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Deuterium retention, desorption and migration in plasma facing materials for fusion devices

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PhD thesis, MEPhI
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Outline

- Introduction
- Experimental methods
  - Thermal desorption spectroscopy
  - Ion-driven permeation
  - Ion beam analysis
- Bulk retention (W, Steels, C)
- Retention in codeposits and erosion products (C, W, Be, Li + JET)
- Summary

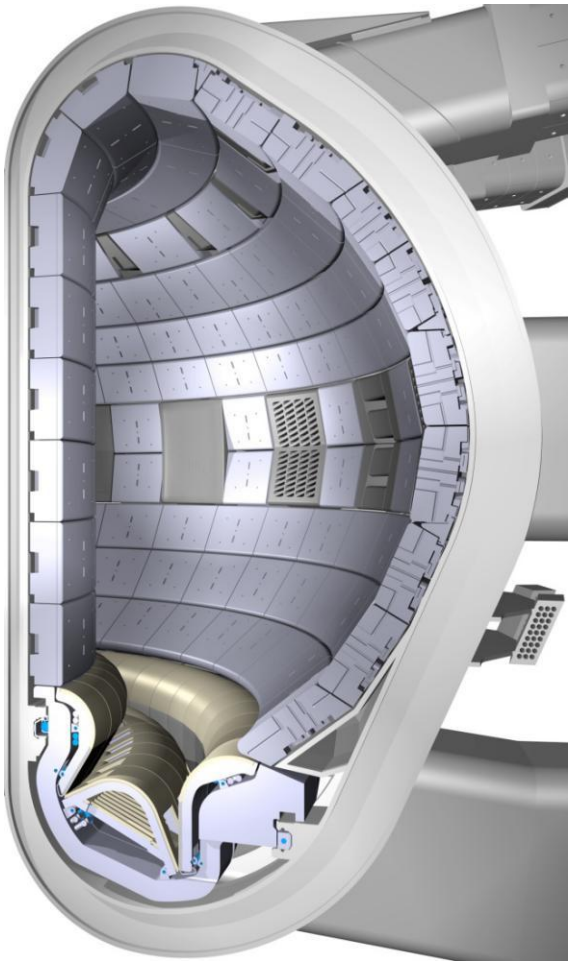
Introduction

Plasma Facing Components (PFC) for fusion devices should survive under extreme heat loads, have a reasonable erosion rate, be compatible with the plasma, and comply with safety requirements.

Investigation of hydrogen isotopes retention, desorption and migration in PFC is very important for the safety issues and for plasma operation.

Main PFC in tokamaks:

- W (high melting point, high sputtering threshold)
- C (low Z, no melting)
- Be (Low Z, good getter)
- Li (Low Z, good getter, renewable surface)



Experimental methods

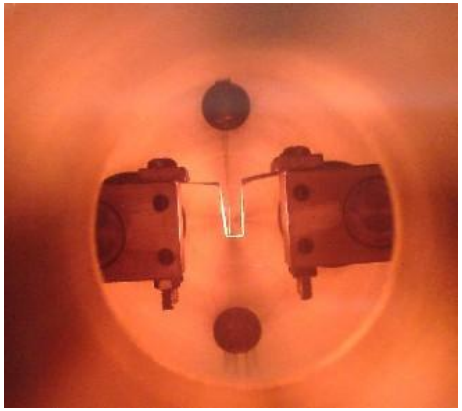
TDS

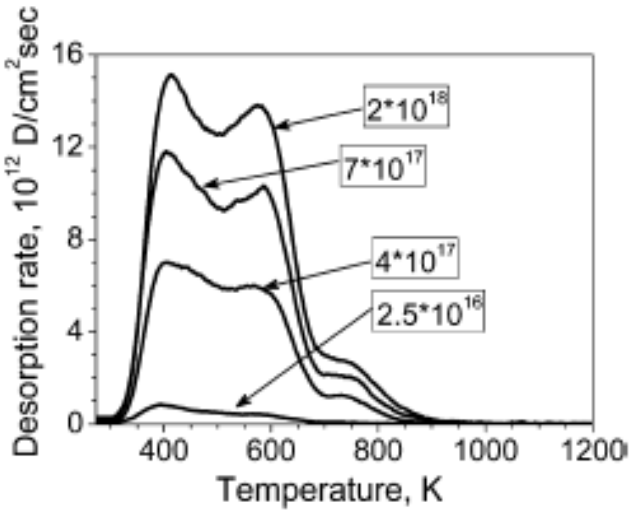
TDS gives the total D retention and characteristic temperatures of D release from material. However, parameters of D interaction with materials can be also derived.

Deuterium desorption from endothermic metals (Q_s<0) is often limited by D detrapping from various kinds of lattice defects. TDS spectra for endothermic metals usually consist of many peaks and one can get information about detrapping energies.

There are some special techniques to investigate parameters of defects and their evolution:

- Probe method
- Heating rate variation

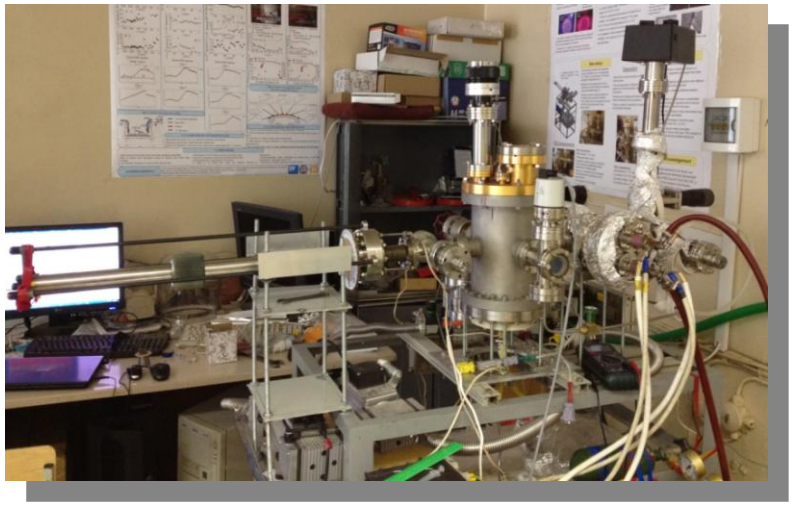




TDS facilities

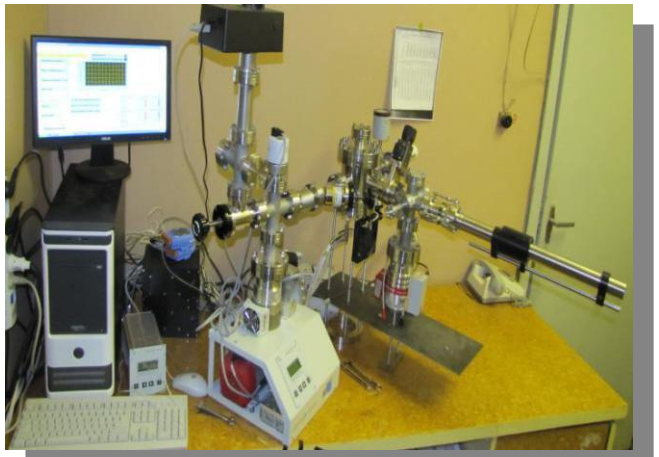
Medion (Ion Beam +TDS)

TDS stand MEPhI (for externally exposed samples)



MD-2 (Deposition + TDS)

TDS stand VNIINM (for Be samples)



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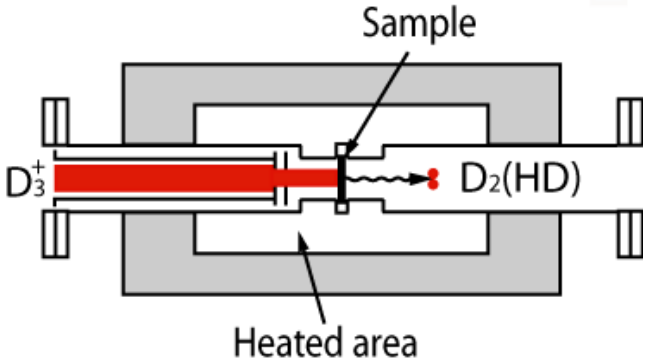
IDP & IBA

Ion-driven permeation

Dynamics of D permeation through materials was analyzed under ion beam irradiation



one can get characteristics of deuterium transport in the material, trapping efficiency of lattice defects and characteristics of surface

Ion beam analysis

RBS: surface composition analysis

NRA: D retention and profile in the top layer of materials

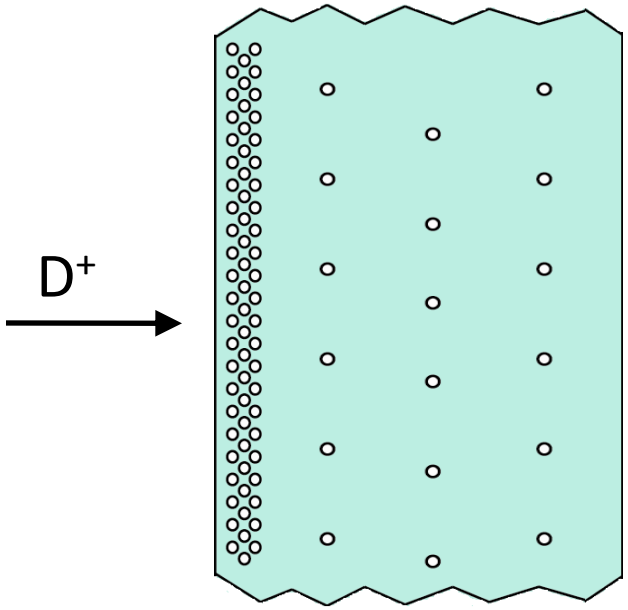
$D(^3He,p)^4He \Rightarrow D$

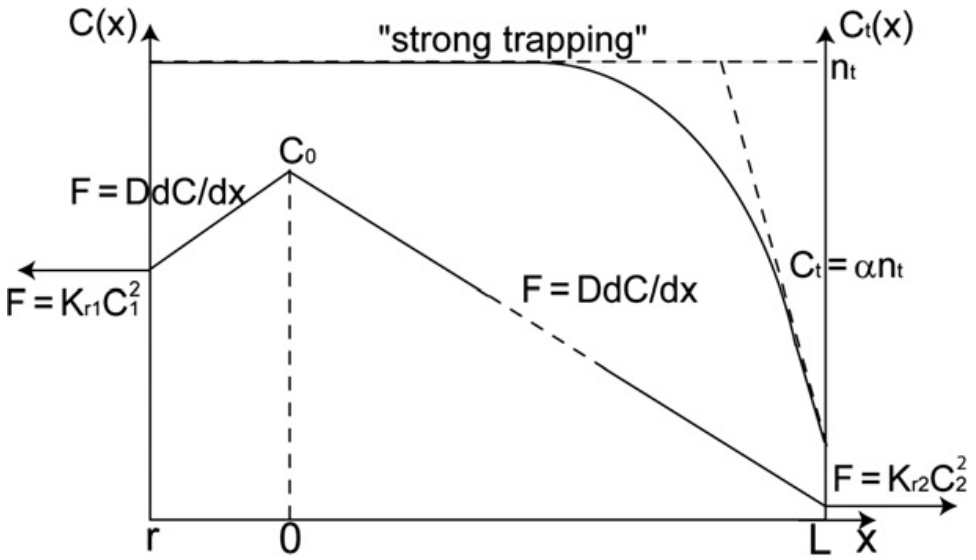
$^{12}C\ (^3He,\ p\ )^{14}N \Rightarrow C$

$^6Li(^3He,p)^8Be \Rightarrow Li$

Max Planck Institute for Plasma Physics (Garching, Germany)

Bulk retention





100 m²×10 μm×10 at.% = 30 g

100 m²×1 cm×0.1 at.% = 300 g

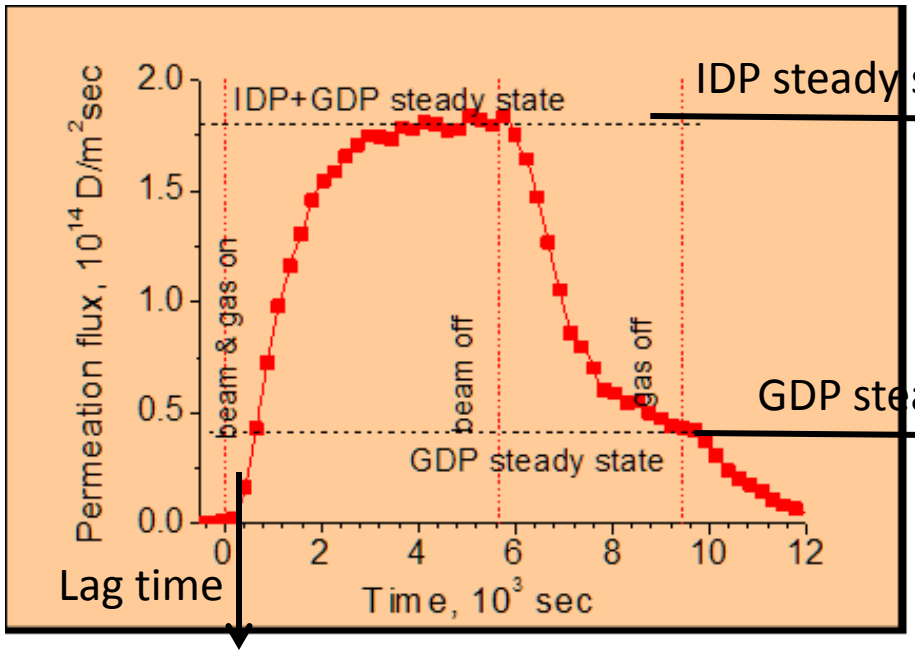
Most of PFC candidate materials are endothermic (Qs>0), however **neutron radiation damage can increase significantly H retention**

The population and the filling rate of radiation defects depends on :

temperature, binding energies and concentrations of traps, diffusivity, surface conditions, and the incident ions flux and energy.

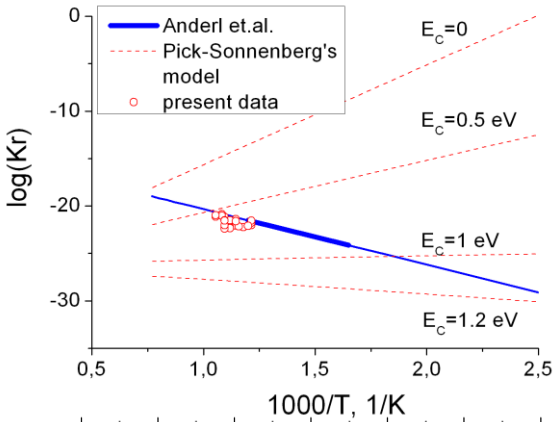
Plasma physics department

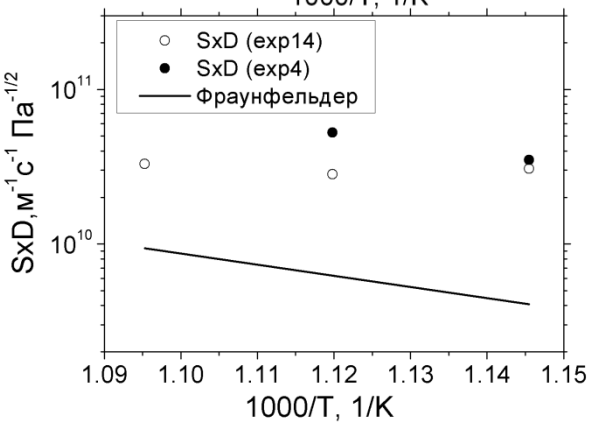
W: IDP experiments

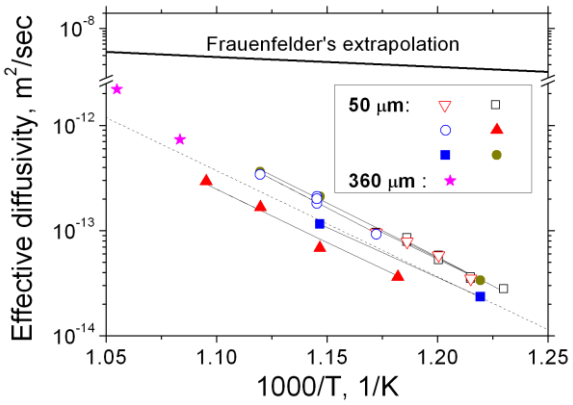


IDP steady state

GDP steady state





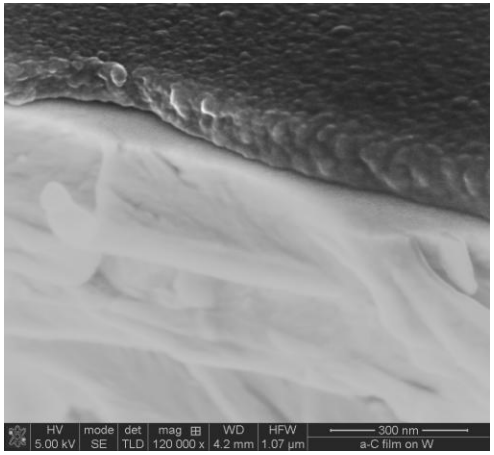


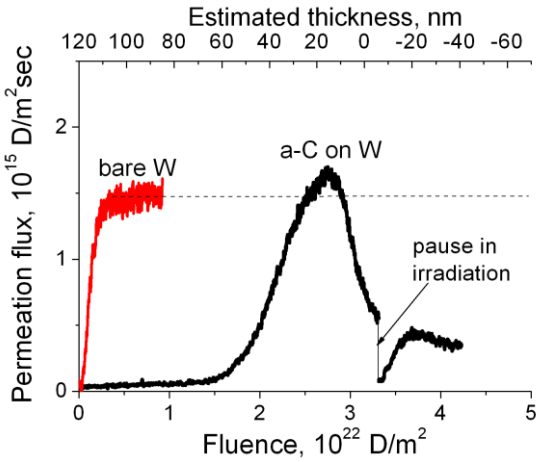
Recombination coefficient, permeability, and detrapping energy for defects has been determined from IDP&GDP experiments. E_{dt} = (2.05±0.15) eV

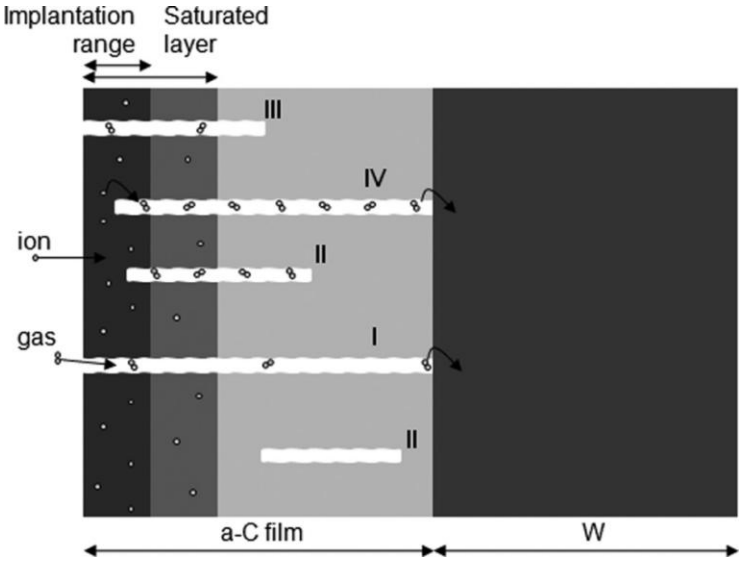
IDP: C-coated W

Influence of the surface conditions on bulk retention has been observed in IDP and TDS experiments.

IDP though C-coated W

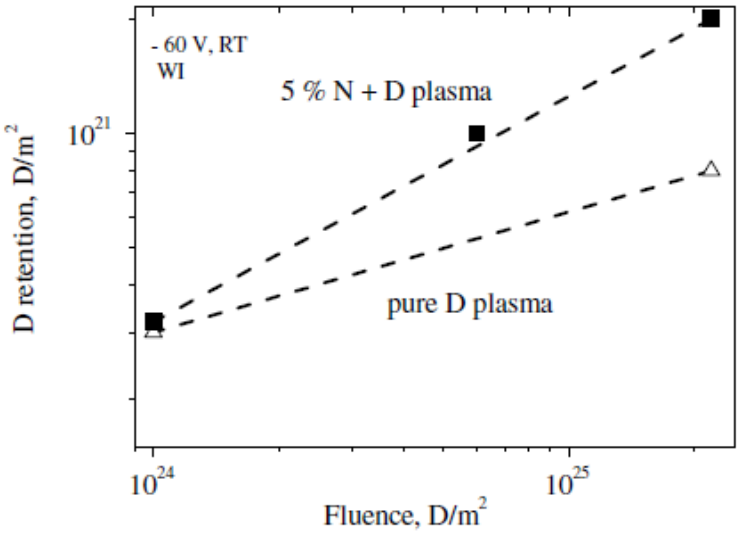


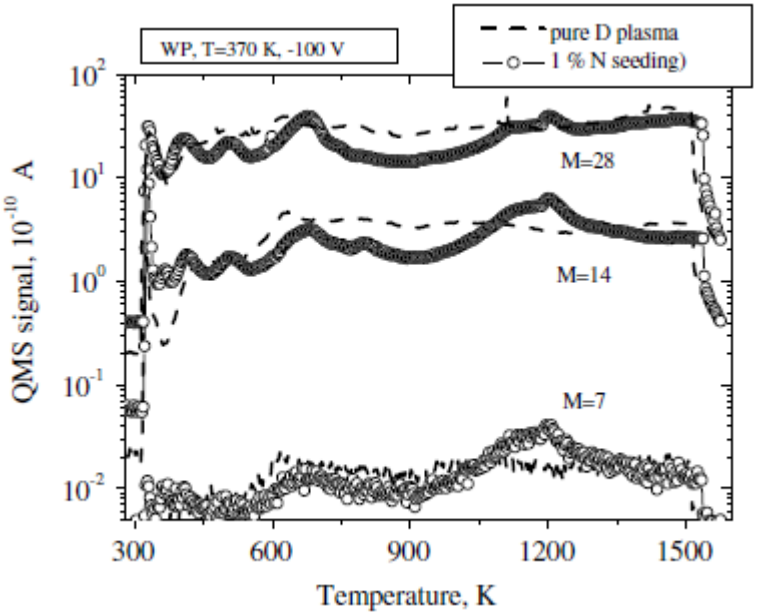




- Thin C film completely changed the permeation flux and bulk retention.
- The increased lag time was observed, but only in first irradiation.
- The steady state permeation flux was much higher than for pure W in some conditions. Presence of system of cracks was suggested.

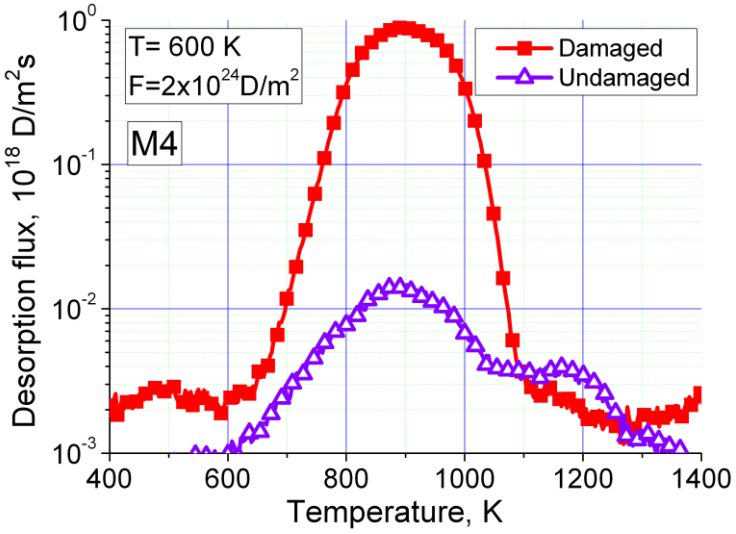
N seeding

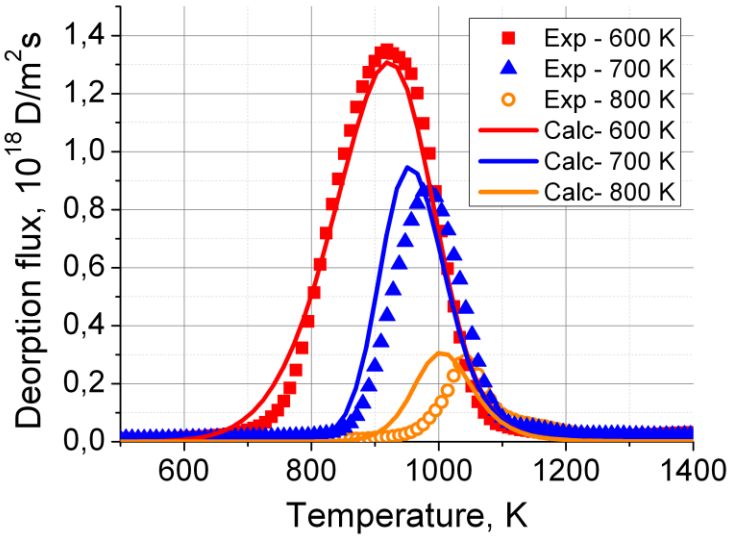




- Seeding of D plasma by several percent of nitrogen led to increase in D retention by 2-3 times at the fluence of 2×10^{25} D/m^2
- The reason is formation the nitrogen-enriched at the surface
- It is stable up to 1100–1200 K, according to TDS data

W: damage by 20 MeV

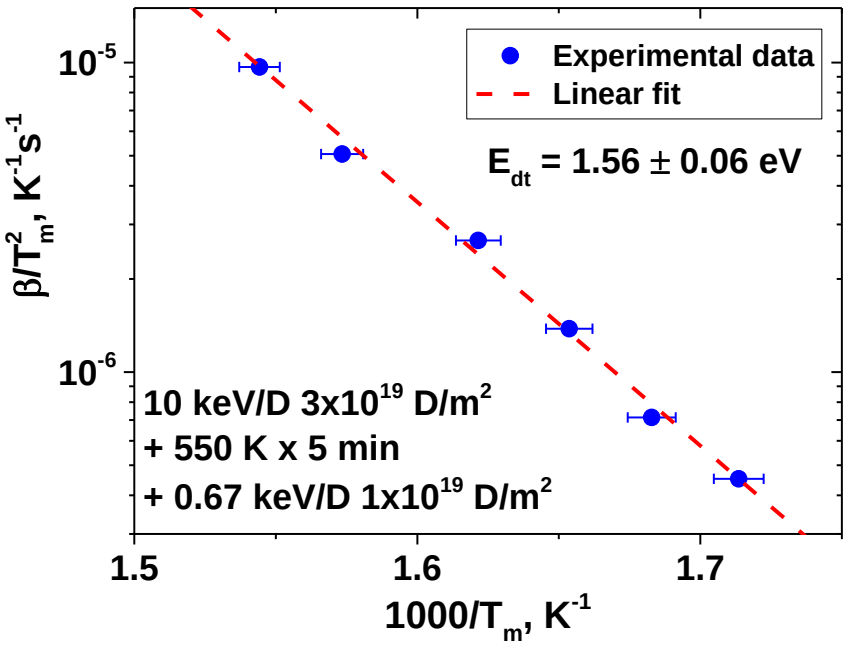




- A good agreement with the experiment was achieved assuming the traps with the detrapping energy of 1.7-2.0 eV.
- Similar values have been derived from ion-driven permeation experiments. These traps are assumed to be the sites at the surface of inner voids or vacancy clusters.

W: Heating rate variation

Detrapping energy can be determined from TDS with different heating rates β

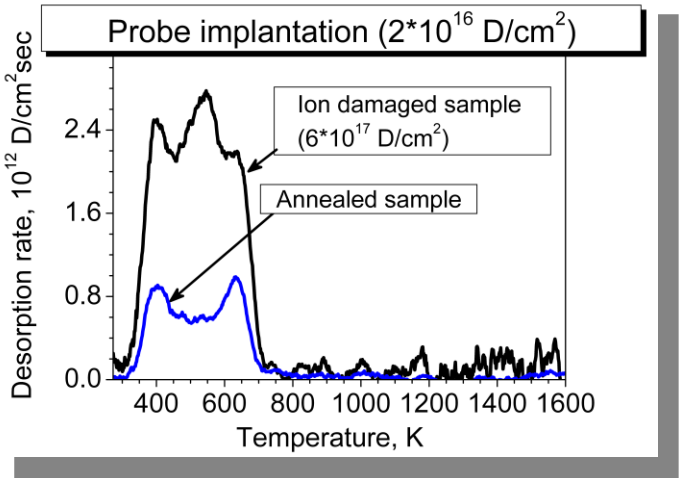


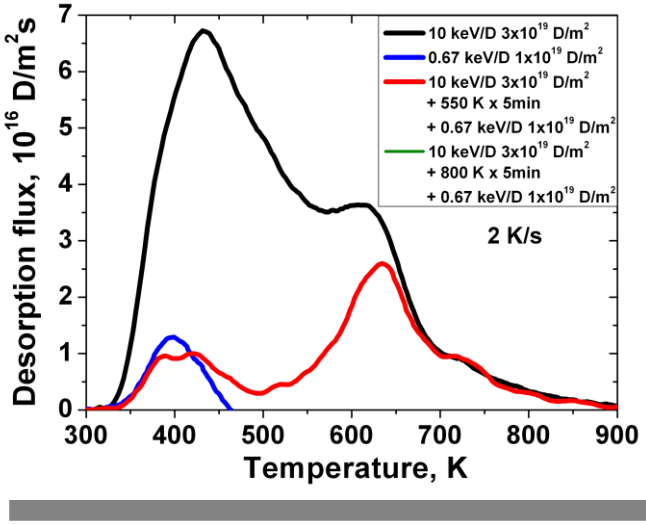
This model was initially proposed for adsorbed species on the surface. In the case of the high recombination rate it can applied for the bulk.

Depending on trap concentration, the detrapping energy E_{dt} or the sum (E_d+E_b) can be determined from the slope.

The detrapping energy for the vacancies in W was determined by this method to be E_{dt} = (1.55±0.6) eV.

W: Probe method





Main idea: Damaged sample is implanted by "probe fluence" that is enough to fill traps by D, but doesn't create additional traps.

Probe TDS reflects types and concentrations of defects

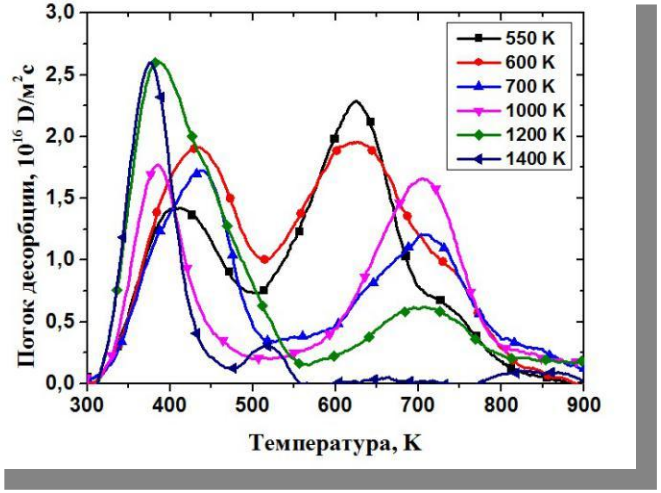
Probe fluence : 2×10^{16} D/cm^2 10 keV/D_3^+ ,
1×10^{15} D/cm^2 2 keV/D_3^+

H atomic implantation is planned in future experiments as a probe implantation

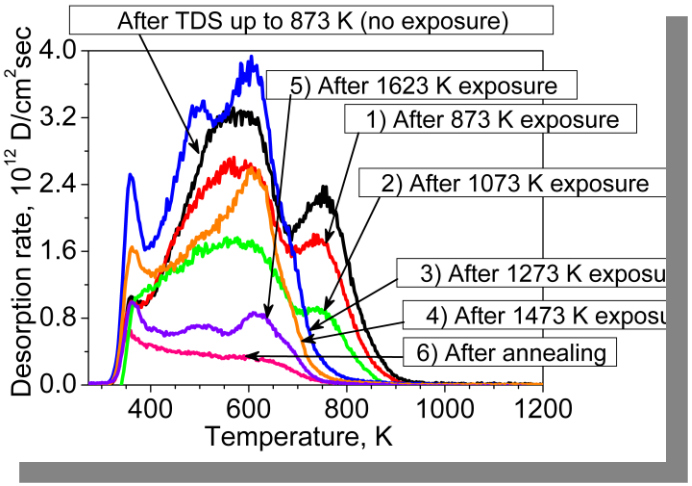
Plasma physics department

W:Damage by keV D ions

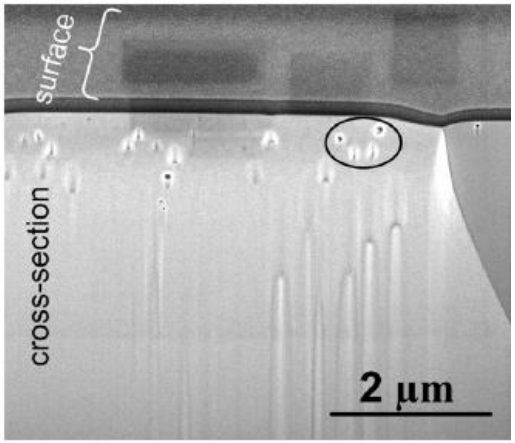
Damaged by 3×10^{15} D/cm^2 , 10 keV D$^+$



Damaged by 6×10^{17} D/cm^2,10 keV D_3^+



- At 600-700 K vacancies are actively agglomerate to complex defects. Further increase of the temperature leads to increase of the clusters.
- At high temperatures above 1400 K, annealing of these vacancy clusters is possible.
- In the case of the high concentration of defects, large pores can be formed which are stable, at least up to 1800 K

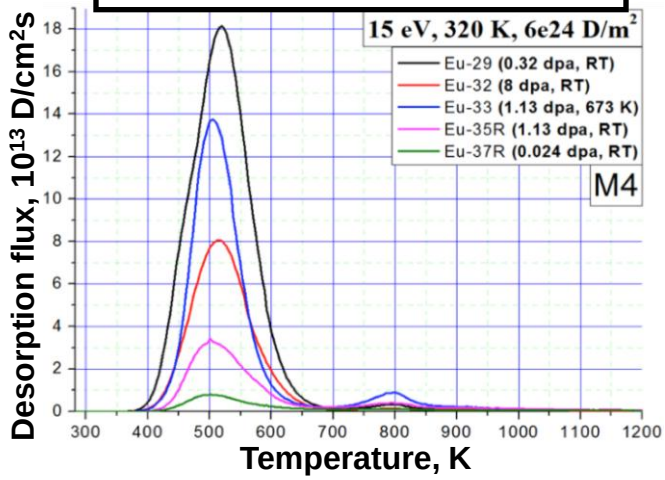


Plasma physics department

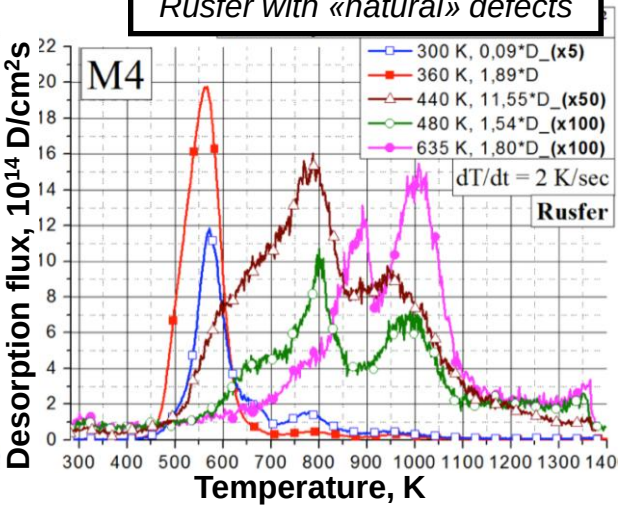
Bulk retention: Steels

- Generally, D retention in stainless steels is rather small. Much lower binding energies than in W
- Strong effect of experimental procedure on H behavior has been observed
- TDS spectra have one major peak at 500-550 K
- This peak was associated with the surface barrier that should be chromium oxide.

Eurofer damaged by 20 MeV W

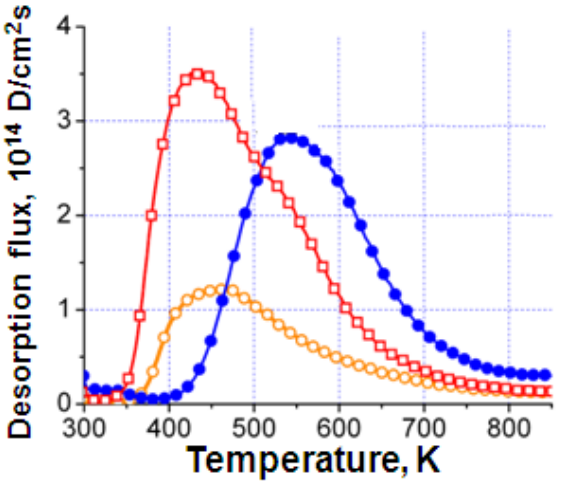


Rusfer with «natural» defects



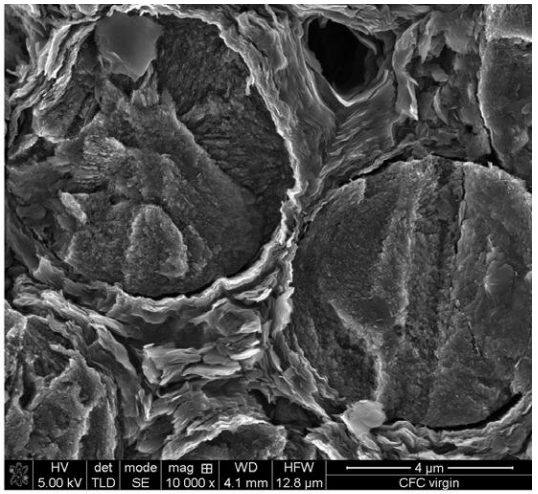
MEDION experiments: with in sity TDS

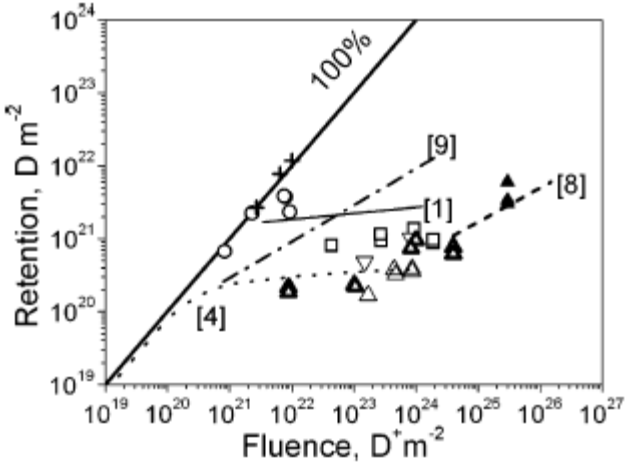
Three different procedures:
Implantation and TDS after 0.5h
Implantation and TDS after 15 h
Implantation, air exposure for 0.5h, and TDS after 15 h

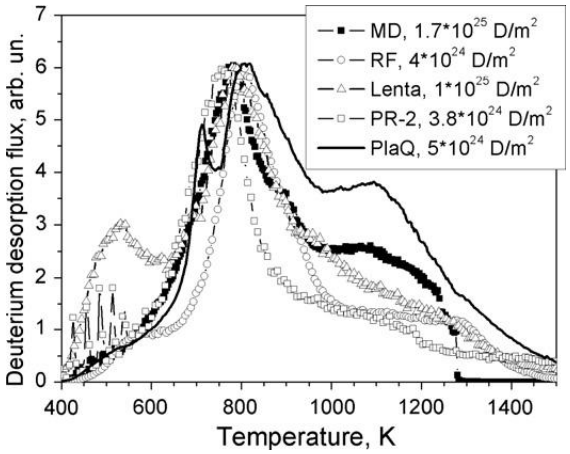


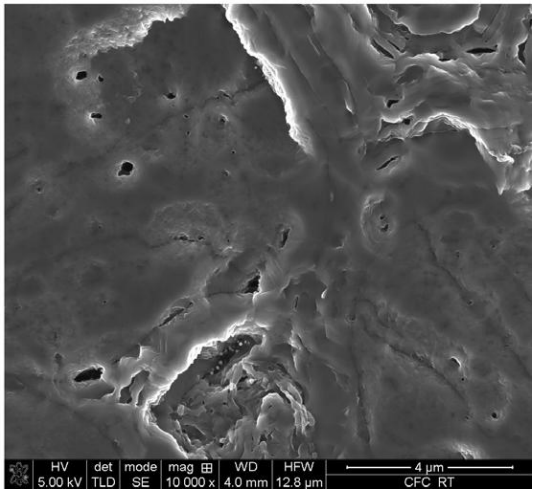
Bulk retention: C

- H diffusivity is very small; however, most of C materials are very porous and H can migrate deeply through the open porosity of the material, where D can be trapped on dangling bounds.
- In addition, an amorphous layer with the high D concentration is formed at the surface.
- A significant part of deuterium stays in C materials up to very high temperature, above 1000 K.







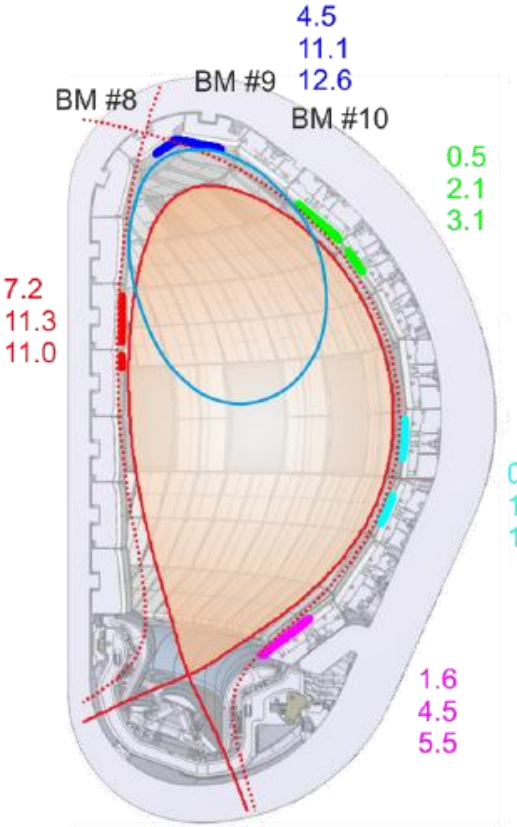


Codeposits & Erosion products

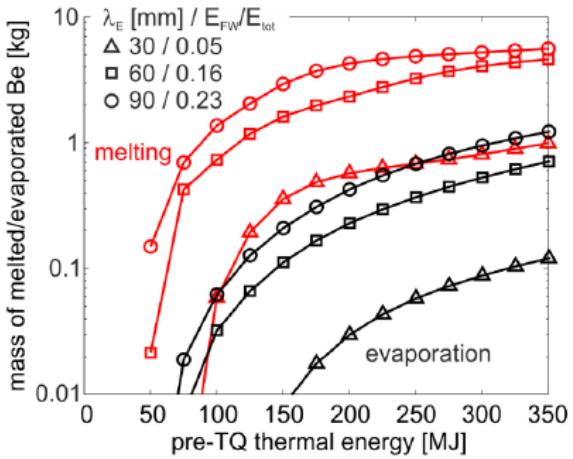
Erosion of PFC and further co-deposition with H isotopes is a serious part of hydrogen inventory in tokamaks.

D concentration in codeposits is usually very high. For RT deposition, it is at the level of tens at.% for the most of materials.

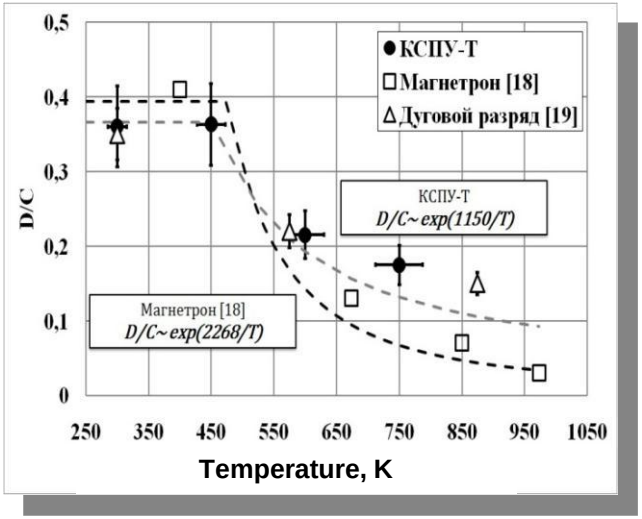
The erosion rate depends strongly on material and the energy and the flux of incident particles.

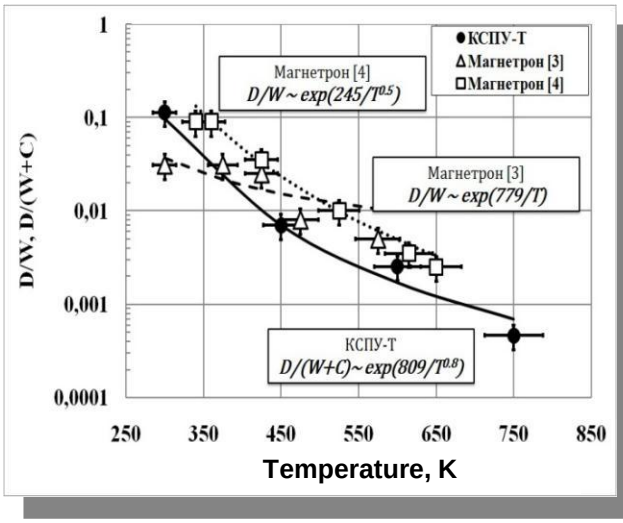


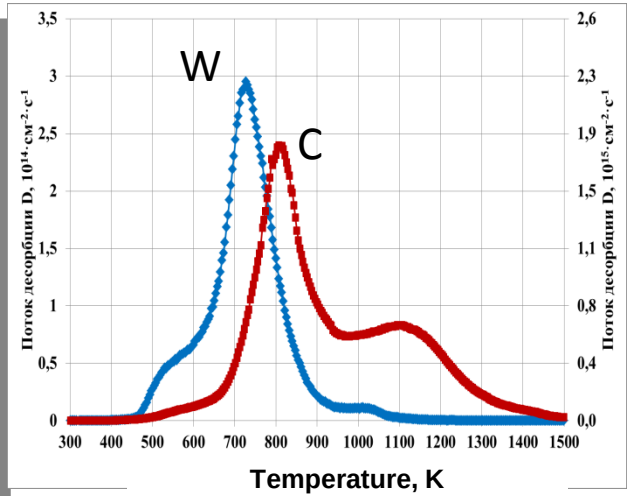
During ELMs and especially abnormal events like (MD and VDE), macroscopic particles can be ejected from the surface. This mechanism can be a large source of Be dust. Unmitigated VDE and MD in ITER can produce up to several kg of Be per one event.



Codeposits: C&W



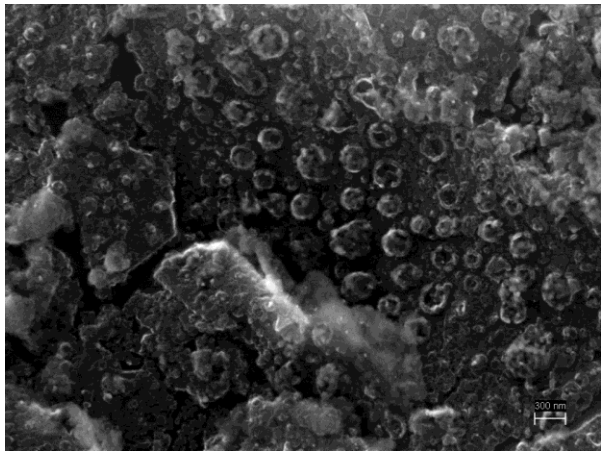


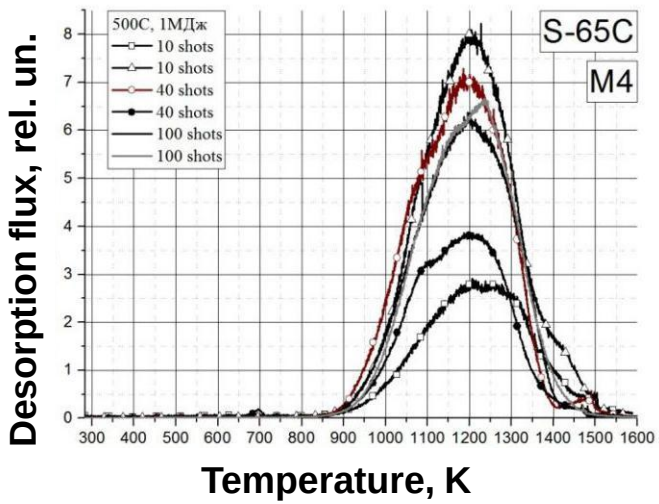


- Codeposits formed during operation with W and C targets at **MD** (stationary) and **QSPA** (impulse, high flux) have been investigated
- W-D films had a significant amount of C impurities in both cases.
- All films deposited at room temperatures had a comparable deuterium concentration (10-40 at.%)
- D retention in W-D films drops strongly with temperature

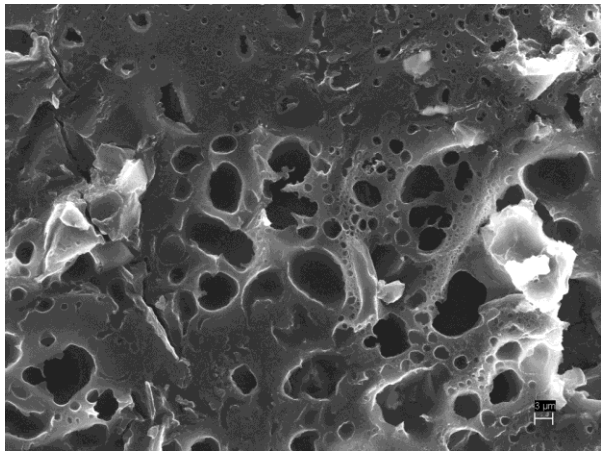
Erosion products: Be

- TDS stand has been built at VNIINM for analysis of Be samples exposed at QSPA-Be
- Beryllium tiles were irradiated by 10-100 pulses of D plasma (1 MJ/m^2, 0.5 ms, T_{base} =250 C, 500 C).
- Droplets were collected from the surface for TDS
- High temperature release of deuterium has been observed 900-1400 K).
- $N_D/N_{Be}=3\times10^{-6}–10^{-4}$.

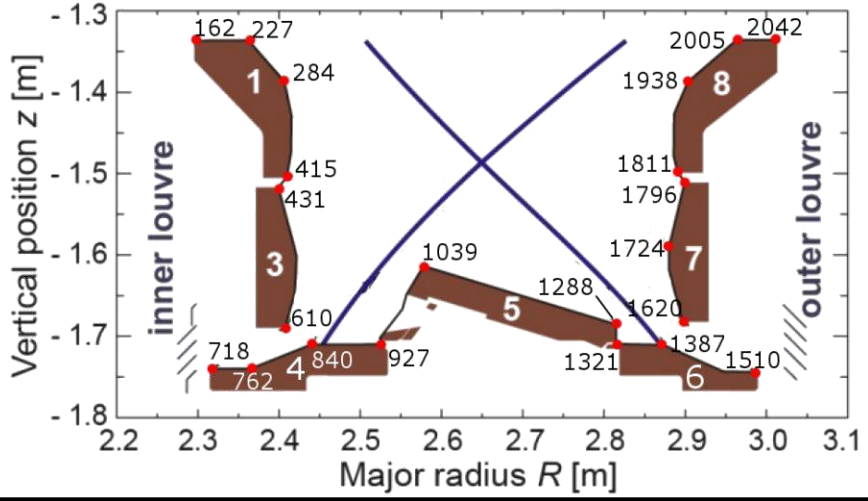


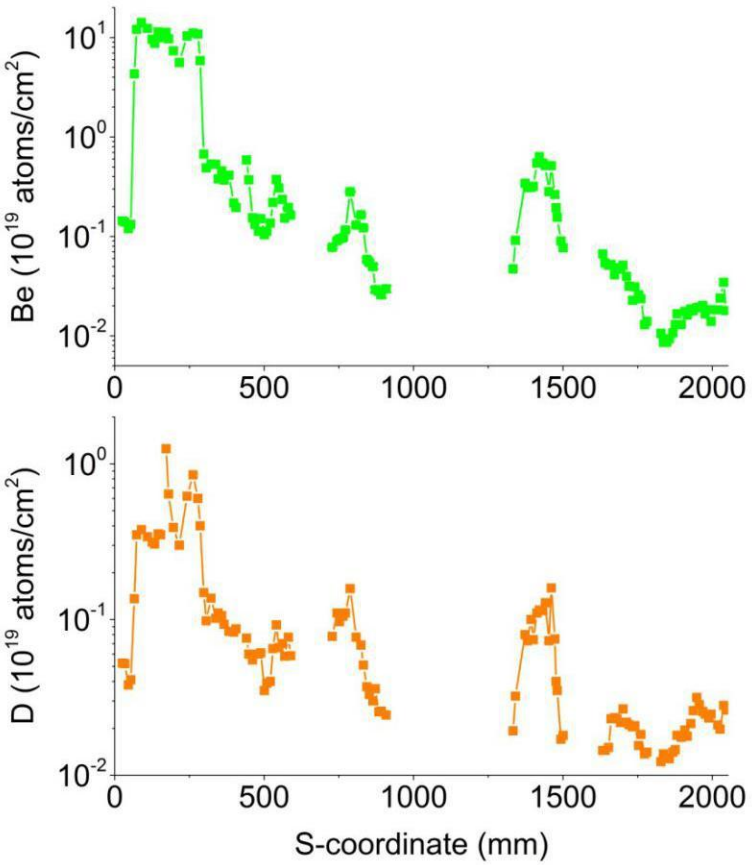


D retention depends on Be grade, it was higher in S-65C than in TGP-56FW by three times in average.



Codeposits: JET



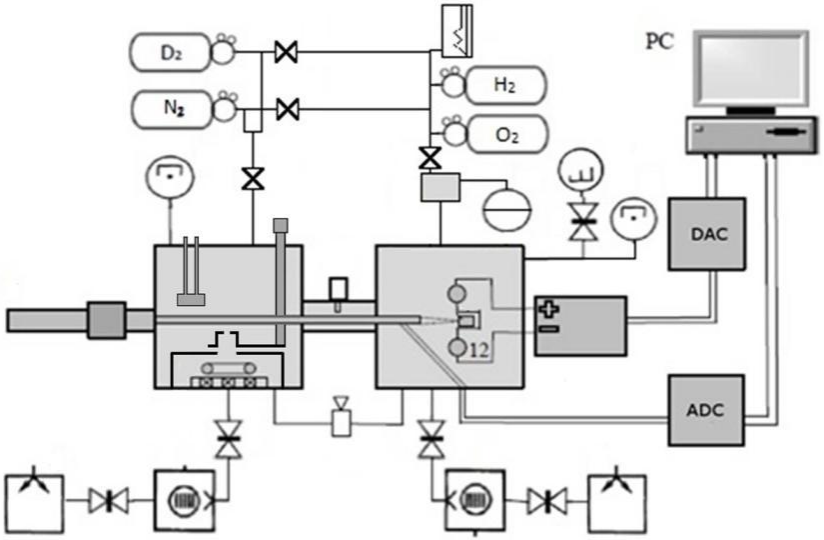


Tiles from JET (UK) were investigated by RBS and NRA in IPP (Garching). Two campaigns, full C machine (2005-2009) and with ITER-like wall (2011-2012) have been compared.

The amount of codeposits and D inventory in them was drastically decreased for ITER-like wall. Chemical erosion of C by low energy particles is the most probable explanation for the comparatively lower Be erosion rate.

Plasma physics department

Codeposits: Li



- Li is a perspective material for future fusion devices
- Li is very chemically active

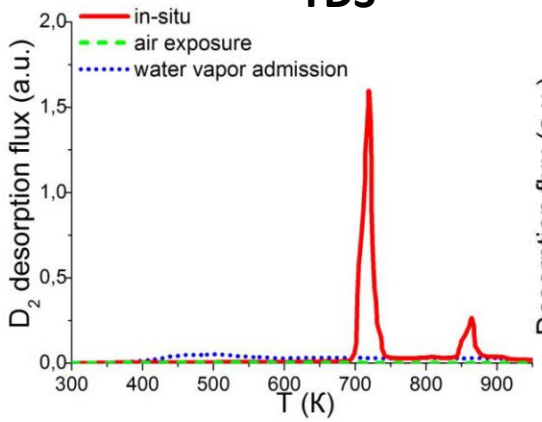
  ⇒ **In-situ analysis** are necessary

Main observations:

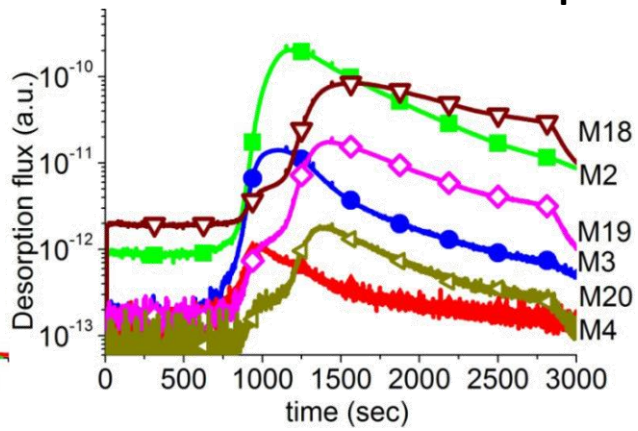
- D/Li ~ 20% in codeposits
- D release in a sharp peak at ~700 K and, sometimes, additional peak at ~850 K
- Intensive reaction with air and water vapor

Water vapor exposure can be recommended for T removal

TDS



Interaction with water vapor



Main results

- Four different TDS installations for invesion of deuteirum retention by means of thermal desorption spectroscopy
- Detrapping energies for deuterium release from radiation damage has been derived from TDS and IDP experiments
- Experimental data on influence of the surface layer on D bulk retention. The model desribing deuterium permeation through the C-coated W
- Experimental data demonstrating the role of the surface on deuterium retention in steels
- Experimental data on deuterium codeposition with varios PFC materials
- Experimental data on deuterium retention and thermal desorption in erosion products of Be, demostrating high energy trapping sites
- Method of hydrogen istopes removal from lithium. Experimental data on low temperature D release during interaction with air and water vapor.