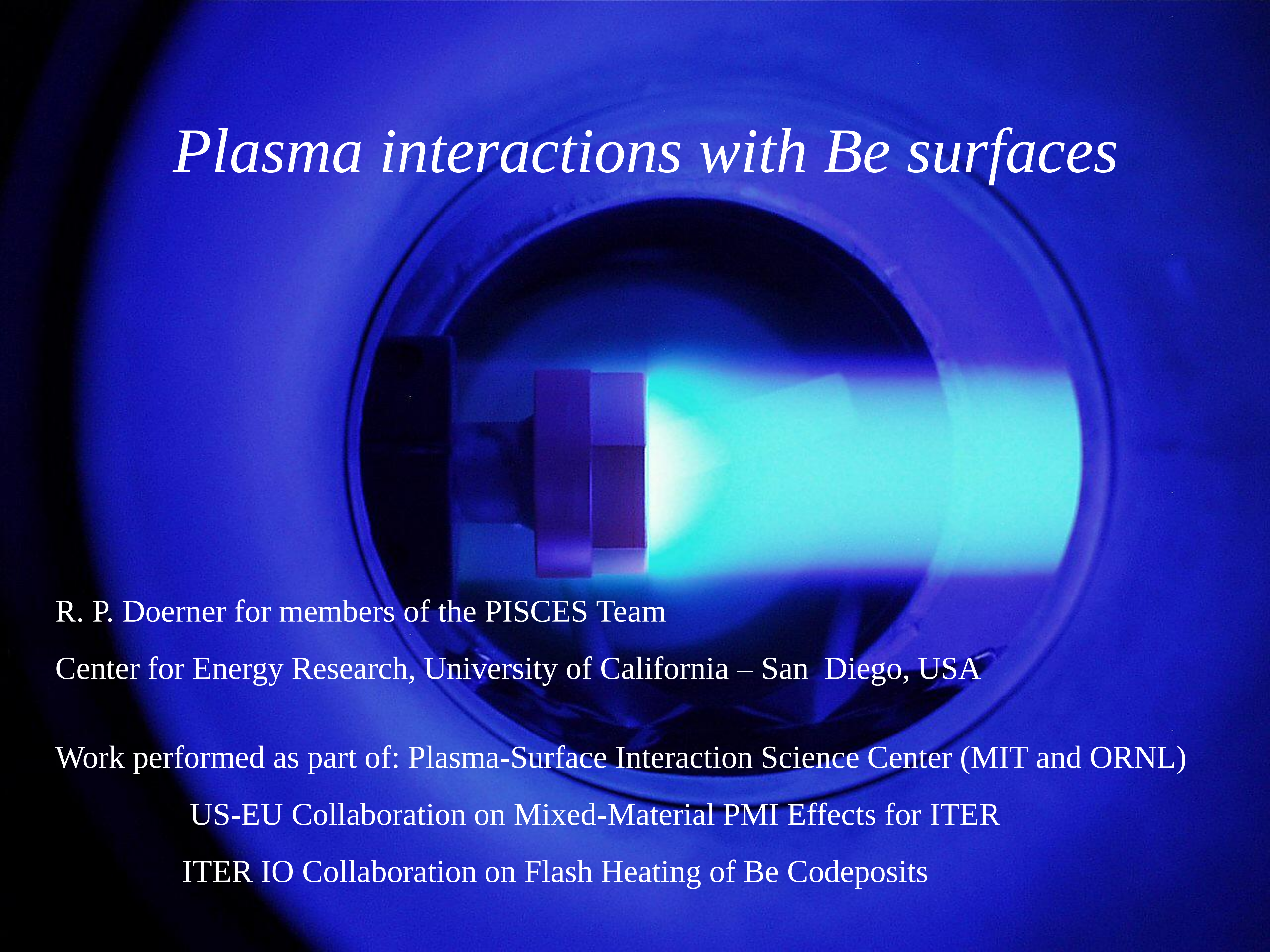


# *Plasma interactions with Be surfaces*

R. P. Doerner for members of the PISCES Team

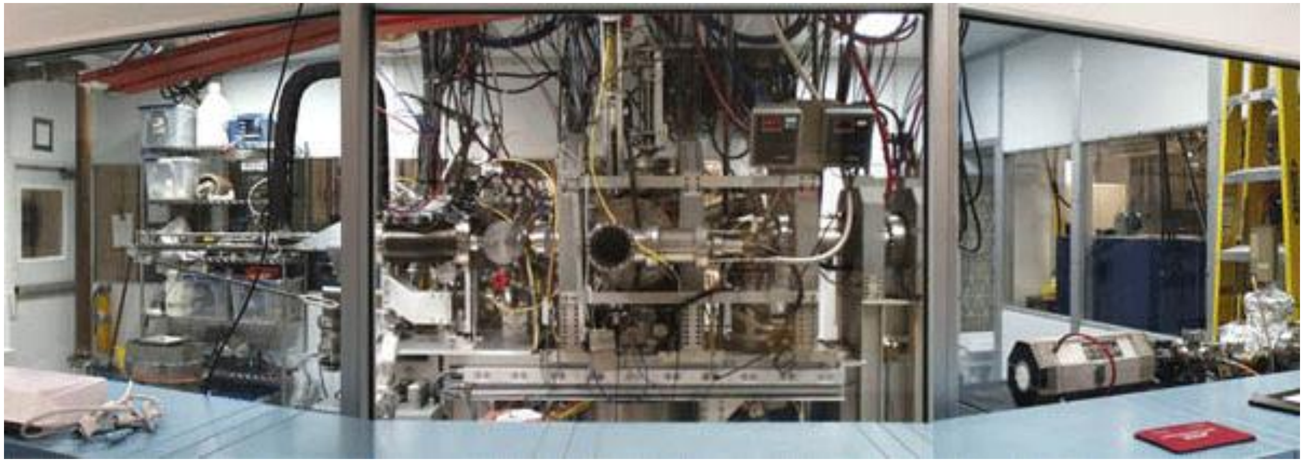
Center for Energy Research, University of California – San Diego, USA

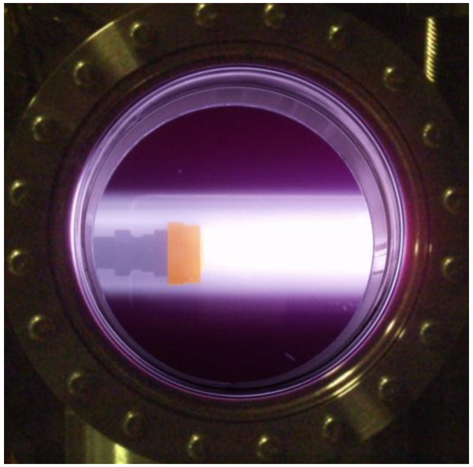
Work performed as part of: Plasma-Surface Interaction Science Center (MIT and ORNL)

US-EU Collaboration on Mixed-Material PMI Effects for ITER

ITER IO Collaboration on Flash Heating of Be Codeposits

# The PISCES-B divertor plasma simulator is used to investigate ITER mixed materials PSI.



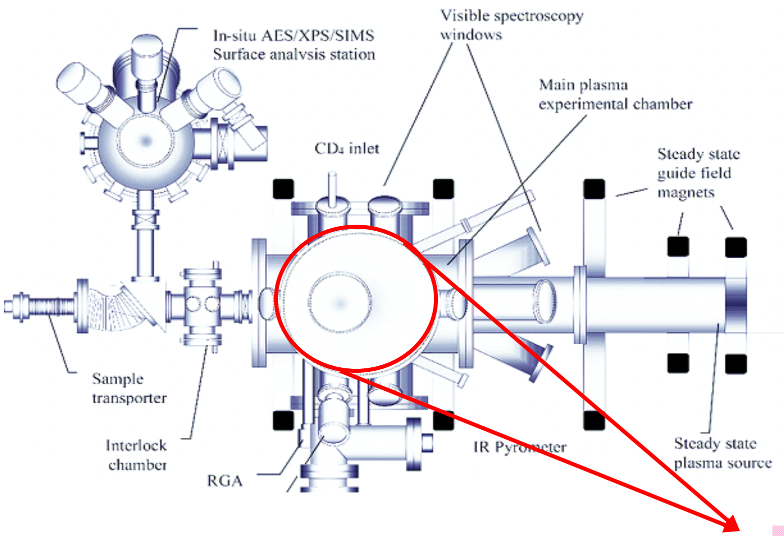


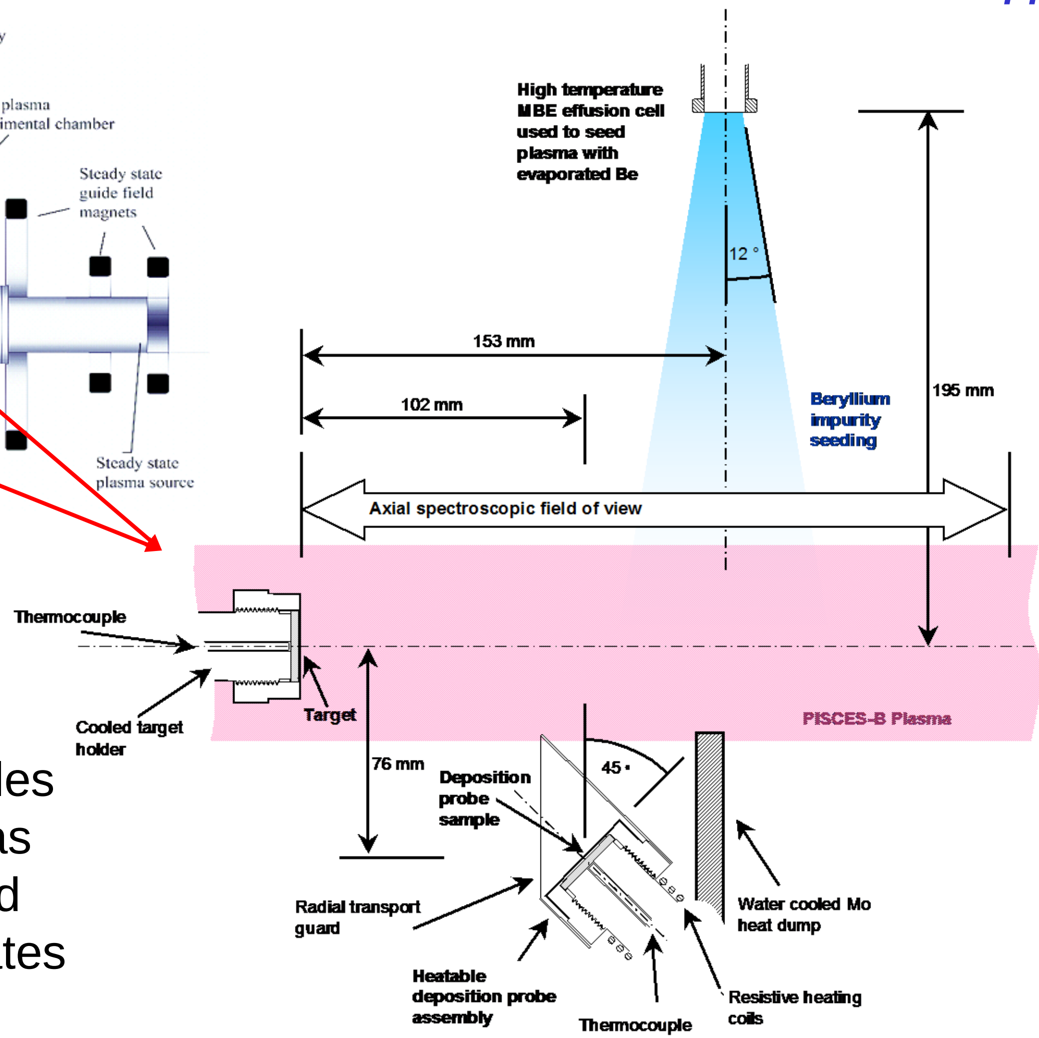
- PISCES-B is contained within an isolated safety enclosure to prevent the release of Be dust.

| | PISCES | ITER (edge) |
|---|---|---|
| **Ion flux ($cm^2s^{-1}$)** | **$10^{17}$–$10^{19}$** | **~$10^{19}$ - $10^{20}$** |
| **Ion energy (eV)** | **20–300 (bias)** | **10–300 (thermal)** |
| $T_e$ (eV) | 4–40 | 1–100 |
| **$n_e$ ($cm^{-3}$)** | **$10^{12}$–$10^{13}$** | **~$10^{13}$** |
| **Be Imp. fraction (%)** | **Up to a few %** | **1–10 (ITER)** |
| **Pulse length (s)** | **Steady state** | **1000** |
| PSI materials | C, W, Be | C, W, Be .. |
| Plasma species | H, D, He | H, D, T, He |

# *PISCES-B has been modified to allow exposure of samples to Be seeded plasma*






P-B experiments simulate Be erosion from ITER wall, subsequent sol transport and interaction with W baffles or C dump plates, as well as investigation of codeposited materials using witness plates

# Outline of Technical Presentation



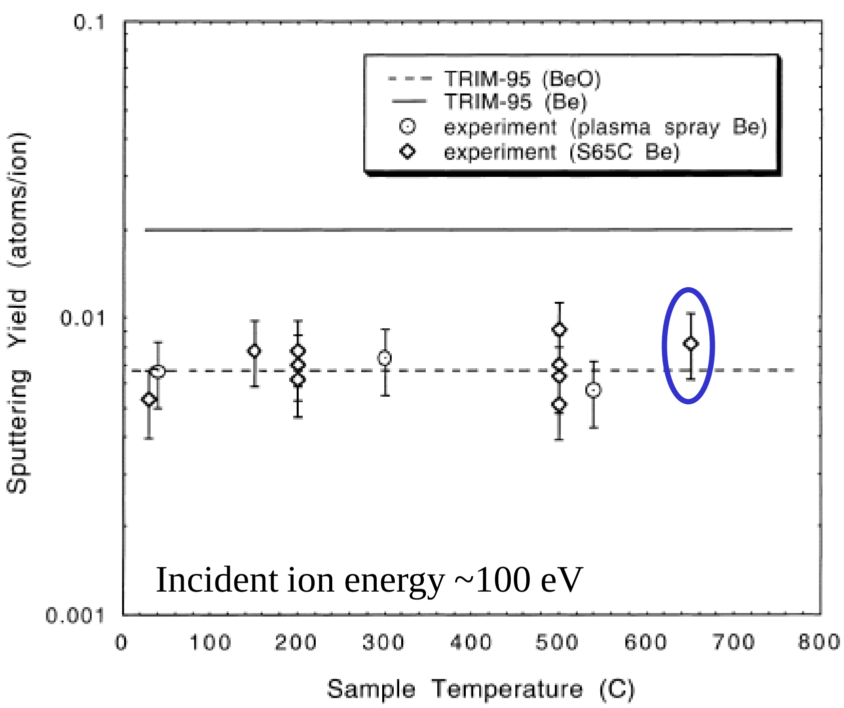
- Erosion in the plasma environment
  - Be erosion from D, He and Ar plasma
  - Chemical sputtering of BeD
  - Redeposition/sticking efficiency
- Retention and release
  - Plasma exposed Be targets
  - Be-rich witness plate codeposits
  - Release due to flash heating
- Be-containing mixed materials (W, C, N, O)
- Spectroscopic issues for Be
- Summary

# Significant variations in the Be sputtering yield are measured



discrepancy between - JET - PISCES-B - ion beam – TRIM - sputter yields
(< 45%) (< 0.4%) (< 8%) (< 3.5%)

R.P. Doerner et al. / Journal of Nuclear Materials 257 (1998) 51–58




J. Roth et al., FED 37(1997)465.

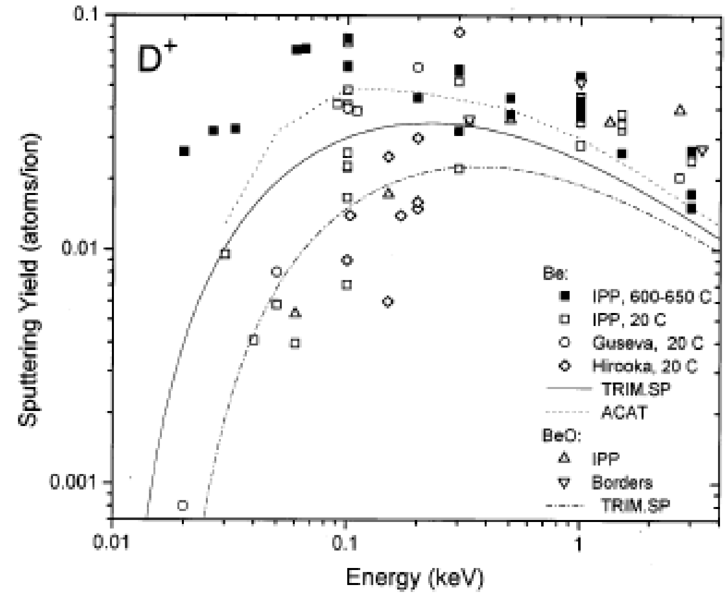
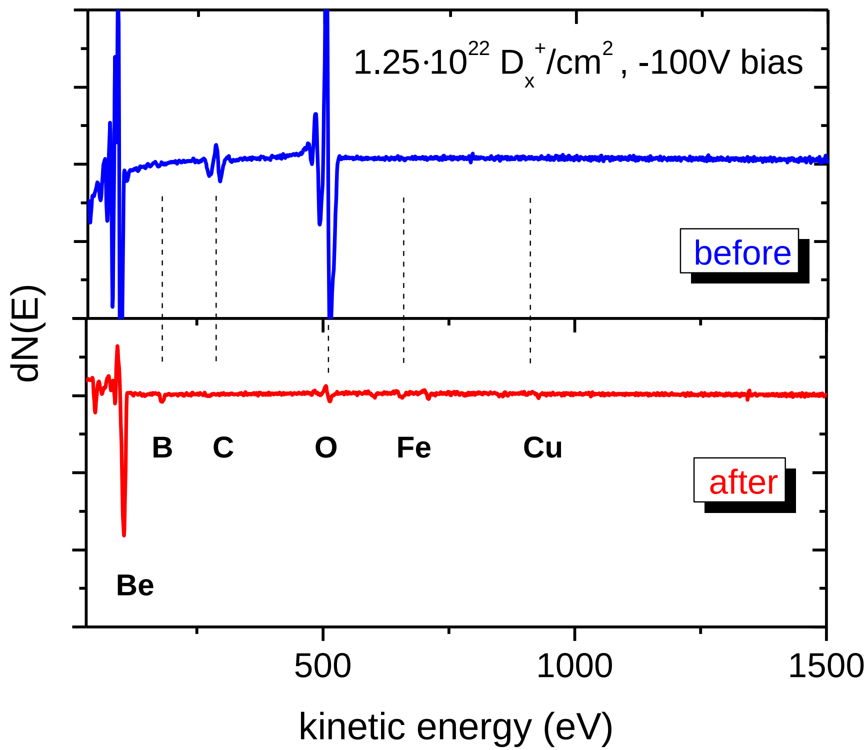
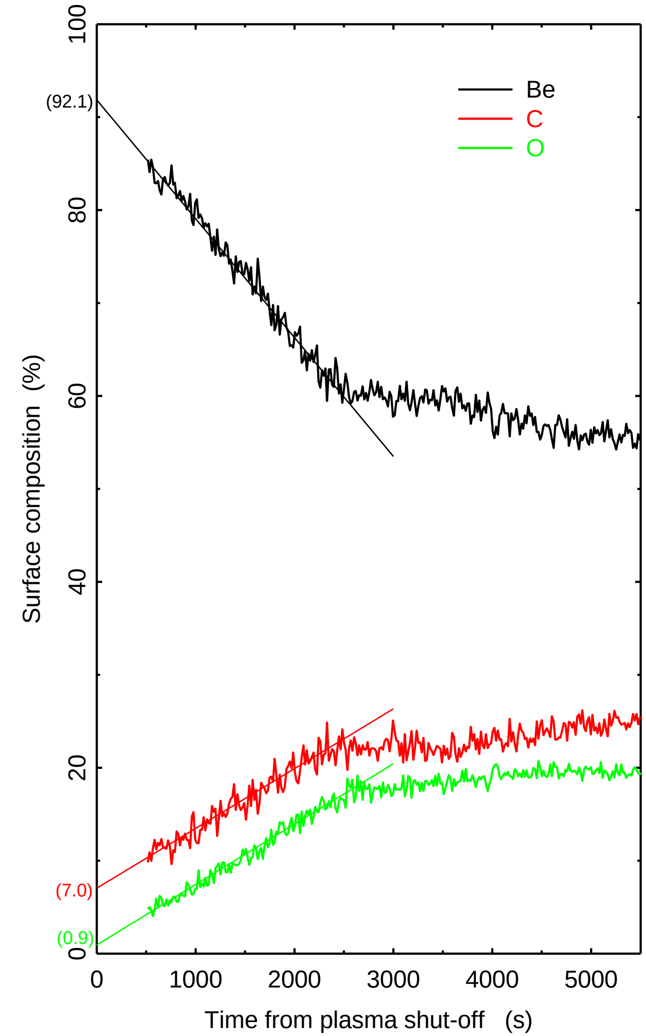



Fig. 2. Energy dependence of the sputtering yield of Be and BeO bombarded with D at normal incidence. Experimental data [6,13,23–26] and results obtained with computer simulation [6,22].

# AES reveals a relatively ‘clean’ Be surface during sputtering yield measurements

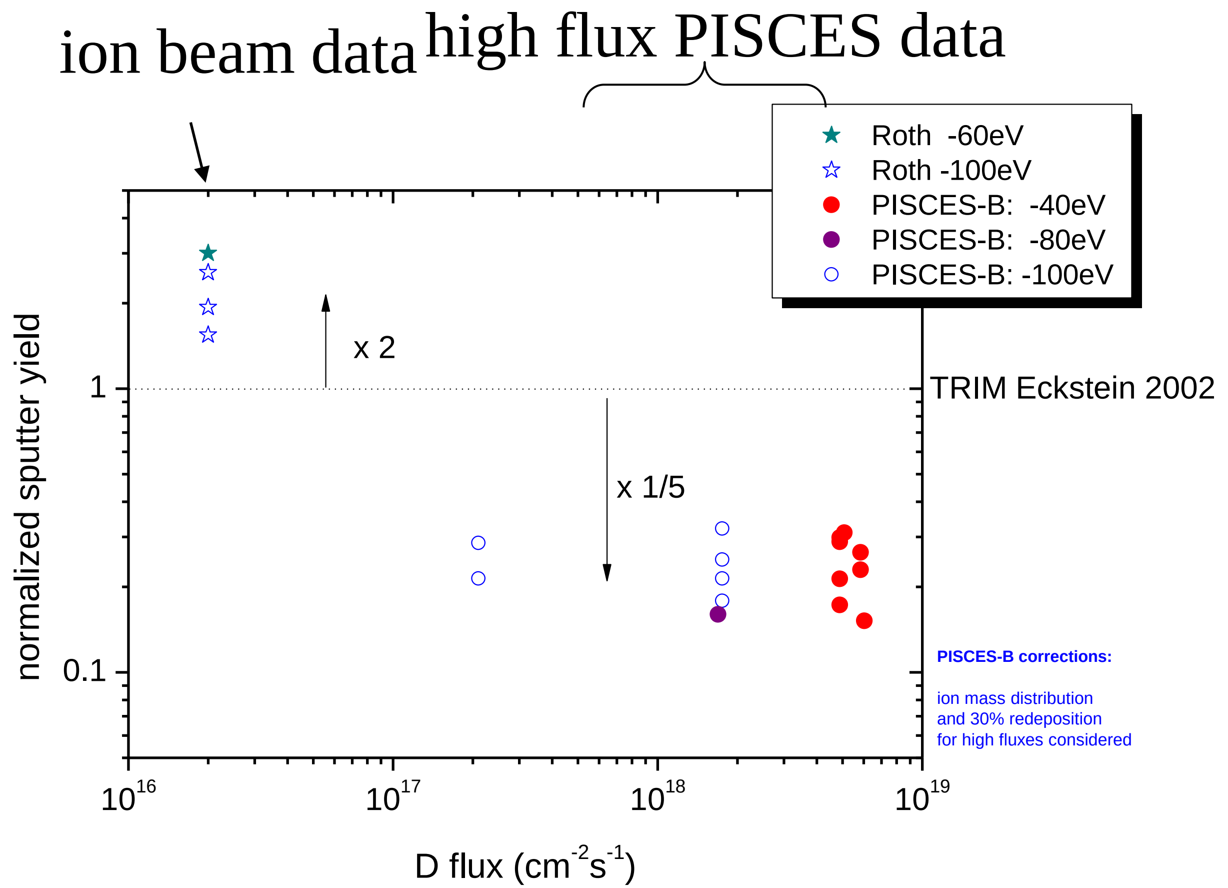
**PISCES**



# Sputtering of Be with D: discrepancy in total yield



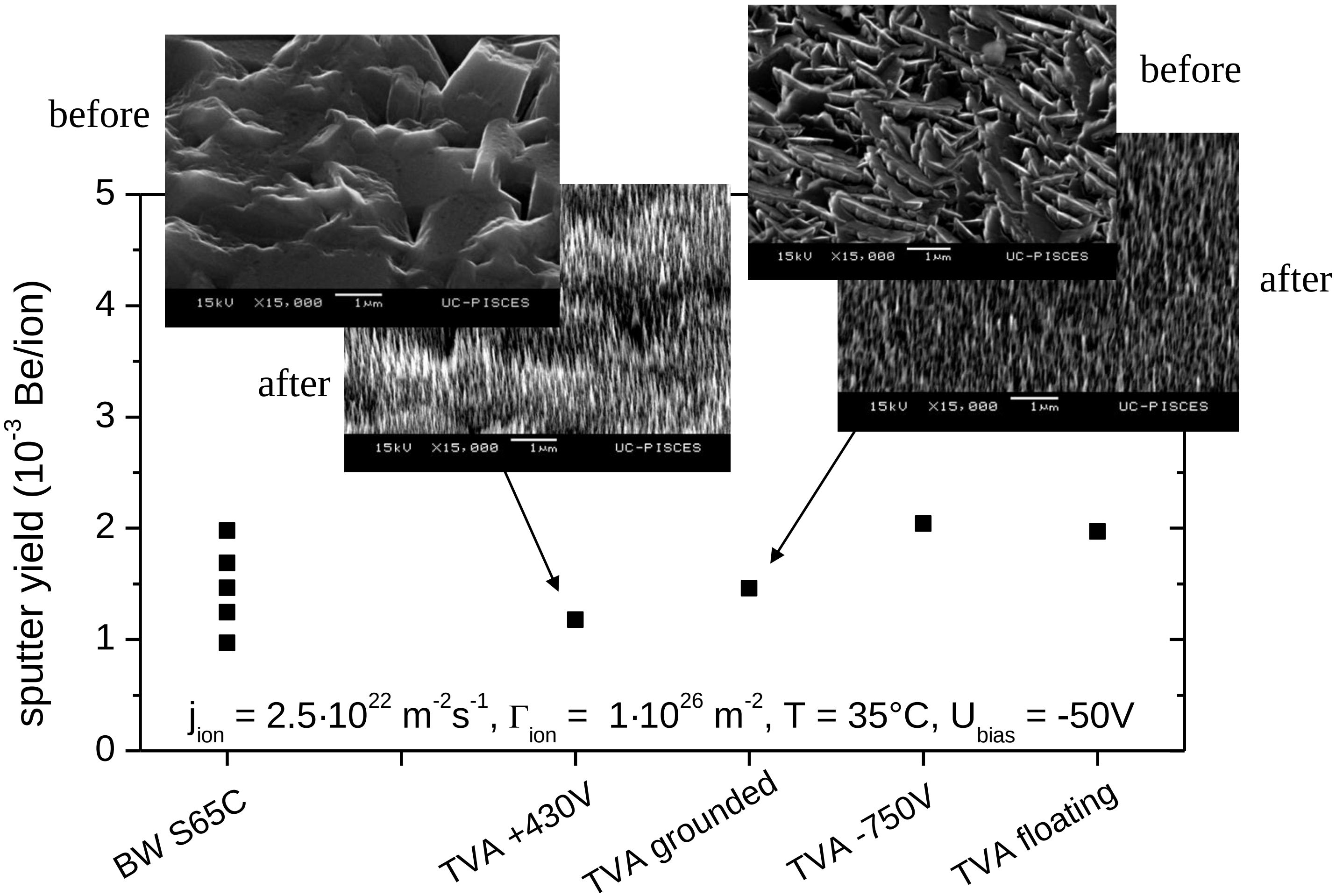



**High JET yield can be explained by angle, Be self-sputtering & impurities.**
**PISCES yields are a factor of 5-10 lower than TRIM. Why?**

# sputtering of Be: influence of initial surface morphology



D exposure -50V, < 330K: $j_D = 1 \cdot 10^{22}\ D^+/cm^2$

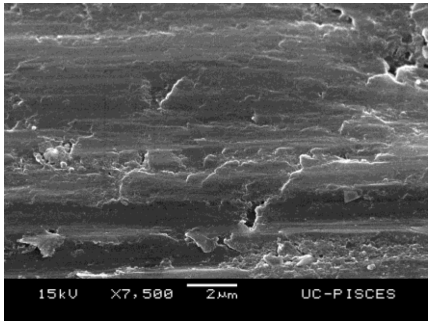



**⇒ no influence within accuracy of the measurement**

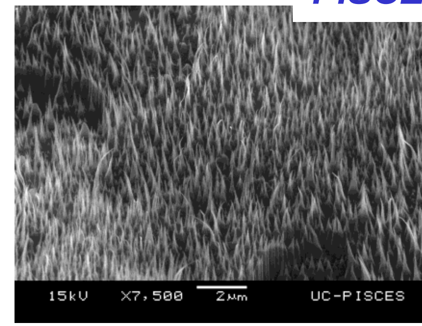
# sputtering yield: evolution with time / fluence



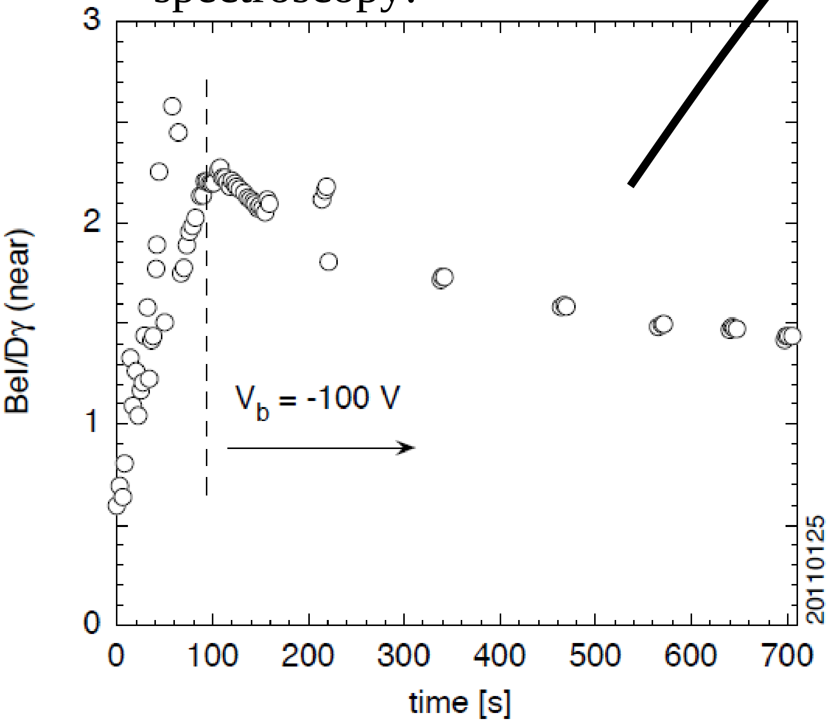
before



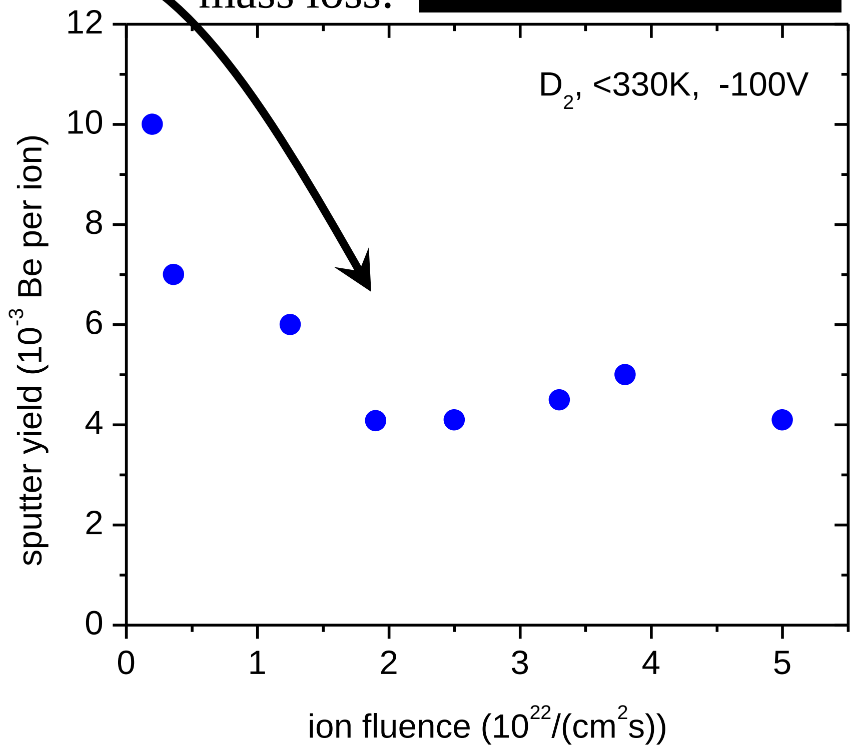

after




spectroscopy:




mass loss:

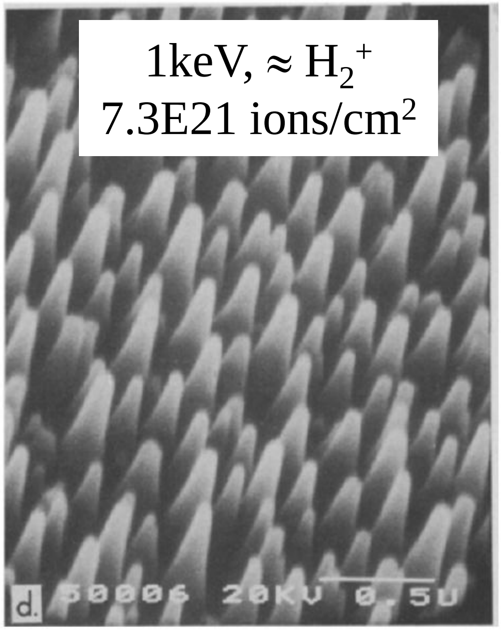


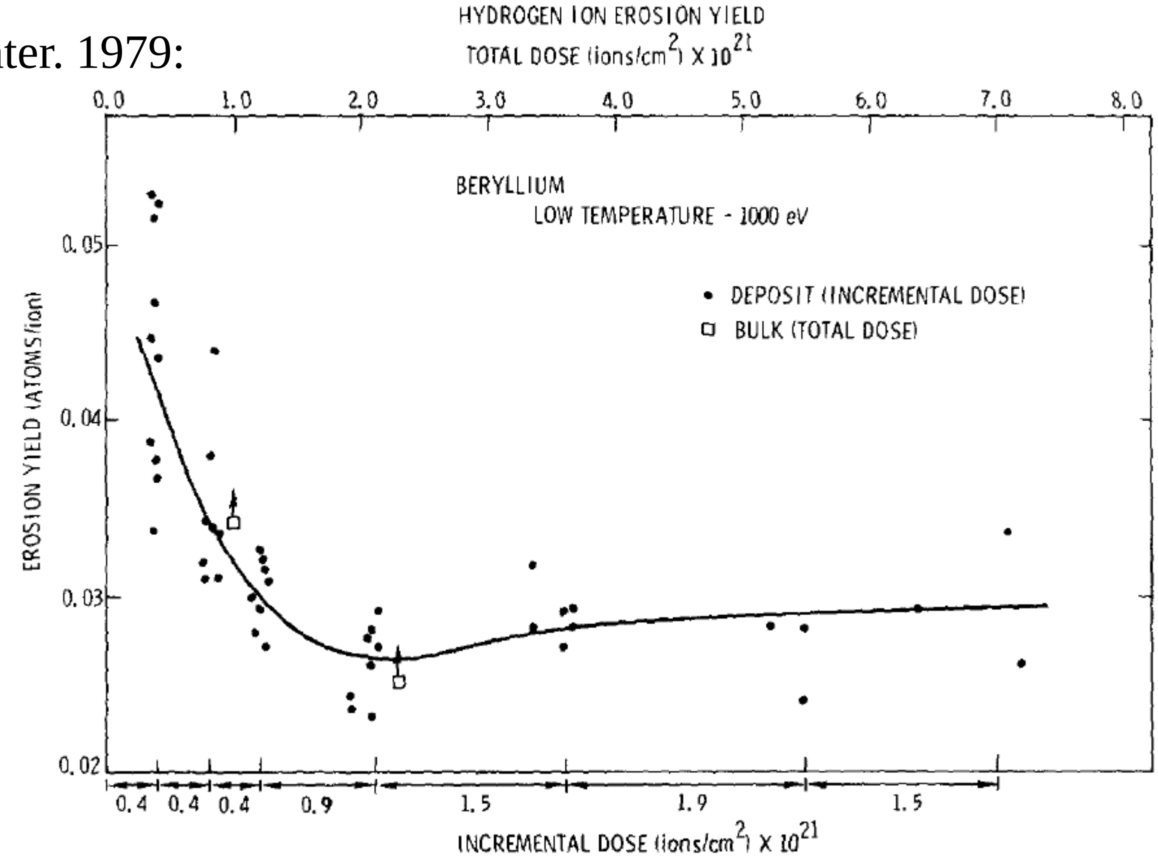

**⇒ morphology change can account for a factor of 2 in reduction of the yield**

# Similar yield evolution with time/fluence is documented in the literature



Mattox and Sharp, J. Nucl. Mater. 1979:

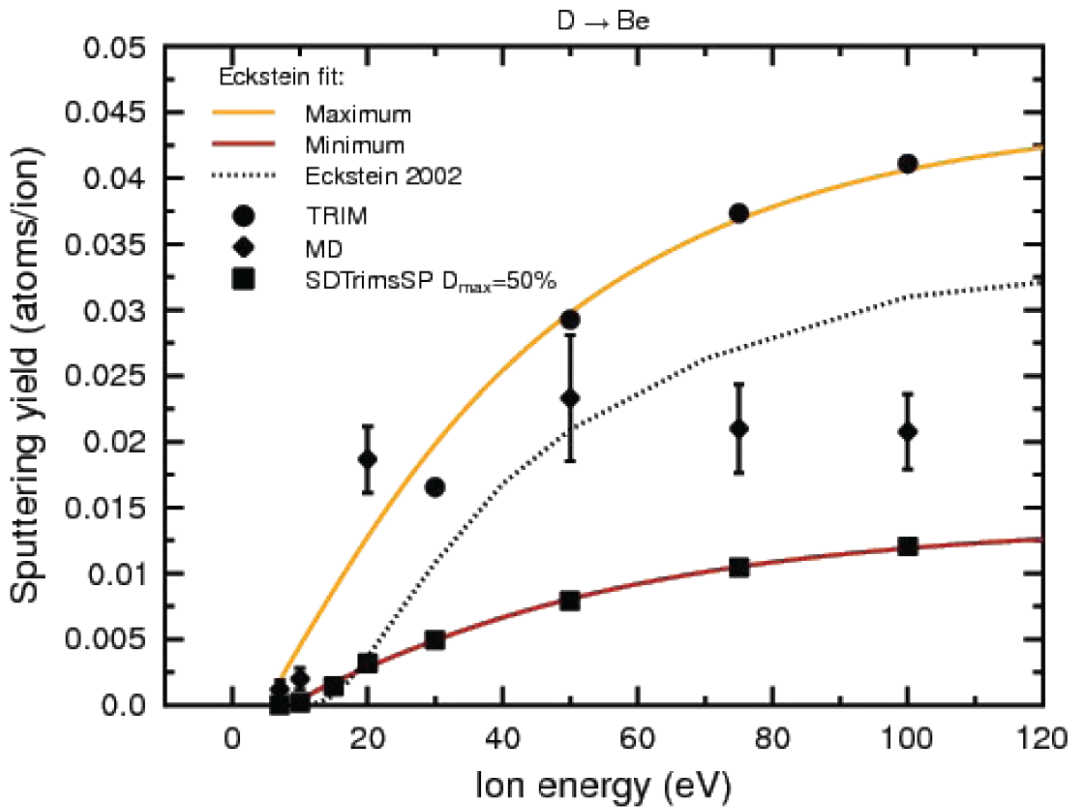



**⇒ morphology change can account for a factor of 2 reduction of the yield**

# Plasma atoms remaining in the near surface also can reduce the sputtering yield by a factor of 3-4





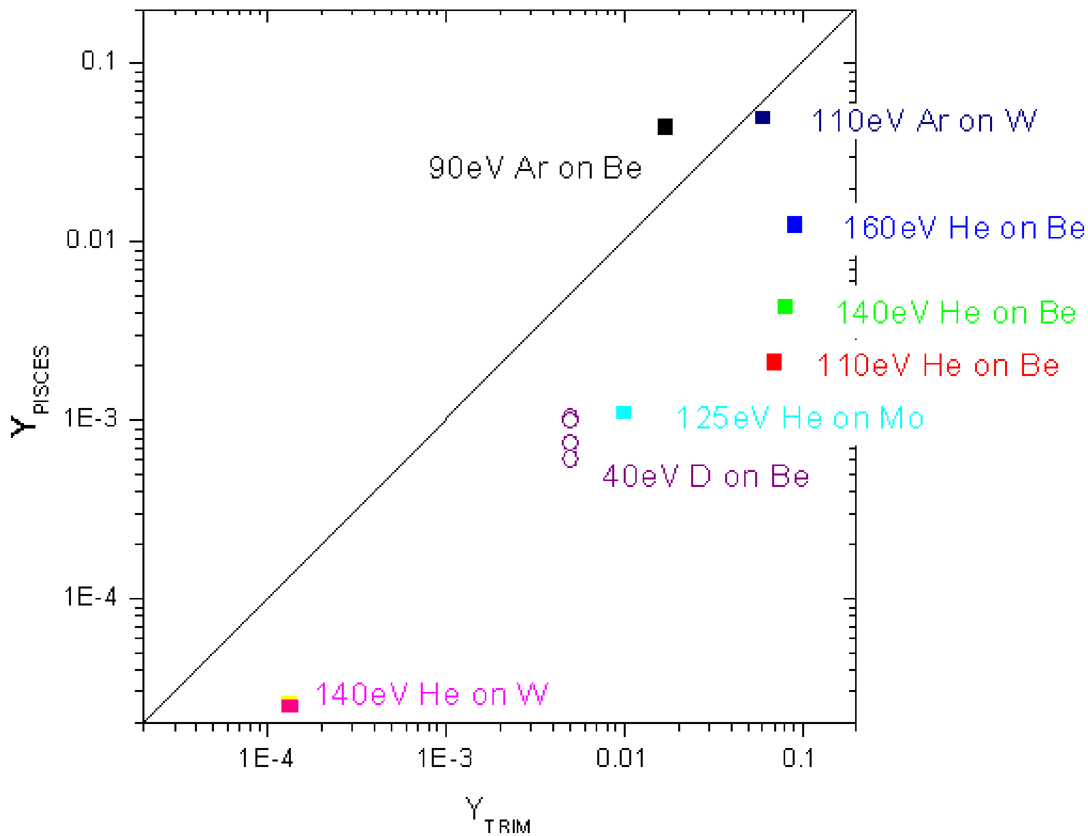

From C. Björkas

1) "maximum" – static TRIM + MD
2) "minimum" – SDTrimSP with 50% of D (reasonable limit)

# Heavy ion bombardment yield agrees with TRIM calculations



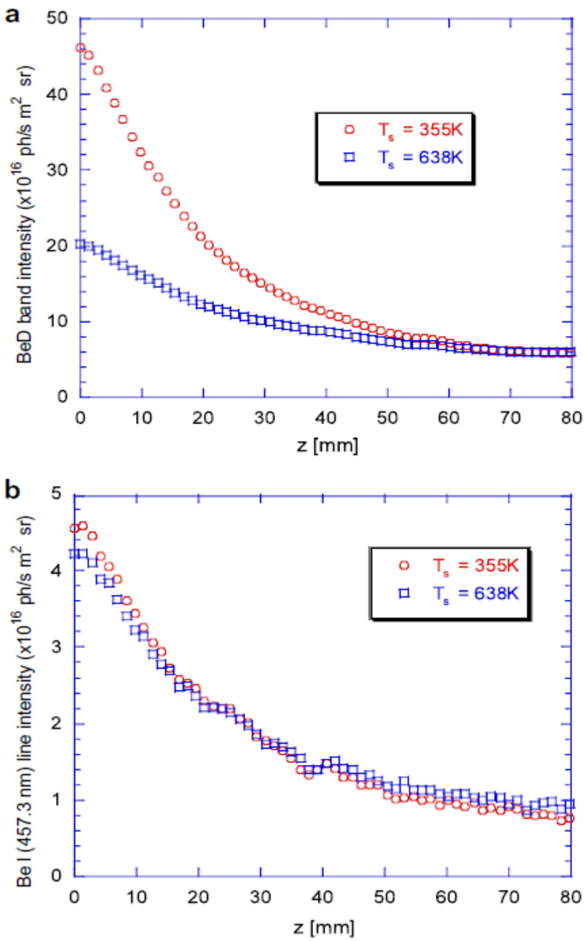
- Reason for this behavior is not understood
- Ar on Be results in smooth surface after sputtering
- Ar implantation depth is shorter
- Reflection coefficient of Ar is lower than He or D (more momentum directed into target)
- Ar diameter is larger, perhaps less likely to reside in the near surface region
- Effect is measured for a variety of substrate materials



# Chemically-assisted physical sputtering of BeD is temperature dependent

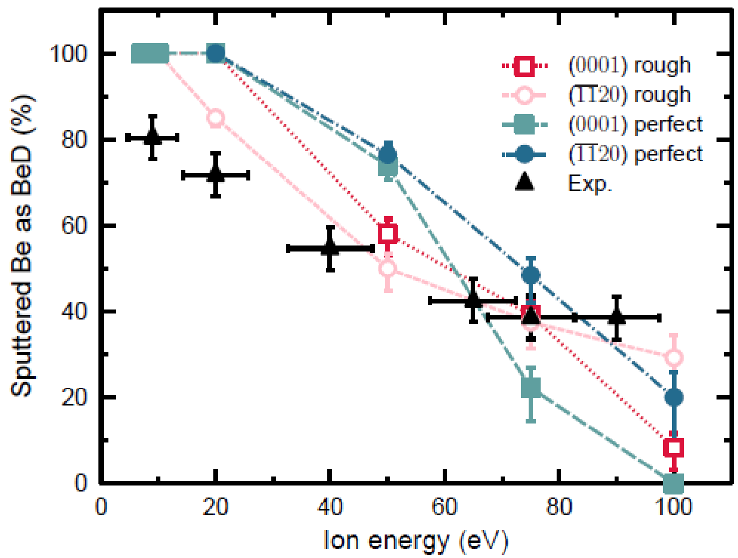


Similar e-folding distance of BeD and Be I intensity indicate BeD is physically sputtered, not chemically eroded. Beryllium deuteride is not volatile.




From R. Doerner et al, JNM 390-391 (2009) 681.

MD simulations of D on Be predicted subsequent erosion measurements

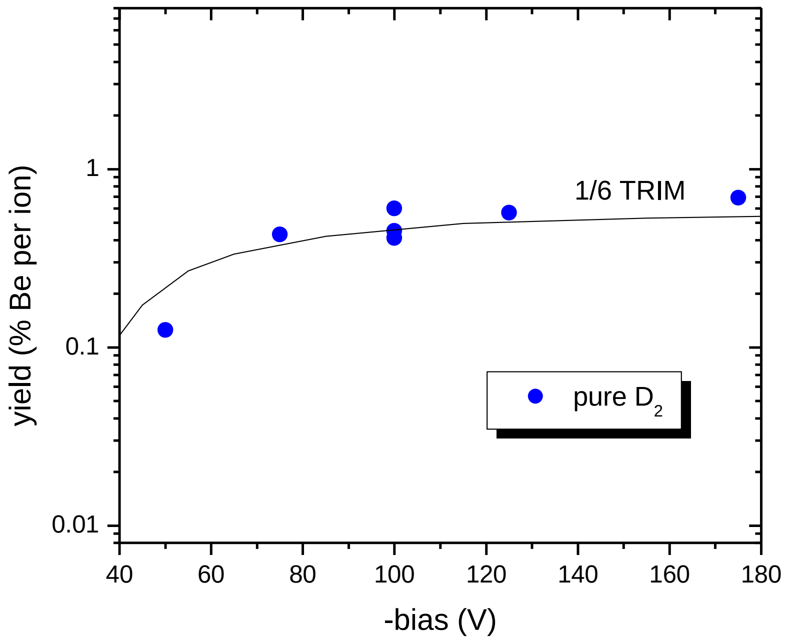



Exp. from D. Nishijima et al, PPCF 50(2008)125007.
Sim. from C. Bjorkas et al., New J. Physics (2009).

# Erosion/deposition balance in Be seeded high flux D discharges

*PISCES*

Mass loss



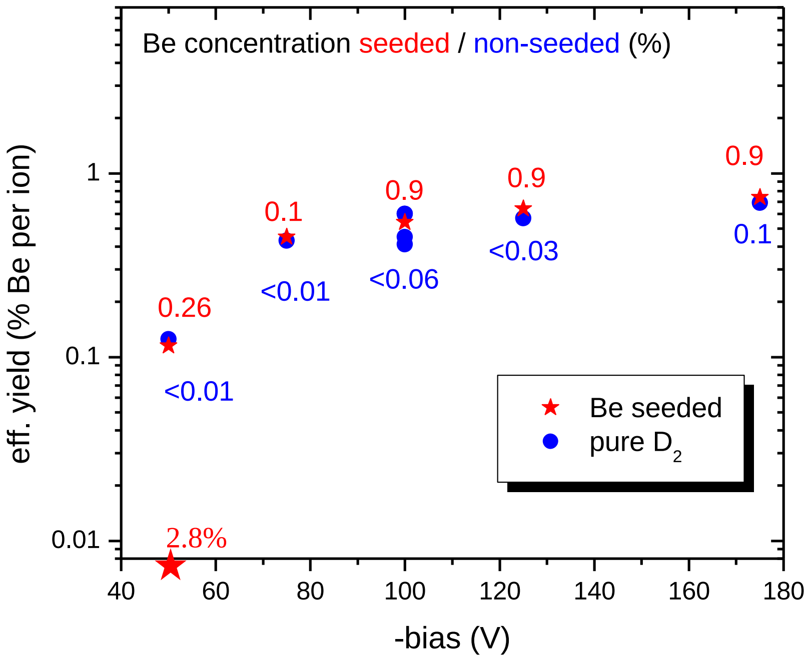

ion fluence: $\approx 10^{22}/cm^2$
target temperature < 320K

- Use Be oven seeding to balance surface erosion to test input parameters of material migration models
- Mass loss measures net erosion
- Spectroscopy measures gross erosion (Be I line)
- $Y_{Be \rightarrow Be} \approx Y_{D \rightarrow Be}$, and low concentration of Be
- When incident/seeded Be ion flux = sputtered flux of Be, net erosion should = 0.

# No change in mass loss is measured when Be seeding flux equals sputtering of Be by D

*PISCES*

Mass loss




ion fluence: ≈$10^{22}$/cm$^2$
D/Be plasma
target temperature < 320K

- ADAS database is used
- Be flux from Be II (313.1 nm) and background plasma flow velocity (E. Hollman JNM, PSI-19)
- Be ion flux is verified during no bias discharges, when weight gain is measured (net deposition)
- Net erosion stays constant, implying gross erosion must increase
- Erosion yield of 0.15% can only be compensated by seeding 2.8% Be

# Beryllium seeded He discharges

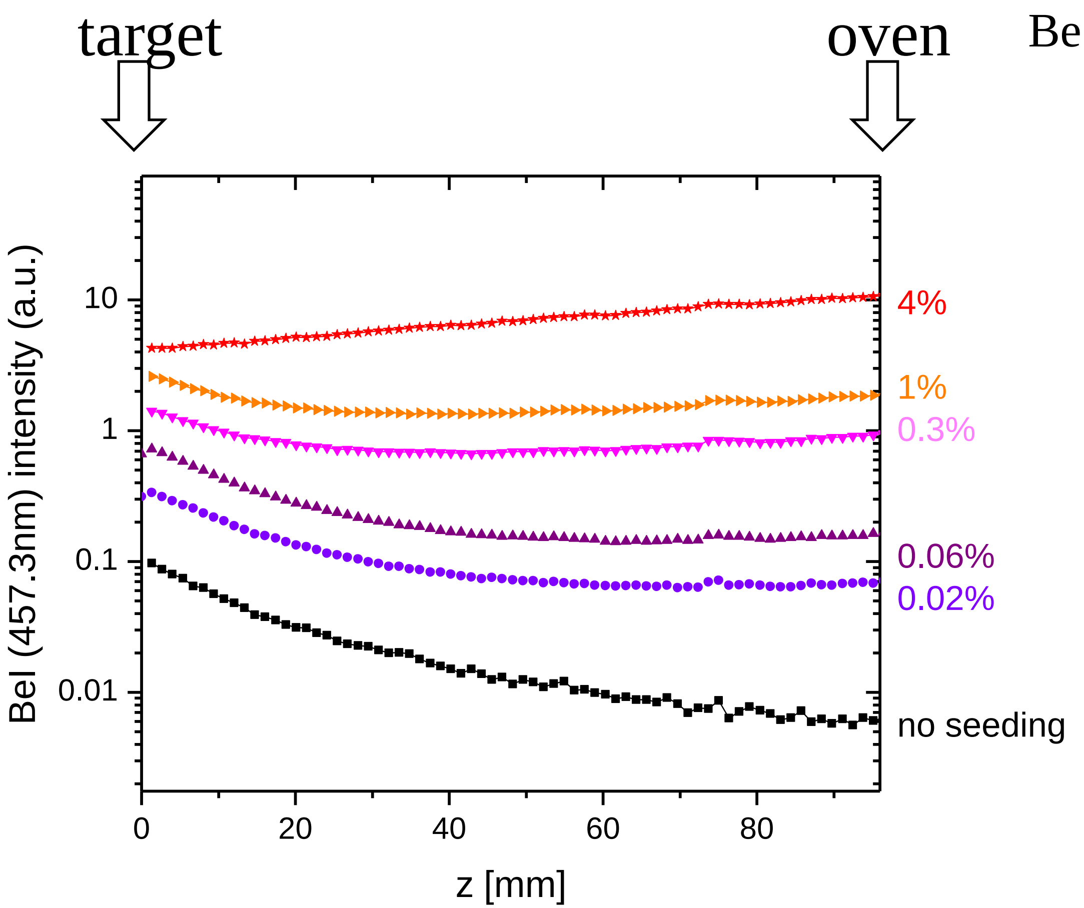


<u>target:</u>

bias: < -40V results in E ≈ 30eV

He ion flux: $5 \cdot 10^{18}$ $cm^{-2}s^{-1}$

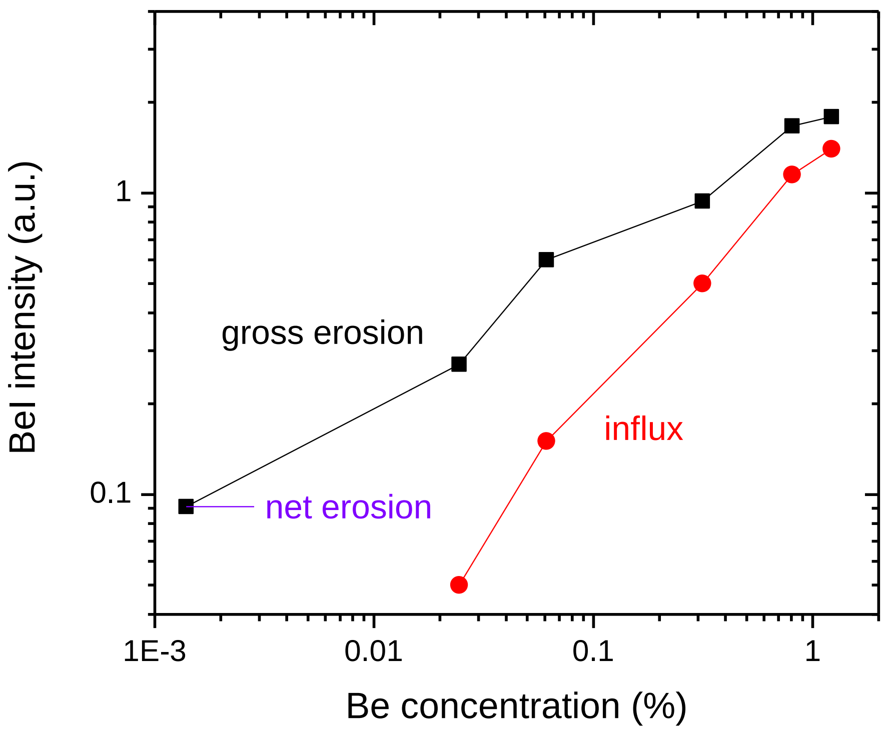
Be seeding: $n_{Be}/n_D = 0 – 4$ %




sputter yield He on Be @ 30eV:
Y = 0.15 %

# Gross erosion increases with increasing Be influx

PISCES



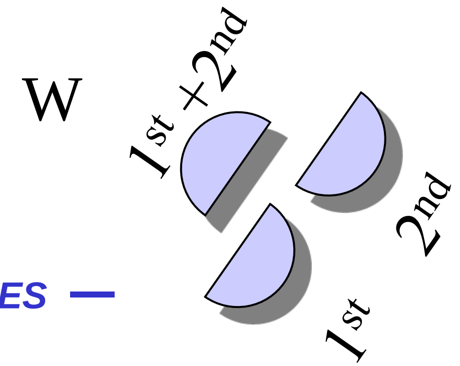

- Gross = net erosion with no Be seeding ($\lambda_{ion}$ is large compared to $r_{plasma}$, so redeposition is small)
- Gross erosion increases as Be influx increases, leaving net erosion unchanged
- At large enough Be influx, net erosion begins to decrease and eventually net deposition occurs
- This implies either low sticking coefficient or high re-erosion of Be influx

# Retention and release of D from Be

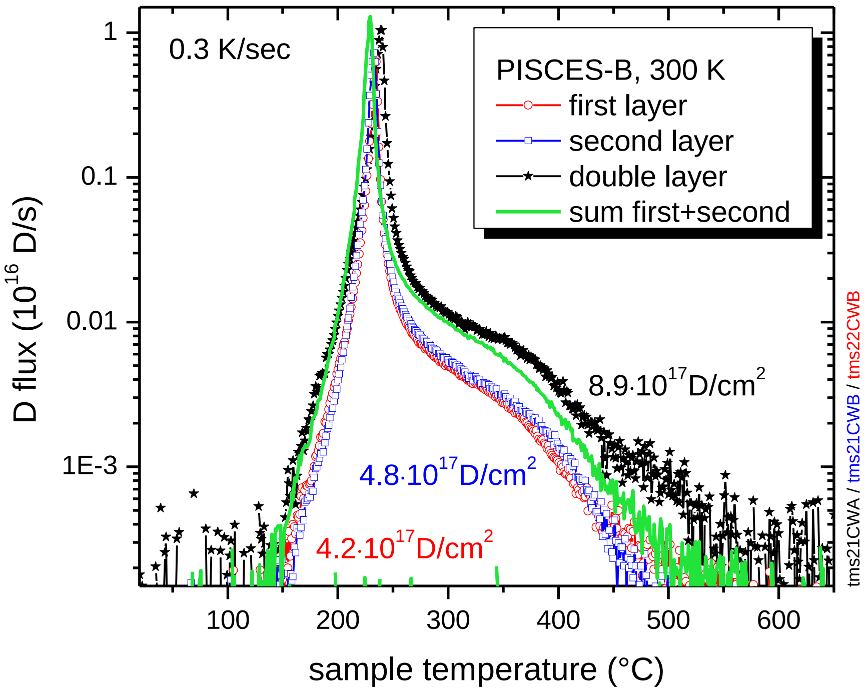


- Quantity retained in plasma exposed surface saturates at low levels ~ $10^{21}$ $m^{-2}$
- Retention in thick BeO layers is not well defined
- In-vessel accumulation will be dominated by codeposition, and hence depends on erosion rates of Be
- Retention in codeposits shows Arrhenius relation, between RT and 300$^{o}$C (is higher temp data needed?)
- Release behavior still has some uncertainties
  - Long term baking
  - Release during fast heating events
  - BeO release

# Do BeO layers influence the D release?




PISCES




- Two Be codeposits were collected while venting to replacing one half the sample between codepostion runs
- Several nm thick BeO will exist between subsequent codeposits
- Release behavior of the multilayer codeposit is almost identical to the sum of the individual codeposits
- Conclusion is that internal BeO layers will not impact the knowledge gained from studying pure Be codeposits

# D retention in clean Be (UHV subML O coverage)



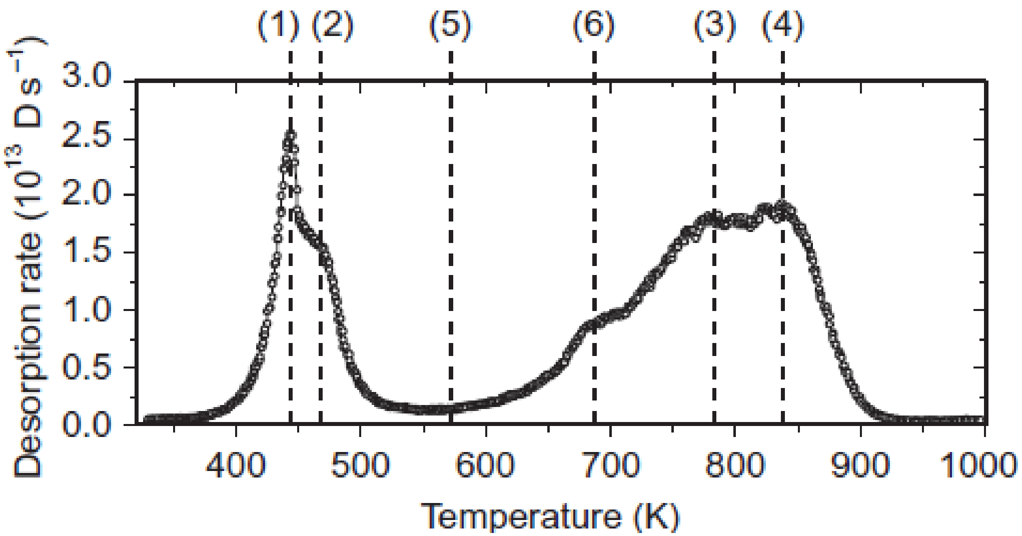
Reinelt et al. New Journal of Physics 11 (2009)
Oberkofler et al. Nuc.l Instrum. Methods 267 (4) (2009)

low flux/fluence implantation at 0.3-3keV with $10^{15}$D cm$^{-2}$ s$^{-1}$:

structural modifications, supersaturated ($D_2$ in bubbles / amorphous Be-D, D/Be up to 1)

defects created by the implantation cascade (vacancies)

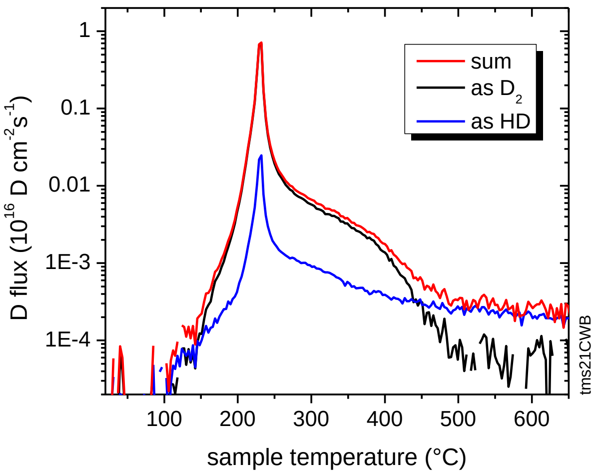
Be-$D_2$ BeO

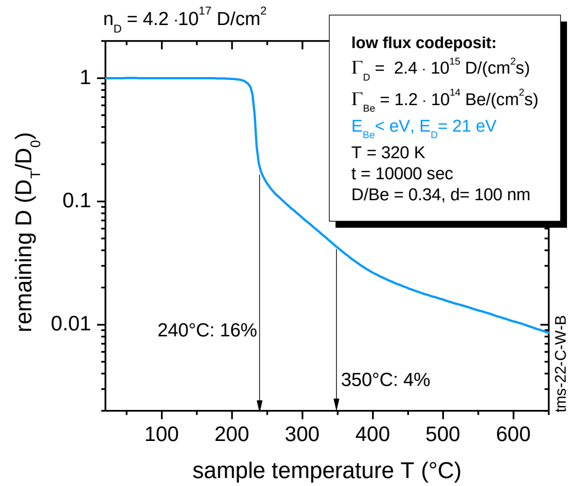


# Codeposits grown in different ways (energies, growth rates) show similar release features

**PISCES**

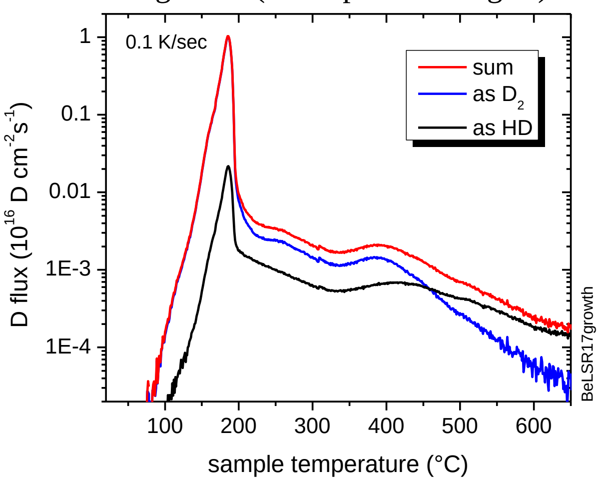
Low flux (witness plate)

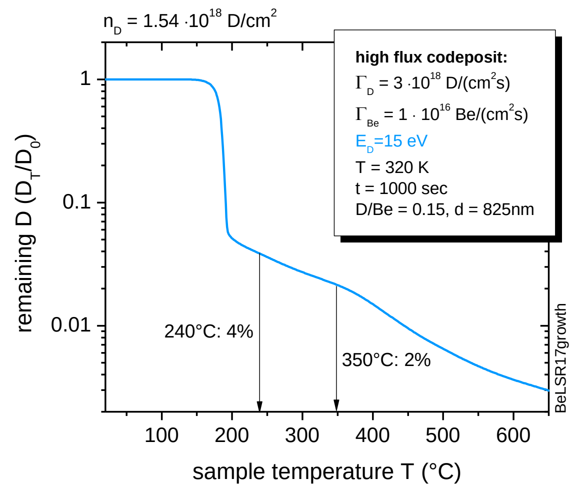



In both cases, D/Be retained in the codeposit after 350ºC is ~ 0.01

High flux (net deposition targets)

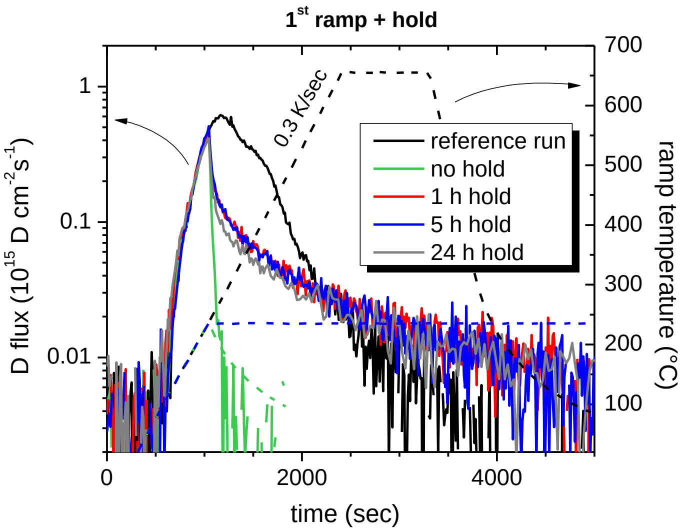


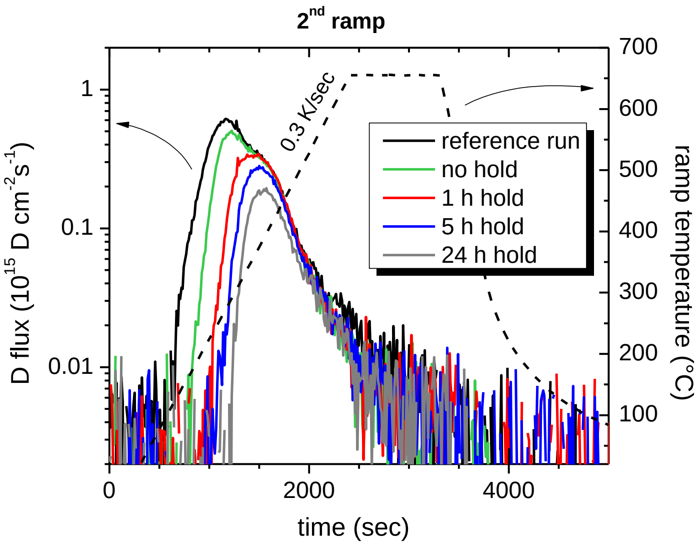



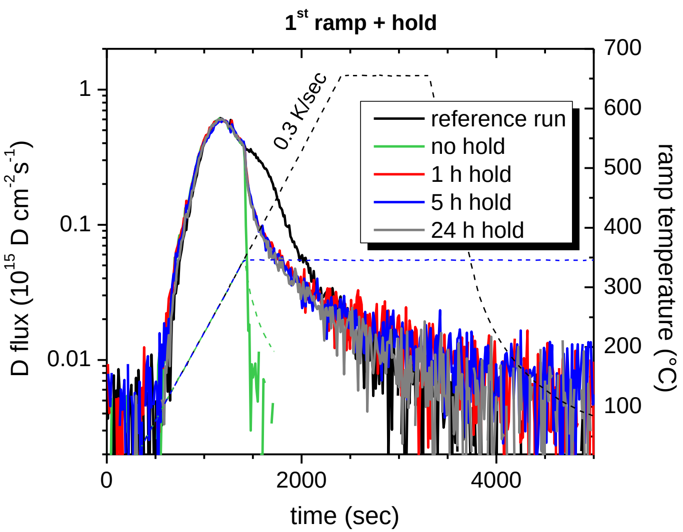

This value also holds for net erosion targets and codeposits made by D/Ar sputtered magnetron targets

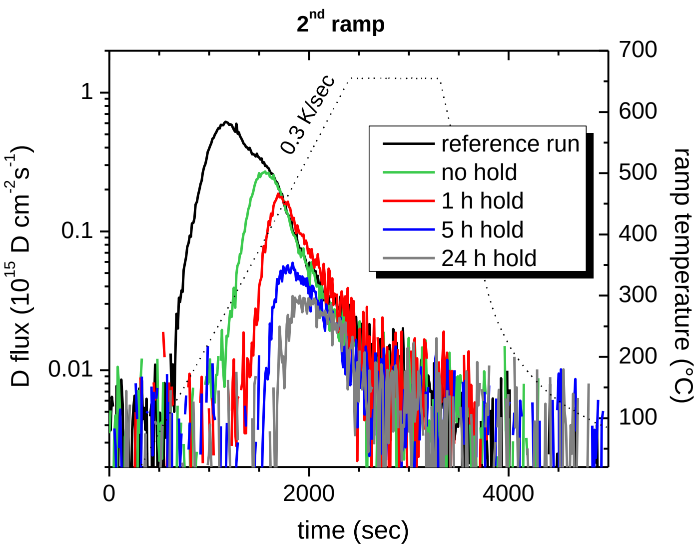
# D release from magnetron sputtered D/Be during long hold baking

PISCES

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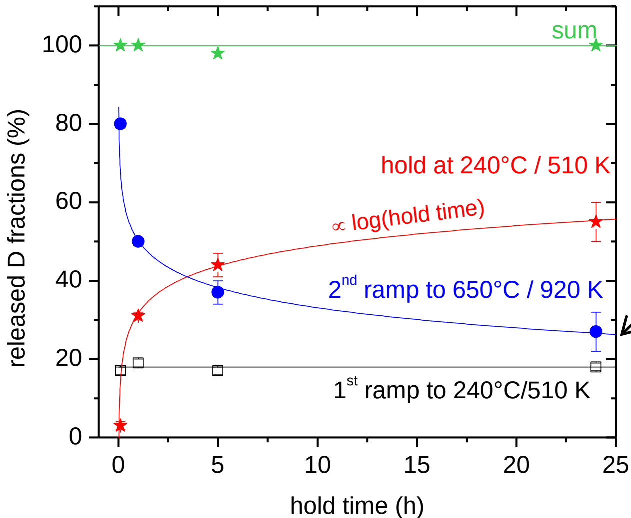
# Slow release (time constant of hours) is detected during long term baking of codeposits

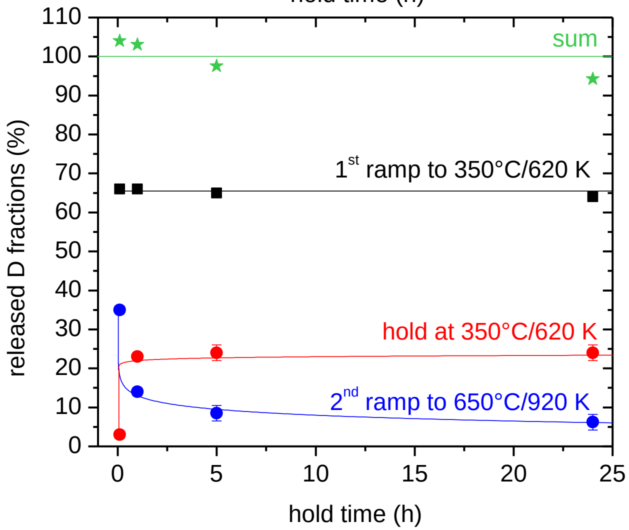
PISCES




After long term bake @ 240ºC (first wall bake temperature)

$1.0 \cdot 10^{17}$ D $cm^{-2}$ remains after hold

or: D/Be = 0.8%




After long term bake @ 350ºC (divertor bake temperature)

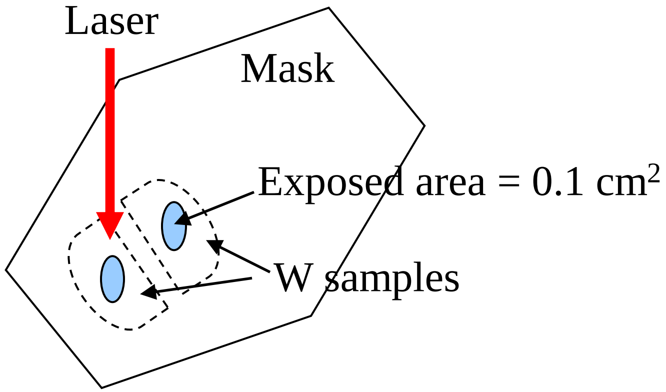
$2.5 \cdot 10^{16}$ D $cm^{-2}$ remains after hold

or: D/Be = 0.2%

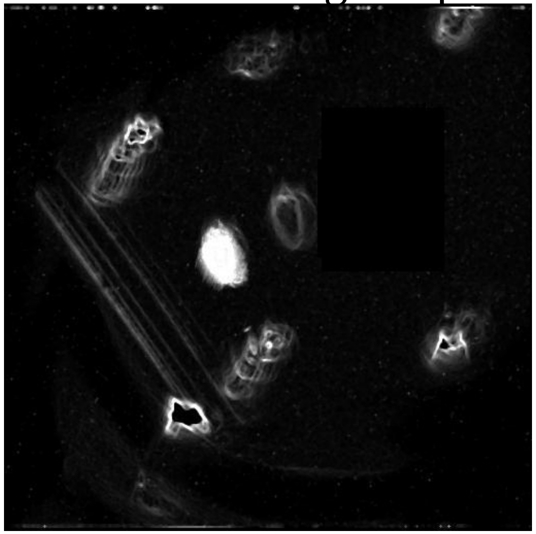
# Measure the effectiveness of various heat pulses associated with radiative plasma termination, as a means of tritium removal from Be codeposits in ITER.



- Side-by-side codeposits are created in PISCES-B, one is then flashed, and TDS of both are compared
- A 50 J laser (@1064 nm) is used to vary the temperature of a Be codeposit formed in PISCES-B.
- Laser has a variable pulse length (up to 10 msec), power and pulse shape
- 4-color pyrometer to measure surface temperature



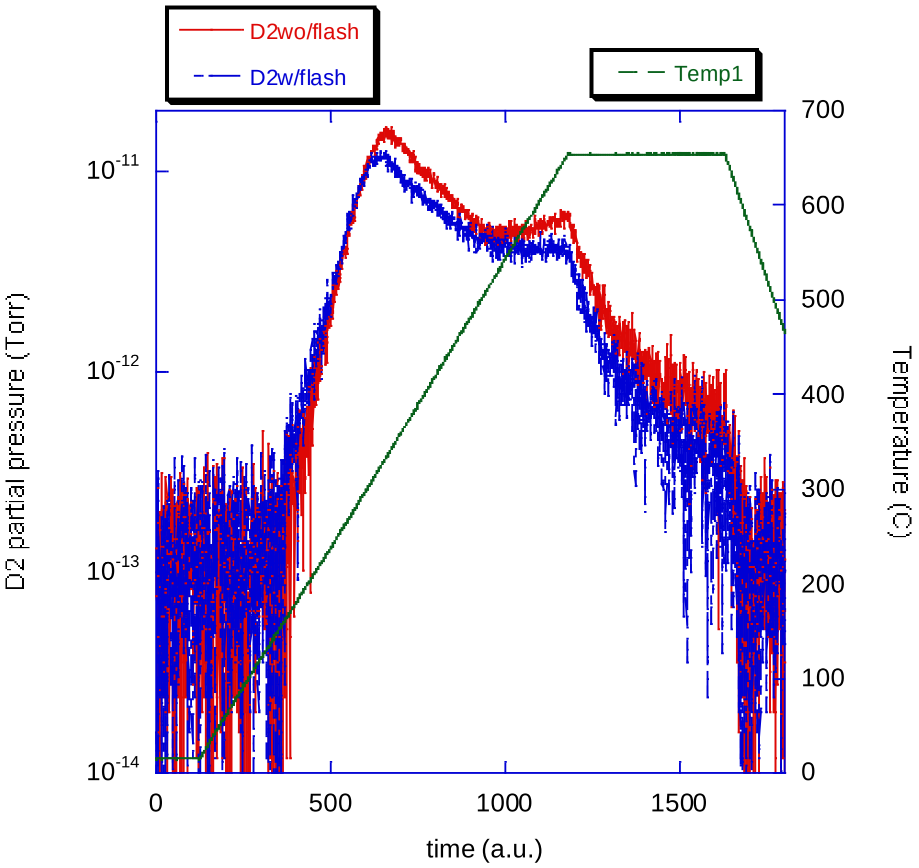

Laser hitting sample



# Flash heating of Be codeposits results in little release of retained D

PISCES

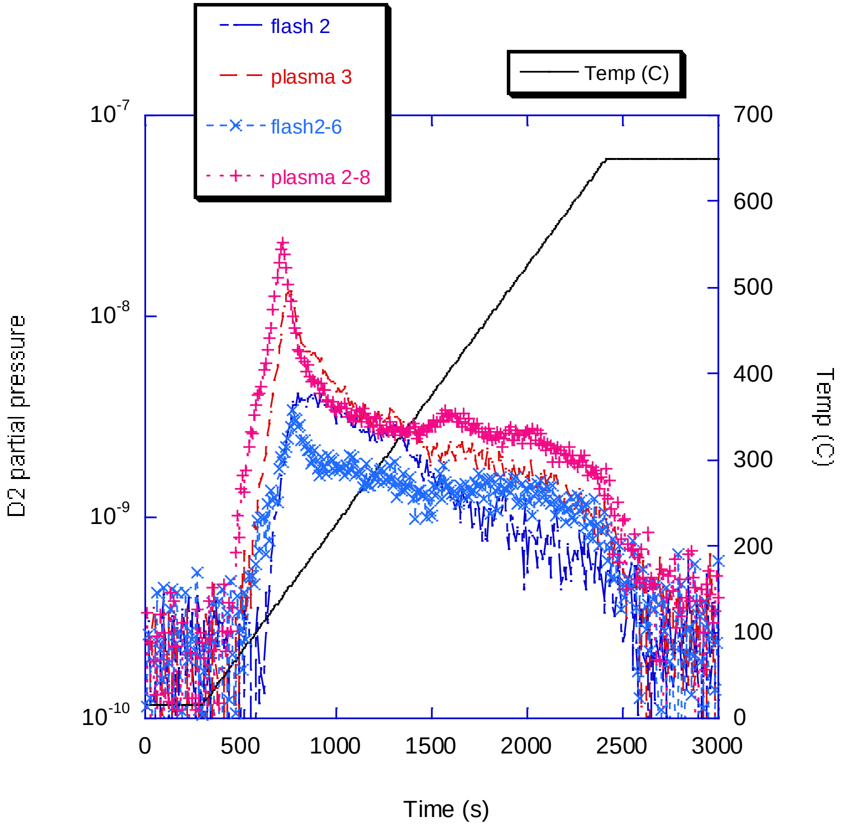
- $\Delta T$ is measured to be ~1000 K
- Shape of release curve is nearly identical
- Integrated retention in flashed codeposit is 80% of retention in un-flashed codeposit
- Consistent with Keroack & Terreault JNM 1994.



# However, TDS of flashed Be target exposures do show reduction in retention

PISCES

- Retention decreases by ~50% due to laser flash after D plasma exposure at ~50°C ($\Delta T \sim 450$°C)
- Low temp release peak is reduced, so may just make vessel baking less effective in ITER
- Similar flash after exposure at 200°C reduces retention by 25%, primarily lower temperature release peak

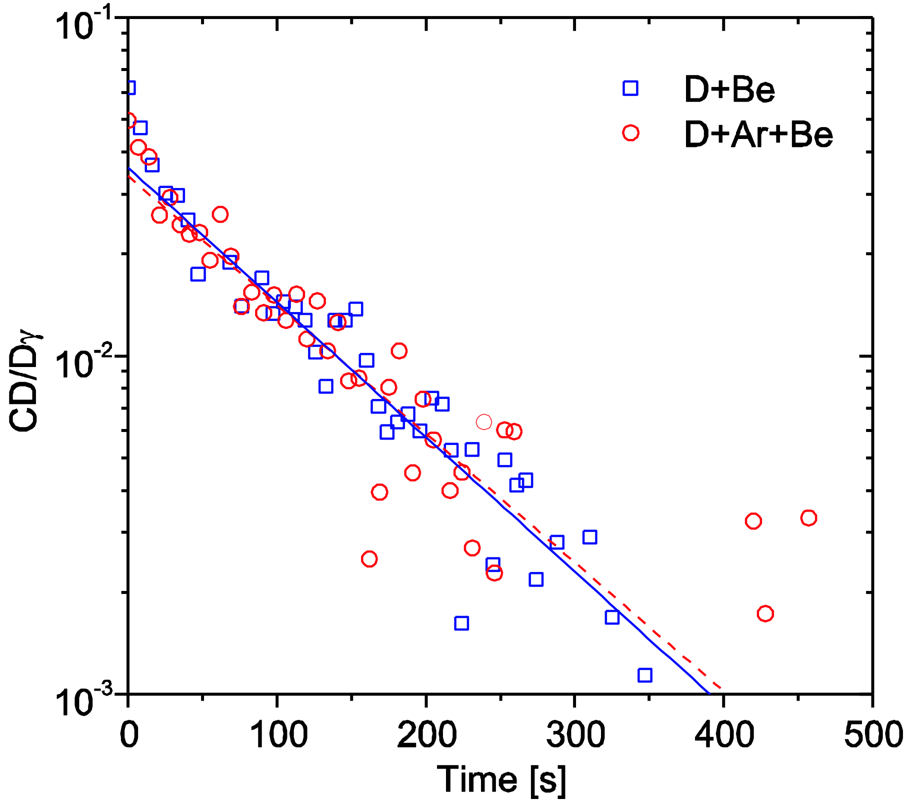


# Be-C experiments

# Argon sputtering does not remove the beneficial effect of Be mitigation of carbon chemical erosion



-50V bias, D plasma, 0.2% $Be^{+}$, 0 or 10% $Ar^{+}$




From A. Kreter et al., JNM (2009)

- Chemical erosion (CD band emission) is mitigated during Be-containing plasma interaction with graphite surfaces due to $Be_2C$ layer formation
- ITER will need to inject a radiating species (such as Ar) into the divertor to detach strike point without carbon radiation
- 10% Ar in incident plasma does not effect formation of $Be_2C$ surface layer
- Chemical erosion mitigation is unaffected by Ar sputtering

# Be-W experiments

# Stable Be-W alloys are known and have melting points closer to that of Be than W.

PISCES

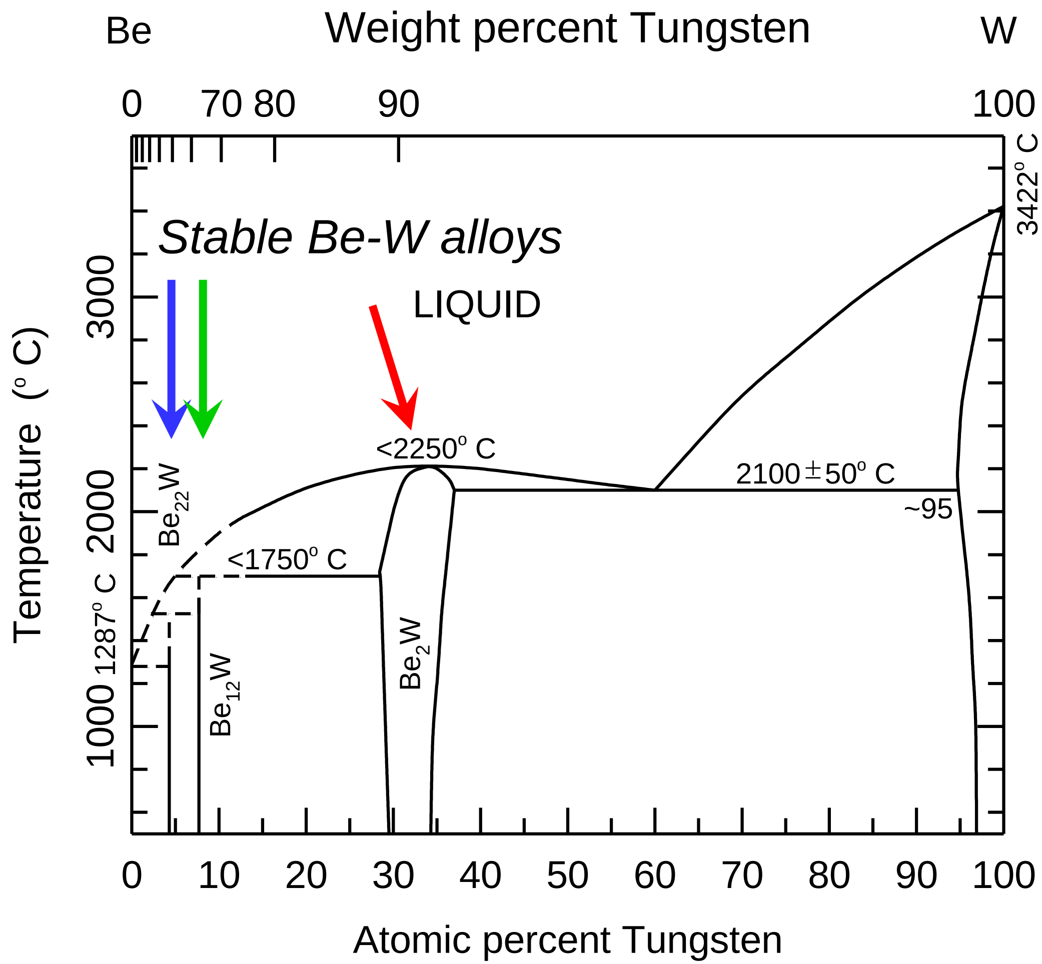
- Stable Be-W inter-metallics are:

~2200°C ($Be_2W$)

~1500°C ($Be_{12}W$)

~1300°C ($Be_{22}W$)

- Supply of Be to hot W surfaces will likely limit growth rates

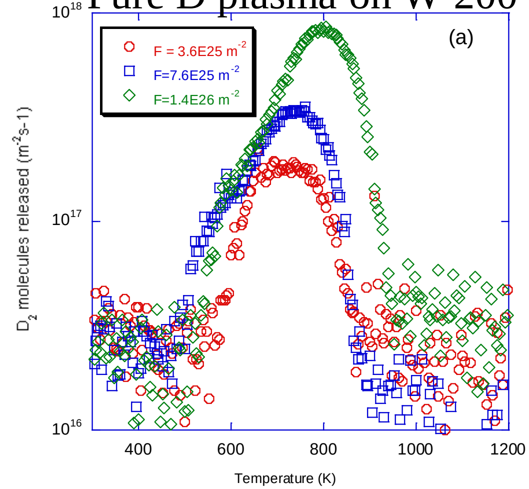


# Thin $Be_2W$ surface layer does not drastically increase retention in W

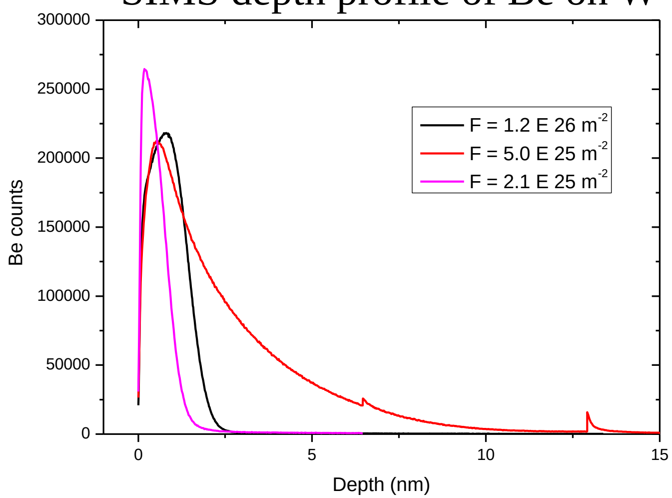
*PISCES*

A thin (few nm) $Be_2W$ layer forms on the surface of W exposed to D+Be plasma when the Be flux is smaller than erosion, but layer does not act as a permeation barrier to prevent release of implanted D

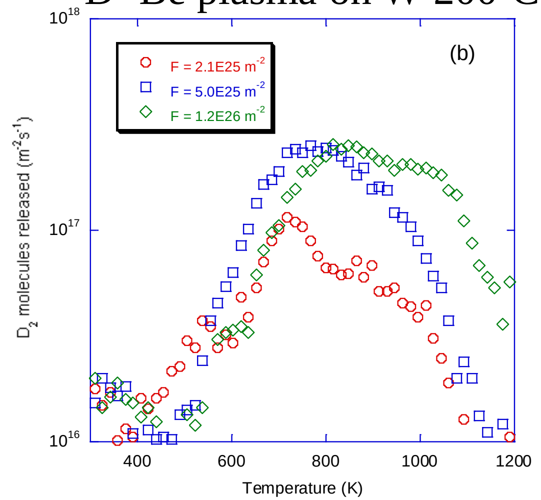
Pure D plasma on W-200ºC




SIMS depth profile of Be on W




D+Be plasma on W-200ºC

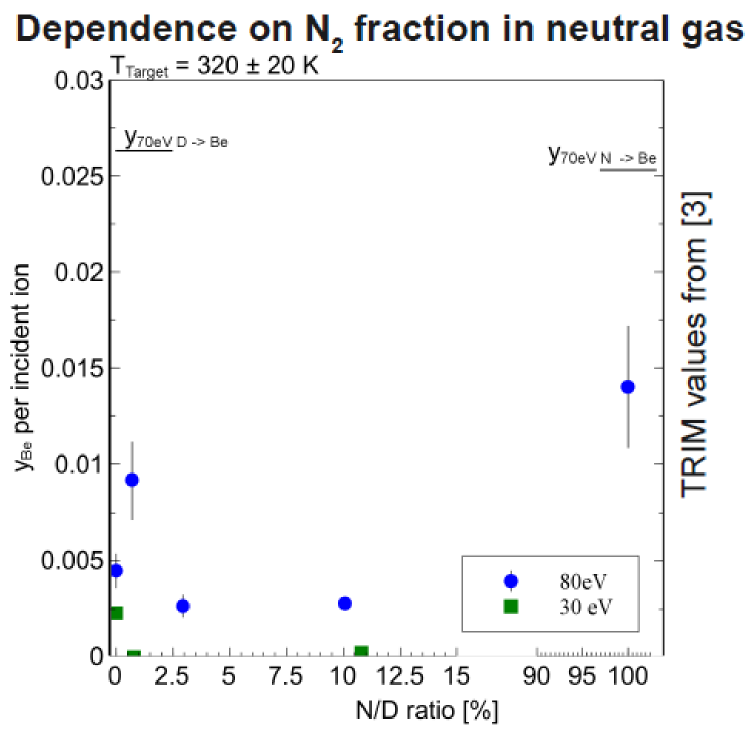


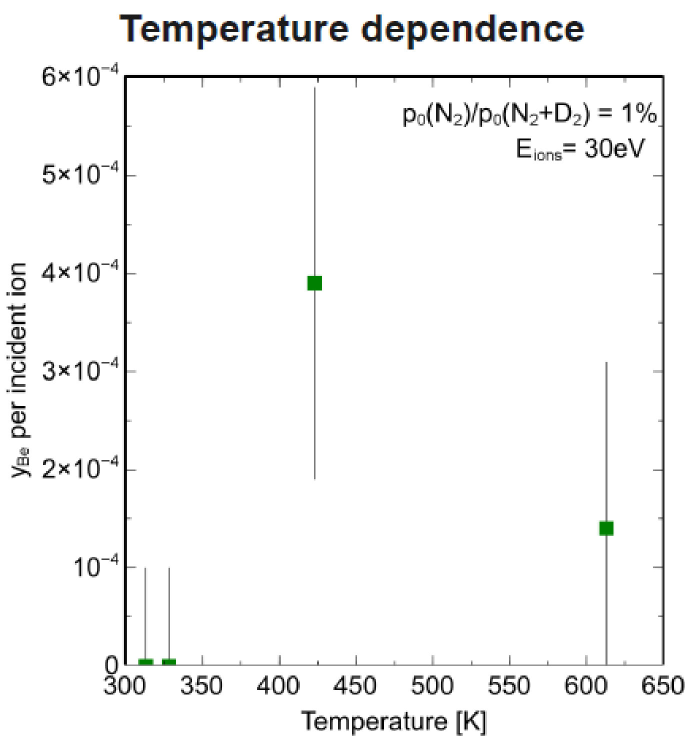

UCSD
University of California San Diego

# Be – N experiments

# Sputter yields are generally lower during D/N plasma, but temperature behavior suggests chemical activity of the surface is important

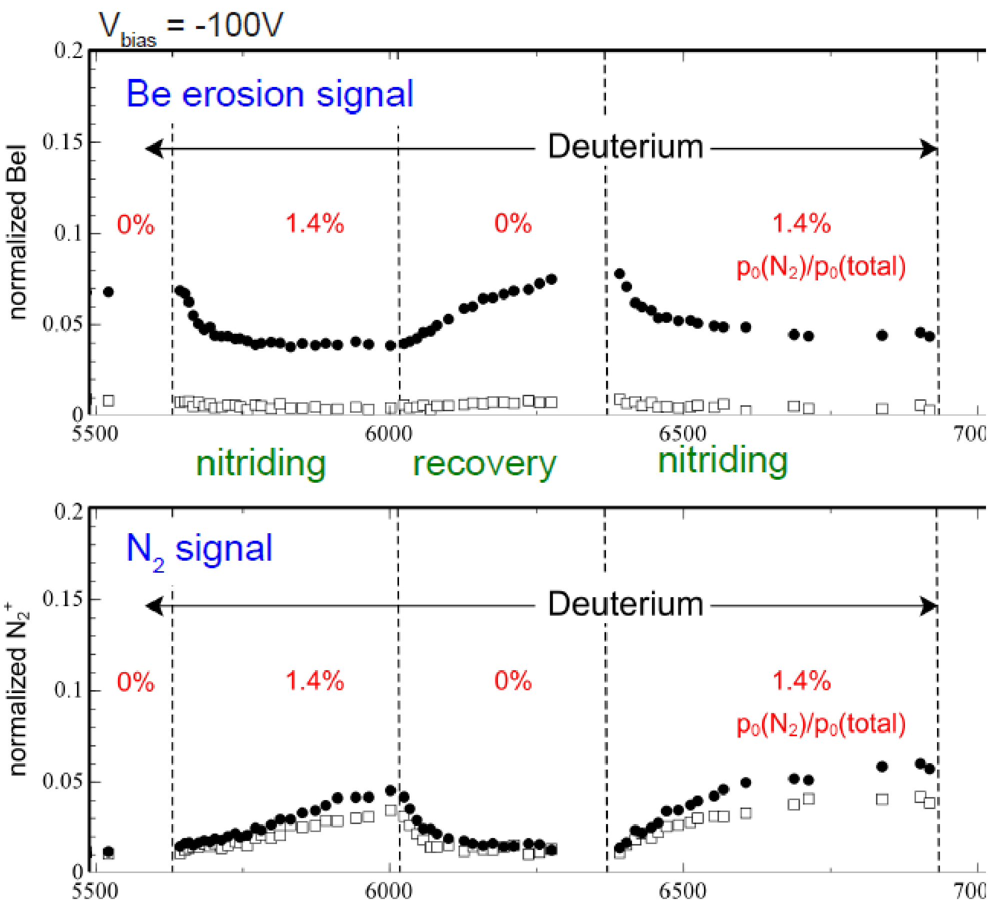
PISCES



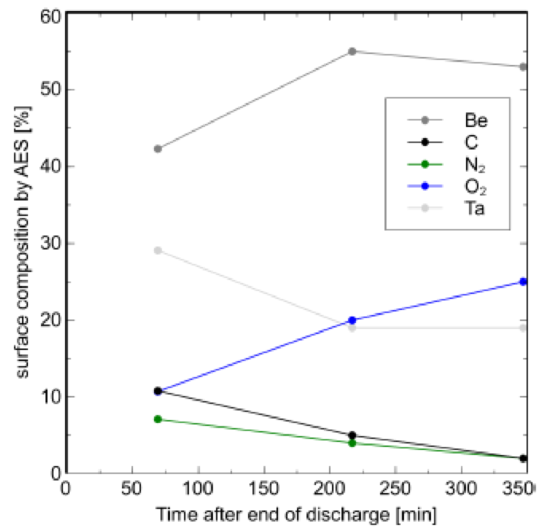



# Recovery from a nitrided Be surface appears possible, oxygen in background gas competes with surface nitride over time

PISCES




time dependence of surface composition after $D_2$&He discharge




1% $N_2$ , D& He plasma, Ta cap

Switchover from nitride to oxide surface also reported by Oberkofler NF 50(2010) 125001.

# Be-N summary from T. Dittmar PFMC poster

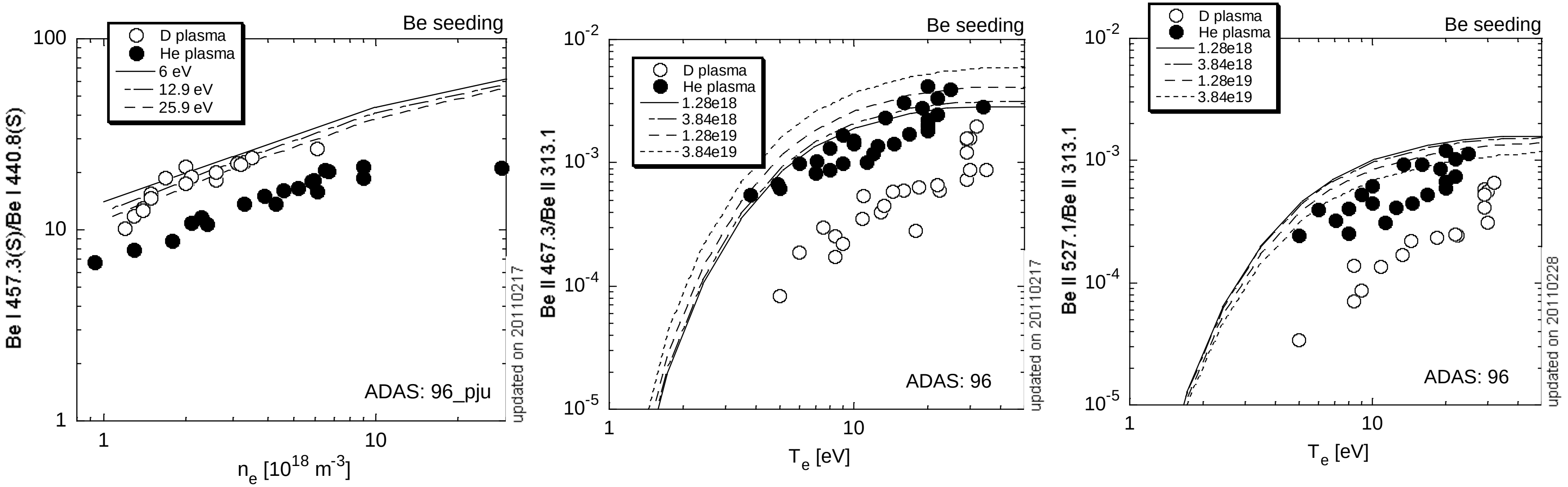


## Conclusions

- Running $N_2$ containing plasmas in the Be environment of PISCES-B is possible without severe consequences regarding machine operation
- Recovery from nitrogen loaded surfaces:
  - possible by prolonged D operation, but not in or after He plasmas
  - in vacuo oxidation of Be can displace/cover nitrided surface
- Chemical processes, probably involving $ND_3$ and $ND_x$ radicals, play significant role in nitriding and recovery.
- Retention:
  - "deuterated" and nitrided target samples show similar $D_2$ retention
  - Potentially less $D_2$ & HD in witness plates deposits, but absolute Be/D ratio and total contribution of $ND_x$ are still to be determined
  - nitriding shifts release to higher temperatures
- No clear evidence of insulating layers on the target samples could be found, whereas Be-N-D deposits on witness plate samples seems to be insulating.

# Be line ratio measurements

PISCES




Work continues to understand difference between D and He plasma measurements. Possibly electron temperature distribution effect.

# What Be PMI issues are still unresolved



- Erosion – physical sputtering yield, chemical erosion (T), reflection coefficient from plasma exposed surface (material migration), role of plasma atoms in surfaces
- Surface morphology – formation causes, effect of impurities, fluence, surface temperature, net deposition exfoliation (dust generation)
- Retention in, and release from, thick BeO surfaces
- Release during thermal excursions, heating rate dependence
- Mixed materials