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Subsurface structures on rolled and re-crystallized W after D bombardment

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1. Motivation

Tungsten is a promising candidate as a plasma-facing material in the main chamber and also for divertor areas in fusion reactors. The erosion of this high-Z material by plasma is acceptably low and the D retention is intensively investigated by many international groups. The absolute D retention in W seems to be tolerable for the next generation of nuclear fusion devices like ITER or DEMO. But still clarification is needed how and under which conditions the D enters the material and where D is mainly accumulated.

2. Experimental

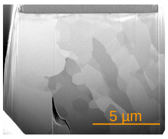
Material

Japanese ITER grade W

- rolled and swaged/forged W polycrystall; purity 99.99 %(wt)
- grain sizes elongated ca 1-5 µm (bcc, fiber texture [110])
- deformation axis perpendicular to surface
- mechanically polished, cleaned in Acetone ultra-sonic bath
- annealed 1473 K 30 min (stress relief)

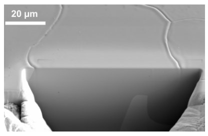
re-crystallized W

- **Japanese ITER grade** additional re-crystallized at 2073 K, 1h, H_2-atmosphere
- grain sizes ca 50 µm (lattice bcc)



SEM cross-section of initial Japanese ITER grade

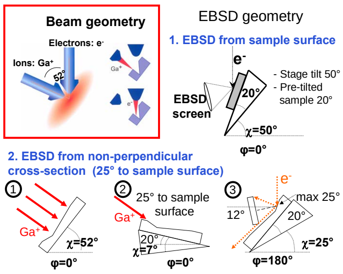
- pieces 10x10 mm², thickness ~2.5 mm
- typical loaded with D-plasma 38 eV/D flux 10^{22} D/m²s, fluence 10^{27} D/m²
- loading temperature range **300-700 K**



SEM picture from re-cryst. initial sample: surface and cross-section

Characterisation / equipment

- Helios Nanolab 600 (FEI): **FEG-SEM** with **FIB at 52°** Pt/C-deposition, stage: +60° …. -9° tilting
- **EBSD camera** (HKL/ Nordlys II Oxford) **detector axis -12° under horizontal line** EBSD-map (Oxford **HKL Channel 5**.0.9.1 Tango)
- NanoSIMS 50L Cameca (*TU-München*[c]) Cs^+, 16 keV; 10^{-10} mbar; pre-sputter $1x10^{20}$ Cs/m²
- Tandem accelerator for NRA
- TDS (thermal desorption spectroscopy)

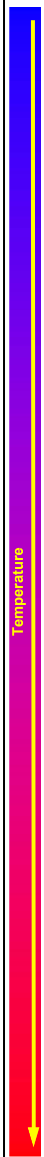


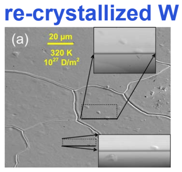
3. Surface structures after D loading

Variations of surface structures after loading temperatures from 300-700 K

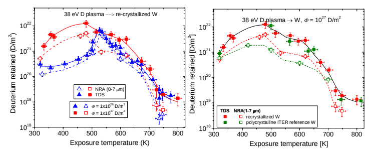
fluence 10^{27} D/m² ; 10^{26} D/m² flux 10^{22} D/m²s energy of D 38 eV

re-crystallized W — **Japanese ITER grade**

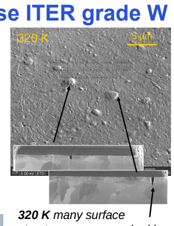




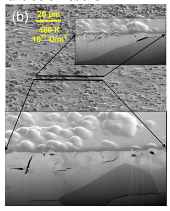
320 K only rare structures and deformations



D-content, measured by ion beam analyses NRA (nuclear reaction analyses) and TDS (thermal desorption spectroscopy)



***320 K** many surface structures, some marked by FIB cuts. Due to marking the blister collapsed. → blisters connected by a gas filled crack system*

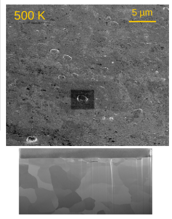


***480 K** cracks, strong deformations, many surf. structures*

Surface structures vary with temperature:
- ~ **300 K** only singular small bubbles appear with weak plastic deformations in the subsurface
- ~ **480 K** many structures and **strong deformations and cracks** are visible (brittle)
- ~ **600 K no cracks, deformations** inside the grain, **but extrusions** appear and a cavity is formed at the first grain boundary (ductile)
- ~ **700 K** no structures are visible

Surface structures do not vary with temperature:
- **size and number** of surface structures nearly **independent** on temperature
- **no deformations, cracks** (only between grains)
- **no extrusion**
- all cavities are formed in the destroyed polished layer.

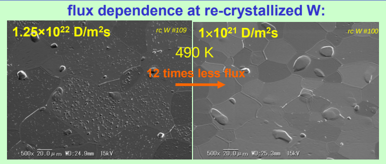
The mechanically damaged layer promotes the cavity formation under D loading
- ~ **700 K structures** are visible



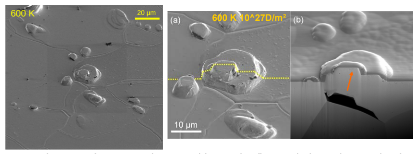
***500 K / 600 K** cross-sections show only cavities along the damaged zone of surface as at 320 K. The number and size of blisters are constant*

***Remark:** Only very rare large grains show also extrusions from the grain boundary to surface as in re-crystalline samples*

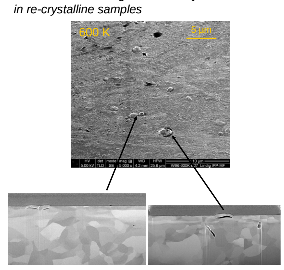
flux dependence at re-crystallized W:



D loading with different fluxes, low flux cause only large extrusions, higher flux cause additionally small structures with cracks as above



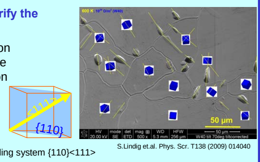
***600 K** large surface extrusions, cavities at the first grain boundary under the surface, no cracks, no bent grains*

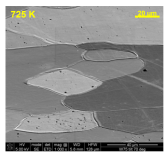


blisters / cavities start always on probably weak boundaries between bulk and damaged surface layer (by mechanical polishing). This damaged surface layer promotes obviously blister formation.

EBSD measurement to clarify the formation mechanism

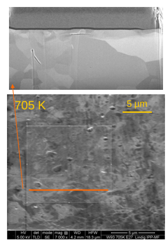
By EBSD the grain orientation could be determinate and the correlation between extrusion shape could be confirmed. The material is moved out along the gliding system {110}<111> (ductile).





***725 K** no surface structures, no features in cross-section*

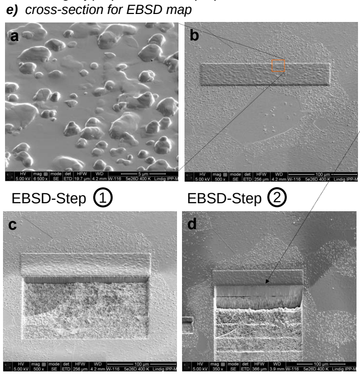
- total D retention is equal in both types of materials, measured by TDS
- D retention in near surface (up to 7 µm) in polycryst. W with damaged surface layer by mechanical polishing (technical surface?) is up to 5 times higher than in re-cryst. W
- number and size of blister in mechanically polished polycryst. W are nearly independent on temperature
- polycryst. W shows blister also at 700 K
- mechanically damaged surface layer promotes blistering



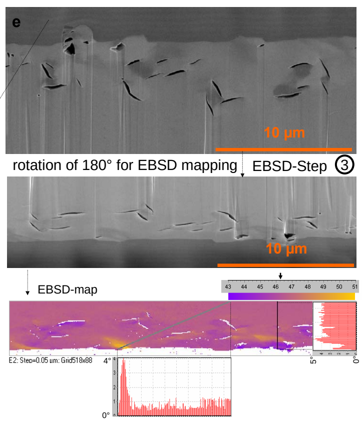
***705 K** many surface structures visible, cross-sections also cavities*

4. EBSD maps of plastic deformation

*Fig **a)** shows re-crystallized W loaded with **5 · 10^{26}** D/m², 400 K, 10^{22} D/m²s, **b)** coated with protection Pt/C mixed layer for FIB-cutting, **c)** prepared for non-perpendicular cross-section-EBSD, **d)** tilt for EBSD-cut, EBSD steps (1-3) see also box 2 **e)** cross-section for EBSD map*



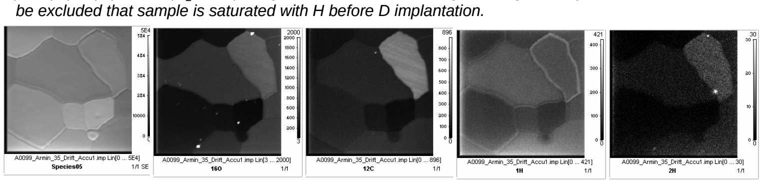
The EBSD maps clearly show a deformation inside of one grain. Here the grain is bent up to 5° by plastic deformation during D loading. Without much doubt the very high transient super-saturation of D causes this strong deformation. Probably the cracks follow the cleavage planes in the grain.

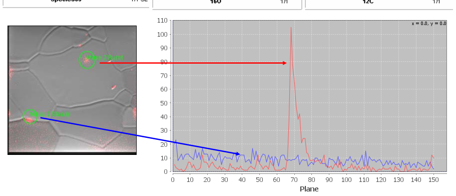


EBSD map (Euler 2) in one grain. The deviation of the main orientation (46.2°) is colour-coded and selected line profiles show the misorientation relating to the first point.

5. NanoSIMS, H localisation in re-cryst. W

First experiments were performed on a D loaded sample to investigate an expected accumulation of D at grain boundaries of re-crystallized W on the surface by Nano-SIMS (fluence 3·10^{19} Cs/m²).
The first series shows a SE-picture made by Cs ions and maps in the light of mass numbers 16 (O^-),12 (C^-),1 (H^-) and 2 (H_2^- or D^-). At grain boundaries the H signal is significantly increased. It cannot be excluded that sample is saturated with H before D implantation.





The second series shows one of the gas bursts during the SIMS scans. In the SE-picture the SIMS-map of mass-2 signal is superposed. The regions of integration of counts is marked in green.
The diagram shows the integrated counts in these areas in respect to scan number (plane) of the burst (red) and of the steady spot (blue).

→ **see also P 51A and P 29B**

6. Conclusion

- **number and size of blister in mechanically polished polycrystalline W is nearly independent on temperature**
- **mechanically damaged surface layer promotes blistering**
- **polycryst. W shows blisters at 700 K in contrast to re-crystallized W**
- **very high transient super-saturation during D exposure causes probably cracks and plastic deformations and plastic bending of grains of about 5° (up to 7°) over 1 µm length**
- **flux dependence of surface structures, cracks, deformations**
- **total D retention is in both types of materials is equal, measured by TDS**
- **D retention in near surface (until 7 µm) is in polycryst. W with damaged surface layer by mechanical polishing (technical surface?) until 5 times higher than in re-cryst. W**
- **hydrogen (H/D) can be localized with NanoSIMS on grain boundaries**
- **gas bursts during NanoSIMS-scans until now not clear explained (opened cavities ?)**

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