \times 10 \times 0.8$ mm^3 = “standard”
  Ø5 mm,
  Ø6mm,
  all from the very same batch and treated in the very same way.

- XRD to identify the (110)-orientation dominated surface

- Grinding with silicon carbide paper from P400 down to P4000

- Electropolishing in 1.5% NaOH

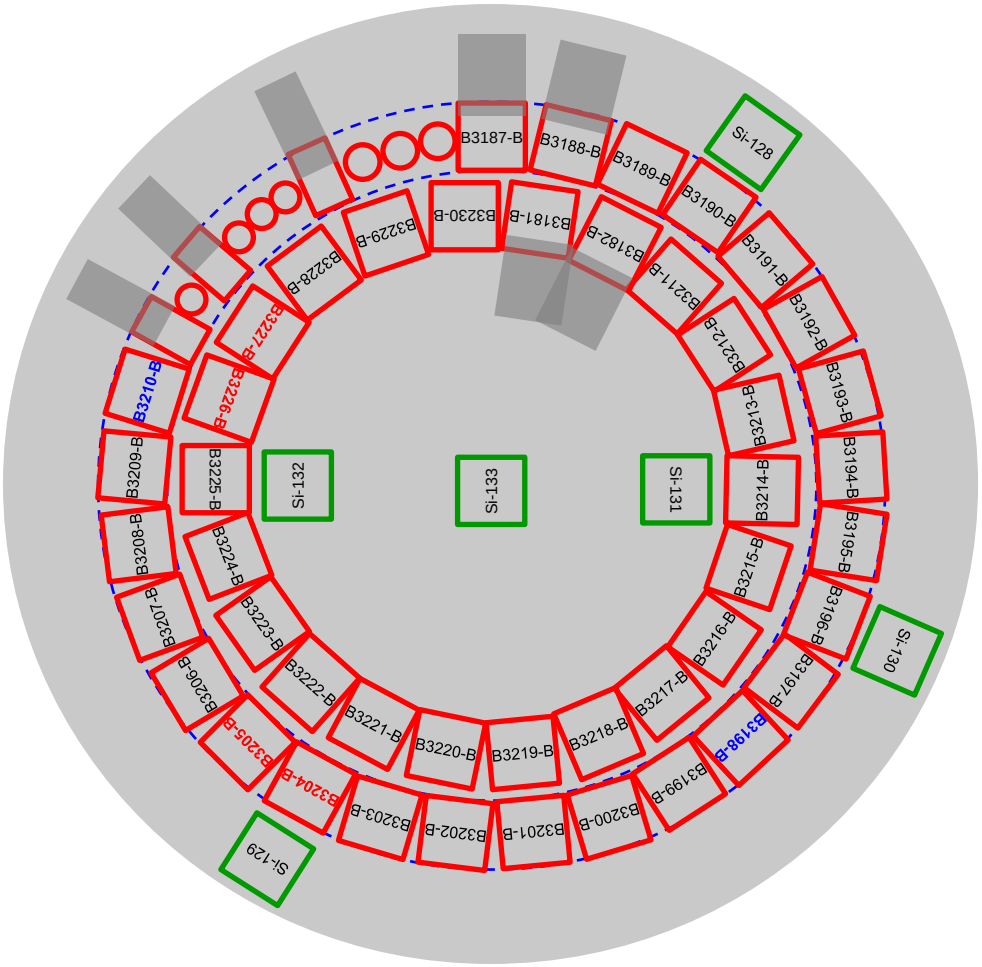
- Degassing and stress relief in vacuum at 1200K for two hours

Sample preparation: film

- Magnetron deposition in Ar/D_2 atmosphere (Denton, Discovery®18, Denton)
- Liquid nitrogen trap to minimize water vapor/ oxygen contamination ($p < 5\times10^{-5}$ Pa)
- Surface etching in Ar with -410V rf bias for 4 min
- Film deposition in 1:1 Ar/D_2 flow at 0.6 Pa, -100 V rf substrate bias for 30 min

- Rotating substrate holder to produce a homogeneous layer
- Silicon substrates for thickness measurement
- All 60 samples in one deposition run
- Samples were positioned in two rings

Sample arrangement



Sample information:

Bulk W samples:

44 regular samples (24 outer + 20 inner)

3 rectangular samples (10x6 mm^2)

7 circular samples (4 times Ø5 mm, 3 times Ø6mm)

RecS6-1, RecS6-2, RecS6-3

CirD5-1, CirD5-2, CirD5-3, CirD5-4

CirD6-1, CirD6-2, CirD6-3

Single crystal Si samples:

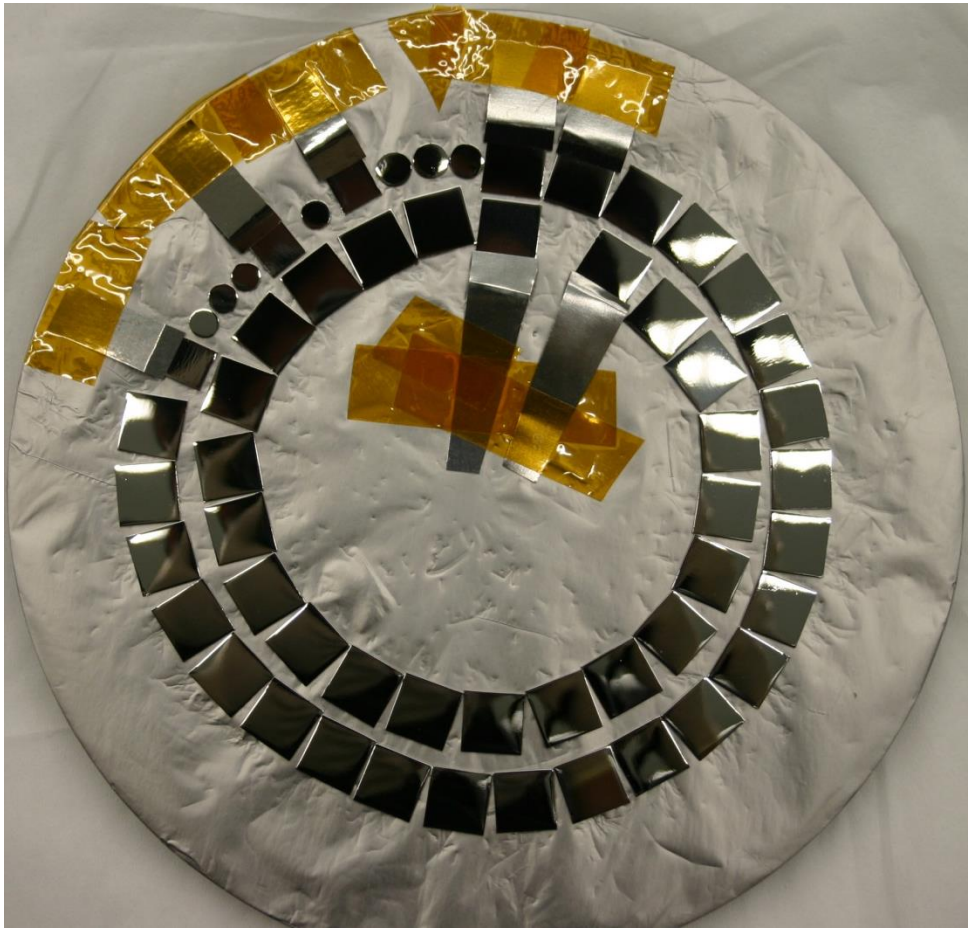
3 outer +2 inner +1 center

Indicated samples underwent TDS

- In October
- In March

Expectation: all samples should show comparable D amounts

Sample arrangement



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Single crystal Si samples:

3 outer +2 inner +1 center

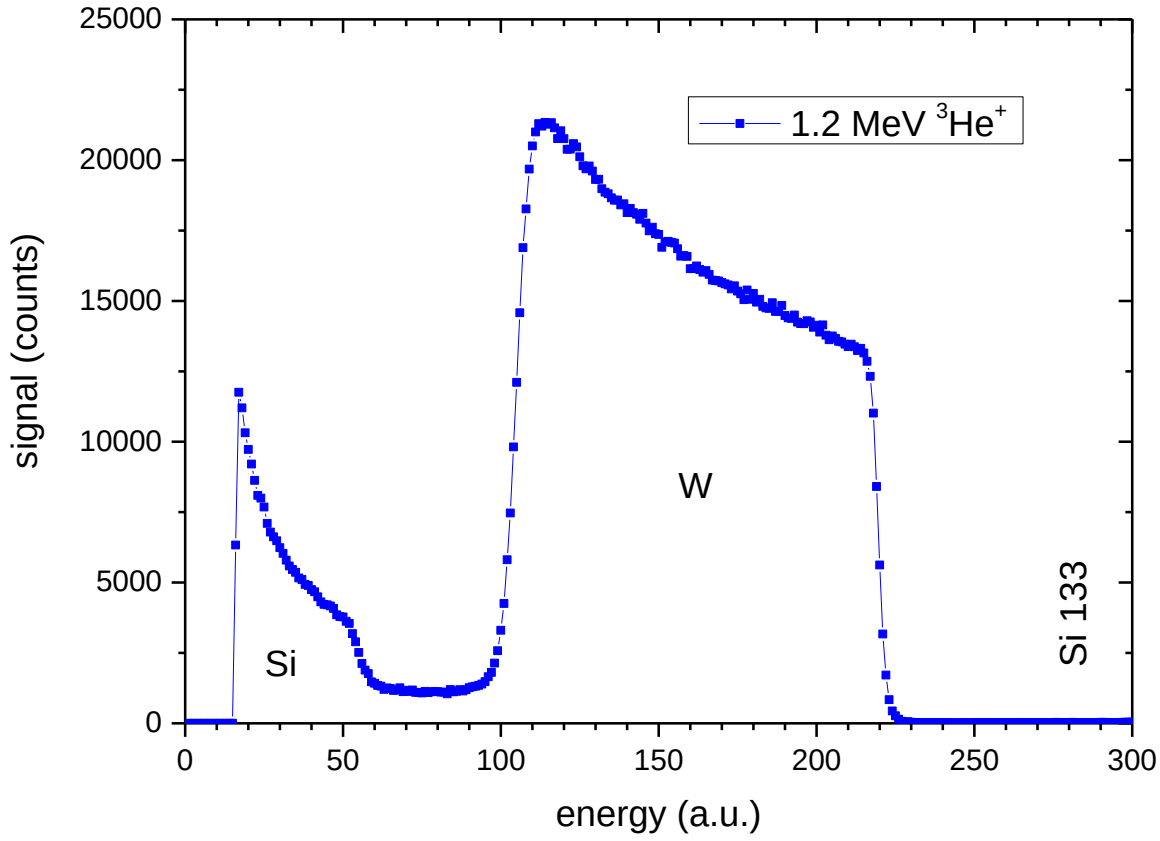
The yellow kapton tape was used to fix the W-foil-clamps partially covering some samples

Photo taken before deposition: without Si samples

All the samples were mounted on the sample holder which was wrapped with 100nm-W-coated Al foil. Deposition on the sidewall will be taken aside first.

Pre-characterization

Rutherford Backscattering of Si witness plates



Areal density: 2.3 x 10^{22} W/m^2

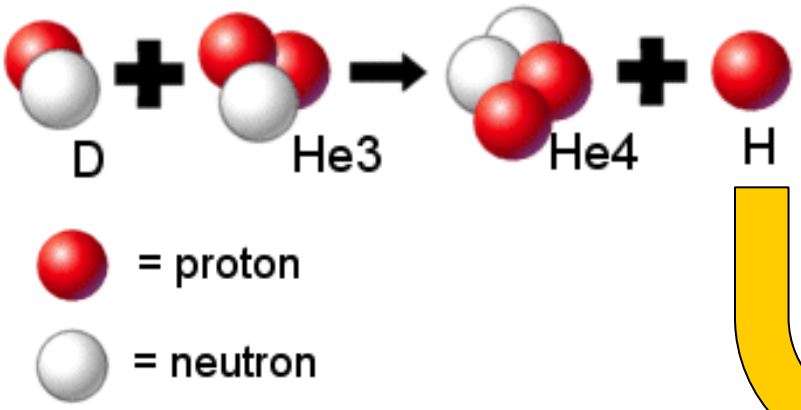
Thickness: ≈ 300 nm

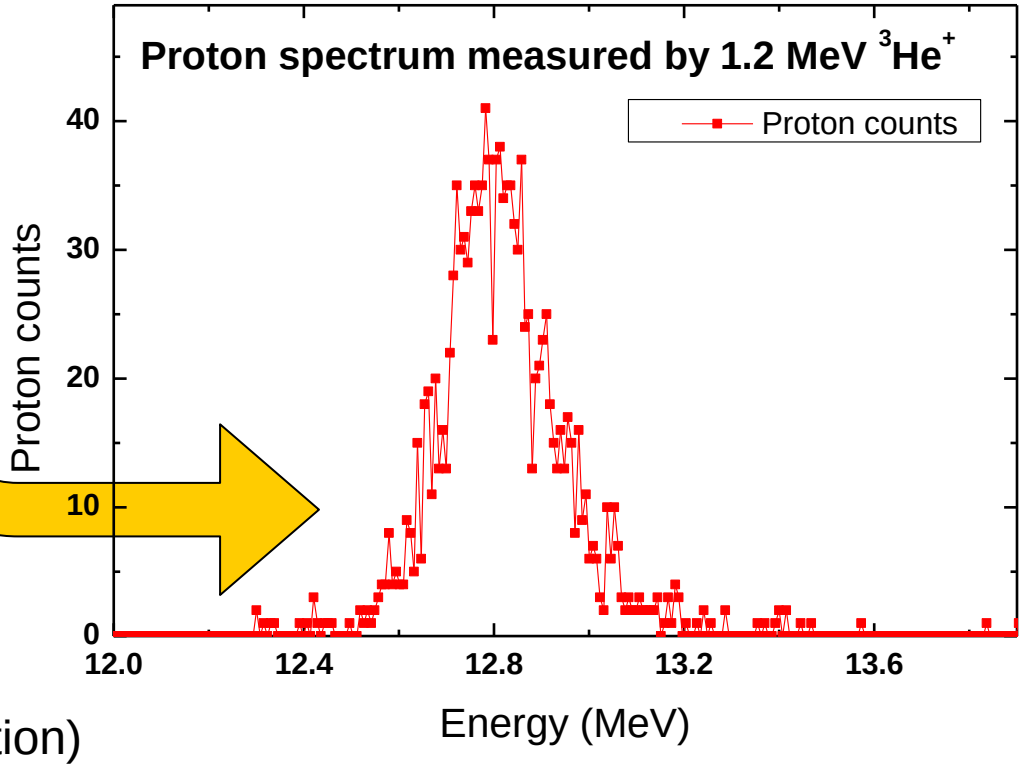
Expected density: ≈ 95 %

Pre-characterization

Nuclear Reaction Analysis (NRA) for D amount measurement

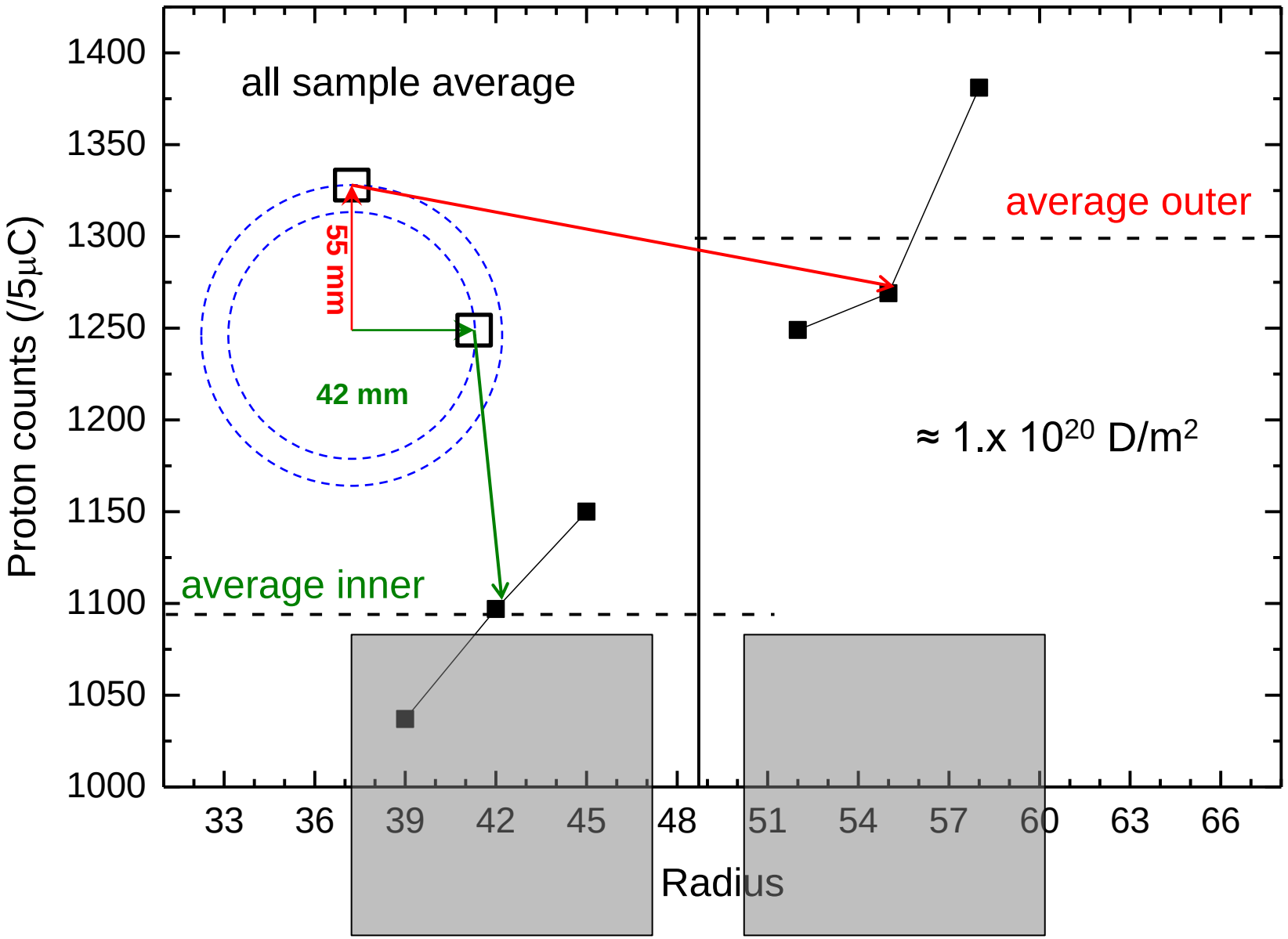
Each sample is measured on **three different (radial) spots** with 1.2 MeV $^3He^+$ beam (NRA).





- Absolute accuracy: 5%? (cross section)
- Reproducibility: 3% (current measurement)

Pre-characterization: NRA



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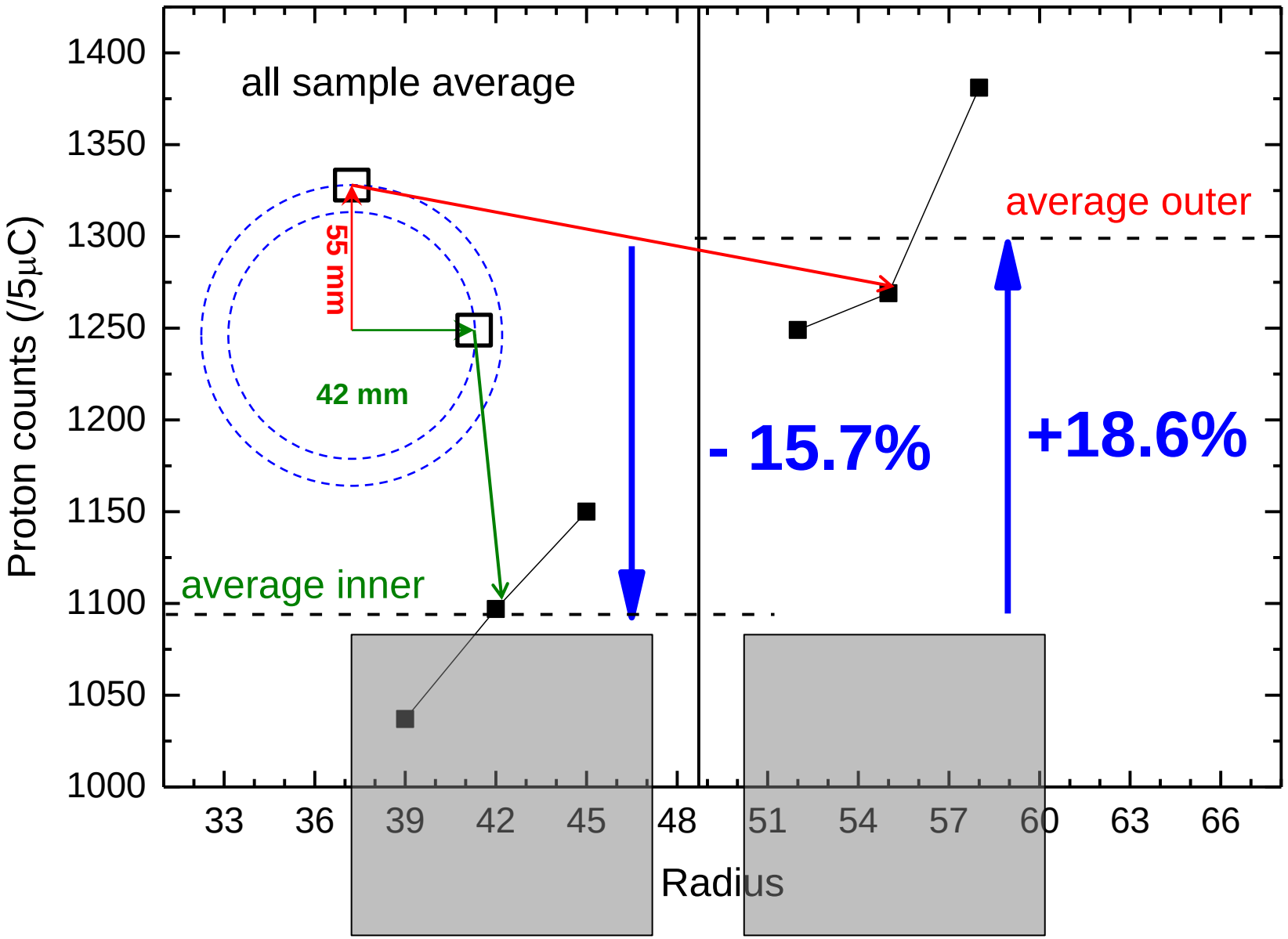
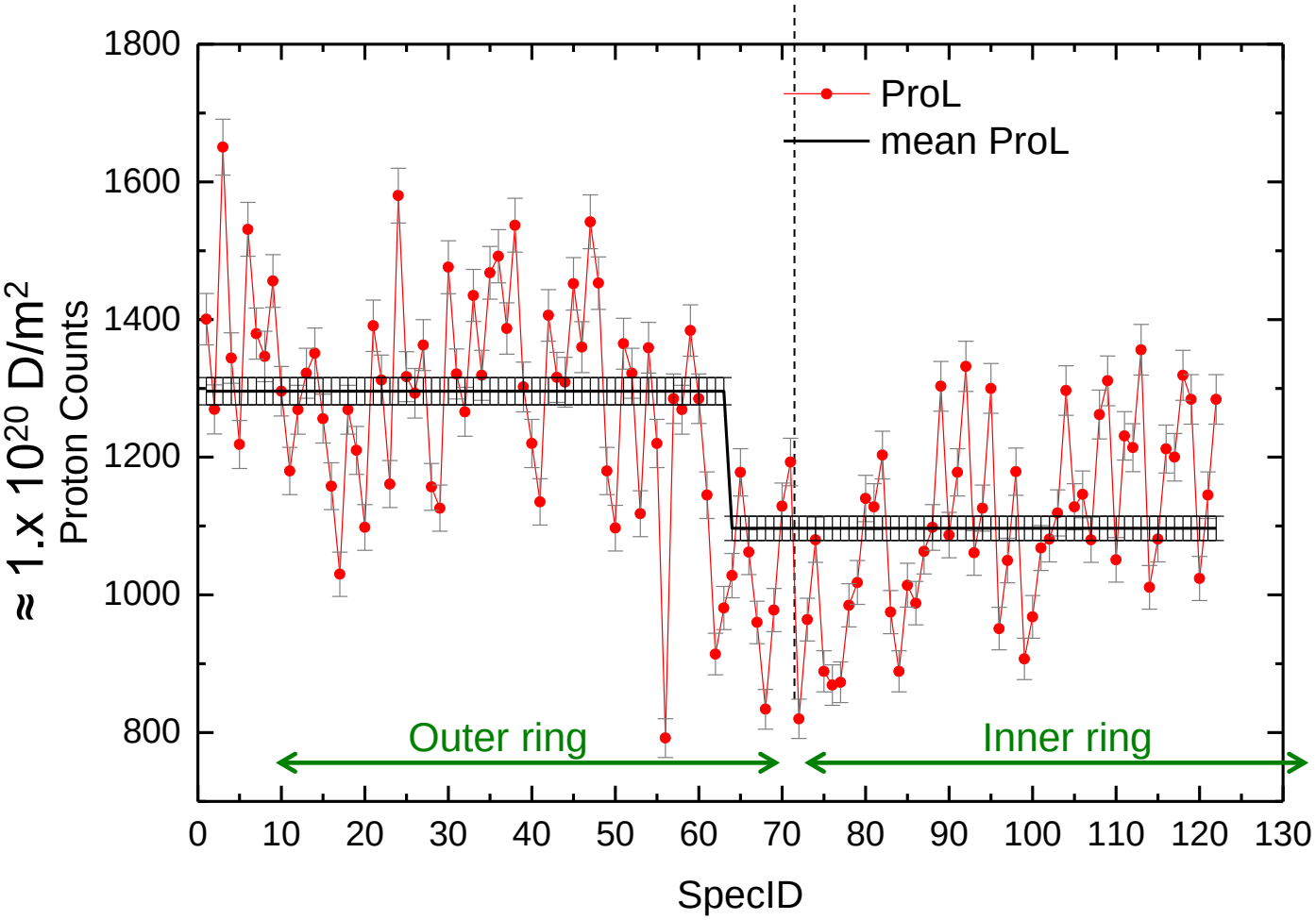


Pre-characterization: NRA

Ion beam analysis on Oct. 5 2016: 1.2 MeV $^3He^+$

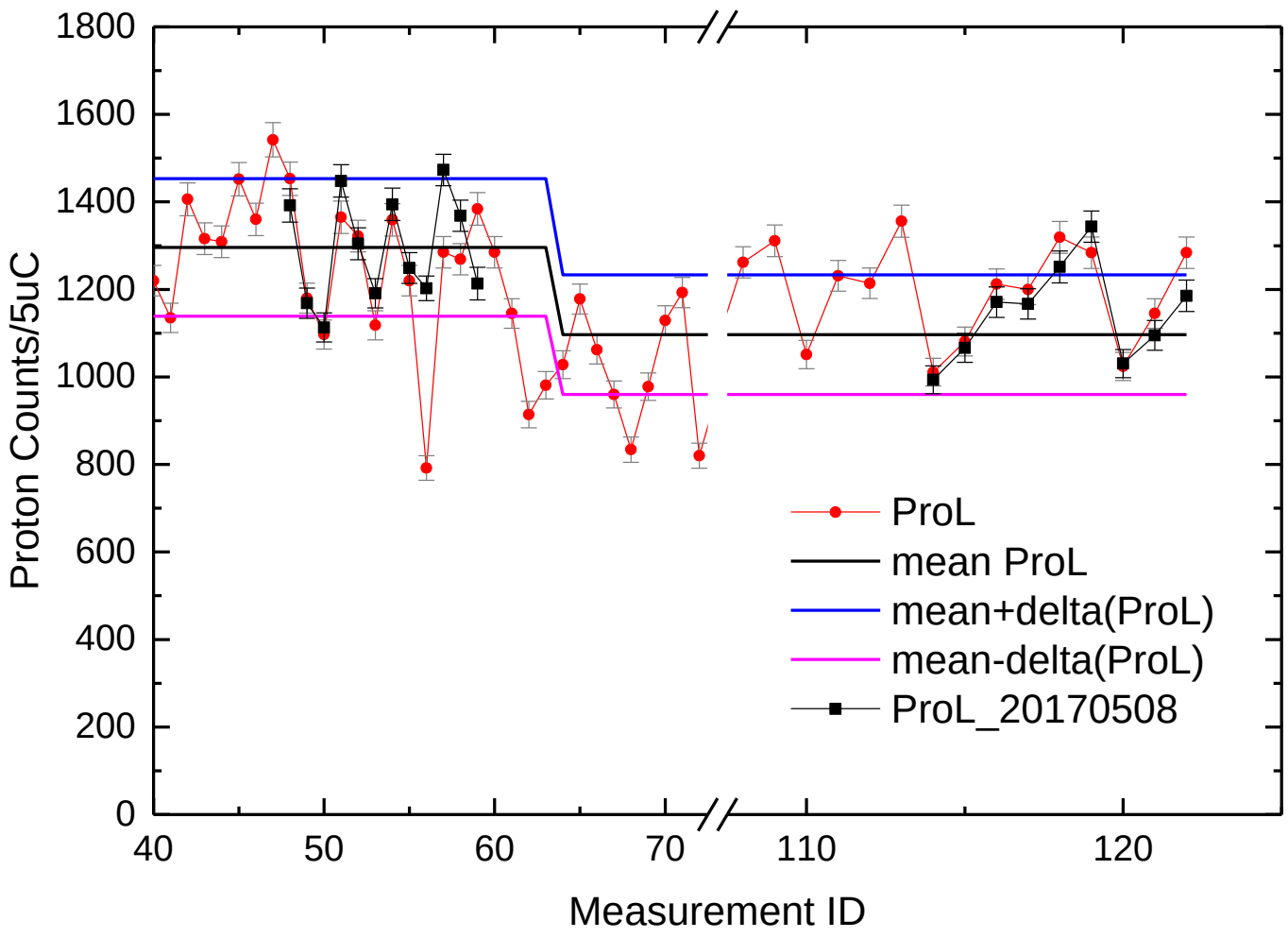


Error bars of individual data points are sqrt(N) = statistical uncertainty of individual data point)

Blue and magenta lines are mean +- sqrt(variance) = measurement scatter (this is significantly larger than the anticipated scatter)

Mean (black line) = mean signal for outer and inner ring samples (indicated error band is sqrt(mean) = anticipated error).

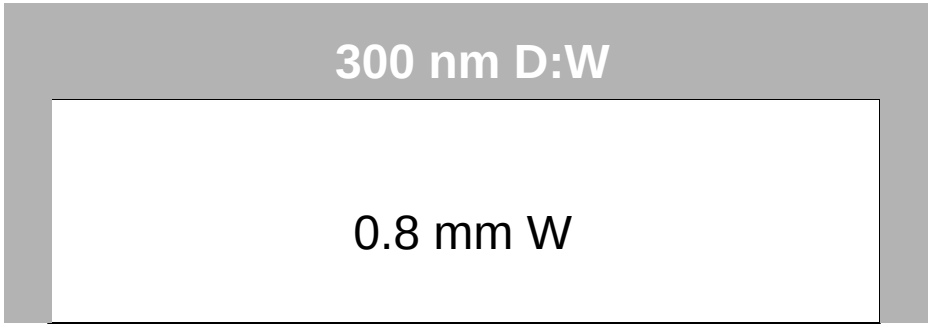
Pre-characterization: 2nd NRA after 5 months



Black data points are from May 08, 2017,
Red data point from October 05, 2016 (measured for the samples still remaining in IPP).
The results agree perfectly within experimental uncertainty.
So we conclude that there is not loss of D during storage!

Always 3 positions on one sample are measured. A clear decreasing trend across both samples is found. The D amount decreases from the outermost position on the outer-ring samples to the inner most on the inner ring samples (the fact that the gradient seems to be opposite in the figure is due to a rotation of the samples).

IBA pre-characterization



Areal density:

2.3×10^{22} W/m^2

1.3×10^{20} D/m^2

D/W ≈ 0.6 at.%
(De Temmerman scaling 0.4%)

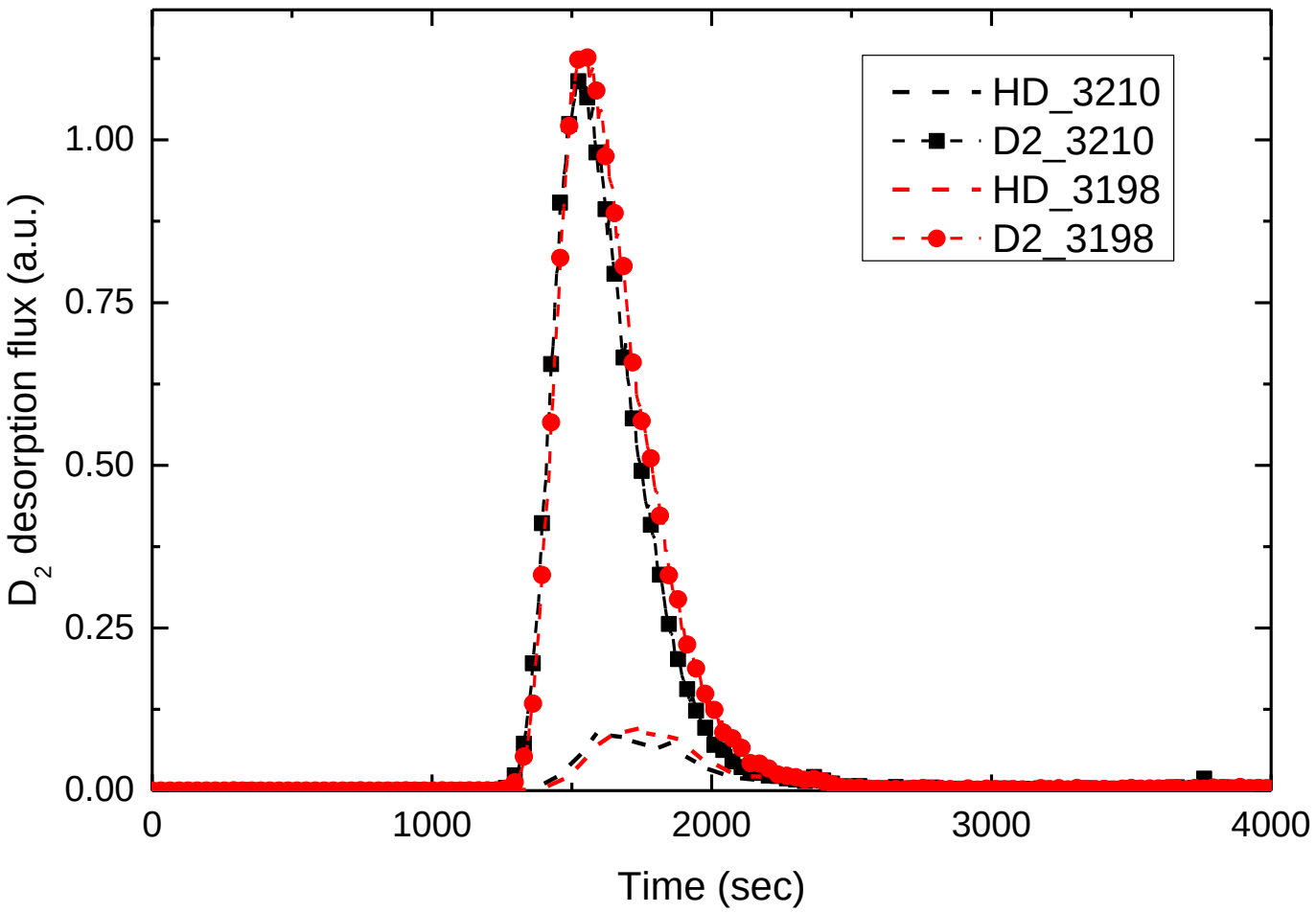
Thickness: ≈ 300 nm

Expected density: ≈ 95 %

Deposition on side walls < 10%

(to be checked)

Pre-characterization: TDS

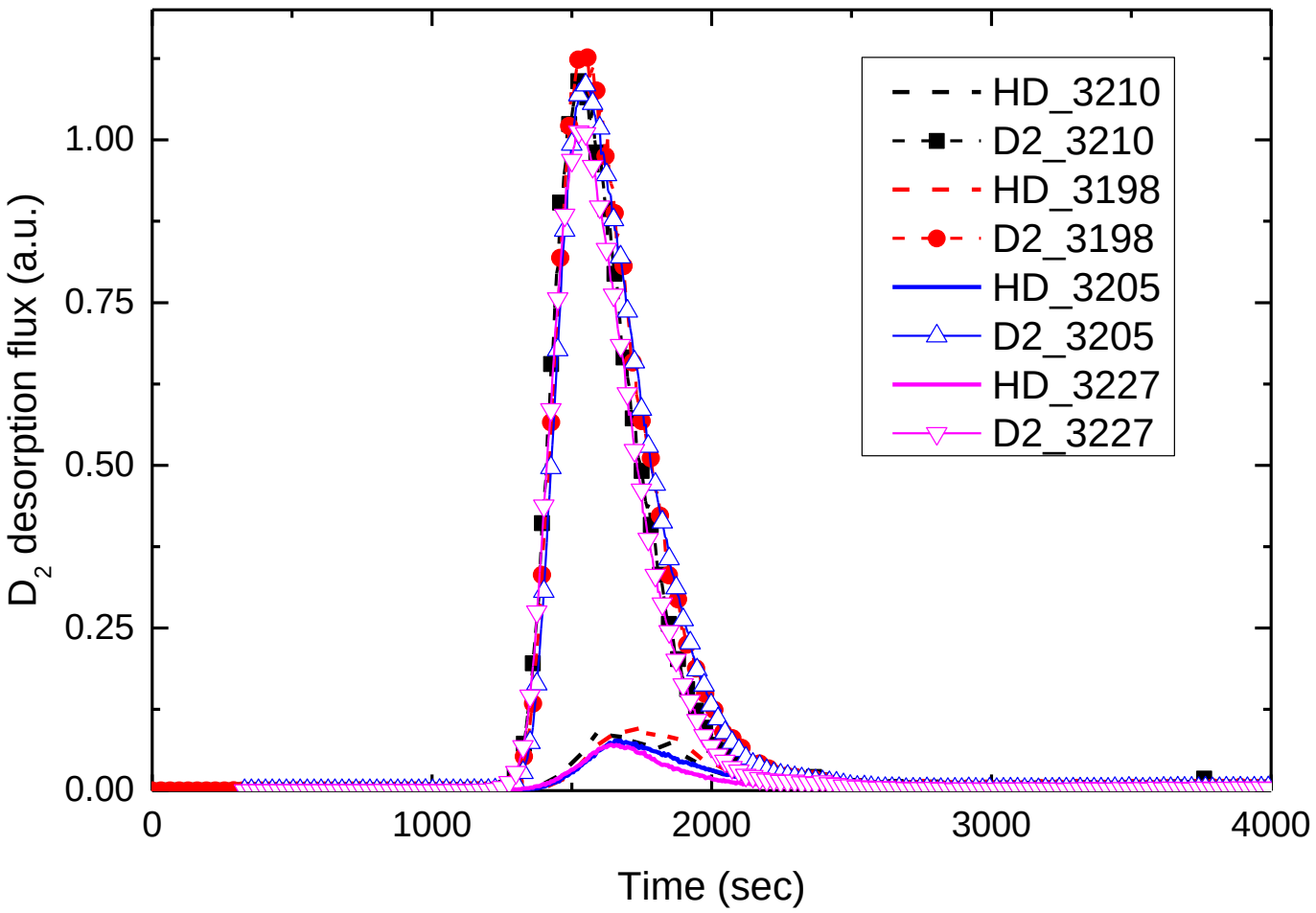


TDS on two outer ring samples in October:

- Same shape and peak position
- Spectra yield the same D amount within 10%

⇒ **reproducible**

Pre-characterization: TDS



TDS on two outer ring samples in October:

- Same shape and peak position
- Spectra yield the same D amount within 10%

⇒ **reproducible**

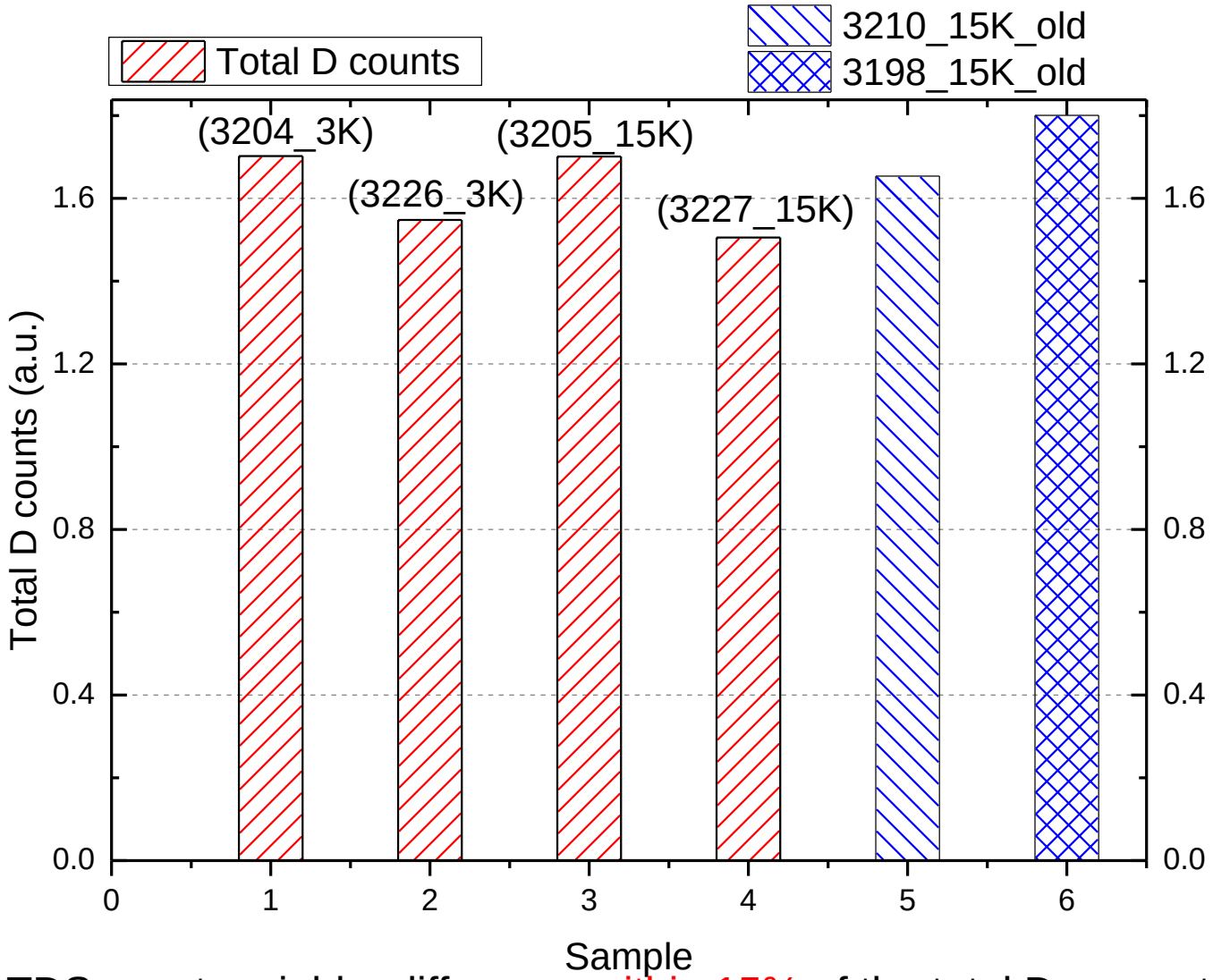
TDS on two more outer ring samples in March:

- Same as above

⇒ **No outgassing**

.

Pre-characterization: TDS



TDS total counts comparison:

In red: TDS in Mar. 2017

In blue: TDS in Oct. 2016

Note that the new samples have 2 different ramping rates. The old TDS measurements are for 2 inner-ring samples

TDS spectra yield a difference within 15% of the total D amount between the inner- and outer-ring samples for both measured sets of samples at different time.

Conclusion

- 60 samples were simultaneously coated in October with a ≈ 300 nm thick D containing tungsten film by magnetron sputtering (50 on W, 10 on Si)
- Each sample was measured by NRA at three locations before shipping
- Samples have a (radial) gradient in D content of 10%
- D content from inner and outer ring samples deviate by 15%
- D content within one ring varies by less than 10%
- There is no outgassing over 5 months observable; neither with TDS nor with NRA

Personal remark

- Final goal: understand and describe hydrogen isotope uptake, transport, retention and release in tungsten
- TDS is one major analysis technique to provide important information
- However, to reach the final goal there are many many more steps to make
  - Compare different TDS setups: How reliable is it what we measure?
  - Compare different diffusion trapping codes: Do we model the same?
  - Identify valuable set of benchmark experiments
  - Move from empirical to microscopic understanding
  - …
- We are only at the very beginning