Max-Planck-Institut für Plasmaphysik

Behaviour of Tin under Low-Temperature Deuterium Plasma Irradiation



Motivation

- Liquid metals as alternative to solid W divertor? (see, e.g. [1])
- Avoid brittle failure, melt damage, neutron damage...
- Tin: low evaporation rate up to about 1000 K, no known solid hydrides, endothermic H solubility [2]
- Promising heat load handling capability [1, 3]
- Very little actual data on D retention
- \succ Indication for chemical erosion by volatile SnD₄ formation [4, 5]
- Systematic study below and above melting point of Sn (505 K):
 - > D retention and release after Sn exposure to D plasma
 - Sn net erosion and nearby re-deposition by mass loss measurements and Mo witness samples

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Evolution of Sn surface

- 300 K:
 - Small bubbles near surface; unstable during electron beam imaging!
 - > Dune-like surface, ridges crowned by Cu- and Fe-rich precipitates -> erosion morphology
- 495 K:
 - \succ Formation of sponge-like, highly porous layer \rightarrow up to 250-500 µm thick after 96 hours
- 515 K:
 - \succ Sn droplet quickly starts wetting W coating, then rises out of crucible \rightarrow large cavity below Sn
 - \succ Gas amount in cavity ~2000x larger than anticipated based on diffusion \rightarrow convection in melt
 - \succ Smaller bubbles found in Sn \rightarrow rise to surface and burst, or get dragged to bottom by melt motion











Experiment details

- Low-temperature ECR plasma source *PlaQ* [6]
 - > Ion flux: ~1.2x10²⁰ D/m²s (97% from D_3^+)
 - \succ -25 V bias (+ 7 V plasma potential): ~11 eV/D from D₃⁺
 - \succ ~100x more D⁰ than D ions
 - Absolute temperature accuracy: ±5 K

Sn samples:

- > 99.999% purity (Kurt J. Lesker Co.)
- \succ Cu crucible with ~2 µm W coating, 10 mm inner diameter
- Pre-molten & degassed in vacuum at 523 K for 2 h, slow heating and cooling
- Typical mass of Sn: ~2.6 g
- Mo witness samples:
 - \rightarrow ~12x15x0.5 mm³, mechanically polished

Mass loss & re-deposition

SDTrimSP 6.00 [7] simulations:

- > Sputtering threshold \geq 50 eV
- ➤ Maximum ion energy (D⁺): ~32 eV



- → expected: <u>chemical</u> erosion (if any)
- 300 K: •
 - Strong mass loss by chemical erosion
 - Small re-deposition on Mo witness sample
- 495 K:
 - Much smaller erosion → sponge-like structure!
 - No measureable re-deposition
 - Erosion of small Sn droplets from 2nd Mo sample
- 515 K:
- Very large mass loss, strong scatter
 - \rightarrow ejection of Sn droplets due to bursting gas bubbles
- Sn droplets found on witness samples

Conclusions

- Large part of D retention in Sn apparently within gas bubbles
 - Small, near-surface bubbles for 300 K exposure
 - Thick, sponge-like, porous layer for 495 K exposure
 - Large gas bubble below Sn droplet for 515 K exposure
- Release practically all D up to melting point (505 K) for solid Sn exposure

deposited

D release until long after melting for liquid Sn exposure





distance from center [mm]

Deuterium retention & release

Deuterium depth profiling: ³**He(D, p)**⁴**He NRA**

- 300 K:
 - > D retention concentrated at surface
 - \succ Increasing fluence \rightarrow growing tail into bulk
 - > NRA total amount corresponds approximately to spike in TDS spectra

• 495 K:

- Larger total inventory, less peaked profiles
- \blacktriangleright Depletion of D at surface \rightarrow sponge-like structure!
- \succ 1 h exposure: D contained mostly within first 20 μ m
- thickness of bubble layer!
- Longer exposure times: significant amount of D beyond analysis range (compare also to TDS!)

• 515 K:

- D concentration in Sn at or below detection limit $(\leq 5 \times 10^{-5} \text{ at.-}\%)$ within NRA range
- D assumed to be mostly within gas bubbles

Thermal Desorption Spectroscopy

Solid exposure (300 and 495 K; Sn without crucible): \triangleright D₂ Release starts already at room temperature





- Strong chemical erosion at 300 K, weaker at 495 K
 - Possibly linked to formation of sponge-like layer
 - Some re-deposition of Sn on Mo at 300 K
 - > 495 K: no measurable Sn re-deposition; erosion of small Sn droplets
- Erosion at 515 K dominated by droplet ejection due to bubble bursts
 - Deposition of Sn spray on Mo sample
- Wetting of W by Sn at 515 K under D plasma exposure \rightarrow oxide removal?

<u>References:</u>

[1] J.W. Coenen et al., Phys. Scr. T159 (2014) 014037 [2] M.B. Bever, C.F. Floe, Trans. AIME 156 (1944), 149-159 [3] A. Vertkov et al., Fus. Eng. Des. 117 (2017) 130-134 [4] D.T. Elg et al., Plasma Chem. Plasma Process. 38 (2018) 223-245

[5] T.W. Morgan et al., Plasma Phys. Control. Fusion 60 (2018) 014025 [6] A. Manhard et al., Plasma Sources Sci. Technol. 20 (2011) 015010 [7] A. Mutzke et al, SDTrimSP Version 6.00, IPP 2019-2 (2019)

- Massive release spike at melting point
- \rightarrow Dominates D₂ release
- D release stops after end of melting phase
- Additional peak at ~400 K for 300 K exposure
- \succ D released almost exclusively as D₂
- Liquid exposure (515 K; Sn in crucible):
 - Continuous signal + spikes
 - D release continues until long after melting point
 - \succ Smaller spikes \rightarrow bubbles rising to surface
 - \succ Large spikes \rightarrow Sn droplet rising due to expansion of large gas bubble, and then collapsing
 - \succ Strong HD release in addition to D₂
 - In contrast to NRA: D retention comparable to sample exposed for 1 h at 300 K

10² 600 700 300 400 500 800 sample temperature [K]





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