

IAEA CRP "Beryllium" final meeting, Vienna 2016

Beryllium-related PSI-studies at IPP Garching

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- Experimental equipment used/still in use for Be related studies
- Recent projects:
 - D retention in and release from Be mixed materials
 - H/D isotope exchange experiments
 - Cavity probes in PISCES and JET
 - JET marker tiles and wall inserts
 - Cross sections for ion beam analysis
 - Modelling material migration

List of recent publications



Experimental facilities at IPP Garching used for Beryllium studies

- UHV preparation chamber (Be thermal vapour deposition) connected to dual ion beam setup and in-situ Photoelectron Spectroscopy (XPS)
- High current ion beam setup "HCS" with in situ mass balance
- Thermal desorption spectroscopy experiment "TESS"
- "RKS" ion beam analysis setup with Rutherford Backscattering Spectroscopy, Nuclear Reaction Analysis and Elastic Recoil Detection Analysis
- "SAK" ion beam analysis setup with air tight glove box equipped with Rutherford Backscattering Spectroscopy and Nuclear Reaction Analysis

IPP High Current Ion Source(s)



D implantation with mass separated ion beam



Features:

- D_3^+ ; D energy 30 eV/D 2 keV / D ٠
- D flux / fluence: ~ 10^{19} D/m²s / < 10^{25} D/m² •
- Sample heating by e-bombardment < 1000K ٠
- Decomissioned, upgraded and duplicated in 2015/2016 ٠
- New in-situ mass balance in operation in autumn 2016, in-situ IBA > 2017 ٠
- No Be related work anymore ۲

Temperature-programmed desorption

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TPD or long term annealing at the TESS facility

- Used for "Normal" TPD of Be mixed TVA layers provided by National Institute for Laser, Plasma and Radiation Physics, Bucharest and implanted at "IPP HCS"
- Typically sample heated up to 1000 K with a ramp rate of 0.25 K/s.
- UHV and tubular oven allows long-term outgassing for days and weeks
- Be safety: Dedicated quartz tube for Be studies with "catcher labyrinth"
- Operational but Be activities on TVA thin films stopped in 2014 due to risk of flaking
 - built their own TDS setup





- In addition to
 - RBS and
 - NRA also
 - Elastic Recoil Detection Analysis (ERDA) for H, D and He detection
- Only "dust free samples"





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 - Elastic Recoil Detection Analysis (ERDA) for H, D and He detection
- Only "dust free samples"
- Dangerous with thin films!

≈ 250 nm Be on W



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- Only "dust free samples"
- Dangerous with thin films!

≈ 250 nm Be on W





Sample manipulator

Analysis chamber

Load lock chamber



- Dedicated chamber for Be contaminated Jet samples (Tritium < 1 GBq)
- Rutherford Backscattering Spectroscopy (typically protons or Helium) and Nuclear Reaction Analysis (³He for D, ⁴He, p for N and D for ³He)
- Regular swipe sampling

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D retention in / release from Beryllium-related mixed material layers

- Work performed under framework of F4E contracts: F4E-OPE-080 / -347 "Deuterium retention and outgassing experiment", towards an evaluation of the tritium removal operation in ITER (baking at 240 °C for the first wall / 350 °C for the divertor).
- Completed and published since the last CRP



Work strategy

- Be containing samples to be investigated: Be, Be-W, Be-C and Be-O mixed layers prepared by Thermionic Vacuum Arc (TVA) deposition method at National Institute for Laser, Plasma and Radiation Physics, Bucharest.
- Layer characterization, **D** ion implantations and subsequent outgassing experiments at IPP Garching.
 - Rutherford Backscattering Spectrometry (RBS) using 2.0 MeV ⁴He⁺ for the layer characterization.
 - D loading to the layers in the High Current Ion Source device.
 - D retention is analysed by Nuclear Reaction Analysis (NRA) using 800 keV ³He⁺ (D(³He, p)⁴He reaction).
 - TDS and **long-term outgassing** experiments in TESS facility.



Layer preparation by Thermionic Vacuum Arc (TVA) deposition

National Institute for Laser, Plasma and Radiation Physics, Bucharest

- Be-W and Be-C mixed layers are prepared by simultaneous deposition of Be and W or C using dual target source configuration.
- Gas inlet system successfully developed -Be-O mixed layer can be deposited by oxygen injection during Be deposition.
- Each deposition batch typically consists of 15 silicon and 2 graphite substrates – Silicon is for the outgassing experiment and Graphite is for the layer characterization.



Schematic view of TVA setup (inside the vacuum chamber)



Layer thickness and compositions determined by RBS

Layer type	Layer areal density	Layer stoichiometry	
Ве	6.9×10 ¹⁸ at./cm ²	(O impurity ≤ 1 at.%)	
Be/W	4.3×10 ¹⁸ at./cm ²	W: 6 ±2 at.% (O: ~ 10 at.%)	
Be/C (low C)	6.2×10 ¹⁸ at./cm ²	C: 13 ±1 at.% (O: ~ 7 at.%)	
Be/C (high C)	4.6×10 ¹⁸ at./cm ²	C: 50 ±1 at.% (O: ~ 8 at.%)	
Be/O (low O)	4.1×10 ¹⁸ at./cm ²	O: ~ 6 ±1 at.%	
Be/O (high O)*	2.6×10 ¹⁸ at./cm ²	O: ~ 50 ±1 at.%	

Layer thickness 340 - 570 nm

D retention in Be-containing layers

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Amount of D retention in Be-containing layers vs. implantation temperatures

- D retention in general decreases with the D loading temperature (above ~400 K).
 - The absolute amount varies depending on the experimental condition.
 - ✓ Reduction is limited in the case of Be-C mixed cases.
 - ✓ Retention in the ITER first wall temperature range is expected to be 10²⁰ - 10²¹ m⁻².



Amount of D retention 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.

D concentration in Be-containing layers

Depth profile of D concentration in Becontaining layers

- Determination of depth profile of D concentration by analyzing the profile of alpha particles emitted from D(³He, α)H reaction.
- C-rich D-C layer keeps relatively high D concentration even at high temperature – likely due to the trapping by C-D chemical bonds.
- Why different penetration depths? Different diffusion or simply morphology?

D retention in mixed materials

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, α)H reaction

- D is dominantly retained in the implantation layer
- At elevated T, some D migrates to the interface

D retention in Be-containing layers

D concentration in Be-containing layers vs. implantation temperatures

Maximum D concentration (shown as D/X) in each Be-containing mixed material layer 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"

- D/X in Be layer agrees 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.

1st Summary

D retention in and release from Be-containing mixed material layers were investigated in view of ITER tritium removal schemes:

- Retention in the mixed layer decreases with increasing the D loading temperature, but the tendency has some variation depending on the admixed element and its amount.
- Admixed impurities seems not to lead to dramatic changes of absolute retention. Only for high C content case total retention and the release temperature increase.
- Removal efficiency by the wall baking procedure(s) will be limited if the wall temperature is already comparable to the baking temperature.

Related publications:

K. Sugiyama, J. Roth, A. Anghel C. Porosnicu, M. Baldwin, R. Doerner, K. Krieger, C.P. Lungu., J. Nucl. Matter. 415 (2011) S731

K. Sugiyama, C. Porosnicu, W. Jacob, J. Roth, Th. Dürbeck, I. Jepu, C.P. Lungu, J. Nucl. Matter. 438 (2013) S1113

K. Sugiyama, C. Porosnicu, W. Jacob, I. Jepu, C.P. Lungu, Nuclear Materials and Energy 6 (2016) 1

D retention in mixed materials: additional work since then

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Preparation of "codeposition" by TVA deposition method at MEdC/NILPRP

- ✓ Layer deposition with TVA under D₂ gas atmosphere.
 - Analysis of N and D containing TVA films for EFDA/EUROfusion tasks
 - Mixed success:
 - D contents << 10 %
 - N containing films had more O than N
 - Very promising results with recent magnetron sputter device (as initially done shown by PISCES lab)
 - Analysis at other European labs (JSI Ljubljana, IST Lisbon)

D/H - Isotope exchange experiments bulk beryllium and Be:H and Be:D thin films

- Work performed at PISCES and IPP Garching (T. Schwarz-Selinger, D. Nishishima, R. Doerner, unpublished)
- Work performed at PISCES, IPP Garching, MePHI, CEA (D. Kogut et al. Phys. Scr. T167 (2016) 014062 (6pp))

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Hydrogen isotopes retention in beryllium

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In general: hydrogen retention in beryllium is even less understood as in tungsten

- material grade (codeposits vs. bulk)
- temperature ~
- D energy

"de Temmerman scaling": Nucl. Fusion 48+49 (2008)

- flux - fluence
- seeding (He, N, Ar, ...)

Investigations are hampered by - its toxicity

- its reactivity
- reduced IBA depth resolution and sensitivity

Strategy: - exchange experiments in plasma-sprayed Be targets

- effectiveness in thick codeposited layers?

To chck its applicability as fuel removal method

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Isotope exchange in PISCES-B on Be with Be walls

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 \Rightarrow complete exchange: $\approx 10^{25} - 10^{26}$ ions/m²

 Φ_{exchange} for Be >> than for W?

issues: - wall effect / recycling?

- D in depth?
- sputtering?
- ITER relevant: codeposits

evolving surface morphology does not allow to judge about that!

Better defined experiment for ion beam analysis (NRA for D + Be; ERD for H + D):

- ➢ 50 nm: PISCES-B Be:D codeposit (D/Be = 0.25) on polished tungsten
- sequentially exposed to H ion beam in Garching "HCS"

 exchange in near surface layers in Be as
 effective
 fast as in W!

> also for thick codeposits?

➤ ≈ 200 nm PISCES-B Be:H codeposit on polished tungsten (H/Be=0.2)

- ➤ ≈ 200 nm PISCES-B Be:H codeposit on polished tungsten (H/Be=0.3)
- exposed to D ion beam

 \Rightarrow only surface near exchange @ 310K \Rightarrow dense material

ERD

ЪD

- ➤ ≈ 200 nm PISCES-B Be:H codeposit on polished tungsten (H/Be=0.3)
- exposed to D ion beam

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- > \approx 350 nm magnetron sputtered Be:H codeposit on polished tungsten (H/Be=0.04)
- exposed to D ion beam

ERD

➤ ≈ 325 nm magnetron sputtered Be:H codeposit on polished tungsten (D/Be=0.04)

ERD

exposed to D ion beam

 \Rightarrow uptake > release! + indication for deeper penetration?

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- More high temperature experiments and experiments on bulk Be were planned but Be operation ended at HCS
- Ideal experiment:

H/D exchange at the same ion energy and sample temperature at which D/H was implanted during growth to see quanitatively the exchange rather than a new equilibrium state

Initiated by D. Kogut and D. Douai CEA

Same procedure as in 2014:

- Be:H preparation on rough W subsrate by magnetron sputtering at PISCES lab
- Exposure to D ion beam at MEPhI
- Analysis at
 - Ruhr Uni Bochum by ¹⁵N NRA for H depth profile and at
 - IPP Garching D content with NRA and exchange with ERDA

Drawback:

- High energy D implantation (2.5keV/D compared to < 100eV/D during growth)
- Inhomogenous implantation spot
- Limited number of samples

- Distinct H peak in the D implantation zone!
- DITMIX diffusion trapping model with Baldwins input data describes observation assuming trap creation by high energy D implantation (2.5keV/D compared to < 100eV/D during growth)

100

200

300

Depth (nm)

400

500

600

- See D. Kogut et al. Phys. Scr. T167 (2016) 014062 (6pp)

0' 0

D/H isotope exchange experiments

- Exchange of H by D (or vice versa) is possible even at room temperature with high rates
- Clear high temperature experiments are lacking
- In terms of tritium removal for ITER:

Despite indications for deeper hydrogen penetration in Be:H in net deposition areas dense Be:T codeposits will be simply covered up by Be:D (until delamination: dust issue?) if only running a high performance shot in D_2 .

Cavity experiments at PISCES

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ülich, Germany

Beryllium seeding experiments at PISCES in 2010/2011

Seeding series at 50 eV ion energy (-65 V bias), 370 K shows:

Erosion yield of 0.22% can only be compensated by seeding 2.2% Be!

The Cavity technique:

adapted from Ch. Hopf et al., JAP 87 (6), 2719 (2000)

Here: $\gamma = 0$ If erosion can be neglected: $\beta = s$

 \Rightarrow Analysing deposition pattern of cavity / Hohlraum / pillbox structures

Measuring the surface loss probability

 \Rightarrow Analysing deposition pattern of cavity / Hohlraum / pillbox structures:

Ch. Hopf et al., JAP 87 (6), 2719 (2000)

> Here also: Comparing total amounts inside and outside of the cavity

PISCES-B witness plate:

- 3.3-10¹⁹ Be m⁻²s⁻¹,
- 2000 sec,
- 300 K

Result:

- Deposition at the bottom is a direct image of the gap (in terms of width and thickness)
- Integral Be amount inside of cavity = expected one from top
- No deposition at the back side of the lid

⇒ Surface loss probability β = 1⇒ No erosion: **sticking s = 1**

PISCES-B witness plate:

- 0.65•10¹⁹ Be m⁻²s⁻¹,
- 3600 sec,
- 300 K

Result:

- Deposit contains D! D/Be = 1%!!!
- Integral Be amount inside of cavity
 = expected one (top)
- ¼ of the total deposition at the back side of the lid
- \Rightarrow Surface loss probability β < 1?
- Deposition at the bottom is spread out due to collisions

PISCES-B target plate:

- 2.6-10¹⁹ Be m⁻²s⁻¹,
- 3770 sec,
- <400 K

Result:

- Integral Be amount inside of cavity
 = ½ of expected one (top)
- no deposition at the back side of the lid
- \Rightarrow Surface loss probability $\beta < 1$?
- Deposition at the bottom is spread out due to collisions

Can a surface loss probability between 0.8 and 1 explain the Be seeding experiments?

Beryllium film deposition in cavity samples in remote areas of the JET divertor during the 2011-2012 ITER-like wall campaign

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 3DB, UK
 * See the Appendix of F. Romanelli et al., Proceedings of the 25th IAEA Fusion Energy Conference

2014, Saint Petersburg, Russia

Cavity probes in JET

- Exposed in the 2011-2012 ILW campaign
- Silicon samples in stainless steel housing
- 2.4 MeV ³He⁺ NRA at inner top and bottom for Be, D, and C

Cavity probes in JET: Inner divertor

- The Be source is almost similar to the C source in JET-C for the inner divertor: Most particles originate from the sloped central area of divertor tile 4, from the direction about 15° below the horizontal plane, near the strike points on this tile
- A large amount of C originates from the rear side of tile 3. Parasitic discharges or chemical erosion of C by deuterium atoms might be the cause
- The D source is a mixture of Be and C sources
- Relatively large amount of C, with C/Be=0.5
- Large D content with D/(Be+C)=0.3
- For D and Be, highly sticking species are • present (S>0.7). Only low sticking species for C (S<0.5)

Cavity probes in JET: Inner divertor

- Relatively large amount of C, with C/Be=0.5
- Large D content with D/(Be+C)=0.3
- How to explain low sticking species for Be?
 Erosion?

	Two-component model	One- component model	Ratio top/bottom
Be	91%*0.33+9%*0.97	0.4	0.46
D	48%*0.49+52%*0.76	0.64	0.68
С	17%*0.01+82%*0.53	0.44	0.53

• At least cavities act as pinhole camera to locate sources

- Film depositon in shadowed areas of the JET divertor was analyzed using NRA.
- The Be deposition rate is **34 times smaller** than the C deposition rate during the 2005-2009 JET-C campaign. D accumulation rate was **45 times smaller**
- Be and C originate mostly from nearby strike point locations, they are likely reflected or re-eroded from W divertor tiles; some C in the inner divertor originates from the rear side of tile 3, where it might originate due to parasitic discharges or erosion by atomic deuterium
- There are indications that re-erosion plays a significant role in particle transport in the outer divertor and under tile 5.
- Low effective sticking coefficients dominate for all components, with s≤0.3. This cannot be explained by atomic reflection or re-erosion.

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Cross sections for IBA

- > $^{9}Be(p,p_{0})^{9}Be EBS between 400keV and 4150keV (non RBS > 230keV)$
- ⁹Be(p,d₀)⁸Be NRA between 400keV and 4150keV
- > ${}^{9}Be(p,\alpha_{0}){}^{6}Li EBS$ between 400keV and 1300keV
- Scattering angle of 165°
- Measured on Be thin films and benchmarked on bulk films

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⁹Be(p,d₀)⁸Be elastic scattering

JET inner wall inserts

S. Krat

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* See the Appendix of F. Romanelli et al., Proceedings of the 25th IAEA Fusion Energy Conference 2014, Saint Petersburg, Russia

Long-Term Samples: Erosion at inner wall cladding

Erosion at inner wall 2011-2012

- Main chamber erosion: Erosion of Be decreased by factor ~5 compared to C
- Decrease of plasma impurity concentration from $\sim 1 2\%$ C to 0.1 0.2% Be
- \Rightarrow Decrease of net impurity source from inner wall
- \Rightarrow Reason: Absence of chemical erosion by low-energy neutral hydrogen

Discharge campaign	Number of discharges	Number of successful discharges (I _p >0.7 MA)	Total discharge time ($I_p>0.7$ MA), 10^4 s	Divertor phase discharge time, 10 ⁴ s	Limiter phase discharge time, 10 ⁴ s
2011-2012	3812	2819	6.41	4.51	1.9
2013-2014	4150	???	7.12	5.09	2.03

Erosion at inner wall 2011-2014

- Erosion of Be in 2013-2014 decreased by factor 1.8 compared to 2011-2012
- Erosion of W remained constant

Future work on long term samples

- Inner wall erosion monitors 2016 201? are forseen
- Inserts to be provided by CCFE
- W coating at IPP (M. Mayer)
- Be coating at NILPRP (C. Lungu)
- IBA pre-analysis at IPP (M. Mayer)

Status: Waiting for inserts

Erosion and deposition in the JET divertor during the first ILW campaign

M. Mayer

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Experimental: Marker layers in JET-ILW

Marker layers for erosion/deposition studies: 4 μm W

- 4 µm Mo interlayer for separation of thick 20 µm W coating from marker layer
- Non-destructive analysis by RBS + NRA before and after exposure
- Exposed 2011 2012:
 13 h divertor plasma phase + 6 h limiter

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Erosion & Deposition in JET-ILW [1]

- Absolute amounts:
 - WallDYN Be dep. Rate on "Apron": (Equilib. value)
 0.5 to 2.8 (10²⁰ Be m⁻² s⁻¹)
 - Experimental (post mortem) [2] Average over many different plasma scenarios and other uncertainties

0.2 to 0.3 (10²⁰ Berra⁻² s⁻¹)

Qualitative match to post mortem results [2,3]

Main chamber is net erosion zone
 Be deposition mainly on tile 1 ("Apron")
 Rest of divertor no net Be deposition

[1] K. Schmid et al. J. Nucl. Mat. 463 (2015) p. 66 [2] J. P. Coad et al. Phys. Scr. T159 (2014) 014012 _[3] M. Mayer et al. PFMC 2015, Physica Scripta, in print

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→ WallDYN too high by trend, but close given the large uncertainties

- PSI studies involving Be sample preparation or Be sample implantation has come to an end at IPP Garching
- Activities are still possible for experiments where no significant Be amounts are mobilized.
- Analysis of JET marker tiles and wall inserts will be continued
- Collaboration with colleagues from PISCES is being continued (mostly ion beam analysis, but also XPS depth profiling)
- Walldyn modeling on Be transport for Jet and ITER will be continued
- Studies on isotope exchange and Be cavities need to be published
- Collaboration with colleagues from MEdC is being largely reduced (mixedmaterial co-deposited D, O or N -containing layers by TVA)

Recent publications

D retention in and release from Be mixed materials

K. Sugiyama, C. Porosnicu, W. Jacob, I. Jepu, C.P. Lungu, Nuclear Materials and Energy 6 (2016) 1

Erosion at the inner wall of Jet

S. Krat, Yu Gasparyan, A. Pisarev, I. Bykov, M. Mayer, G. de Saint Aubin, M. Balden, C.P. Lungu, A. Widdowson, JET-EFDA contributors, J. Nucl. Metr. 456 (2015) 106

Erosion and Deposition in JET

M Mayer, S Krat, W Van Renterghem, A Baron-Wiechec, S Brezinsek, I Bykov, P Coad, Yu Gasparyan, K Heinola, J Likonen, A Pisarev, C Ruset, G de Saint-Aubin, A Widdowson and JET Contributors, Physica Scripta, T167 (2016)

Cross sections for IBA analysis

S Krat, M. Mayer, C. Porosnicu, Nuclear Instrum. Meth B 358 (2015) 72

Modelling beryllium migration in JET

K. Schmid , K. Krieger, S.W. Lisgo, G. Meisl, S. Brezinsek, , J. Nucl. Mat. 463 (2015) 66