Multi-method approach towards a detailed understanding of Be for fusion

13th August 2014 | M. Köppen

Outline
1. Motivation
2. Surface experiments and methods
   - General approach
   - Experimental capabilities with examples
   - Planned experiments II
   - Work plan
3. Laser-based techniques
4. High heat flux experiments on beryllium and beryllium components
   - Steady state loads
   - Transient heat loads

Motivation
- ITER material mix: Be and W
  - 700 m² 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. Be₅W 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

<table>
<thead>
<tr>
<th></th>
<th>Be</th>
<th>BeO</th>
<th>Be₃N₂</th>
<th>W</th>
<th>WO₂</th>
<th>WO₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting Point [K]</td>
<td>1551</td>
<td>2780</td>
<td>2473</td>
<td>3683</td>
<td>1773</td>
<td>1746</td>
</tr>
<tr>
<td>ΔH° [kJ mol⁻¹]</td>
<td>--</td>
<td>-609</td>
<td>-558</td>
<td>--</td>
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<td>--</td>
</tr>
</tbody>
</table>

Surface experiments and methods for Be and Be-based mixed materials

Outline
1. Motivation
2. Surface experiments and methods

Experimental approach
- Chemical analysis:
  - XPS, ARXPS, ERXPS
  - RBS
  - NRA
  - CRDS
  - TDS
- Hydrogen retention and release:
  - NRA
  - TDS
  - DFT
- Bonding mechanisms:
  - UPS
  - XPS
  - Raman
  - DFT
- Erosion and implantation yields:
  - QMB experiments
  - SDTrim.SP
  - TDS

Multi-method approach: Solutions
- Be-mixed materials
- Hydrogen isotope analysis (amounts, profiles)
- Sputter depth profiles
- Chemical binding states, amounts,
Nuclear reaction
Mass-spectrometer for
Work plan
Rutherford
Planned experiments II
UV source
2 electron sources
2 ion sources, one with
General approach
Standard-X-ray Source
Ion beam analysis
Sample size:
heating: 2.5 cm
Experimental capabilities with examples and plans
Monochromatic x-ray
Mass spectrometer
Be-N-phase II at 113.1 eV
XPS
3 electron evaporators for
Implantation experiment of nitrogen ions into
Mitglied der Helmholtz-Gemeinschaft
1. Define all substances
2. Elementary reactions
3. Define equations for reaction fluxes
\[
\Gamma \left( \frac{\partial n}{\partial t} \right) = \int n_i \Gamma_i \left( \frac{\partial n_i}{\partial t} \right) dt
\]
with border conditions
4. Define balances
\[
\frac{\partial n}{\partial t} = -n_i \Gamma_i + \int n_i \Gamma_i \left( \frac{\partial n_i}{\partial t} \right) dt
\]
Set of ordinary differential equations
\[
y(t) = f(t, y(t)), \text{ with initial value } y(0) = y_0
\]
Experiments: Overview XPS
Main chamber
- XPS
  - Monochromatic x-ray source
  - Hemispherical analyser
- 2 ion sources, one with a
  - 2 electron sources
  - UV source
- Sample size:
  - heating: 2.5 cm
  - Non-heating: 5 cm
Be-mixed analysis (NRA)
Erosion and implantation yields:
- QMB-experiments
- SDTrim.SP
- TDS
Bonding mechanisms:
- UPS
- XPS
- Raman
- DFT
Example X-ray photoelectron spectroscopy:
N-Implantation in Be
- Implantation experiment of nitrogen ions into
diamond beryllium at different fluences up to 5.5 x
- Deconvolution of spectra shows three species:
  - Metallic Be
  - Be-N-phase II at 113.1 eV
  - Be-N-phase I at 113.8 eV
- Be-N-Phase I is an intermediate compound at
low fluences
- Be-N-Phase II is a metastable compound

<table>
<thead>
<tr>
<th>N-fluence</th>
<th>Be</th>
<th>Be-N I</th>
<th>Be-N II</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.4</td>
<td>15.1%</td>
<td>54.5%</td>
<td>30.4%</td>
</tr>
<tr>
<td>3.8</td>
<td>24.4%</td>
<td>47.7%</td>
<td>27.9%</td>
</tr>
<tr>
<td>5.5</td>
<td>22.4%</td>
<td>31.1%</td>
<td>44.3%</td>
</tr>
</tbody>
</table>
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 x 10^{-11} mbar
- Electron beam evaporator

Experiment: Methods in ARTOSS

- Chemical analysis:
  - XPS, ARXPS, ERXPS
  - RBS
  - NRA
  - CRDS
  - TDS
- Erosion and implantation yields:
  - ODME experiments
  - SDTrim.SP
  - TDS
- Hydrogen retention and release:
  - NRA
  - TDS
  - RAMAN
  - DFT
- Bonding mechanisms:
  - UPS
  - XPS
  - Raman
  - DFT

Example: Thermal desorption spectroscopy of D in Beryllium

Deuterium-release and retention of Be(0001)

- Low temperature release peak: Assigned to structurally modified BeD_{2-x}
- High temperature release peak: Retention in ion induced defects

TPD of D implanted Be

Full TPD spectra of D implanted in Be

- Peaks 1 and 2 – structural changes (over-saturation with D, fluence threshold)
- Peaks 3 and 4 – trap sites produced by collision cascades
- Peak 5 – hydride formation (BeD_{2}), decomposition at ~500-600K
- Peak 6 – release from an oxidized Be surface layer

Modelling of D retention:

- CRDS (Charge Resonant Detachment Spectroscopy)
  - Assumption: desorption diffusion limited!
  - Flux at both side surfaces of the sample:
    \[ \Gamma_{\text{side}} = \frac{\Gamma_0 \exp(\frac{z - z_m}{\lambda}) \cdot x_f \cdot \lambda}{x_f} \]

D-release anisotropy at low fluences on Be single crystals

- Temperature shifts in D release peaks due to:
  - Different implantation depth
  - Different crystal orientation (release is from Be planes)
  - Retained amount of D smaller for Be(0001)

Experiment: LAICA

- Experiments at HZB-BESSY II at “SurICat”
  - Preparation chamber “LAICA” is interconnected
    - 3 Evaporators: Be, C and W
    - Heater for temperatures up to 1370 K
    - 5 kV ion gun for implantation and sputtering
    - Auger system
    - Base pressure: 5 x 10^{-10} mbar
  - ERXPS measurements in SurICat
    - Excitation energies from 30 to 1300 eV
    - Base pressure: < 1 x 10^{-10} mbar
Experiment: Methods in LAICA

- Chemical analysis:
  - XPS, ARXPS, ERXPS
  - RBS
  - NRA
  - CRDS
  - TDS

- Erosion and implantation yields:
  - QMB-experiments
  - SDTrim.SP

- Hydrogen retention and release:
  - NRA
  - TDS
  - DFT

- Bonding mechanisms:
  - UPS
  - XPS
  - Raman
  - DFT

Be-mixed materials

Example: Energy-resolved XPS at synchrotron

- Information depth depends on the kinetic energy of the photoelectron

- The kinetic energy depends on the energy of the photon

- Variation of photon energy varies information depth

- Allows chemical depth-profiling of the first nanometers

Experiment: Methods in LID/QMS

- Chemical analysis:
  - XPS, ARXPS, ERXPS
  - RBS
  - NRA
  - CRDS
  - TDS
  - LID-QMS

- Erosion and implantation yields:
  - QMB-experiments
  - SDTrim.SP
  - TDS

- Hydrogen retention and release:
  - NRA
  - TDS
  - DFT

- Bonding mechanisms:
  - UPS
  - XPS
  - Raman
  - DFT

Versatile beryllium analysis facility

- Design and construction of a JET-oriented beryllium analysis facility (inside the controlled area of HML but outside the hot cell)

- Thermal Desorption Spectrometry (TDS)
- Laser-Induced Desorption of complete JET tiles using Nd:YAG laser
- Surface composition analysis by glow discharge spectroscopy

Already operational
Will be moved to hot cells asap

Isotope exchange

- Few experimental data on H isotope exchange in Be

- Beryllium has not an “ideal” hcp structure. Be-Be distances are closer in <0001> than perpendicular to it (e.g. <11-20>) → different diffusion constants

- What role does dissociation play

Confirmation of BeDₓ species

- Observation of specific BeD line (OES) in JET, PISCES ...
- Predicted by calculations (C. Björkas 2009)
- Existence not yet proven in laboratory experiments
- Sputtering of BeD or BeD₂

Planned experiments: ion vs. atom

- Until now only experiments with D ion implantation performed → trapping of D in trap-sites produced during implantation
- Molecular hydrogen does not adsorb on Be → atomic hydrogen needed
- Open questions:
  - Temperature dependency (absorption barrier)?
  - Retention in intrinsic defect sites?
  - Does Ne/N etc. ion bombardment create additional trap-sites for D in Be?
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Work Plan

- Investigation of the system Be-N
- Investigation of hydrogen retention and release behaviour of Be-N
- Investigation oxidation behaviour of Be-N
- Experiments on the interaction of He-ions with Be-N
- Isotope exchange in Be-based mixed materials
- Understanding BeD₂-formation and properties
- Investigation of different behaviour of atomic and ionic hydrogen

Laser-based techniques

Conceptual design of JULE-PSI linear plasma device

Future Program

- Time resolved physics of LIBS plasma
- Investigation of LIBS plasma physics under:
  - vacuum
  - gaseous (Ar, N, He) environments
  - magnetic field
- Impact of the laser wavelength on the LIBS plasma
  - Combination of two laser beams (Dual LIBS)
- Ablation physics: Energy distribution (ToF), species distribution, formation of cluster, reproducibility (particles/pulse),
  - Modelling of the laser-material interaction
High heat flux tests on Be-components

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Repeated thermal shocks on beryllium

Energy density: \( E = 1.0 \text{ MJ/m}^2 \)
Base temperature: \( T_b = 250^\circ\text{C} \)

Pulse duration: \( \Delta t = 5 \text{ ms} \)
Heat flux factor: \( P = v(\Delta t) = 14 \text{ MW/m}^2 \text{s}^{1/2} \)

Stress assisted grain boundary oxidation effect

High heat flux test facility JUDITH 1

Electron beam facility JUDITH 1 is used to simulate transient high heat loads

Electron beam parameters:
- max. power: 60 kW
- acc. Voltage: 120 kV
- pulse duration: \( \geq 1 \text{ ms} \)
- beam diameter: \( \sim 1 \text{ mm} \)

ELM-like loading conditions
- power density: 200 - 800 MW/m²
- pulse duration: 1 ms
- pulse number: up to 1000

Surface finish influence

abs. power density: 0.8 GW/m²
pulse length: 1 ms, pulse number: 100

No clear coincidence of cracks and lathe faced cavities observable

R_s [µm] unload loaded
rough 0.7 2.6
fine 0.4 2.4

Cracks might coincide with grain orientation/microstructure
Further investigations valuable

Assembly of the mock-up in JUDITH 2

• 2 mock-ups are tested at once
• For the thermal insulation both mock-ups are fixed with sharpened screws
• Each mock-up is equipped with 2 thermo-couples

For the protection of the base plate and mock-up holder beam-dumps are used

High heat flux experiments on beryllium and beryllium components

• Steady state loads
• Transient heat loads

Expected transient power loads
(off-normal events have to be considered and tested as well)

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18 August, 2014 IAEA Be-CRP Meeting 2014, Vienna
Thermal shock loading of beryllium

\[ E_{\text{abs}} = 5.2 \text{ MJ m}^{-2}, \Delta t = 5 \text{ ms}, T_0 = 20^\circ \text{C} \text{ (to simulate plasma disruption)} \]

**S65C**
- Hot pressed in vacuum
- Punched, relative high oxygen content

**TR-30**

Thermal shock testing of neutron-irradiated Be samples

- Neutron irradiation: \( T_{\text{irr}} = 700^\circ \text{C}, 0.35 \text{ dpa} \)
- Electron beam loading: \( E_{\text{inc}} = 15 \text{ MJ m}^{-2}, \Delta t = 5 \text{ ms}, n = 5 \)

- Beryllium is transmuted into tritium and helium during irradiation
- Pore formation during electron beam loading of neutron irradiated beryllium
- He concentration is 55 ppm