Max-Planck-Institut für Plasmaphysik, EURATOM Association

Deuterium implanted into polycrystalline tungsten: Novel TPD investigations

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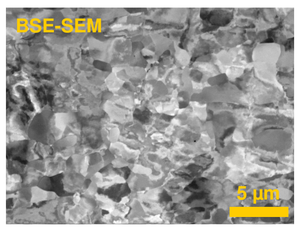
Introduction

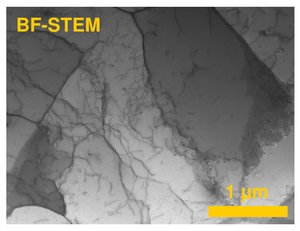
- **Temperature Programmed Desorption** (TPD) in order to study **D binding energies in W**
- Challenge: TPD spectra influenced both by **binding energy distribution** and **D depth profile / diffusion**
- Solution: **"Ramp and Hold" experiments** with large set of identical samples
  - Temperature ramps with **different heating rates**
  - **Interrupted temperature ramps** with **different holding times** at constant temperature
  - **Modeling** of all spectra with **one single binding energy distribution**

Experiments

Samples

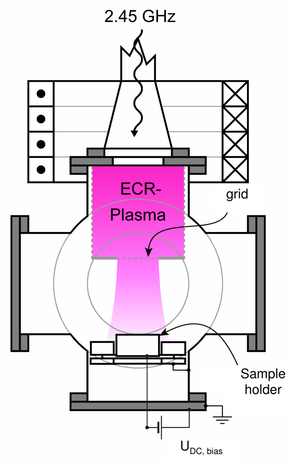
- Polycrystalline, hot-rolled tungsten (PLANSEE, 99.97 wt.% purity, 12×15×0.8 mm^3)
- Metallographic surface finish (i.e., **no distortion layer** at polished surface)
- Stress-relieved and degassed for **1 hour at 1200 K in high vacuum oven**
- Microstructure: **small grains** (~1-5 µm), dense **network of dislocations**





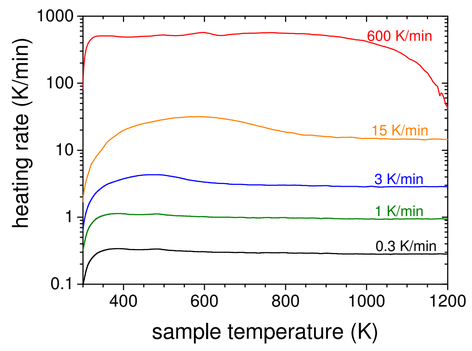
Plasma exposure

- ECR plasma ion source with freely expanding plasma beam
- **Fully quantified** for D_2 plasma [1]
- Ion energy: **38 eV/D**
- Ion flux: **10^{20} D/m²s**
- Implantation fluence: **6×10^{24} D/m²**
- Sample temperature: **370 K**
- **2 months resting time** in vacuum exsiccator after D implantation



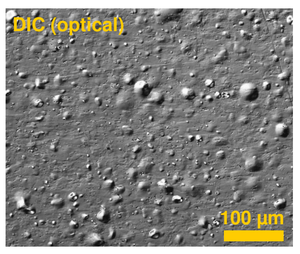
TPD ramps

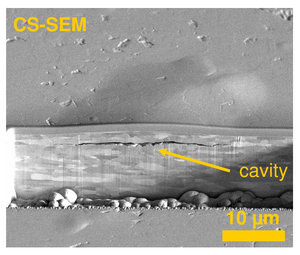
- TPD analysis in TESS set-up (quartz tube) [2]
- **Heating rates between 0.3 and 600 K/min**
- Linear ramp of oven temperature
- Sample temperature calibrated with thermocouple for each ramp



Blister formation

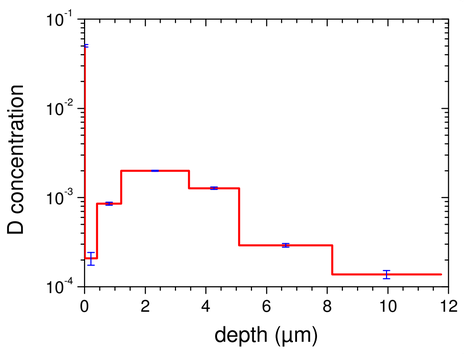
- **Strong blister formation** at sample surface
- Blister **diameter between 1 and several 10 µm**
- Blister **height ≤ 1 µm** (depending on diameter)
- Cap extends over many grains
- Cap thickness up to several µm





D depth profile

- Analysis of D depth profile with **D(^{3}He,p)^{4}He nuclear reaction**
- **^{3}He energy variation** between 500 and 6000 keV
- **Deconvolution of all proton energy spectra simultaneously** by NRADC program [3]

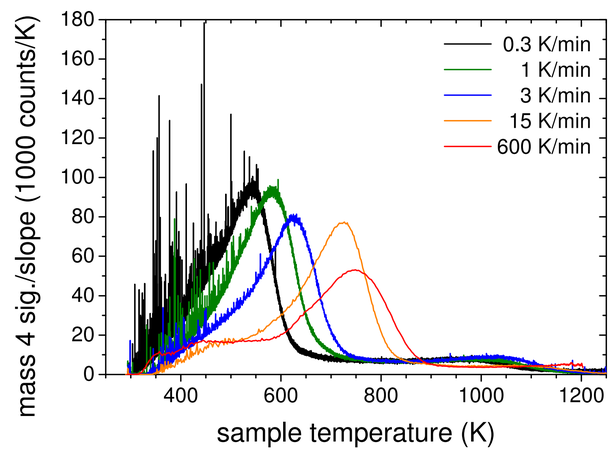


Summary

- TPD for large number of **identical, homogeneously implanted samples**
- D **depth profile known** from nuclear reaction analysis
- **Blister bursting** visible in TPD spectra, contributes **~5% to total release**
- **Variation of heating rate** by more than **three orders of magnitude**
- **High temperature shoulder** (up to ~1050 K) present for **all heating rates**
- **Holding for up to 90 minutes** at temperatures 470 or 660 K does **not remove high temperature part** of release spectrum ⇒ **desorption spectrum not dominated by diffusion for high temperatures**
- First conclusion: **broad binding energy distribution** has to be assumed for D in W!
- **Outlook: ready to start modeling of TPD data**
- **Goal: one single binding energy distribution to match all spectra**

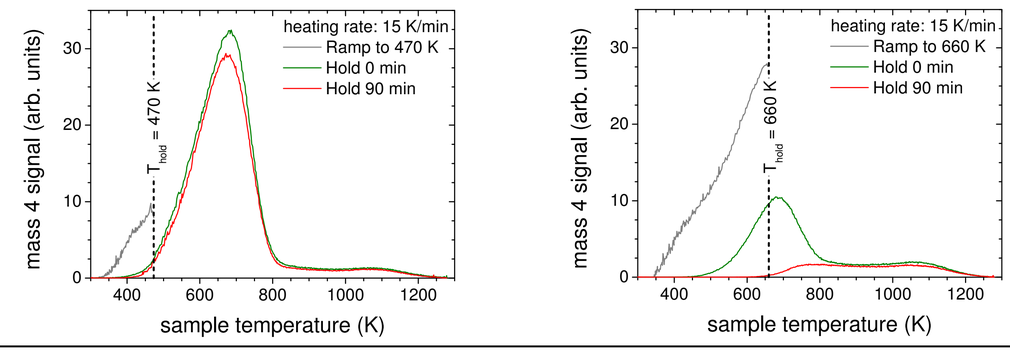
TPD ramp variation

- Main **desorption peak shifts** to lower temperature for slower ramp rate
  - ⇒ allows calculation of **desorption pre-factor** (to be determined by modeling)
- **Low-temperature peak only** clearly visible **for fast ramps**
- **High-temperature shoulder** (~1050 K) present even for very slow ramps
- **Total desorbed amount** nearly equal for all samples: **$6.8 \pm 0.3 \times 10^{16}$ D_2 molecules**
- Negligible contribution of HD, HDO and D_2O



Interrupted TPD ramps

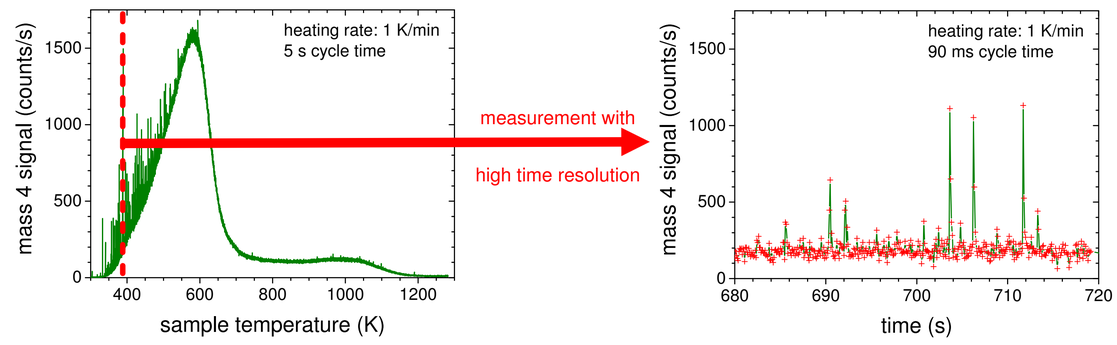
- **Interruption of ramp** as well as **holding for 90 minutes** at **470** and **660 K**
- **Second, uninterrupted TPD ramp** after sample has cooled down to 300 K
- **High-temperature part** of spectrum **barely affected** by partial desorption



Blister bursting

- **Short, intense bursts of mass 4 (D_2)** in low-temperature part of TPD spectrum
- Bursts do not occur for any other mass ⇒ **release of gaseous D_2 from rupturing blisters**
- Burst size distribution follows **exponential distribution**
- **Contribution to total inventory: ~ 5%**
- **~2×10^{11} D_2 molecules per burst**
- Blister volume ~10 µm³ ⇒ **pressure ~0.1 GPa**

  ⇒ in accordance with estimations based on opening of single blisters by FIB [4] (→ see also P 51A)



References

[1] Manhard et al., Plasma Sources Sci. Technol. 20 (2011) 015010
[2] E. Salançon et al, J. Nucl. Mater. 376 (2008) 160–168
[3] K. Schmid et al., to be published
[4] Balden et al., J. Nucl. Mater., article in press (doi: 10.1016/j.jnucmat.2011.04.031)

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