Beryllium-related PSI-experiments in IPP Garching

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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.

Ion-beam Analysis

Example:

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

S. Krat, I. Bylikov, W. Van Renterghem, M. Mayer, A. Widderow, 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 LW 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.

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.

- 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 $10^{22}$ 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.

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.

IAEA CRP “Beryllium”, Vienna 2014
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)

H retention in mixed materials

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

Layer thickness and compositions determined by RBS

<table>
<thead>
<tr>
<th>Layer type (plotted concentration of Be)</th>
<th>Layer Thickness estimated by RBS</th>
<th>Concentration of Be in the layer determined by RBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Be-W (W = 5 at %)</td>
<td>300 nm</td>
<td>W: 6.1 at % (5 ± 10 at %)</td>
</tr>
<tr>
<td>Be-C (10 at %)</td>
<td>300 nm</td>
<td>C: 3 ± 10 at % (20 ± 40 at %)</td>
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</table>

D implantation in the High Current Ion Source

Loading condition:
- D energy: 600 eV, D2+ (≈ 200 eV / D)
- D flux / fluence: 10¹⁸ D/m² / 1.0 x 10¹⁸ 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 (125°C, 250°C and 350°C))

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/He α 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

![Graph showing D retention in Be-containing layers vs. implantation temperatures.](image)

Amount of D retained in each Be-containing mixed material layer as a function of implantation temperature, together with some literature data on pure Be after plasma exposure obtained at SNL/LANL, TPE, PISEC-B, PIESL, and SIMMS. For comparison:

- Bi: Bi(C1,3%)
- Be/Al: Be/C (9%)
- Be/Al: Be/C (9%)
- Be/W: Be/C (9%)
- Be/O: Be/C (9%)

D concentration in Be-containing layers vs. implantation temperature

![Graph showing D concentration in Be-containing layers vs. implantation temperature.](image)

Maximum D concentration (shown as sox) in mixed material layers as a function of implantation temperature. The areas labelled as C, Be, or W indicate results from a data compilation of experimentally-obtained DIX values for the D concentration in "codeposition layers" (H.P. Emmer et al., Nucl. Fusion 45 (2000) 000000)

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 "superstabilised" regions in Be [12]
  - 500-590K: Desorption of Be-dimer (Be2+) [14]
  - 650-750K: Complex trap, attributed to the desorption of Be-methane [22]
  - 660-1000K: D trapped in defects at least of 3 different layers in Be [2]

H retention in mixed materials

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

![Graph showing D desorption from pure Be (with O) during isothermal annealing.](image)

Long-term annealing at 513 K (240°C) Long-term annealing at 623 K (350°C)

- D implanted at 523 K
- D implanted at 623 K

If 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.

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 PISEC is being continued (mostly ion beam analysis, but also XPS depth profiling)
- Collaboration with colleagues from MEDOC is being continued (mixed-material co-deposited D-containing layers by TVA)