Max-Planck-Institut für Plasmaphysik, EURATOM Association

Hydrogen gas filled cavities under surface extrusions on hydrogen-implanted tungsten

M. Balden [a,*], S. Lindig [a], A. Manhard [a], V.Kh, Alimov [b], O.V. Ogorodnikova [a], J. Roth [a]
[a] Max-Planck-Institut für Plasmaphysik, EURATOM Association, 85748 Garching, Germany, [b] Tritium Technology Group, Japan Atomic Energy Agency, Tokai, Ibaraki 319-1195, Japan

Motivation

- The assessment of the tritium inventory in future thermonuclear fusion devices (e.g. ITER) and, therefore, the **retention of hydrogen isotopes** in plasma-facing material, e.g. tungsten, are of major interest.
- Hydrogen-implanted tungsten is extensively investigated, e.g., regarding hydrogen retention and **surface morphology changes**. The reported surface extrusions vary from spherical, more classical blisters [1] to stepped, high-domed structures [2,3]. Furthermore, on in the subsurface, a spectrum of different crack and cavity features are observed [4]. The presence and absence of features under similar exposure parameter (fluence, flux, ion energy, implantation temperature, specimen pre-treatment) as well as the dramatic variation in their size draw a confusing picture of the morphological modifications described in the literature.
- In this study, the three-dimensional morphology of surface extrusions on polycrystalline tungsten and the **gas filling of cavities** beneath the surface are analyzed using confocal laser scanning microscopy (CLSM) and scanning electron microscopy (SEM) assisted by cross-sectioning with a focused ion beam (FIB) and by calibrated residual gas analysis with a quadrupole mass spectrometer (QMS).

[1] B.M.U. Scherzer, in: R. Behrisch (Ed.), Sputtering by Particle Bombardment II (Berlin: Springer, 1983) pp. 271-355.
[2] W.M. Shu, E. Wakai and T. Yamanishi, Nucl. Fusion 47 (2007) 201.
[3] S. Lindig et al., Physica Scripta T136 (2009) 014040.
[4] M. Balden, S. Lindig, A. Manhard, J.-H. You, J. Nucl. Mater., (2011) in press.

Experimental details

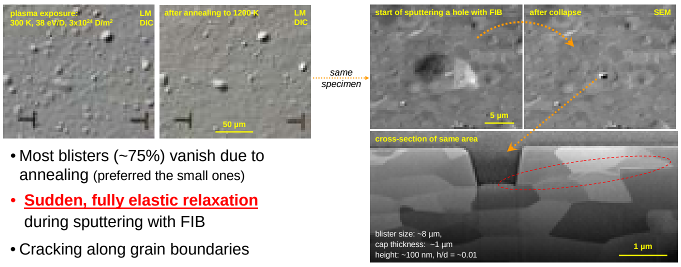
- **Specimens**: Polycrystalline tungsten (Plansee): rolled / polished grain size of ~1-5 µm
- **Deuterium plasma exposure**: surface temperature: 300-500 K 10^{24} - 10^{25} D/m² (10^{20} D/m²s) of 38-200 eV D
- Confocal laser scanning microscopy (**CLSM**, *VK-9700, Keyence / LEXT OLS4000, Olympus*)
- Scanning electron microscopy (**SEM**, *Helios NanoLab 600, FEI*) combined with focused ion beam (**FIB**)
- Calibrated mass spectrometry (**QMS**, *PrismaPlus, Pfeiffer*)

⇨ 3-dimensional surface morphology
⇨ subsurface morphology analysis by cross-sectioning
⇨ degassing behavior of blisters and their relaxation

Relaxation of individual blisters

300 K: small blisters

Relaxation by thermal treatment — *Relaxation by puncturing with FIB*

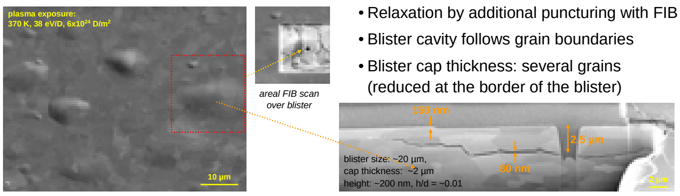


cross-section through hole sputtered to degas in cap of collapsed blister

- Most blisters (~75%) vanish due to annealing (preferred the small ones)
- **Sudden, fully elastic relaxation** during sputtering with FIB
- Cracking along grain boundaries

370 K: small & larger blisters

Relaxation by removing the top of the implantation layer? **No relaxation**

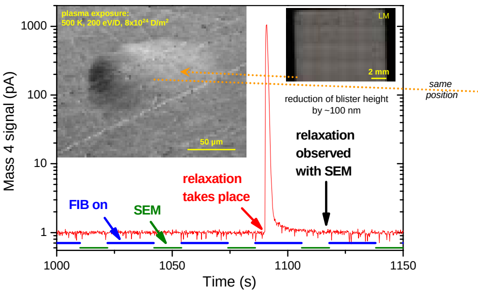


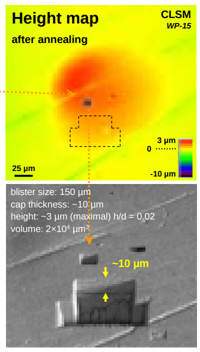
cross-section through blister after degassing

- Relaxation by additional puncturing with FIB
- Blister cavity follows grain boundaries
- Blister cap thickness: several grains (reduced at the border of the blister)

500 K: giant blisters

Relaxation by puncturing with FIB + simultaneous QMS





- Partial relaxation of blister ⇨ **plastic** deformation
- **D_2 gas release** (uncalibrated)

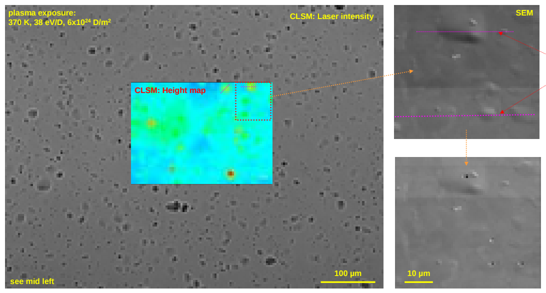
➔ Correlation: exposure temperature ⇔ size ⇔ cap thickness ⇔ plastic deformation

Conclusion

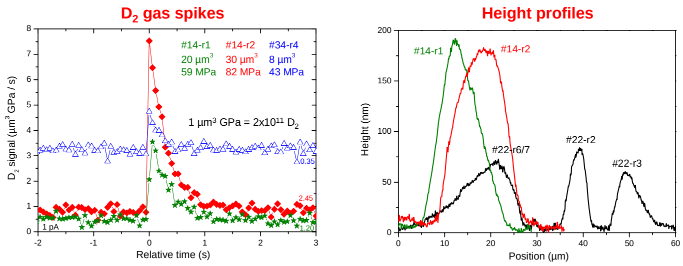
- **Spherical blisters** on polycrystalline hot-rolled W formed by D bombardment up to several 10^{24} D/m². Their **size** depends on implantation temperature (300–600 K) and ranges from **several µm up to a few hundred µm**. The volume of individual blisters was determined.
- Individual blisters were sequentially punctured with FIB and imaged with SEM. A **sudden relaxation** was observed. Smaller blisters appear to be fully elastic. A **D_2 spike** was observed simultaneously with relaxation if volume of the blister cavities was large enough (>3 µm³).
- By determining the blister volume and the amount of released D_2 molecules, the **D_2 gas pressure** can be determined to be of the order of **several 10 MPa** (40–90 MPa).
- The subsequent cross-sectioning shows that **delamination along grain boundaries** roughly parallel to the surface occurred. The blister cap thickness correlates with blister size and the cap contains **between 1 and about 10 layers of grains**.

Pressure determination of gas filled blisters

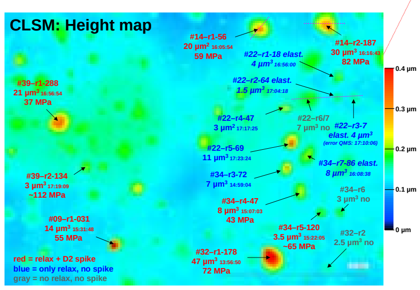
First direct measurement of gas pressure of single blister from
- Blister volume determined by CLSM
- Amount of D_2 molecule obtained by calibrated QMS (open individual blister with FIB)



- Three fully elastically & one plastically deformed blisters after puncturing. The latter one shows D_2 spike *(see below)*



Similar spikes observed also in thermal desorption spectra
Poster Manhard P46A



Overview over punctured blister in height map (CLSM) with
- Identifier
- Volume *(x18)*
- Relaxation with time *(x15)*
- Fully elastic behavior *(x4)* *(no plastic deformation)*
- Pressure if D_2 spike was observed *(x8)*

⇨ Pressure < 100 MPa

Finite element (FE) calculations based on continuum mechanics in the elastic assumption [4]:
- Pressure of 100 MPa is sufficient to reproduce observed cavity height for blister with **cap thickness to diameter of 0.1 (height to diameter of 0.01)**
- Von-Mises stress reach values far above yield strength of W ⇨ beyond elastic assumption

⇨ Further FE calculations for plastic case are ongoing *(J.-H. You, IPP)*

Side remark: Recrystallized tungsten

- Other type of extrusion observed for higher flux plasma on recrystallized tungsten

⇨ Subsurface strongly cracked inside large grains *(more details see **Poster Lindig P63B**)*
⇨ Calibrated QMS simultaneous to slicing with FIB leads to **D_2 gas spikes with 0.2–0.4 µm³ GPa**

- First Nano-SIMS investigations to analyzing the lateral distribution of hydrogen (H, D) promising

⇨ Indications of hydrogen accumulated at grain boundaries
⇨ Gas spikes *(see **Poster Lindig P63B**)*