Modification of the structure and optical characteristics of the surface of tungsten mirrors under bombardment with 38 eV deuterium ions

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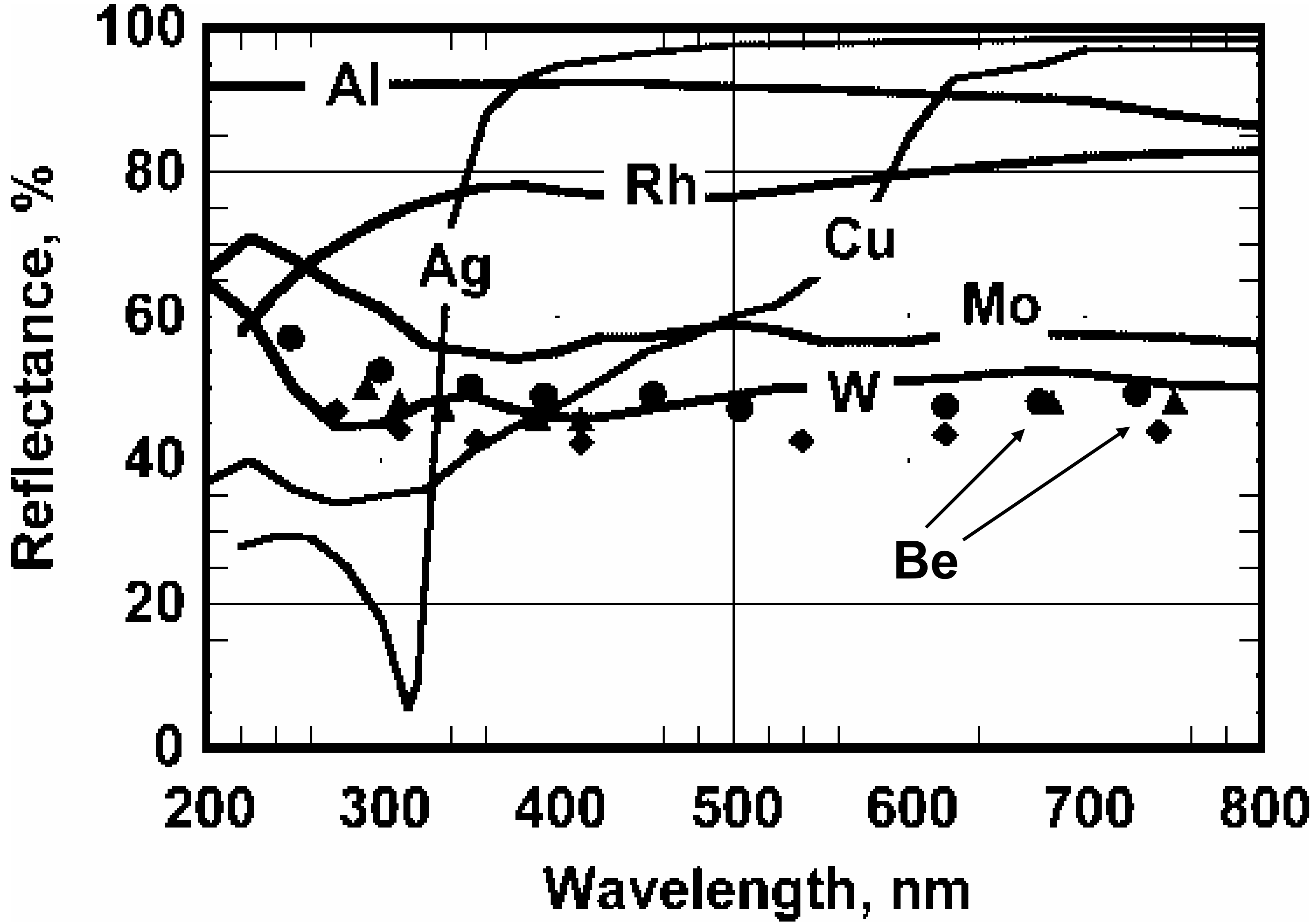
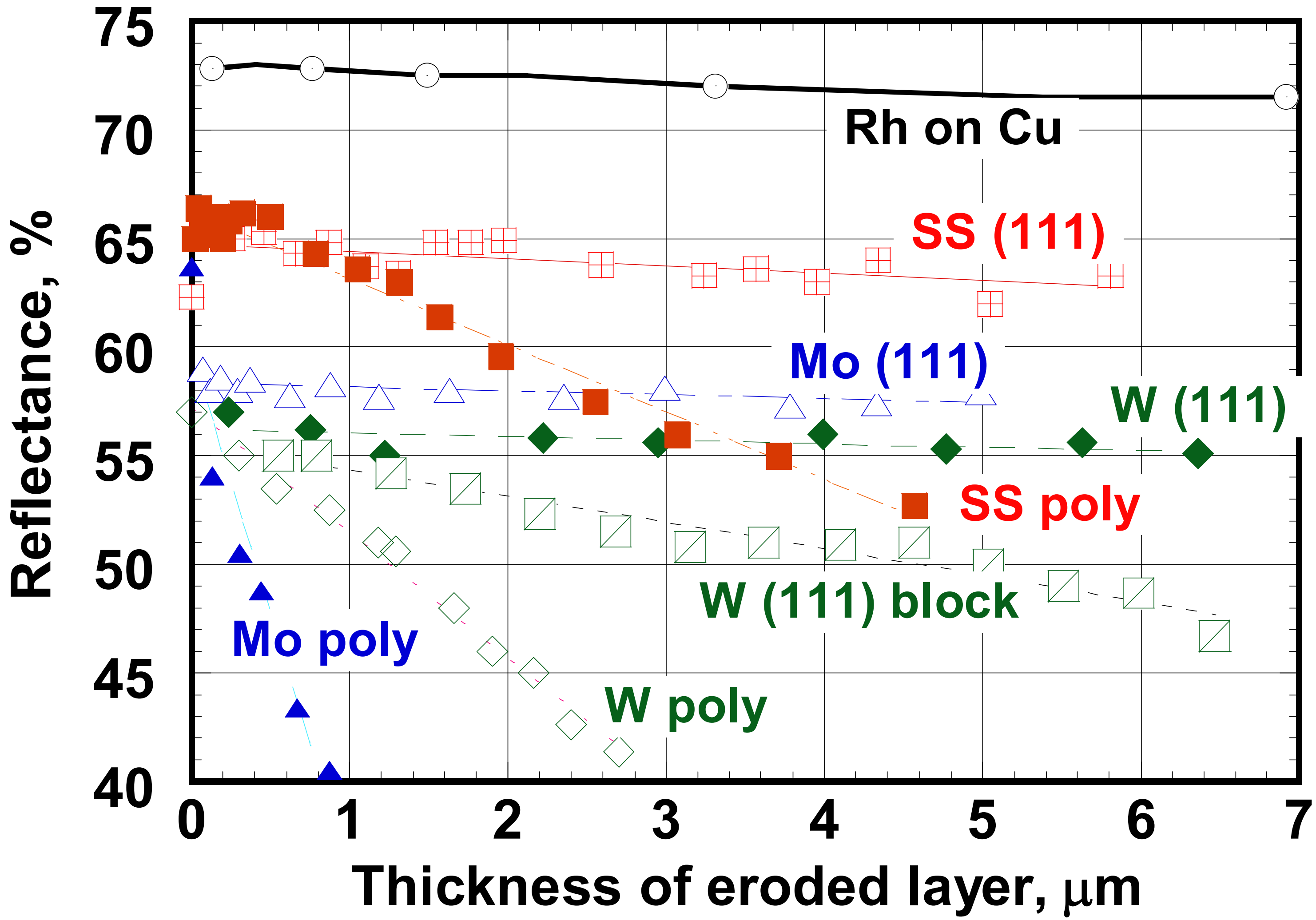
Spectral dependence of reflectance for several metals



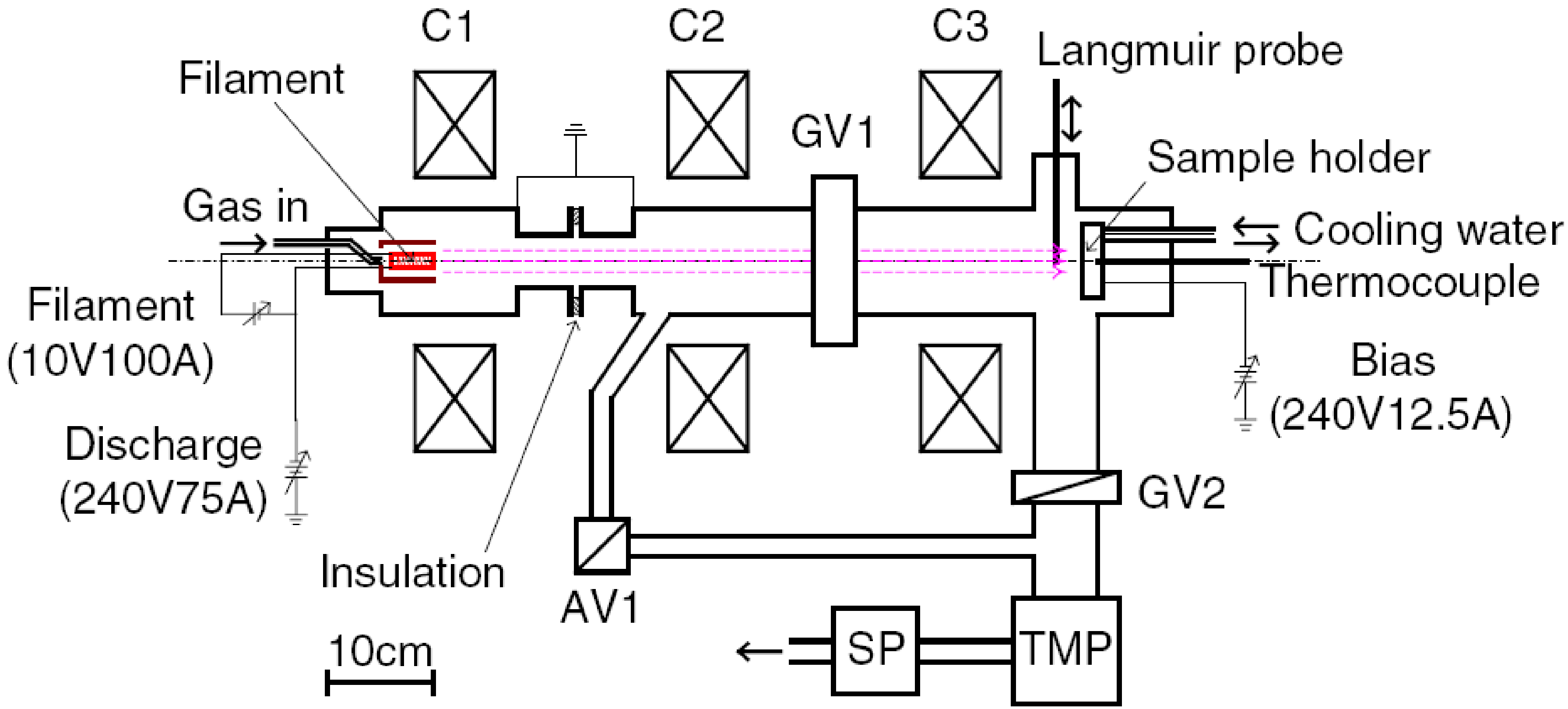
Coefficient of mirror quality, i.e., the ratio of reflectivity to sputtering yield, **R/Y**, for different metals

Metal	Sputtering yield (Y) 10^{-2} at/at	Reflectivity (R)			R/Y		
		250nm	500nm	800nm	250nm	500nm	800nm
Be	5.0	0.61	0.54	0.56	12	11	11
Al	4.3	0.92	0.92	0.87	21	21	20
V	1.1	0.50	0.54	0.53	45	49	48
SS	2.0	0.38	0.67	0.72	19	34	36
Cr	2.6	0.56	0.68	0.63	22	26	24
Ni	2.5	0.44	0.58	0.68	18	23	27
Cu	5.2	0.37	0.60	0.94	7	12	18
Nb	0.24	0.47	0.51	0.60	196	212	250
Mo	0.24	0.69	0.59	0.56	286	246	233
Rh	0.7	0.65	0.77	0.82	93	110	117
Ag	4.0	0.29	0.96	0.98	7	24	25
Ta	0.011	0.41	0.38	0.74	3727	3455	6727
W	0.008	0.49	0.49	0.49	6125	6125	6125
Re	0.035	0.57	0.51	0.63	1629	1457	1800
Ir	0.08	0.57	0.68	0.74	712	850	925
Pt	0.12	0.37	0.61	0.71	308	508	592
Au	0.8	0.34	0.50	0.97	43	63	121

Behavior of reflectance (λ=600 nm) under long-term sputtering of polycrystalline and single crystalline (111) mirrors of indicated metals



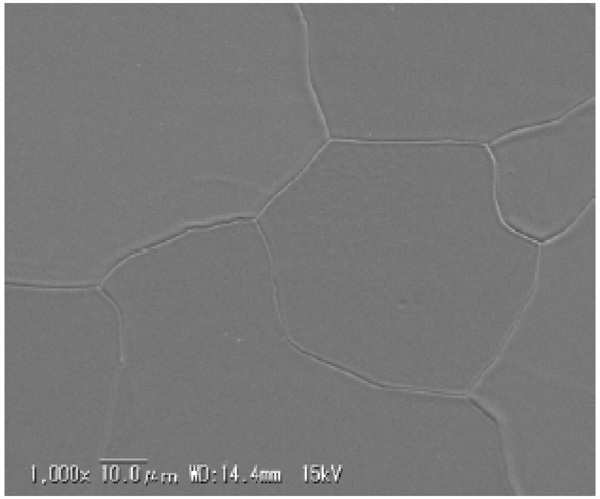
Linear Plasma Generator used in JAEA (Tokai, Japan) for irradiation of W samples with D+ ions



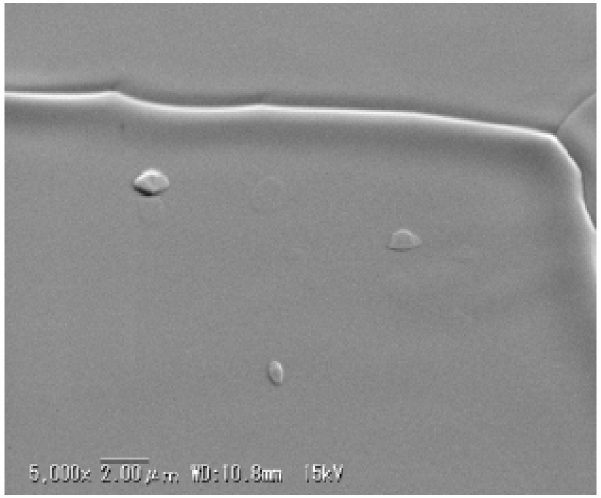
D ion energy:	**76 eV D_2^+**
D ion flux:	**10^{22} D/m^{2}s**
Ion fluence:	**10^{26} D/m^2**
Target temperature:	**320-695 K**

Plates of polycrystalline tungsten, 10×10×2 mm^3 in size, **recrystallized at 2073 K in hydrogen after cutting and polishing**, with a purity of 99.99 wt%, were used as **W** mirror samples

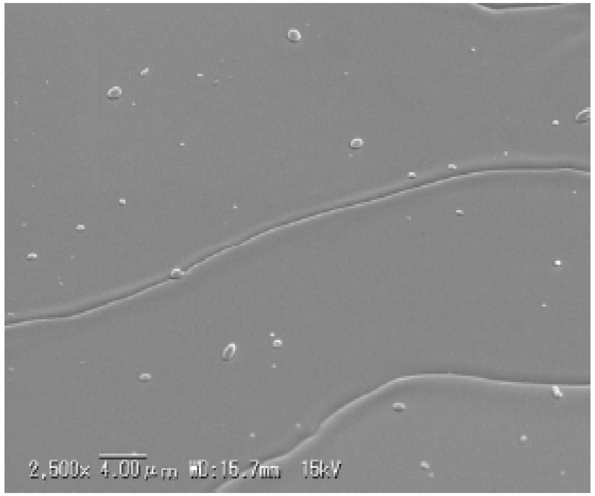
SEM data for recrystallized W mirror samples exposed at various temperature to D^+ ions; 38 eV/D, fluence $10^{26}/m^2$



W42, T = 320 K

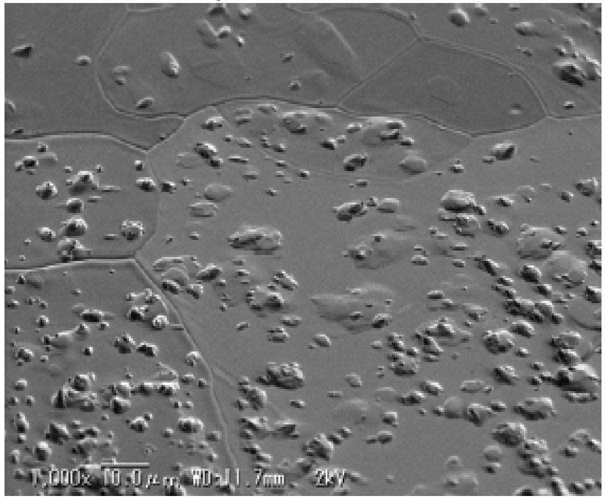


W29, T = 405 K

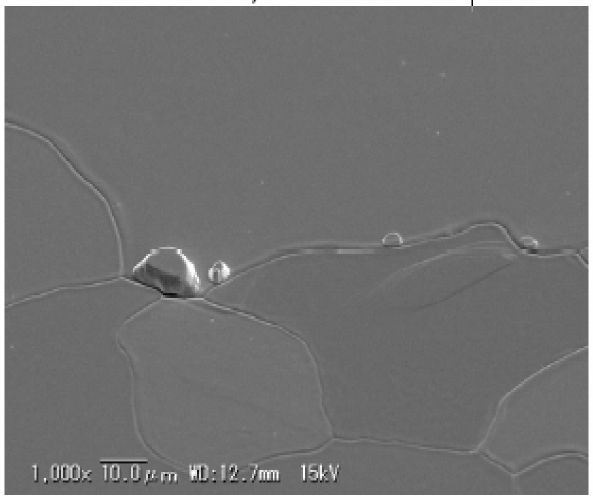


W30, T = 483 K

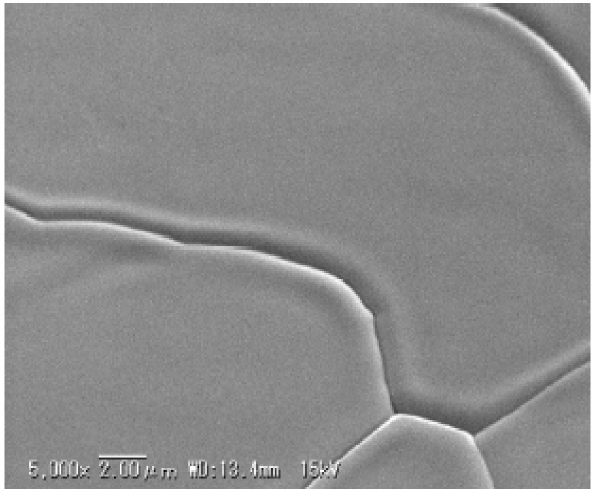
W43, T = 535 K



W35, T = 600 K



W20, T = 695 K



- In Kharkov samples were cleaned by exposure to a low-energy (~60 eV/ion) deuterium plasma ions with a fluence of $\sim 2.5\times10^{23}$ ion/m^2 to remove contaminants that could appear during storage of the samples in air.
- After that there were provided:

(1) direct measurements of the specular reflectance $R(\lambda)$ in wavelength interval 220-650 nm at normal incidence;

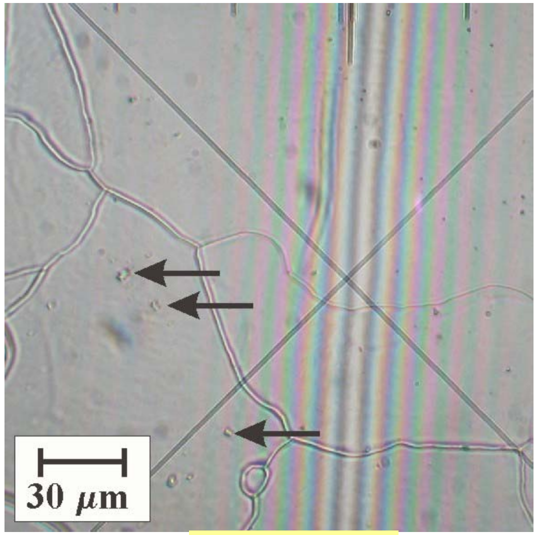
(2) Measurements of the optical refractive index *n* and extinction coefficient *k* with the use of the ellipsometric technique;

(3) Calculation of the reflectance at normal incidence, $R(\lambda)$, using the data on ellipsometric parameters Ψ and Δ;

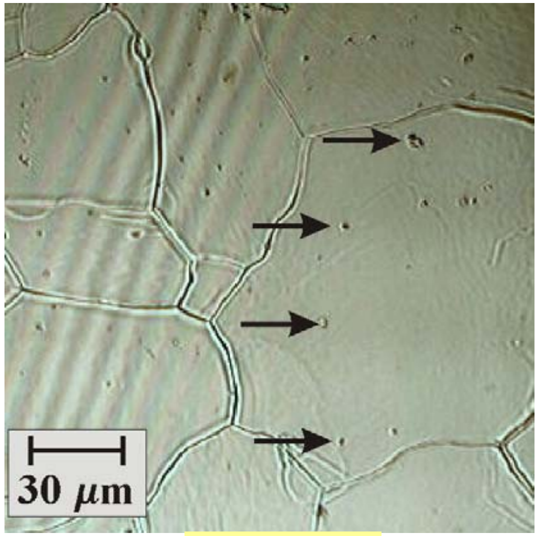
(4) The surface morphology of the W samples was examined by scanning electron microscopy (SEM) and optical microscopy;

(5) The surface relief was controlled by the interferometric microscopy.

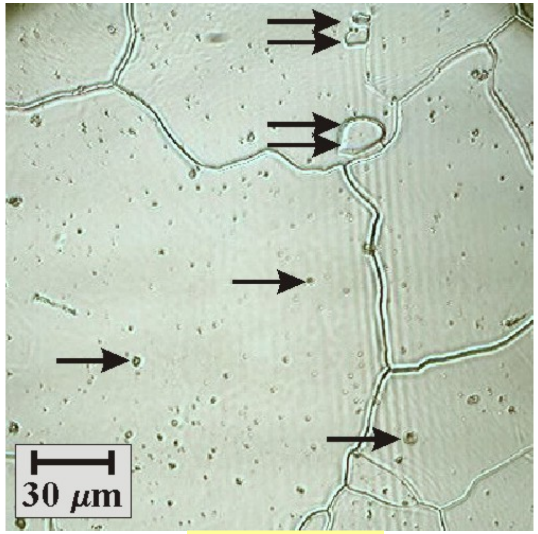
Optical microscope data for W mirror samples



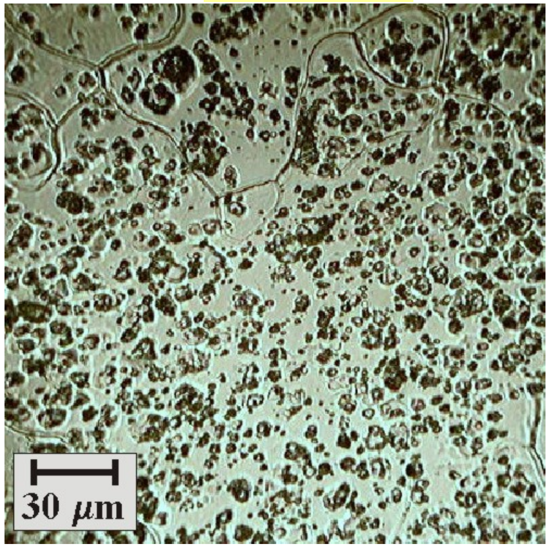
320° K



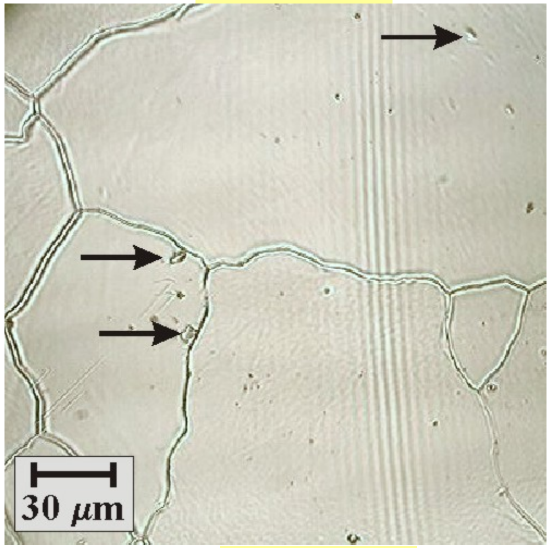
405° K



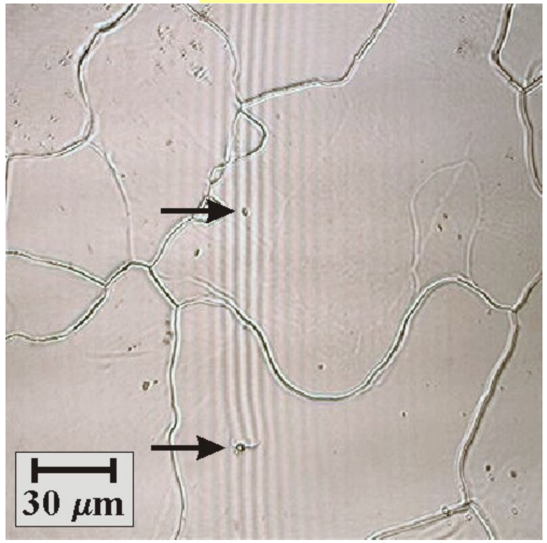
465° K



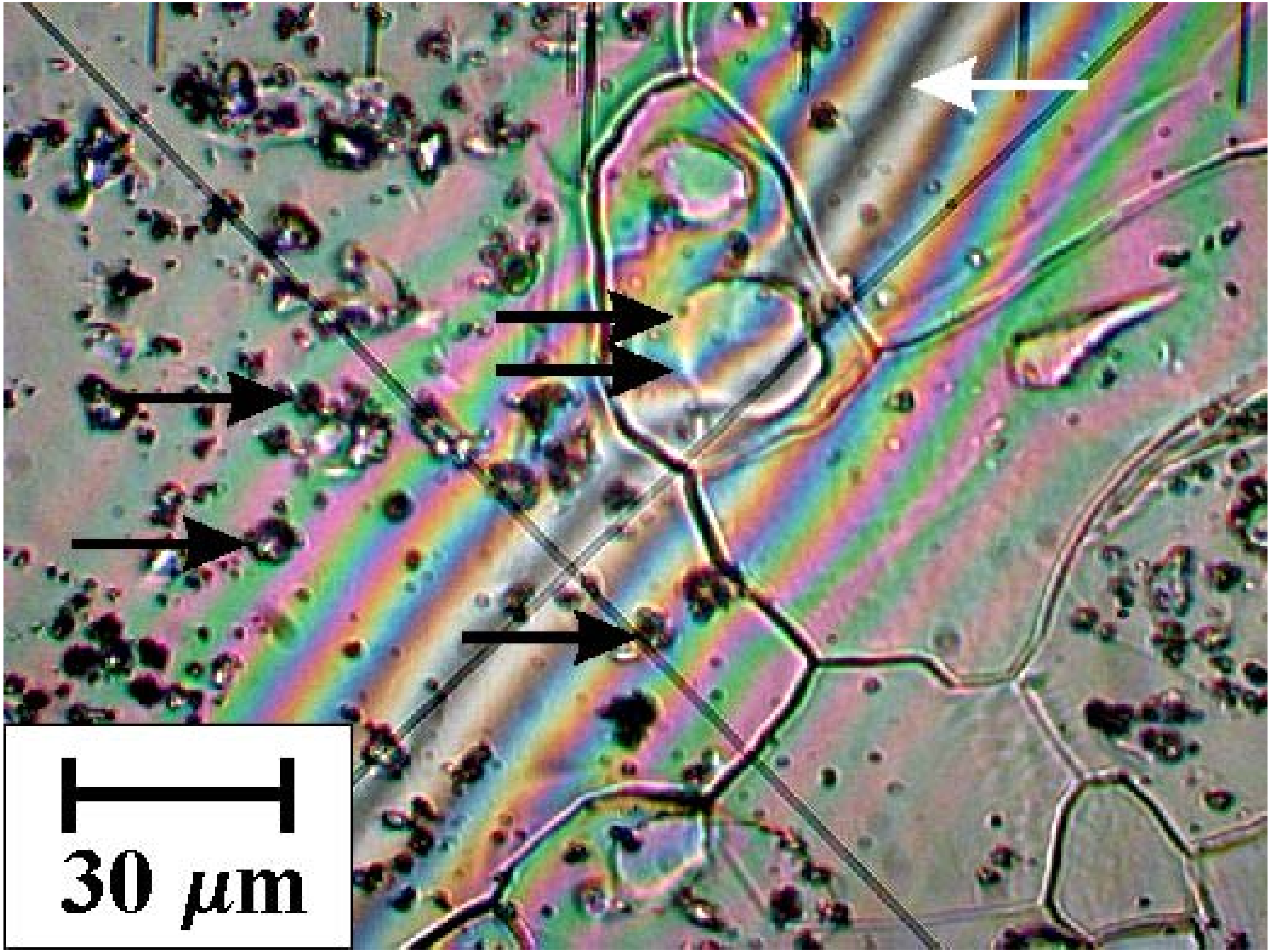
535° K



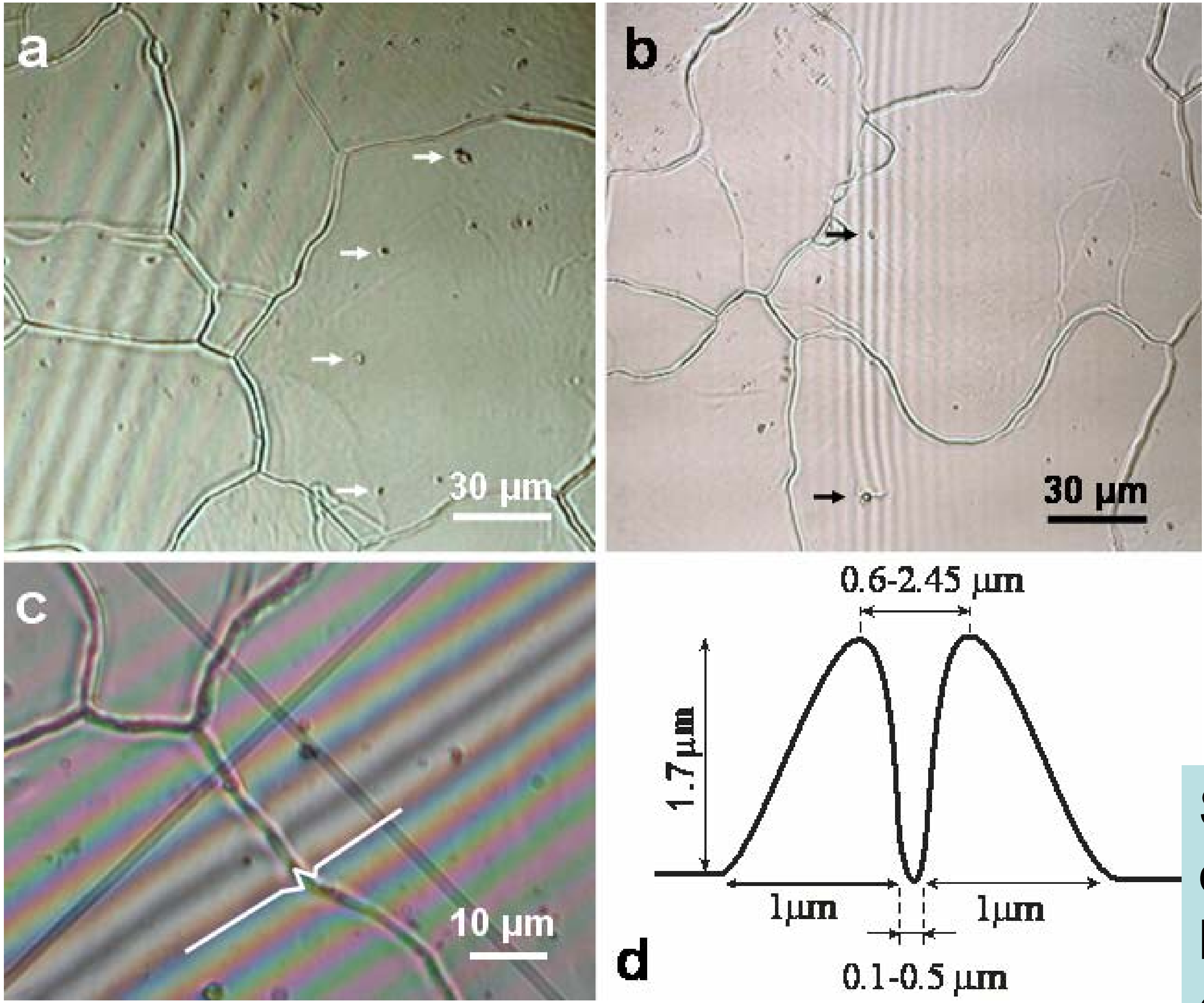
600° K



695° K



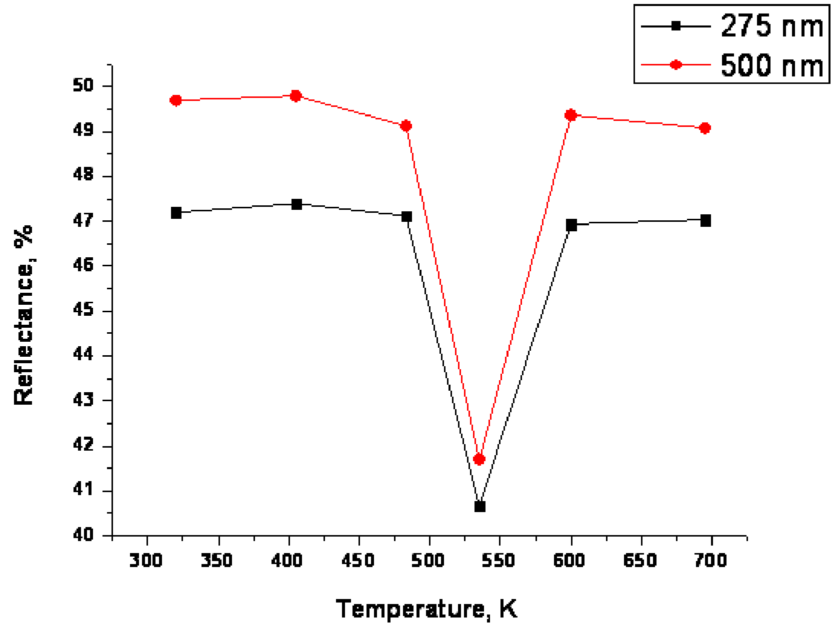
Interference microscope data for 535 K mirror sample

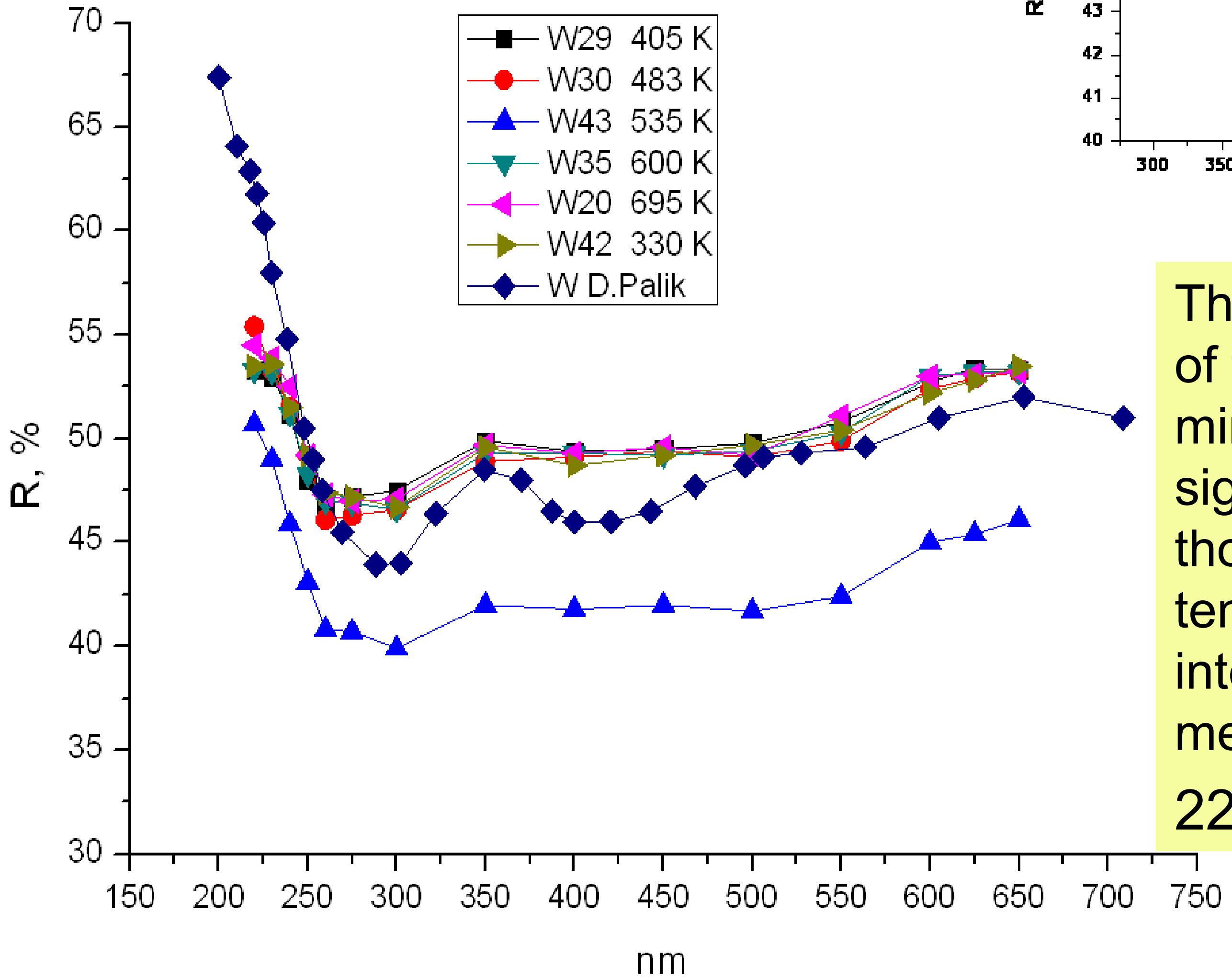


Strange shape of grain boundaries was observed.

No difference between levels of different grains, i.e. no sputtering (E_i is much less the sputtering threshold).

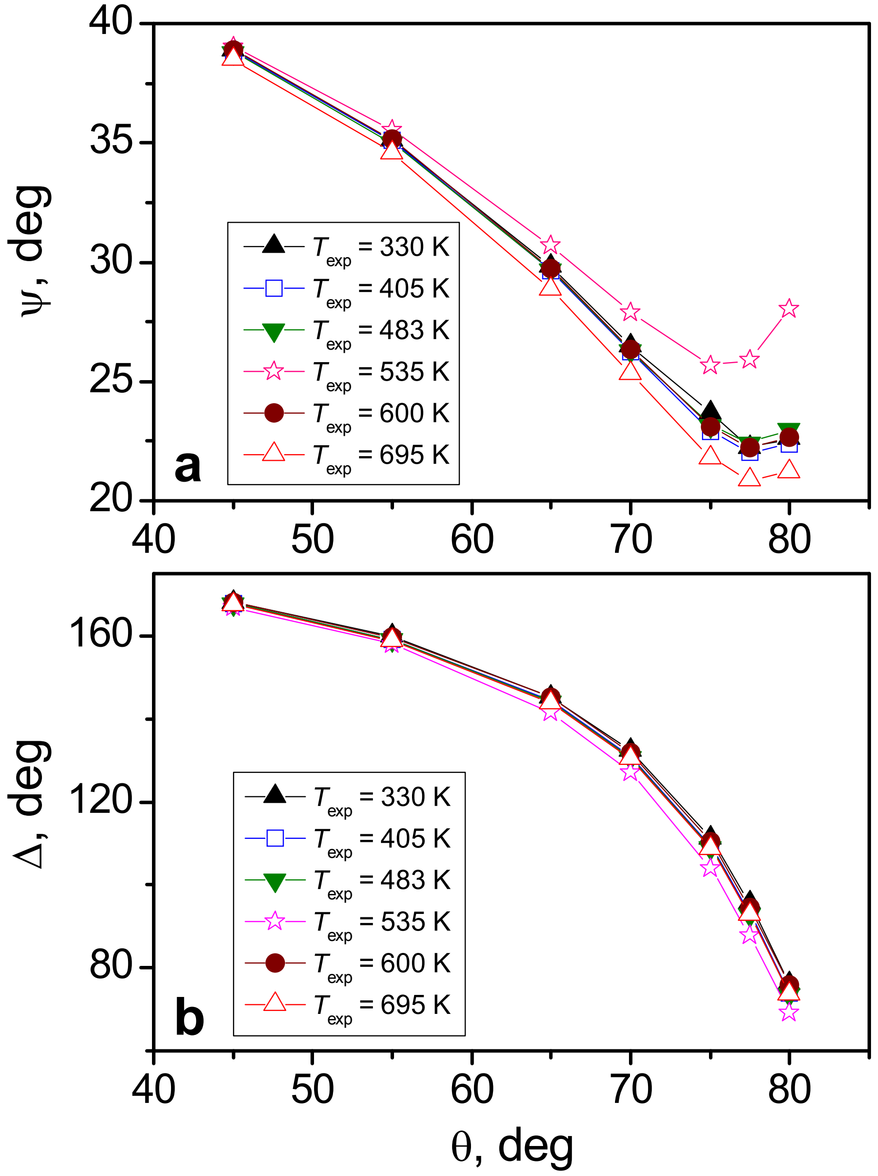
Reflectance at normal incidence of all **W** samples exposed to E_i=38 eV **D^+** ions



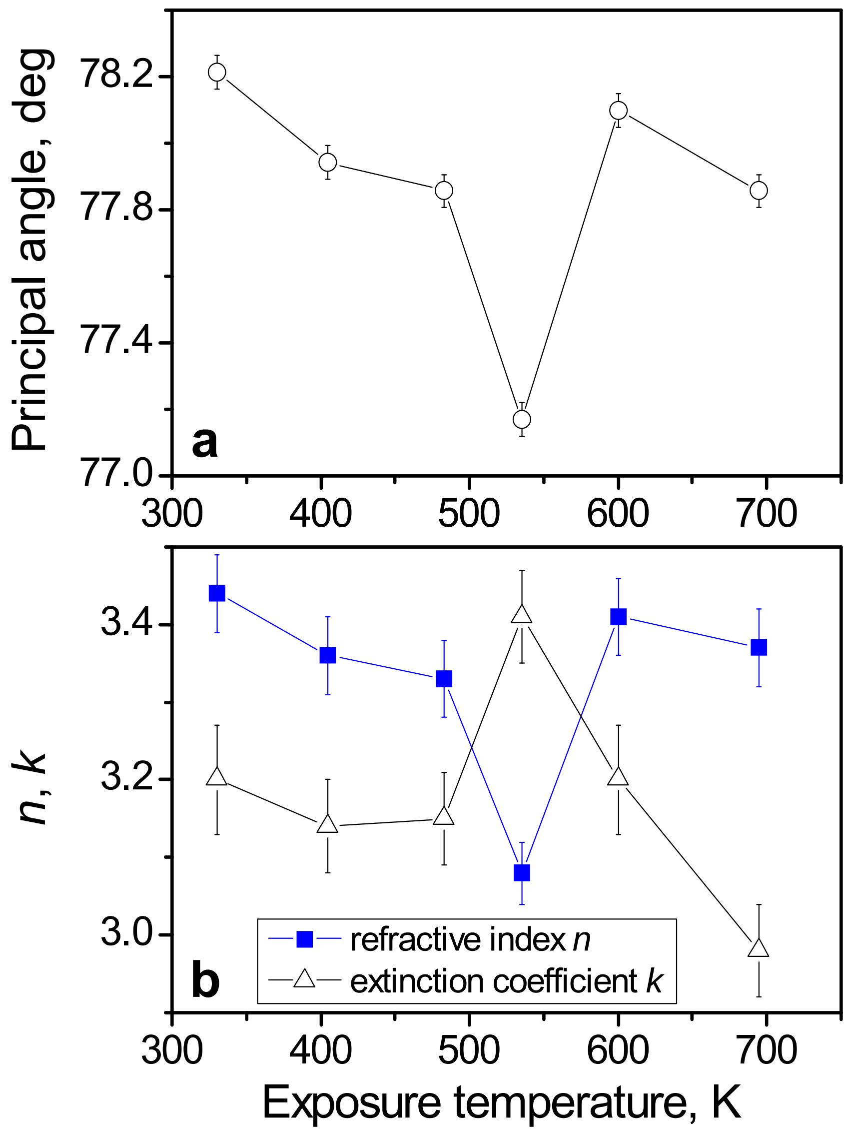


The specular reflectance of the most blistered mirror sample (**535 K**) is significantly below than of those exposed at other temperature in the whole interval of spectral measurements:

220-650 nm.



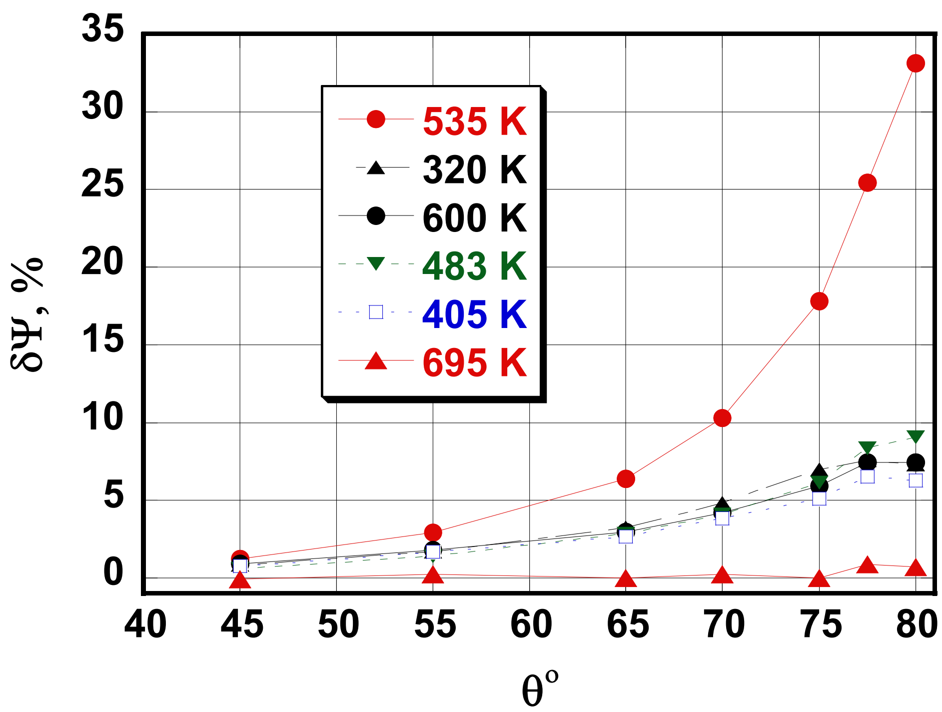
Ψ and Δ versus angle of incidence for all specimens

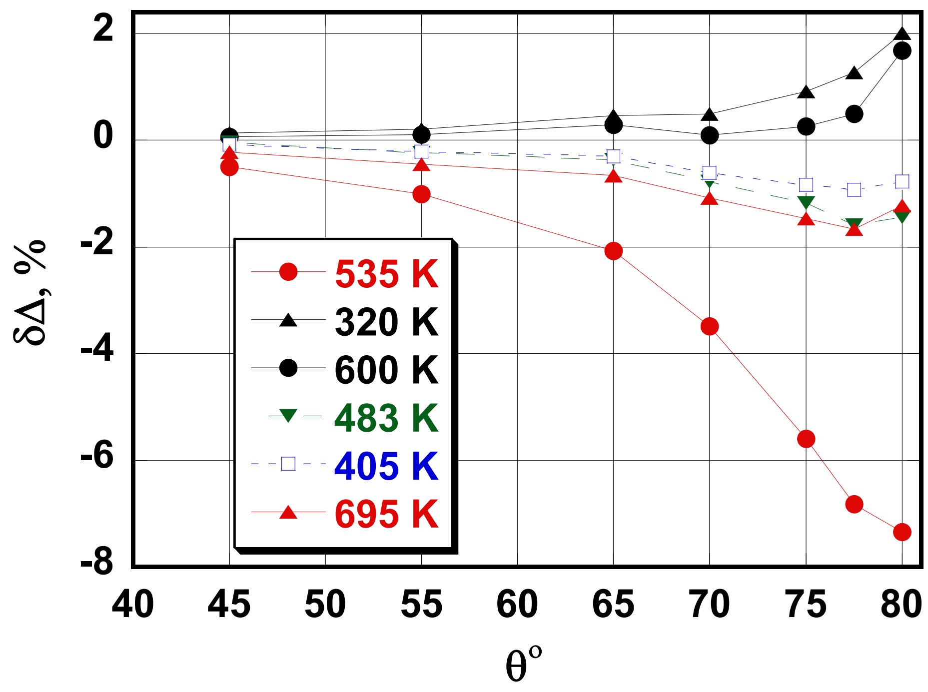


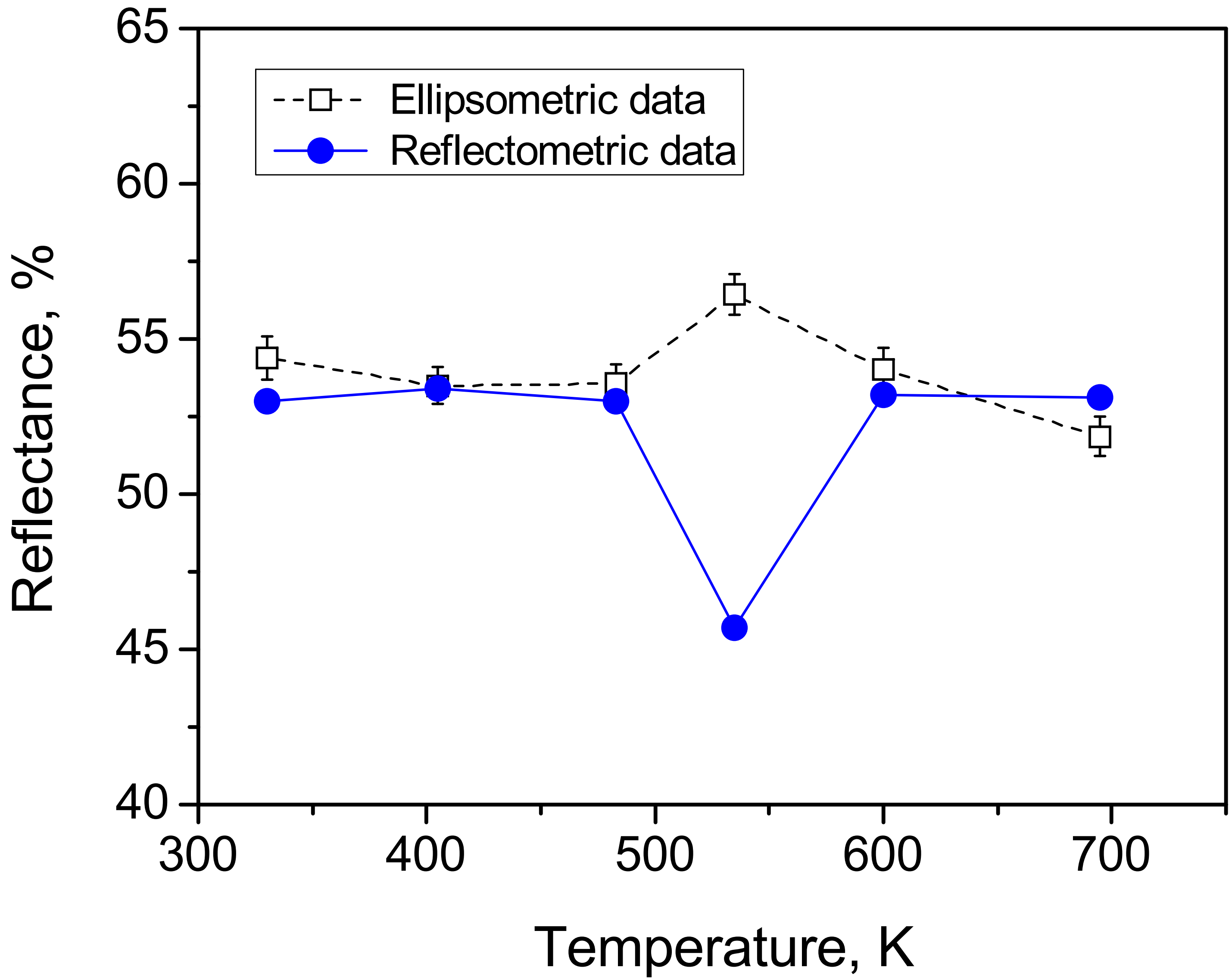
Principal angle and optical indices for all specimens

Much stronger effect of temperature is seen if one compares the relative variations of ellipsometric parameters Ψ and Δ :

$$\delta\Psi(\theta)=\frac{\Psi(\theta)-\Psi_{w0}(\theta)}{\Psi_{w0}(\theta)}\cdot 100\% \text{ и } \delta\Delta(\theta)=\frac{\Delta(\theta)-\Delta_{w0}(\theta)}{\Delta_{w0}(\theta)}\cdot 100\%$$







There is a principal difference between behavior of R(λ) found by reflectometry and ellipsometry for sample exposed at **535 K**.

The reason of this difference is that both methods are based on different physical effects:

- In reflectometry of specular reflection, the **full energy** specularly reflected from the sample is measured, thus the surface defects result in **increase of diffusive component** and, correspondingly, to **decrease of the specular** reflectance.

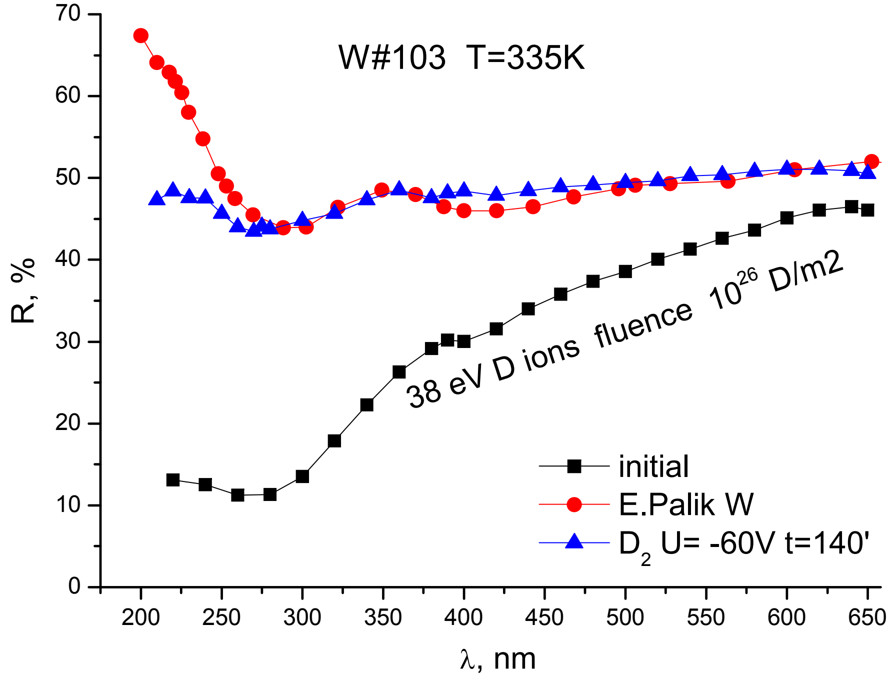
- The ellipsometry methods are based on investigation of changing the polarization state of the **specular component only** and therefore they give the information on the specular reflecting parts of the surface, without taking into account the parts which scatter the light.

In the case of strongly blistered surface (exposure at **535 K**) the ellipsometry brings information about the parts of surface that is still **free from blisters**. Thus the strong modification of ellipsometric characteristics means some **modification of the electronic structure** for this particular specimen as distinct from those exposed at other temperatures.

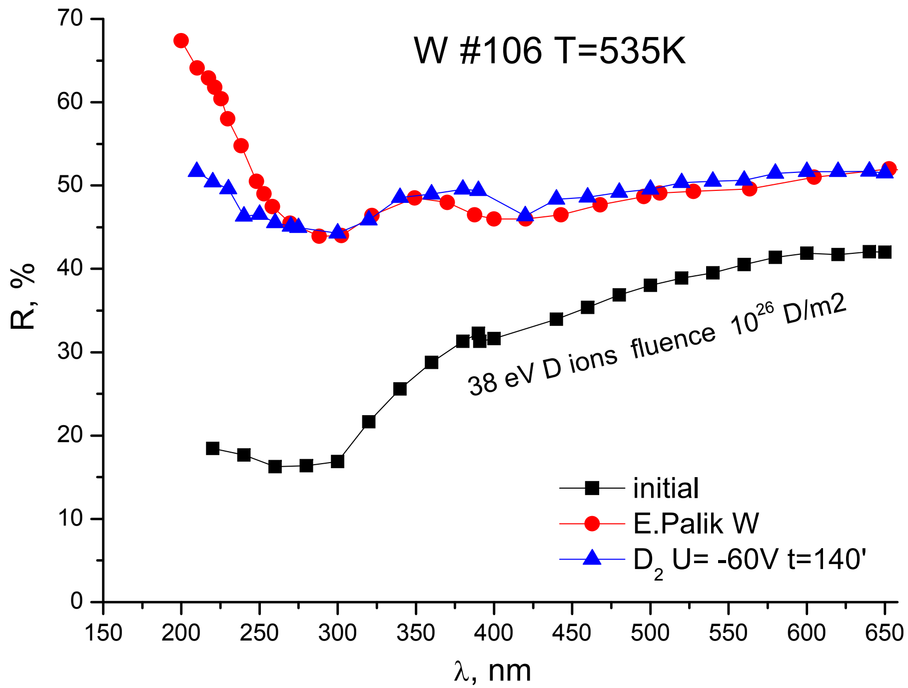
Similar experiment was provided with **polycrystallized ITER-reference W grade samples**

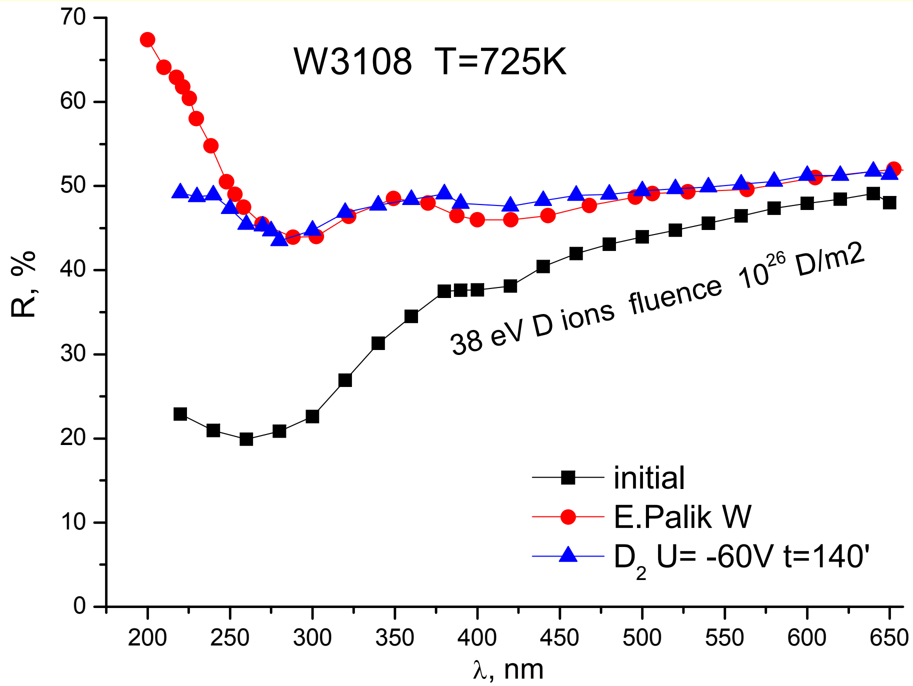
Polycrystallized ITER-reference W grade samples were prepared by other technology. The billets were deformed (rolled, swaged and/or forged) then heat-treated appropriately to obtain better mechanical properties, e.g., strength and toughness, following the sintering process. The microstructure consists of anisotropically elongated grains along the deformation axis and the **grain size is around 1 μm in section and up to 5 μm in length**. The elongated grain orientation is defined to be parallel to the heat transfer direction what corresponds to the ITER specification.

The **W** material has a purity of 99.99 wt.% with principal impurities (in weight **ppm**) of **Mo** and **Fe** ~**10**, **C** and **O** <**30**. Square shaped specimens with a size of **10x10 mm^2** and a thickness of 2 mm were prepared with irradiated surfaces perpendicular to deformation axis. The specimens were double-side polished and cleaned in an acetone ultrasonic bath prior to being placed into the deuterium plasma exposure chamber.



Mirror samples of **ITER-reference W grade** behave very differently: on all samples the number of **blisters** is much less and they have much smaller size (1-5 μm). After cleaning in low temperature D^+ plasma **no any difference in reflectance mirror samples exposed to plasma ions at different temperature was found**.





Conclusions

Summarizing, we can conclude that **two processes** are realized on the surface of **crystallized W mirrors** exposed at **535 K**:

- **(i) strong blistering,**
- **(ii) modification of the electronic structure in the upper-most layer**.
- The fact that the model of a clean surface was found to be optimal, indicates that the modified layer is thicker than the light penetration depth or it has no a sharp boundary.
- Ellipsometry and reflectometry are effectively supplementing each other when such complicated phenomenon like blistering occurs on the surface.
- The **ITER-reference W grade** mirrors are **highly resistant to blistering** independently **on exposure temperature**, and, correspondingly, **no difference was found between reflectance.**