Max-Planck-Institut für Plasmaphysik

IAEA CRP "Beryllium", Vienna 2014

Beryllium-related PSI-experiments in IPP Garching

Wolfgang Jacob

Plasma Edge and Wall Division

Introduction

For a long time IPP was engaged in Plasma-Surface-Interaction laboratory experiments using Beryllium and Be-containing materials.

Much of these laboratory studies were moved from IPP to FZ Jülich in the course of the appointment of Prof. Ch. Linsmeier as director of the PSI department in FZJ (see presentation of Martin Koeppen).

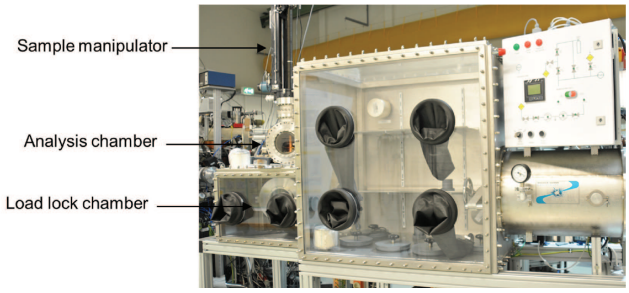
This presentation will summarize the remaining activities in IPP Garching.

Introduction

- Ion-beam analysis station for the handling of Be-containing samples (predominantly for handling of JET tiles)
- XPS depth profiling (very restricted use for Be-containing samples)
- High current ion source: Exposure of Be-containing samples had to be stopped (mid 2013). Some concluding work is about to be finished (data analysis still ongoing)
- Thermal desorption spectroscopy (TDS) (very restricted use for Be-containing samples)

All these activities are possible only for experiments where no significant Be amounts are mobilized.

Ion-beam Analysis



View of the SAK device, showing the beryllium-compatible glove box, the load lock chamber, the analysis chamber, and the sample manipulator.

Ion-beam Analysis

Example:

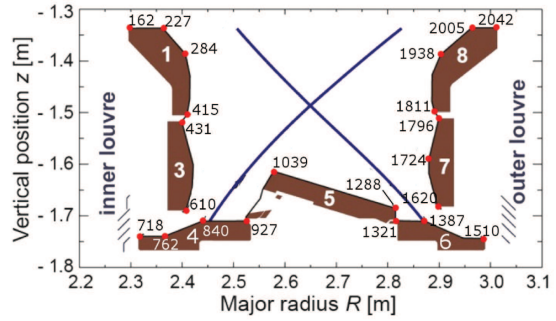
Erosion and deposition analysis of JET divertor during 2011-2012 campaign

S. Krat, I. Bykov, W. Van Renterghem, M. Mayer, A. Widdowson, JET-EFDA contributors

- Work in the frame work of the Fusion Technology Task Force at JET (JW13-FT-4.30, Analysis of marker samples after ILW operation)
- Sachet samples and core samples from divertor tile 1, 3, 4, 6, 7, 8 coated with marker stripes were analysed after exposure during the campaign 2011-2012 as well as deposition monitors from remote areas. All analyses were performed using ion beam analysis methods.

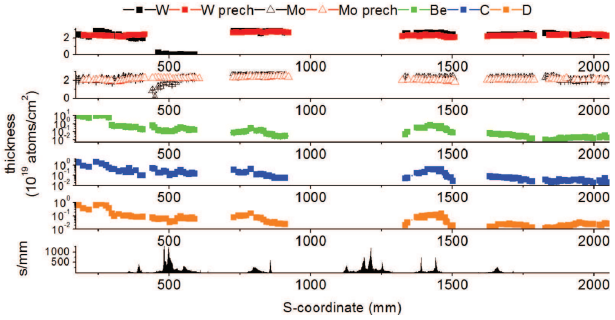
Stepan Krat, Yu Gasparyan, A Pisarev, I Bykov, M Mayer, G de Saint Aubin, M Balden, C Lungu, A Widdowson, JET-EFDA contributors, "Erosion at the inner wall of JET during the discharge campaign 2011-2012 in comparison to previous campaigns" , submitted to Journal of Nuclear Materials

Ion-beam Analysis



The JET divertor during the 2011-2012 discharge campaign. Tile numbers (1, 3, 4, 5, 6, 7, 8) are indicated. Small numbers indicate the s-coordinate system, in mm.

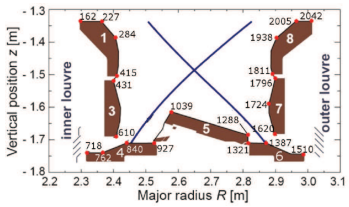
Ion-beam Analysis



Thicknesses of the W and Mo marker layers before and after exposure during the 2011-2012 campaign, and deposition of Be, C and D. Black line: Integrated strike point position during the 2011-2012 campaign.

Ion-beam Analysis

- The W marker layer shows no noticeable erosion in the outer divertor.
- Some erosion can be observed on the lower part of tile 1 and strong erosion of Mo can be observed on the upper part of tile 3.
- Large amounts of Be (up to 10^{20} Be-atoms/cm^2) are redeposited on the horizontal part of tile 1, together with some W.
- Some amounts of carbon are observed on all areas, with the largest amount on the horizontal part of tile 1.



Manuscript submitted to J. Nucl. Mater.

XPS
X-ray Photoelectron Spectroscopy

No detailed example

Some collaboration with PISCES (see presentation by R. Doerner)

- Sputter depth profiling
  (few keV Ar ion sputtering combined with XPS surface analysis)

High-current ion source (HSQ) and TDS

Work performed in the framework of an F4E contract:

F4E-OPE-347: "Deuterium retention and outgassing experiment"

- Contract ended in Summer 2012
- First results published in PSI proceedings 2012
- Some additional implantation experiments were carried out until summer 2013
- Further TDS analysis is still ongoing
- Data analysis ongoing

K. Sugiyama, C. Porosnicu, W. Jacob, J. Roth, Th. Dürbeck, I. Jepu, and C.P. Lungu: "Study of Deuterium Retention in/Release from ITER-relevant Be-containing Mixed Material Layers Implanted at Elevated Temperatures ", Journal of Nuclear Materials, 438, S1113–S1116 (2013). doi: 10.1016/j.jnucmat.2013.01.245

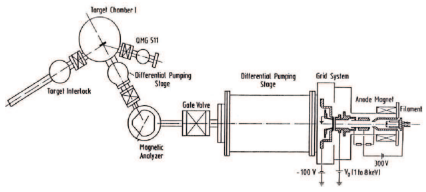
Experimental strategy

- ✓ Samples to be investigated: Be, Be-W, Be-C and Be-O mixed layers prepared by Thermionic Vacuum Arc (TVA) deposition method at MEdC (Bucharest)
- ✓ D implantations and subsequent outgassing experiments at IPP
- ✓ Ion beam analysis before and after TDS
  - Layer composition and thickness are quantitatively determined by Rutherford Backscattering Spectroscopy (RBS) using 2.0 MeV $^4He^+$
  - Deuterium retention in the layer is analyzed by Nuclear Reaction Analysis (NRA) using 800 keV $^3He^+$ ($D(^3He, p)^4He$ reaction)
- ✓ Quantitative analysis of TDS

Layer thickness and compositions determined by RBS

Layer type (planned concentration of admixed element)	Layer Thickness estimated by RBS	Concentration of admixed element(s) in Be layer determined by RBS
Pure Be	570 nm	(O impurity ≤ 1 at.%)
Be-W (W: ~ 5 at.%)	360 nm	W: 6 ±2 at.% (O: ~10 at.%)
Be-C (C: ~10 at%)	350 nm	C: 13 ±1 at.% (O: ~8 at.%)
Be-C (C: ~ 50 at%)	380 nm	C: 50 ±1 at.% (O: ~9 at.%)
Be-O (O: ~ 10 at%)	340 nm	O: ~ 6±1 at.%

D implantation in the High Current Ion Source



Schematic view of High Current Ion Source at IPP-Garching

Loading condition:

- ✓ D energy: 600 eV D_3^+ (= 200 eV / D)
- ✓ D flux / fluence: ~ 10^{19} D/m²s / 1.0 x 10^{23} D/m² (this is well above the saturation fluence)
- ✓ Sample can be heated by a filament heater behind the sample
  (Implantation temperatures: 398 K, 523 K and 623 K (125ºC, 250ºC and 350ºC))

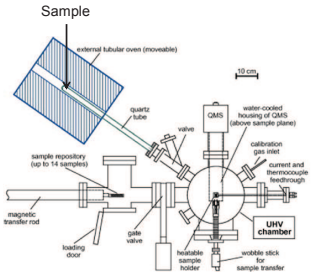
Thermal desorption spectroscopy (TDS) at the TESS facility

Normal TDS:

- ✓ "Normal" TDS provides information about D trapping and the corresponding release temperatures.
- ✓ Sample was heated up to 1000 K with the ramp rate of 0.25 K/s.

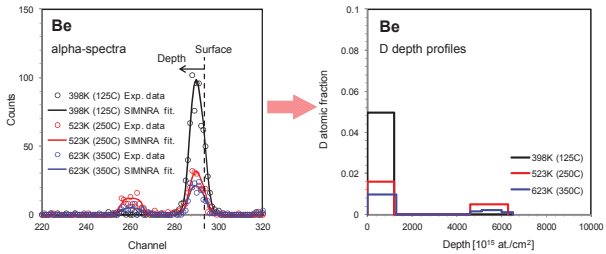
Outgassing by long-term annealing

- ✓ Sample was heated up to 513 K / 623 K with the ramp rate of 0.25 K/s.
- ✓ Isothermal annealing for up to 20 hours at 513 K / 623 K.



Deuterium depth profile in Be-containing layers

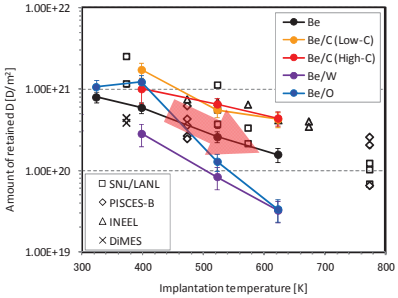
Determination of depth profile of D concentration by analyzing the profile of alpha particles emitted from $D(^3He, \alpha)H$ reaction



- D is dominantly retained in the implantation layer (≈120 nm in Be)
- At elevated T, some D migrates to the interface

H retention in mixed materials

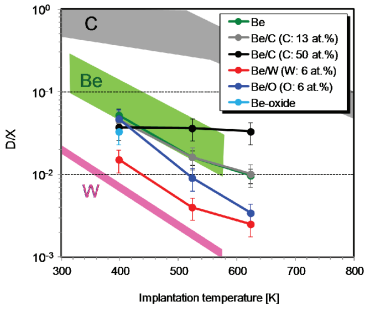
D retention in Be-containing layers vs. implantation temperatures



Amount of D retained in each Be-containing mixed material layer as a function of implantation temperature, together with some literature data of pure Be after plasma exposure obtained at SNL/LANL-TPE, PISCES-B, INEEL and DiMES for comparison.

H retention in mixed materials

D concentration in Be-containing layers vs. implantation temperature



- ✓ D/X in Be layer agrees well with the data obtained from Be-D **codeposition**
- ✓ C-rich D-C layer keeps relatively high D/X even at high temperature – probably due to the trapping by C-D chemical bonds
- ✓ Low fraction of W, O impurity can reduce the D concentration compared to pure Be

Maximum D concentration (shown as D/X) in mixed material layers as a function of implantation temperature. The areas labeled as C, Be or W indicate results from a data compilation of experimentally-obtained D/X values for the D concentration in "codeposition layers" *(R.P. Doerner et al., Nucl. Fusion **49** (2009) 035002)*

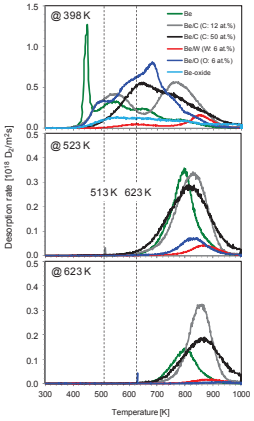
H retention in mixed materials

D desorption from Be-containing layers by normal TDS procedure

- ✓ Variety of D desorption behaviour due to the different D trapping states in each mixed material layer

- 440-470K: D trapped in "supersaturated" region in Be [1][2].
- 500-600K: Decomposition of Be-hydride (BeD_2) [3][4].
- 650-750K: O-related trap, attributed to the decomposition of Be-hydroxide [2][5].
- 800-900K: D trapped in defects (at least 2 different types) in Be [2].
- 600-1200 K: C-D chemical bonds

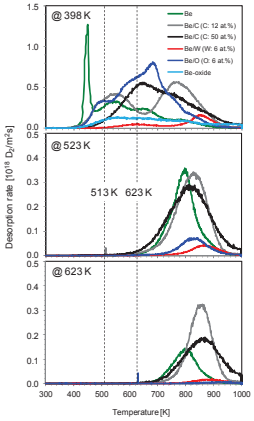
[1] A.V. Markin et al., J.Nucl.Mater. **233-237**(1996)865
[2] M. Reinelt et al., New Journal of Physics **11**(2009)043023
[3] P.E. Barry et al., J.Nucl.Mater. **173**(1990)142
[4] R.P. Doener et al., J.Nucl.Mater. **390-391**(2009)681
[5] A.A. Haasz et al., J.Nucl.Mater. **241-243**(1997)1076



H retention in mixed materials

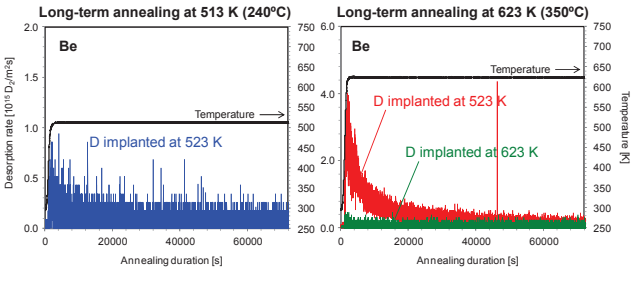
D desorption from Be-containing layers by normal TDS procedure

- ✓ Variety of D desorption behaviour due to the different D trapping states in each mixed material layer
- ✓ **At moderate temperatures**: D retention by some trapping states, Be-hydride/-hydroxide, can be enhanced. Baking efficiency strongly depends on the material mixture.
- ✓ **D implantation > 523 K**: the desorption below the ITER baking temperature is significantly suppressed.
- ✓ **D implantation at higher temperature**: C-D bonds can play a dominant role for D trapping in Be-C mixed layer.
- ✓ **D implantation at higher temperature**: small fraction of W, O in Be can suppress the D retention compared to pure Be



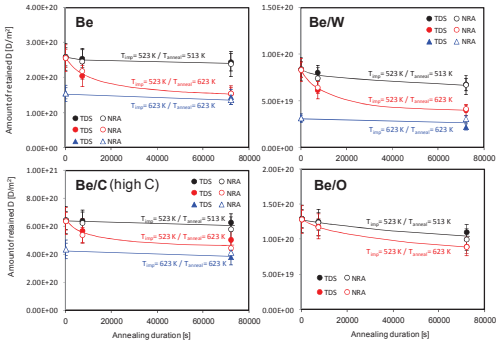
H retention in mixed materials

D desorption from pure Be (≈1% O) during isothermal annealing at 513 K / 623 K



H retention in mixed materials

Reduction of retained D in mixed layers by long-term isothermal annealing



- ✓ It needs higher temperature than implantation temperature for better removal efficiency

H retention in mixed materials

Temperature dependence of H isotopes retention in Be-containing mixed materials was thoroughly investigated in view of ITER removal schemes:

- Data for implanted D compare well with data for co-deposited layers
- D retention decreases with increasing sample temperature
- Admixed impurities (W, C, O) do not lead to dramatic changes. Only for high C content total retention and release temperatures increase to values comparable to those of C layers.
- For ITER: For release by baking, substantially higher temperatures than those during implantation/co-deposition are required.

- This work is being continued, but data are not yet fully analyzed.
- Colleagues at MEdC succeeded to produce co-deposited D containing Be films by TVA method. This allows further annealing studies (e.g., layer thickness dependence).

Conclusions

- PSI studies involving Be and Be-containing samples are still possible at IPP Garching,
  but these activities are possible only for experiments where no significant Be amounts are mobilized.
- Analysis of JET tiles and samples
- Thermal desorption spectroscopy
- Collaboration with colleagues from PISCES is being continued (mostly ion beam analysis, but also XPS depth profiling)
- Collaboration with colleagues from MEdC is being continued (mixed-material co-deposited D-containing layers by TVA)