

# Experimental possibilities in Jülich – an update

15<sup>th</sup> June 2016

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| P. Dollase, J. Du, Y. Ferro, A. Kreter, J. Linke, Ch. Linsmeier, D. Matveev

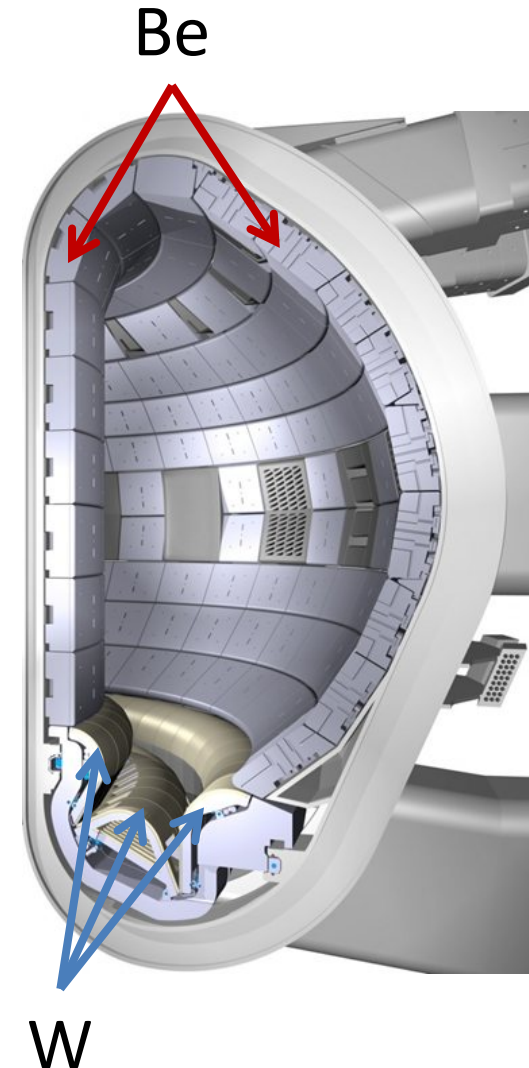
| C. Pardanaud, B. Spilker, B. Unterberg, T. Weber

# Outline

1. Motivation
2. Update on experiments
  - XPS
  - Raman
  - ARTOSS
  - FREDIS
  - JULE-PSI
  - JUDITH 1 & 2
3. Concluding Remarks

# Motivation

- ITER material mix: Be and W
  - 700 m<sup>2</sup> Be
  - W in divertor region
- Plasma impurities
  - Seeding gas, e.g. Nitrogen
  - Contaminations, e.g. Oxygen
- Material transport by:
  - Erosion
  - Plasma transport
  - Re-deposition

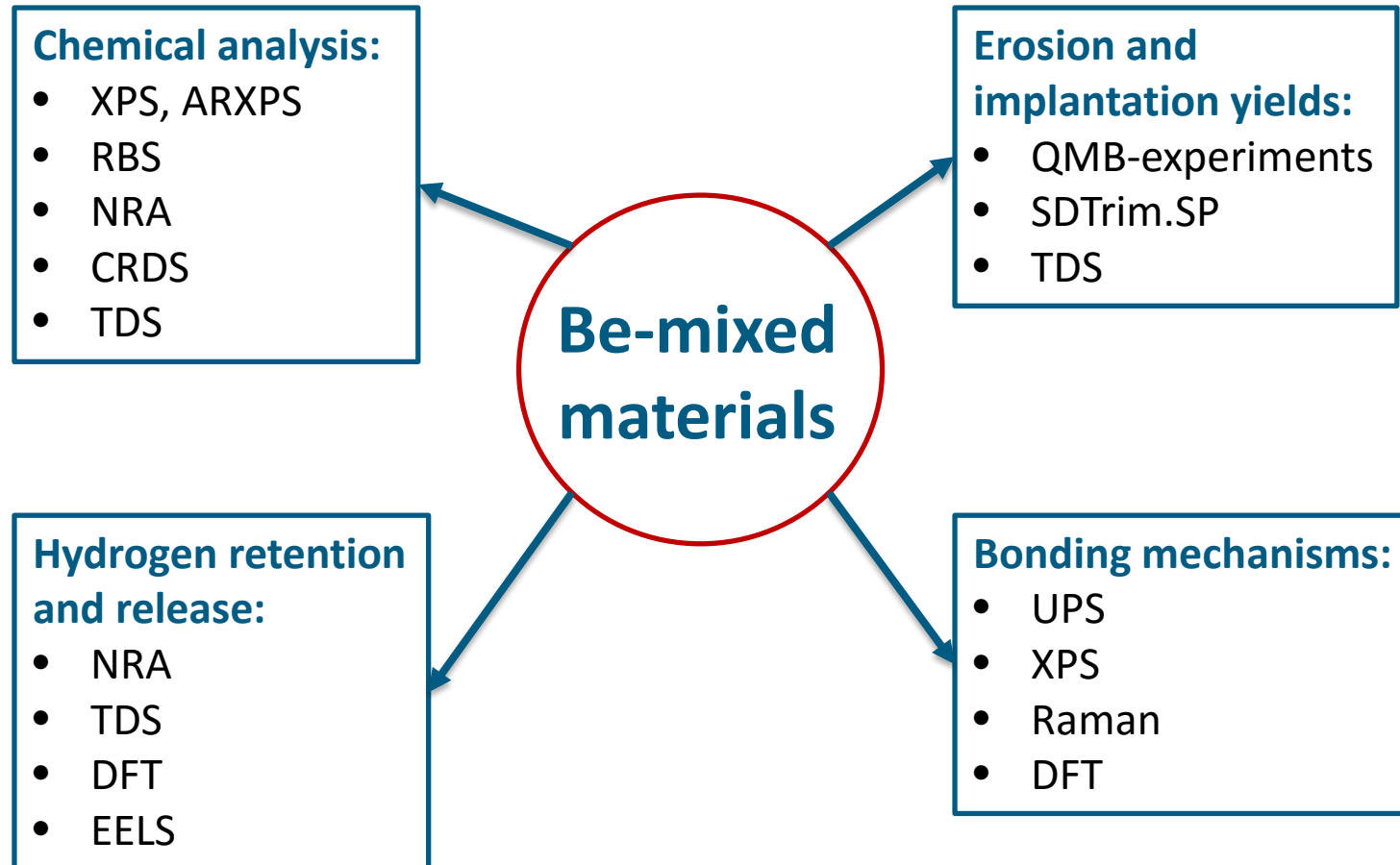


# Why is compound formation of interest?

- Elevated temperatures and ion irradiation lead to solid state reactions
  - e.g. Be<sub>2</sub>W alloy formation in divertor region
- Be-based mixed materials have new properties compared to Be:
  - Isolating / different conductivity
  - Altered hydrogen retention
  - Co-deposits
  - Ammonia formation
- Physical and Chemical Data needed as input data for e.g. ERO, SOLPS, WallDyn to predict first wall properties for future devices

	Be	BeO	Be <sub>3</sub> N <sub>2</sub>	W	WO <sub>2</sub>	WO <sub>3</sub>
Melting Point [K]	1551	2780	2473	3683	1773	1746
$\Delta_f H^\circ$ [kJ mol <sup>-1</sup> ]	--	-609	-558	--	-590	-843

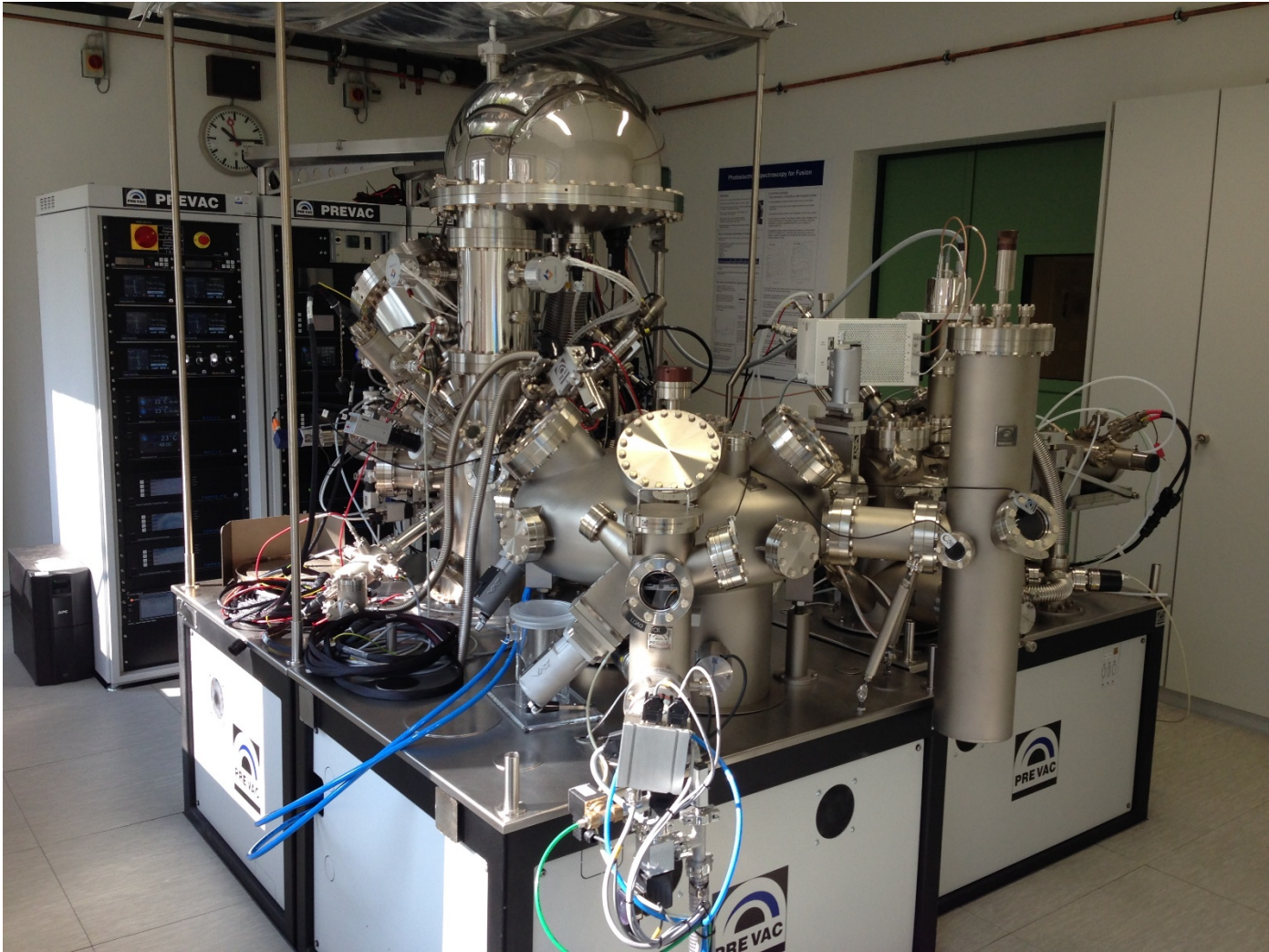
# Multi-method approach: Solutions



# XPS setup

Investigation of Be-based mixed materials

# Experimental Setup



R4000 analyser

# Experiments: Overview XPS

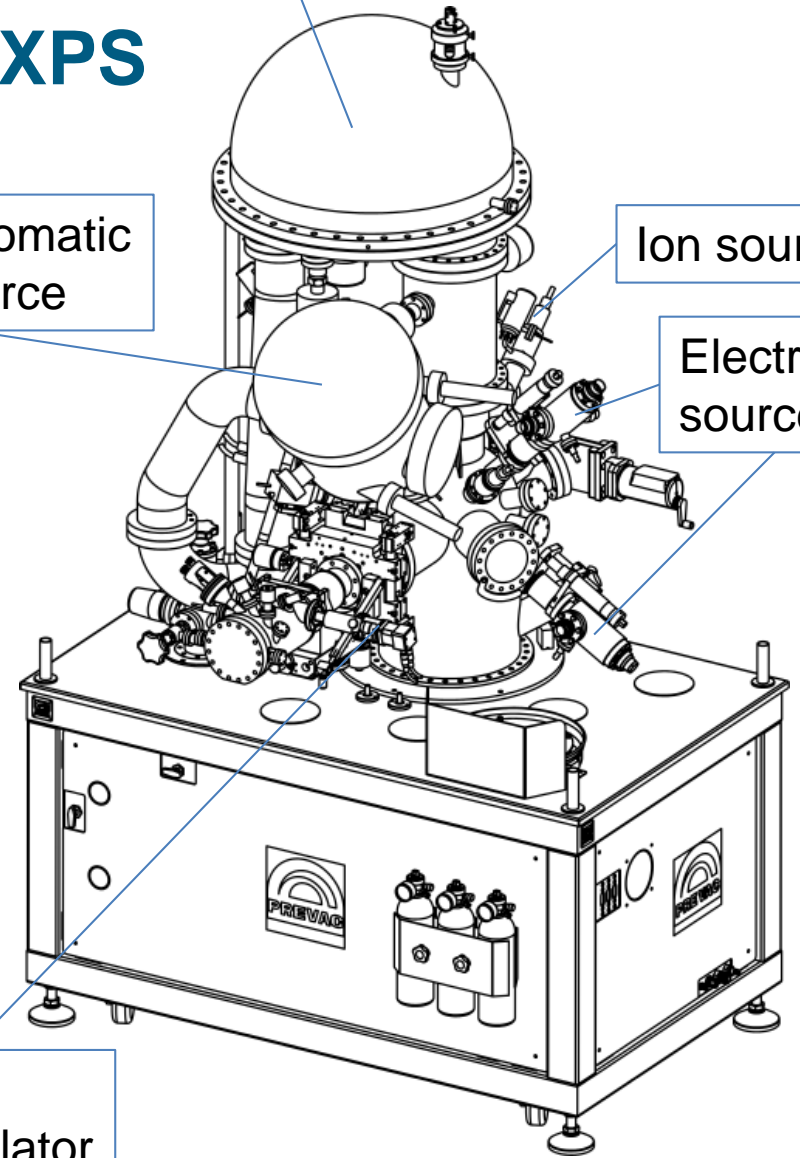
## Main chamber

- XPS
  - Monochromatic x-ray source
  - Hemispherical analyser
- 2 ion sources, one with a Wien Mass Filter
- 2 electron sources
- UV source
- Sample size:
  - heating: 2,5 cm
  - Non-heating: 5 cm

Monochromatic x-ray source

Ion source

Electron sources

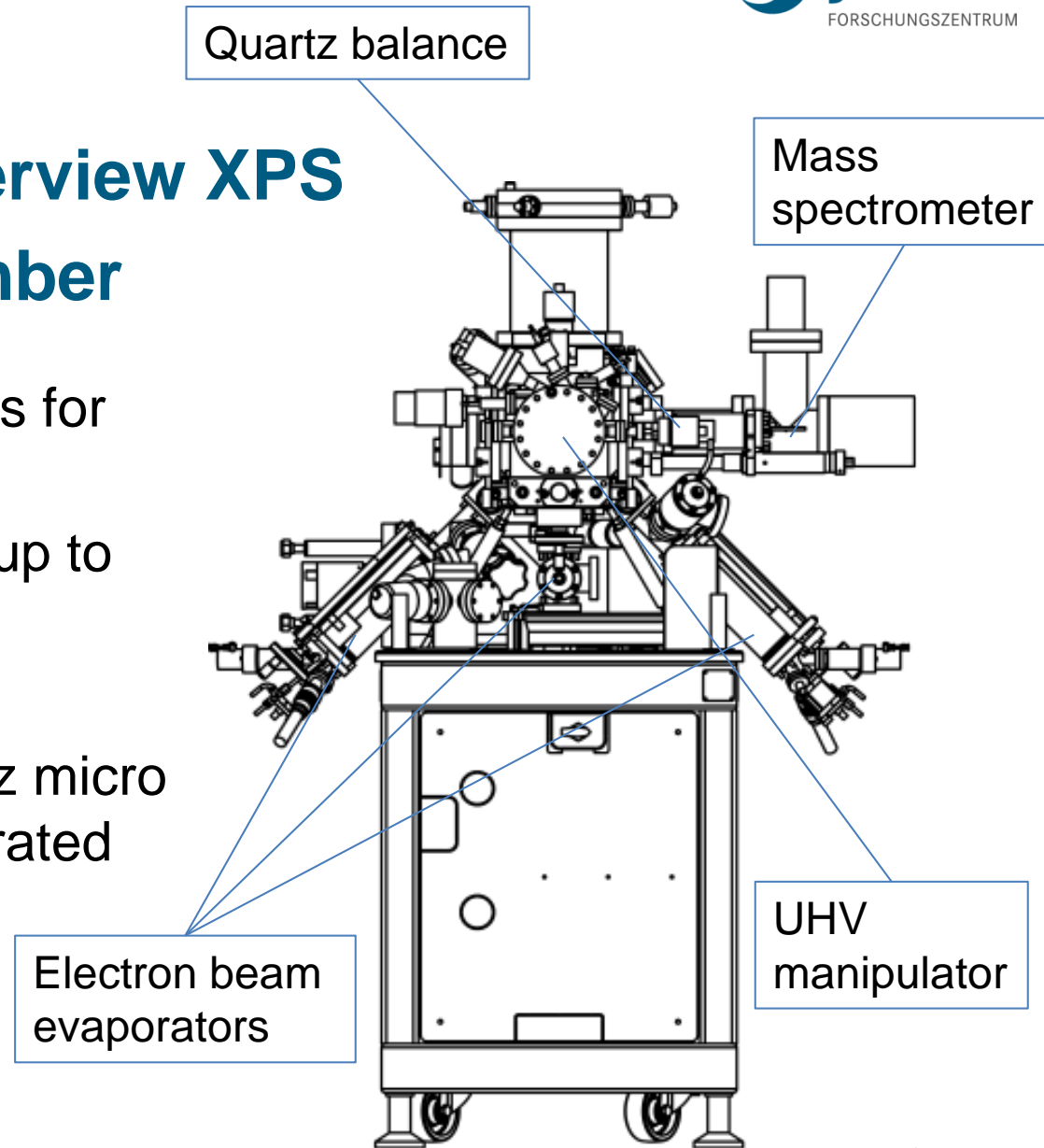


4-axes manipulator

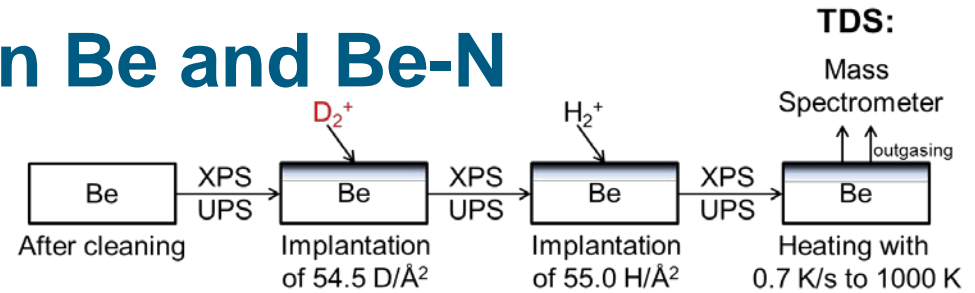
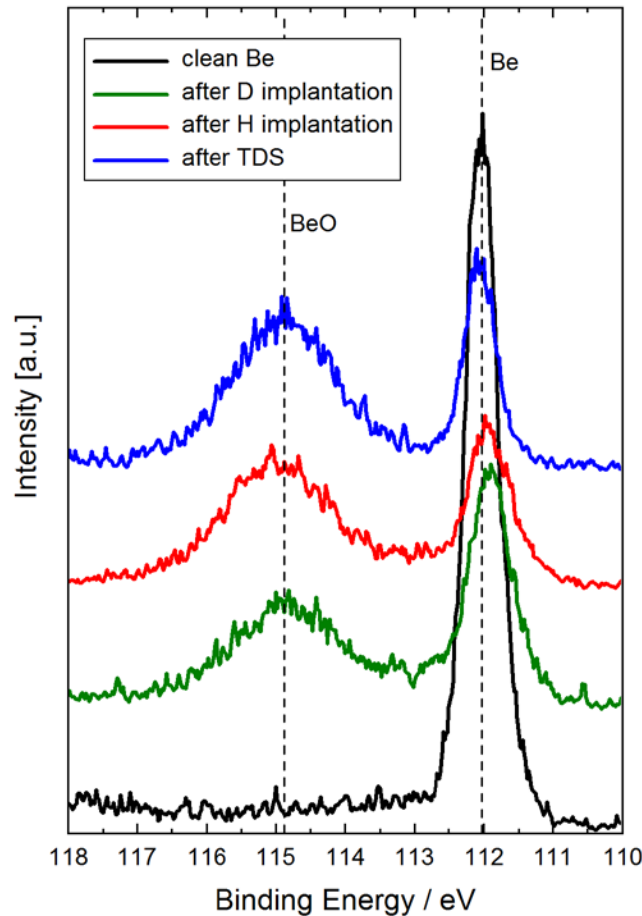


# Experiments: Overview XPS Preparation Chamber

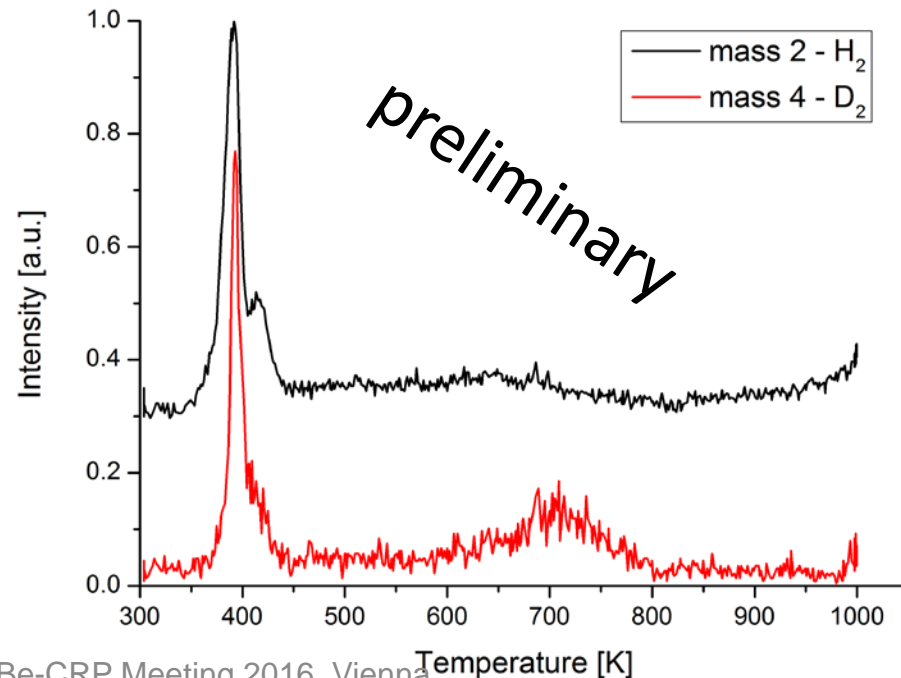
- 3 electron evaporators for W, C, Be
- Manipulator, heating up to 2500 °C
- Mass spectrometer
- High-resolution quartz micro balance can be integrated



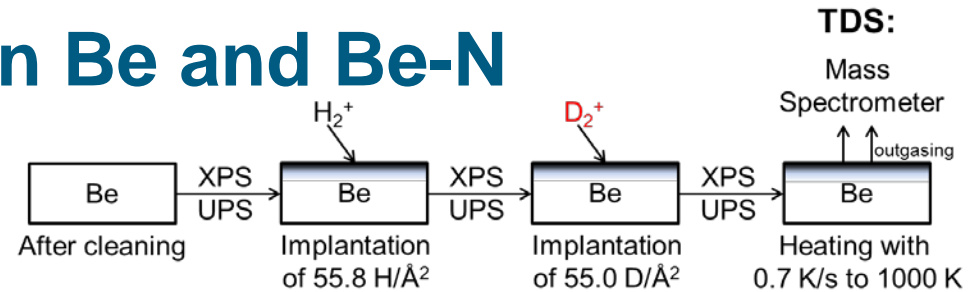
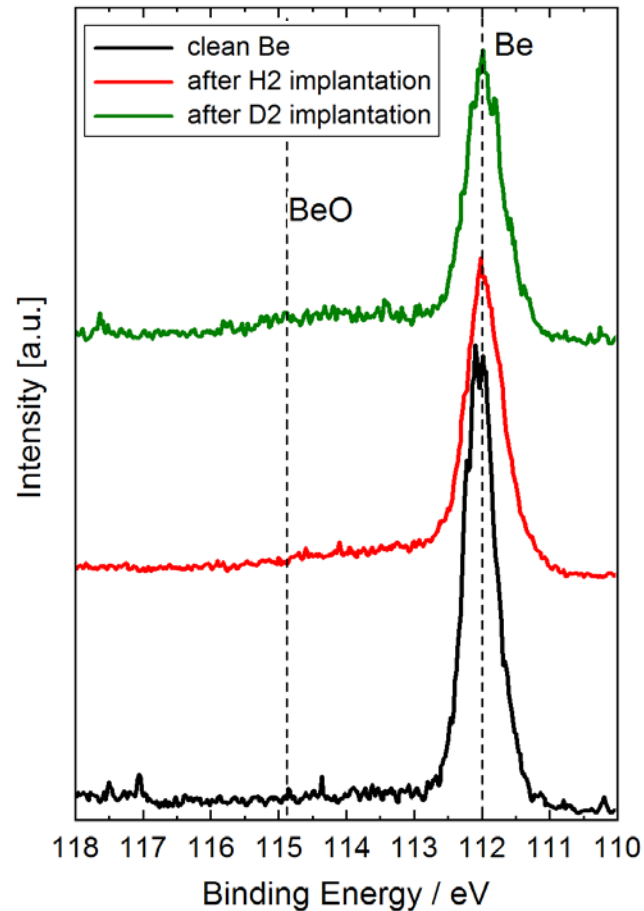
# Isotope exchange on Be and Be-N



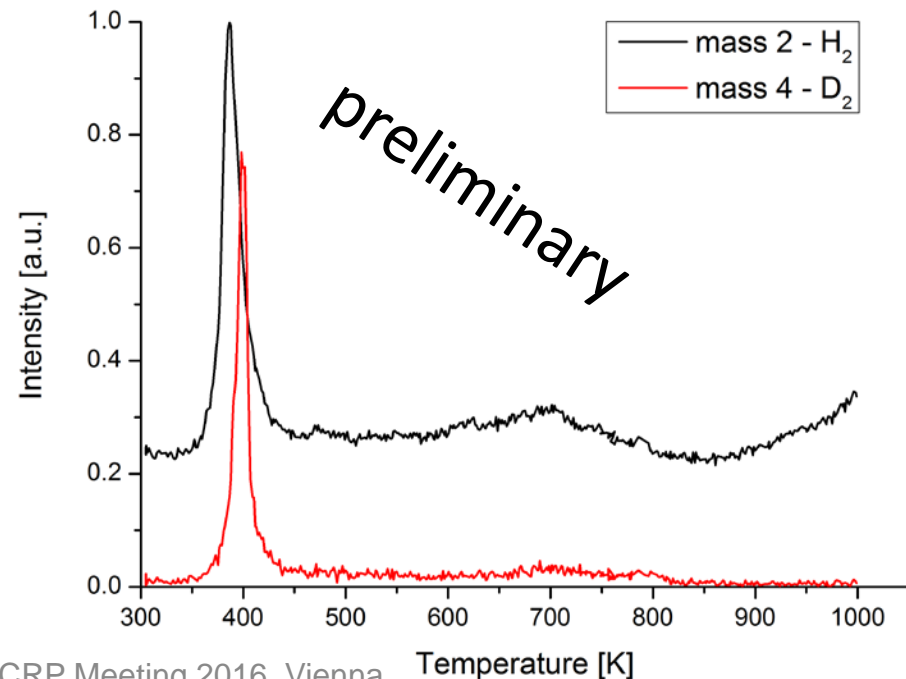
Implantation conditions: 3 kV → 1.5 keV/D or H  
 Area: 2.5 x 2.5 mm<sup>2</sup>; Sample current: 0.6 – 1 μA



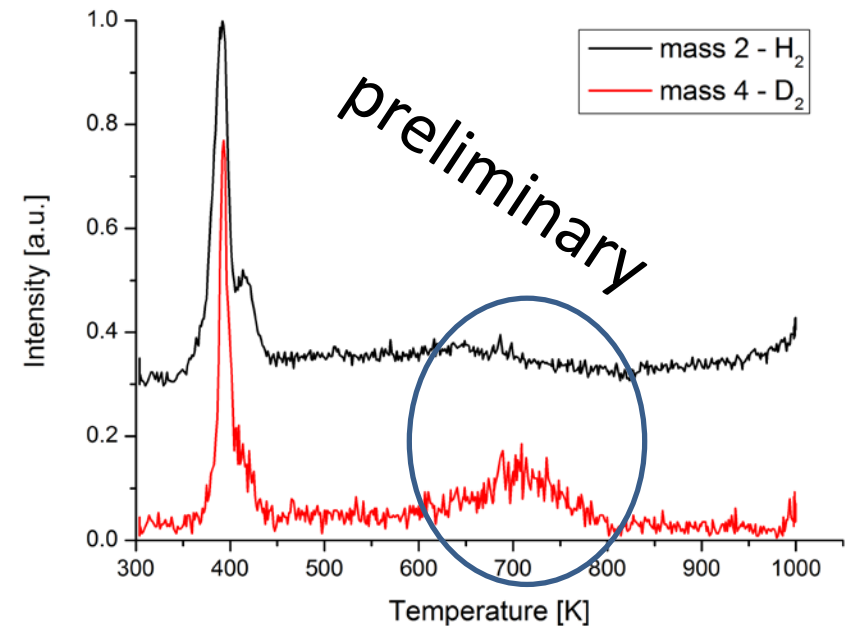
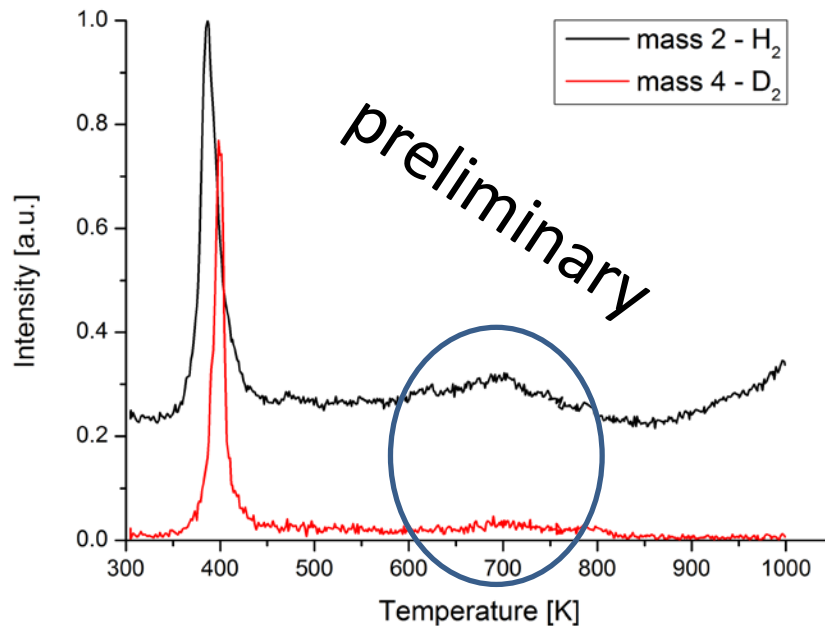
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Area: 2.5 x 2.5 mm<sup>2</sup>; Sample current: 0.6 – 1 μA



# TDS Results



First implanted species occupies energetically favoured trap sites

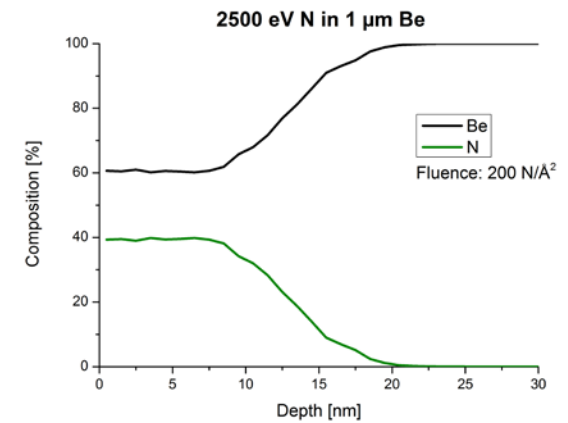
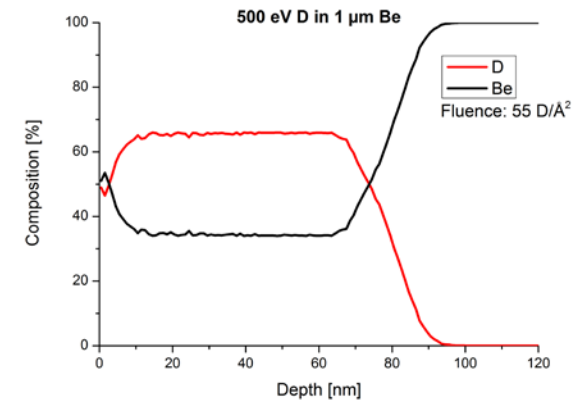
# Isotope exchange: Status & Outlook

## Done:

- Construction, installation and commissioning of the experimental setup
- First experiments for deuterium/hydrogen isotope exchange on beryllium

## To do:

- Further experiments with the ion source with Wien filter
- Use different implantation energies (500 eV to 5,000 eV)
- Do the same experiments on  $\text{Be}_3\text{N}_2$

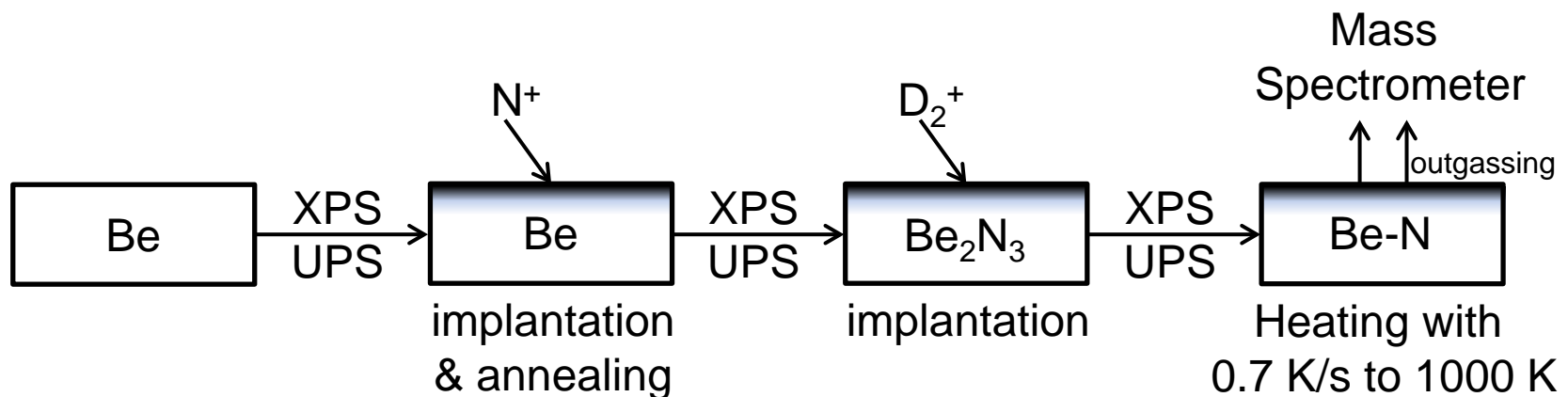


# Outlook: Ammonia formation on Be-N

Exposure of thin Be-N films to hydrogen ions & gas under:

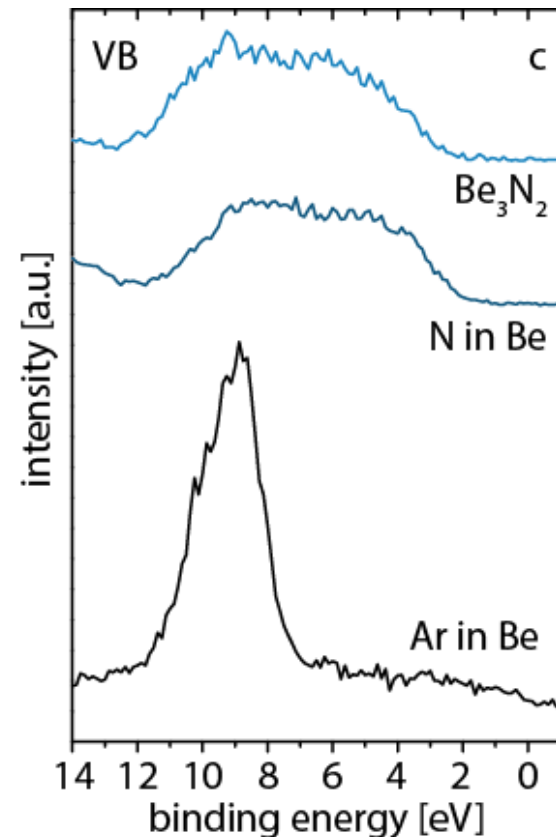
- Different surface temperatures (ions & gas)
- Different hydrogen partial pressures (gas)

=> Address the issue of mobilized tritiated ammonia



# Outlook: Interaction of Be and noble gases

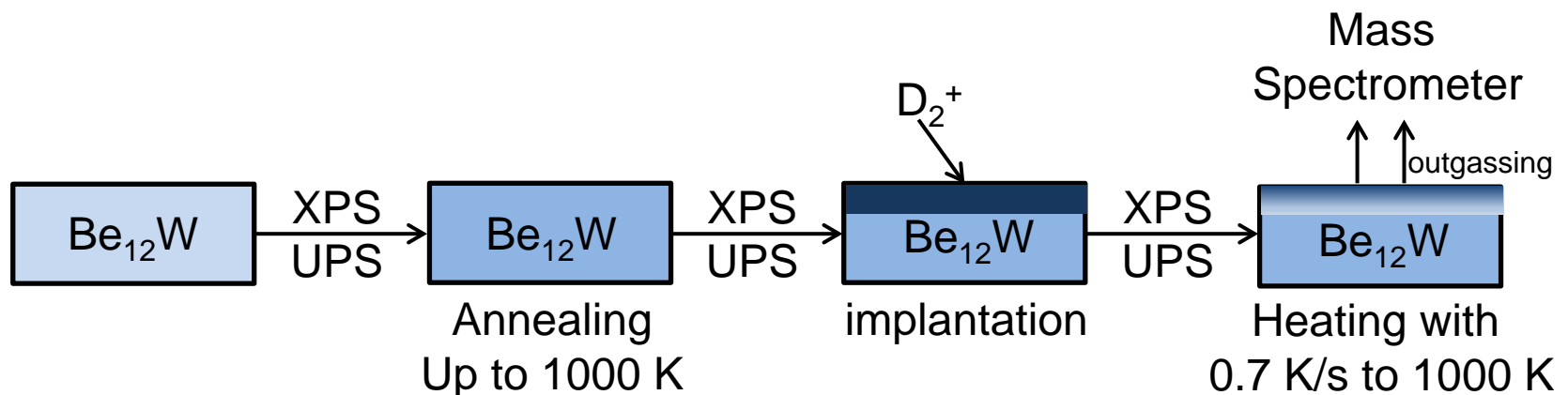
- Influence of noble gas ion bombardment on:
  - Be and its electronic structure (UPS and maybe DFT investigation)
  - H-retention behaviour
  - Noble gases to investigate: He, Ne, Ar, Kr, Xe



See Allouche et al., 2015

# Beryllides

- Understanding beryllides is important as they are used in the breeding blanket
- DFT study on beryllides performed
- Experiments on thermal behaviour and H-retention of beryllides start in october 2016
- Beryllides:  $\text{Be}_{12}\text{Cr}$ ,  $\text{Be}_{12}\text{Ta}$ ,  $\text{Be}_{12}\text{W}$ ,  $\text{Be}_{12}\text{Ti}$ ,  $\text{Be}_{12}\text{V}$



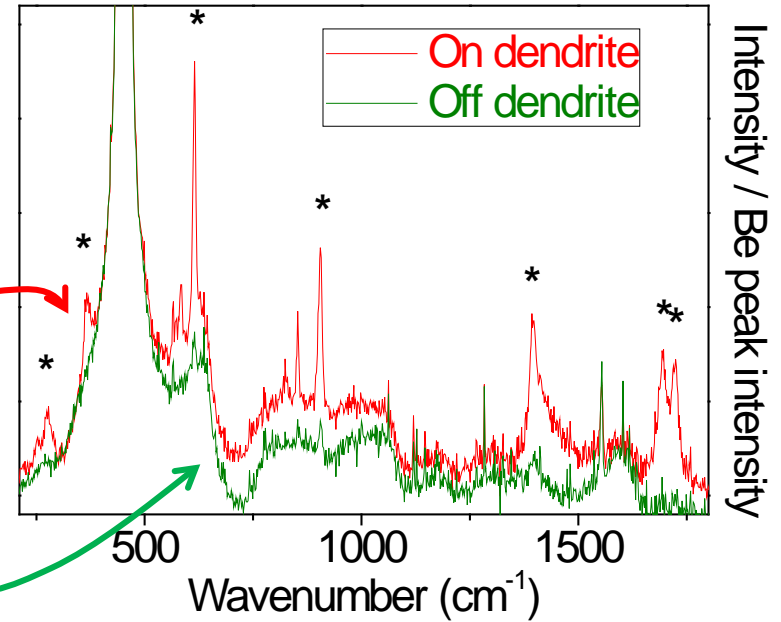
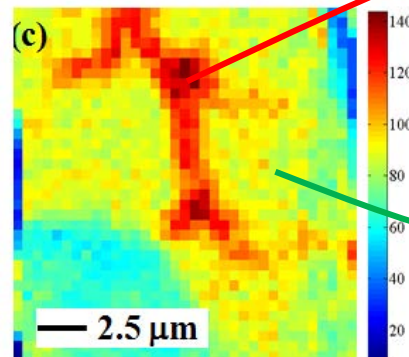
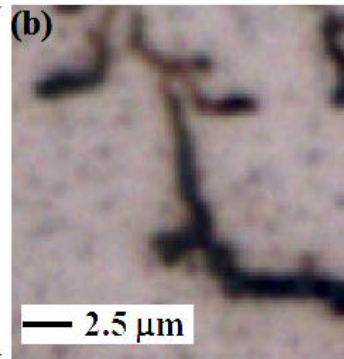
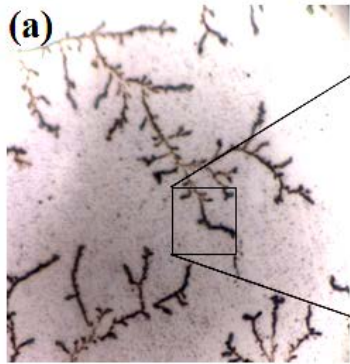


# Raman Spectroscopy

Collaboration with Aix-Marseille  
Université

# Example 3: Be implanted by D

( $\phi > 2 \times 10^{17} \text{ cm}^{-2}$ ,  $E_c = 2 \text{ keV}$ )



*To reach a good S/N ratio: 1 spectrum takes few hours*

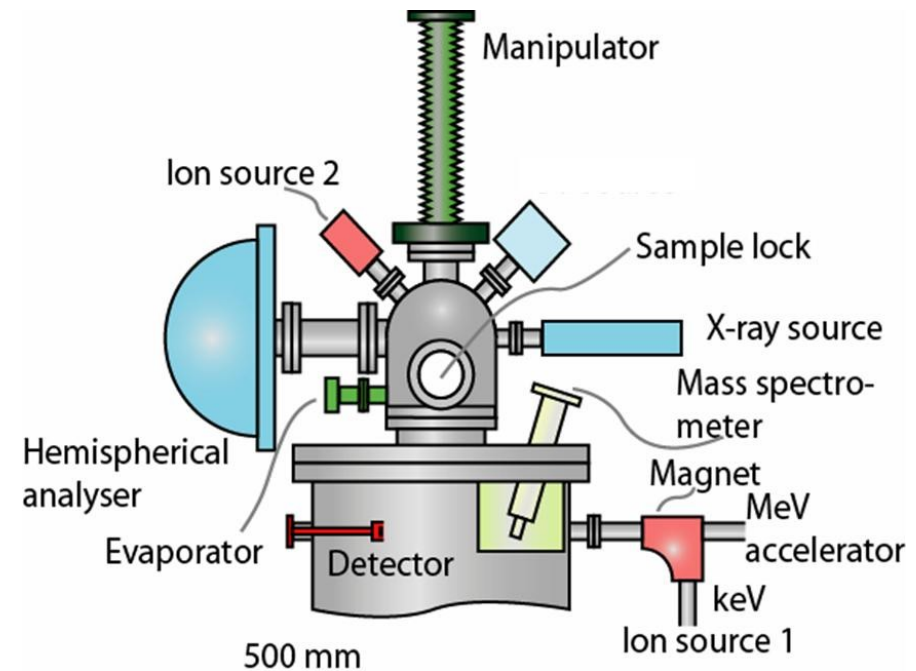
**Be-D vibrational modes evidenced on dendrites !**

# ARTOSS

Dedicated to H-retention experiments

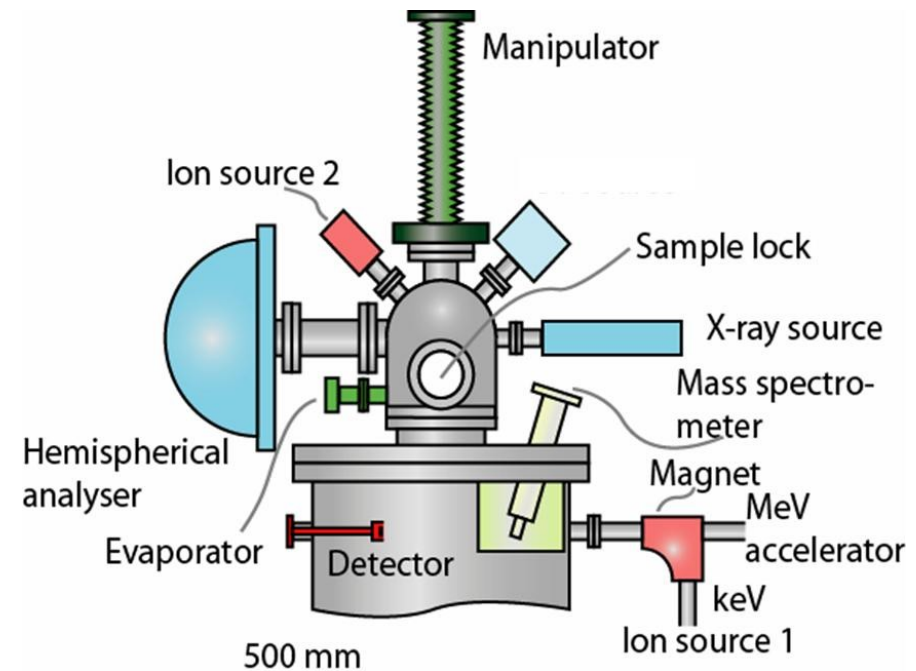
# ARTOSS

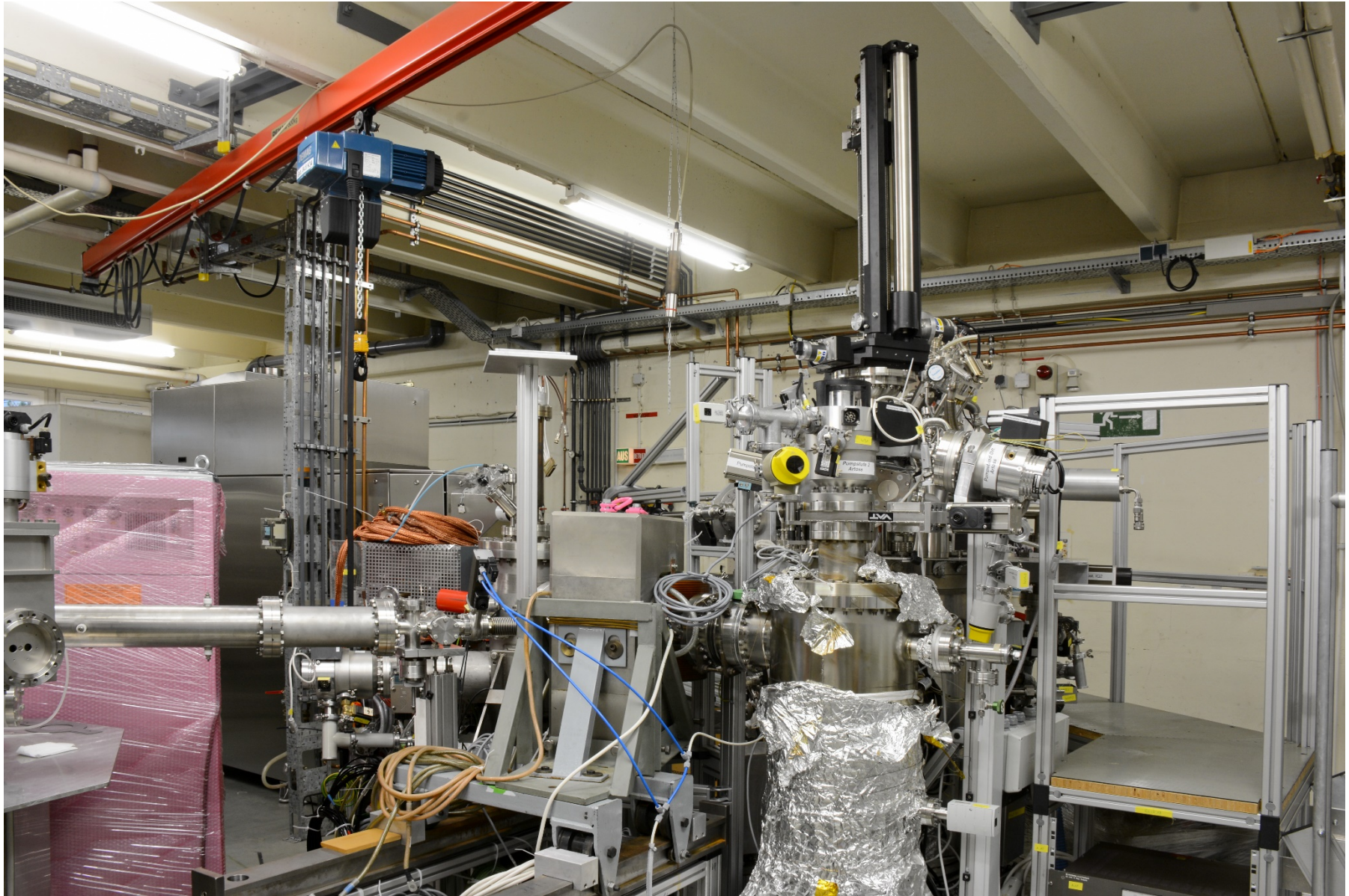
- Ion beam analysis
  - Nuclear reaction analysis (NRA)
  - Rutherford backscattering spectroscopy (RBS)
  
- Mass-spectrometer for thermal desorption spectroscopy (TDS)
  
- Standard-X-ray Source



# ARTOSS

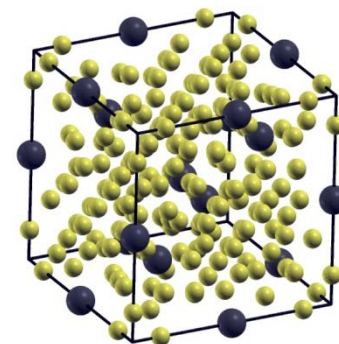
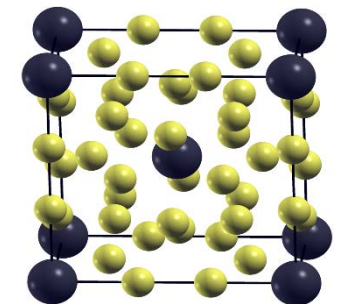
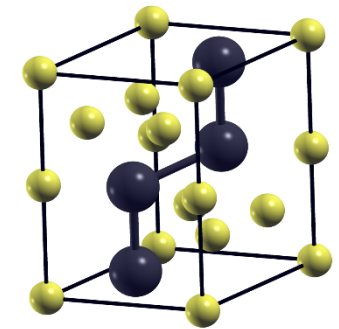
- Mass-separated low energy ion source (0.1-10 keV)
- Thermal atomic H-source
- Base Pressure:  $< 5 \times 10^{-11}$  mbar
- Electron beam evaporator





# Achievements & Outlook

- ARTOSS commissioning is complete (without bake-out)
  
- Start with Beryllium phase:
  - Dedicated studies on the retention behaviour of the beryllium tungsten alloys  $\text{Be}_2\text{W}$ ,  $\text{Be}_{12}\text{W}$  and  $\text{Be}_{22}\text{W}$
  - Comparison to DFT calculation by A. Allouche & L. Ferry and J. Bröder
  - Modelling with CRDS by D. Matveev

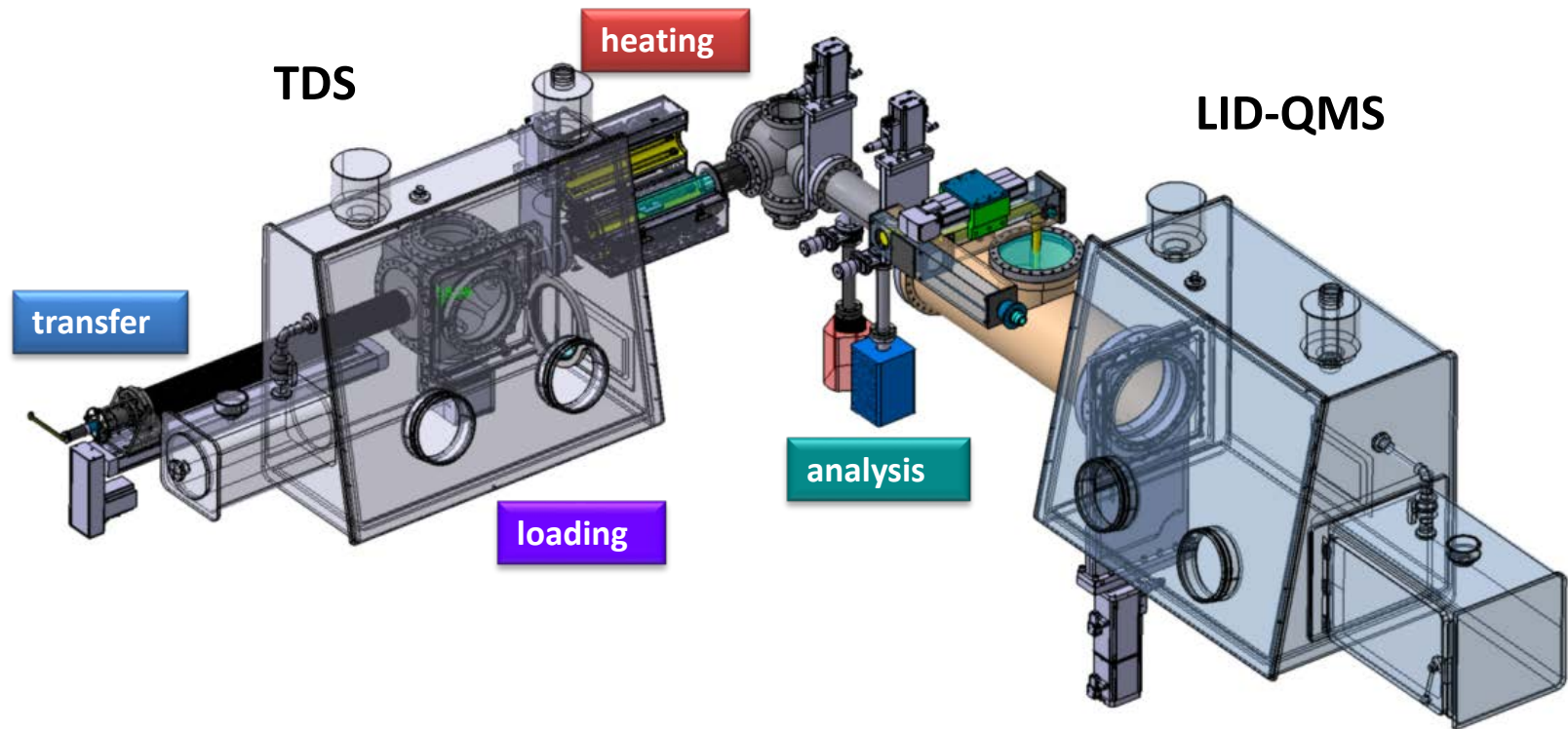


From Bröder 2015

# FREDIS



# FREDIS: Designed for JET-Tiles



- Combines classic TDS with laser-induced mass spectrometry (LID-QMS)
- Operation in hot materials lab => investigation of
  - Beryllium samples
  - Irradiated samples
  - Tritium-contaminated samples

# FREDIS: Fuel Retention Diagnostic Set-up



# Status & Outlook

## Done:

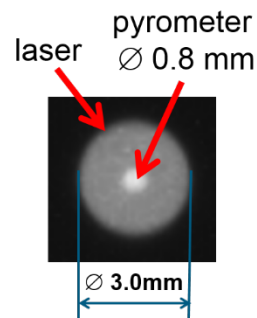
- Device tested outside HML
- First measurements on test samples were successful
- TDS: precise measurements up to 1400 K
- LID-QMS: Spatial resolved H release

## To do:

- Transfer to HML & re-commissioning
- Comparison of TDS and LID-QMS

# Test measurements using LID-QMS for large scale tiles outside of HML

JET CFC tile  $246 \times 142.5 \times 26 \text{ mm}^3$  loaded with deuterium by RF discharge in PADOS facility.

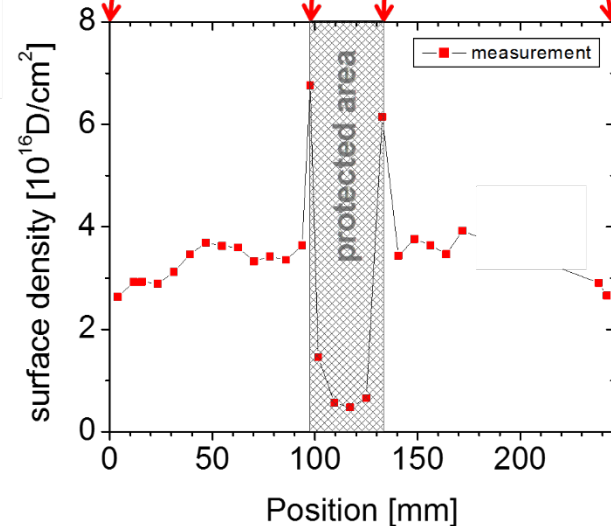
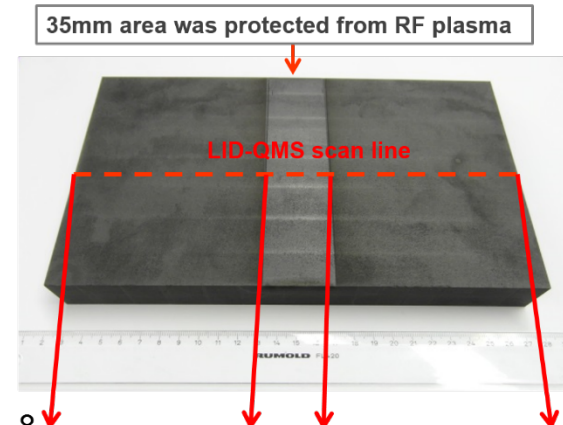
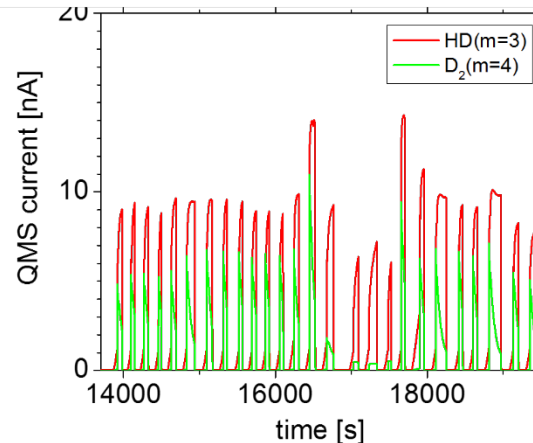
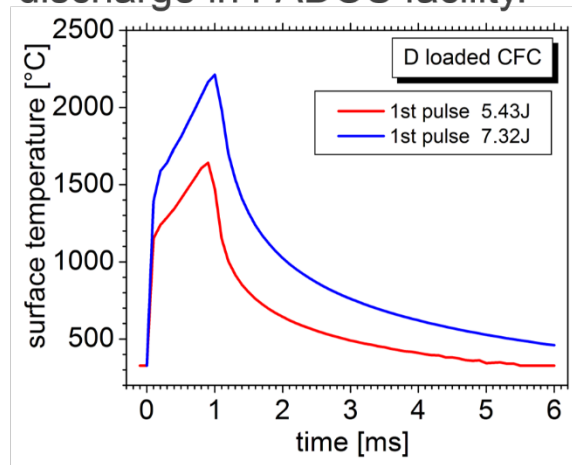


laser and pyrometer spots

$T_{\text{max}} \approx 2200^\circ\text{C}$



IR light from the laser spot (laser energy 7.3J)



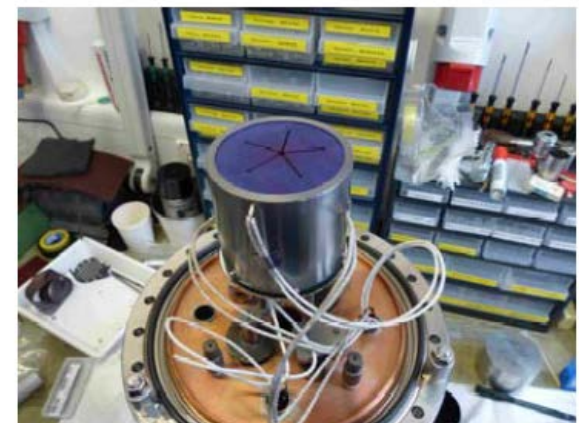
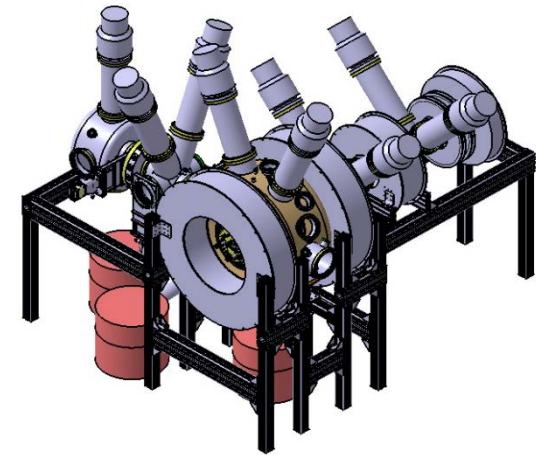
# JULE-PSI

# JULE-PSI – a new linear plasma device to investigate PSI processes with Be

Build to study erosion and fuel retention on Be and Be-based mixed materials

## Status:

- Detailed feasibility study and design review finalized Feb. 2016
- JULE-PSI is currently being assembled outside of the controlled area
- first plasma operation expected for fall 2016 with a new plasma source following PISCES-B concept
- relocation to HML end of 2017



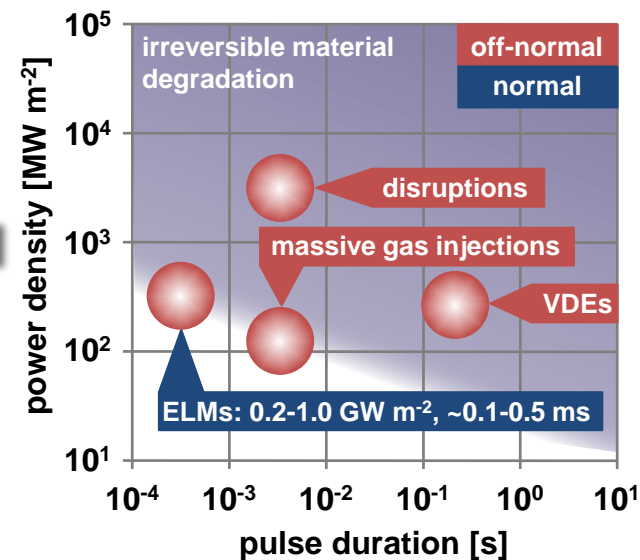
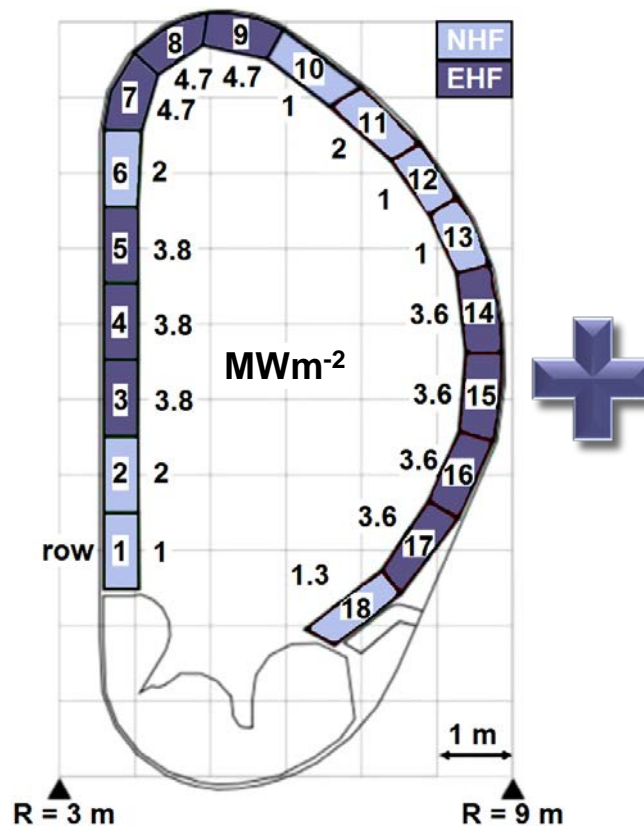
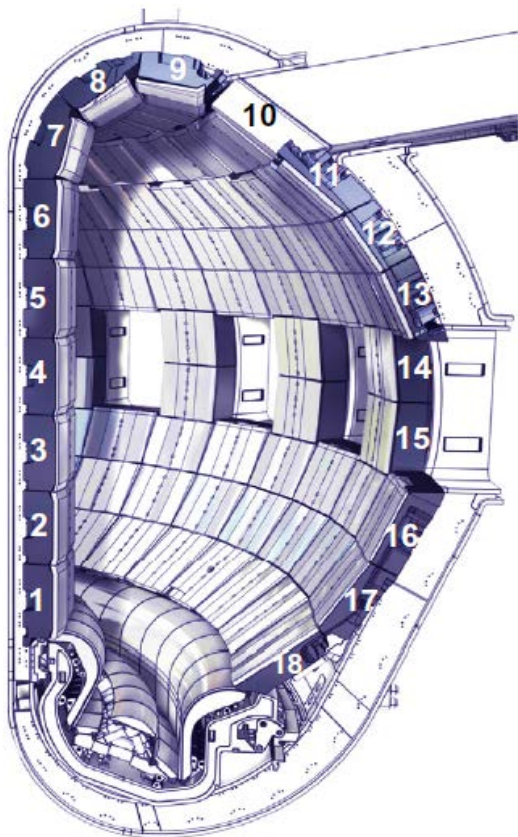
## JULE-PSI: Tech Specs

- Steady-state plasma exposure with plasma flux densities of  $10^{21} - 10^{23} \text{ m}^{-2}\text{s}^{-1}$  and power flux densities of 0.1-2 MW  $\text{m}^{-2}$
- Transient heat loads simulated by means of a Nd:YAG laser with pulse energies up to 100 J and pulse lengths 0.1-20 ms on top of the plasma load
- Measurement of fuel retention by LID-QMS, assessment of net erosion by weight loss measurements

# Judith 1 & 2

Behaviour of Be under ITER relevant  
cyclic heat fluxes





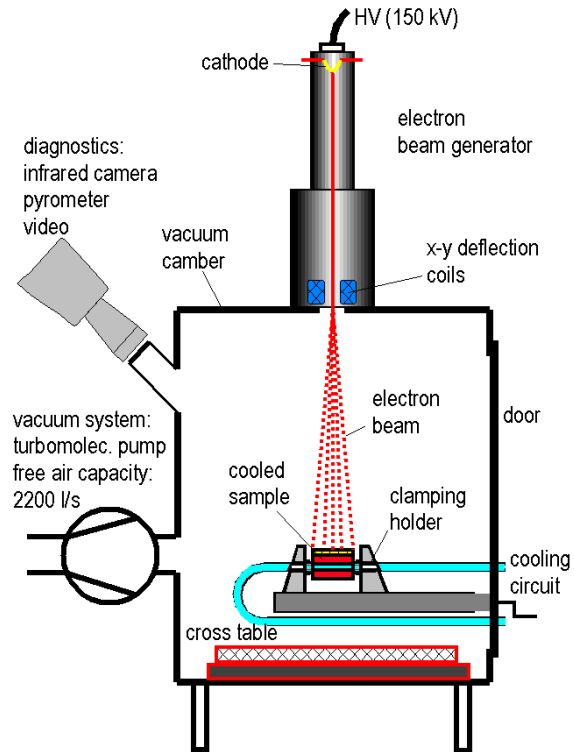
ITER first wall (FW):  
Be armour tiles

Expected maximum  
steady state power loads  
per row

Additional transient  
power loads (thermal  
shocks, TS)

M. Kočan et al., J. Nucl. Mater. (2015), <http://dx.doi.org/10.1016/j.jnucmat.2014.11.130>  
 A.R. Raffray et al., Nucl. Fusion 54 (2014), 033004 (18pp)  
 A. Loarte et al., Phys. Scr. T128 (2007), 222-228

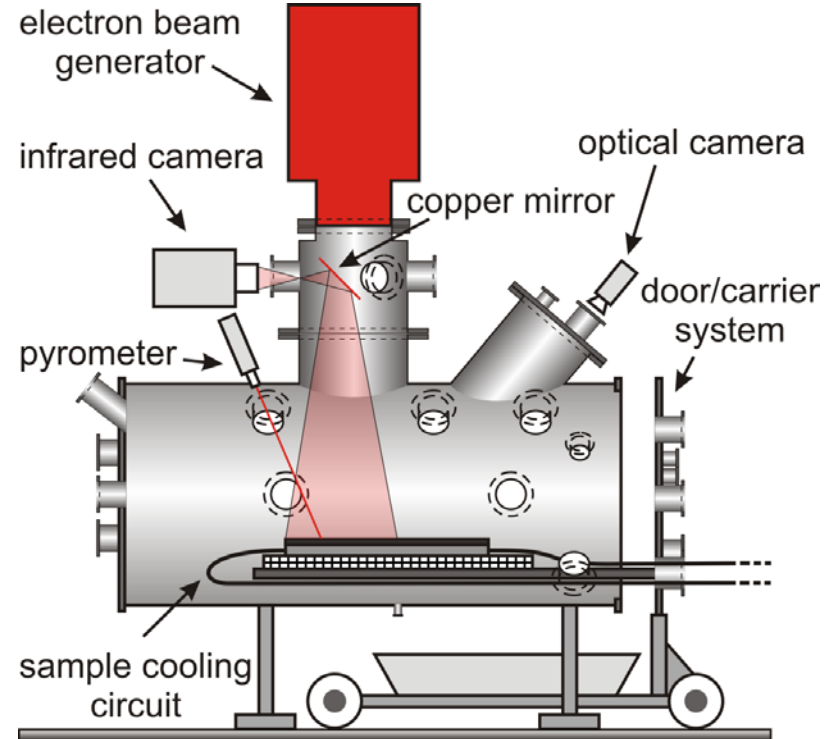
## JUDITH 1



### JUDITH 1 – machine parameters

Max. beam power:	60 kW
Acceleration voltage:	≤ 150 kV
Irradiation area:	4 mm <sup>2</sup> ... 100 cm <sup>2</sup>
Power density:	≤ 15 GW m <sup>-2</sup>
Pulse duration:	1 ms – cont.
Licensed for materials:	toxic; radioactive

## JUDITH 2



### JUDITH 2 – machine parameters

Max. beam power:	200 kW
Acceleration voltage:	40 – 60 kV
Irradiation area:	40 × 40 cm <sup>2</sup>
Power density:	≤ 2 GW m <sup>-2</sup>
Pulse length:	5 μs – cont.

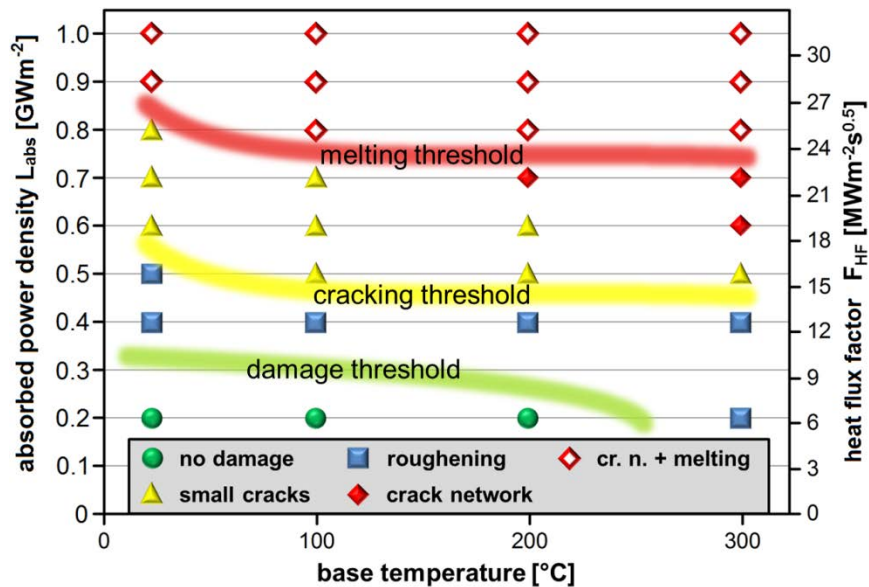
## Last CRP (19.05.2014)

- **Influence of the surface finish on the cracking behavior / thermal shock performance of beryllium (polished is best up to 1000 pulses)**  
[doi:10.1016/j.fusengdes.2015.10.028](https://doi.org/10.1016/j.fusengdes.2015.10.028)

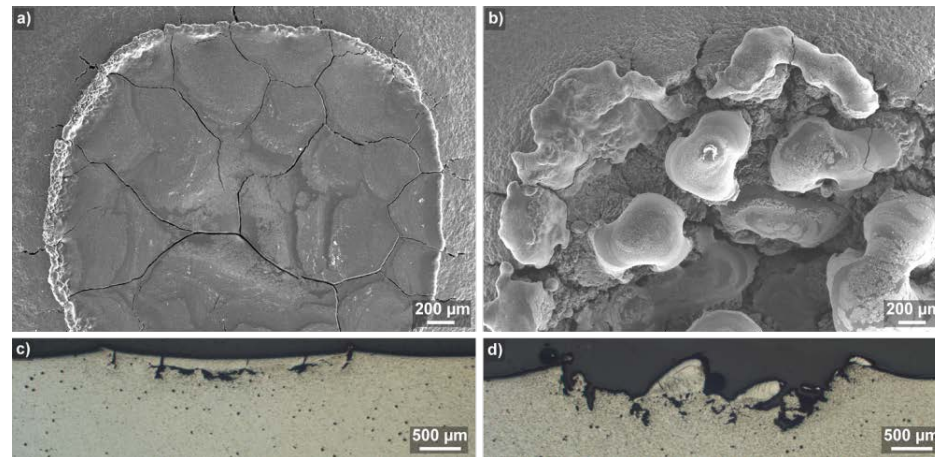
## Progress since then

1. **Damage mapping for transient heat loads**  
[doi:10.1088/0031-8949/T167/1/014024](https://doi.org/10.1088/0031-8949/T167/1/014024)
2. **Experimental simulation of massive gas injection induced heat loads**
3. **ITER/ELM relevant high pulse number experiments**
4. **Transient heat load induced oxide segregation**
- (5.) **Planned: Transients simulated by laser (important and valuable comparison to e-beam, answering various open questions)**

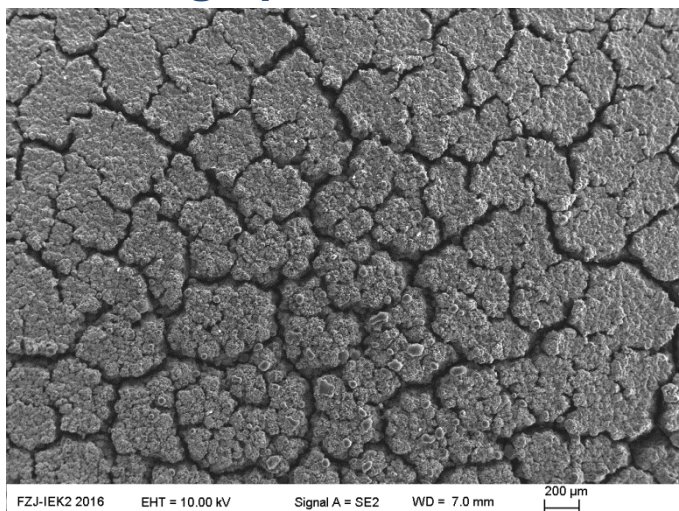
## 1. Damage mapping



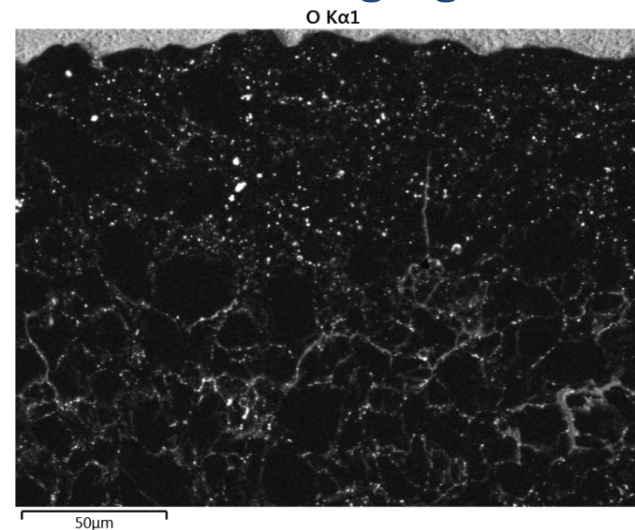
## 2. Massive gas injections



## 3. High pulse numbers



## 4. Oxide segregation



# Conclusion

## Conclusion

- JUDITH 1 & 2 work as expected
  - ARTOSS will start operation soon
  - XPS-Setup successfully finished first experiments
  - FREDIS & JULE-PSI are foreseen to start operation in hot materials lab in 2017
- ⇒ Most experimental setups are fully operational just now or will be operational soon
- ⇒ From my point of view, the Be-CRP should be prolonged to reap scientific results