# In-situ materials research and X-ray diffraction

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## In-situ X-ray diffraction

- In gas environment
  - Material for $H_2$ separation from gas mixtures
- During tensile test
  - Transformation-induced plasticity
- Damp – heat – voltage conditions
  - Degradation of transparent conductive oxide

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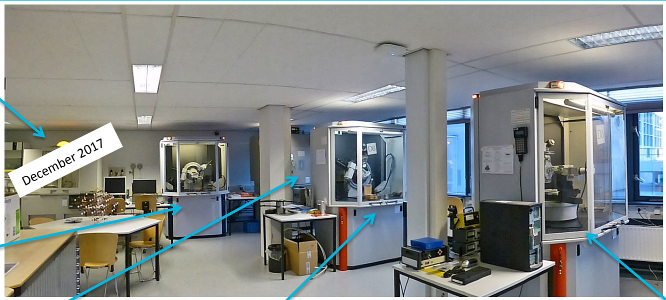
# X-Ray facilities at Materials Science




**December 2017**
**theta/theta : 2D detector**
**HT attachment + tensile test set-up**

**theta/theta : 1D detector + sample changer.**
***identification***

**theta/theta : 0D detector**
**HT attachment (LN2 – 1500 °C) + humidity generator**
***phase transformations, gas material interaction***

**theta /2theta:**
**1D detector + home made diffracted beam mono-chromator.**

**parallel beam: 0D detector + diffracted beam mono-chromator + Eulerian cradle**
**tensile test set-up + HT dome**
***stress and texture in steel, thin films, solar cells, gas material interaction***

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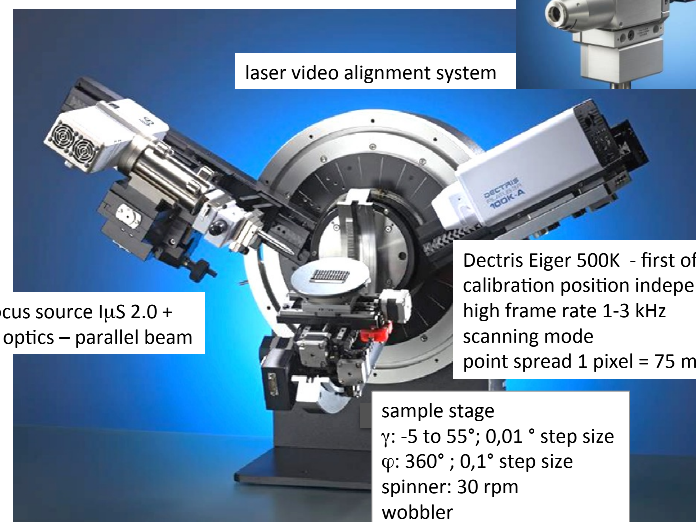
# Instrument



laser video alignment system

microfocus source IμS 2.0 +
Montel optics – parallel beam

Dectris Eiger 500K - first of v3
calibration position independent
high frame rate 1-3 kHz
scanning mode
point spread 1 pixel = 75 micron

sample stage
γ: -5 to 55°; 0,01 ° step size
φ: 360° ; 0,1° step size
spinner: 30 rpm
wobbler

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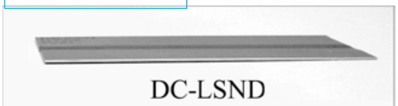
# X-ray Source: IμS – high brilliance

- Micro focus tube with Montel optics


output 4x classical rotating anode

Flux: 3-6 $10^8$ cps/$mm^2$
Beam divergence:
0.06°
Beam size on sample: 0,1 x0,1 to 1,9x1,9 $mm^2$

Local analysis




weld: position dependent phase identification
stress analysis

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# In – situ devices and applications

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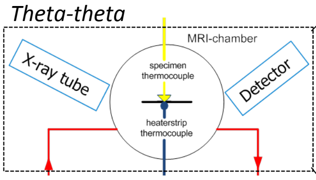
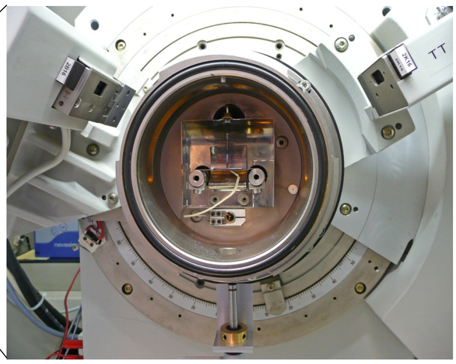
# In gas environment – LT & HT

*Effect of gas environment and temperature on micro- and macro-stress (0D)*

- In-situ changes in stress in thin films and coatings ($\omega$ –tilt)
- Phase transformations

*Theta-theta*



Set-up for reactive gas environment from $LN_2$ to 1500 °C

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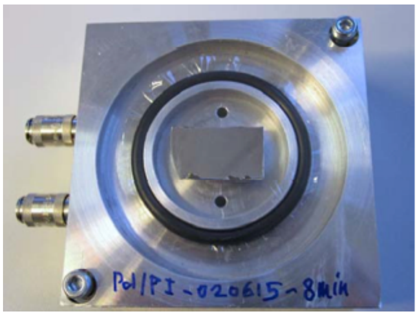
# In gas environment - instruments

*Effect of gas environment on micro- and macro-stress and texture evolution (2D)*

- Anisotropic behavior, stress tensor analysis and delamination




Set-up for reactive gas environment at RT


Set-up for inert gas environment RT to 1100 C°

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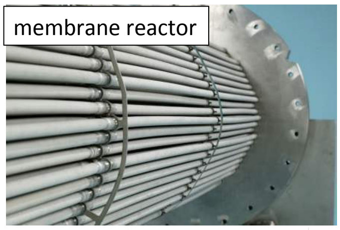
# Membranes for gas separation

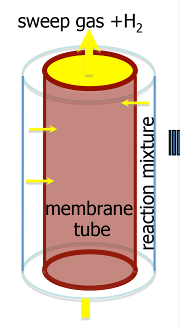


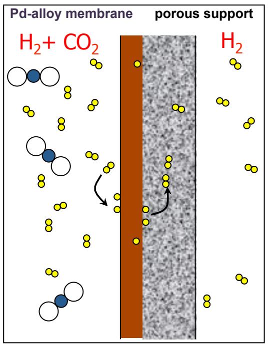

Steam reforming: $CH_4 + H_2O \rightleftharpoons 3\,H_2 + CO$ ($\Delta H$ = 206 kJ/mol)

Water-gas shift: $CO + H_2O \rightleftharpoons H_2 + CO_2$ ($\Delta H$ = - 41 kJ/mol)

$CH_4 + 2\,H_2O \rightleftharpoons 4\,H_2 + CO_2$

remove $H_2$ from reaction mixture




Membrane requirements:

- Stable alloy
  - *no delamination*
- Catalytic: $H_2$ dissociation
- Process Conditions
  - T: 550 – 950 K
  - P: 10 - 50 bar
  - Impurities:
  - $H_2O$, $H_2S$, $CO$, $CO_2$

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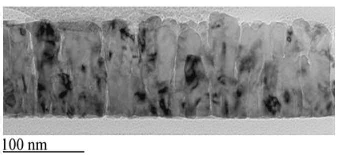
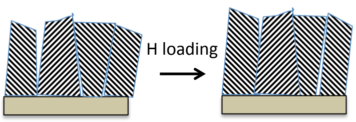
# Membranes for gas separation

- Failure by delamination
  - uptake H leads to $\Delta V/V \sim 10\%$ for Pd-based alloys
- Nano-structuring
  - Quasi-free expansion

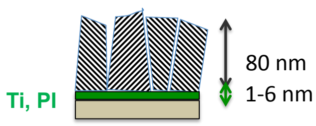



Brush membrane concept




Loose nano-sized columnar structure : sputter conditions

- adhesive layer:

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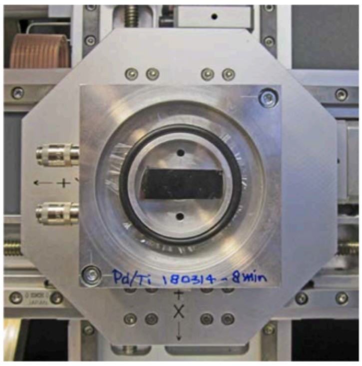
# Improve adhesion

- Hydrogen loading and de-loading cycles
- Analysis of:
  - Phase transformation
  - Stress
  - Texture

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# Improve adhesion

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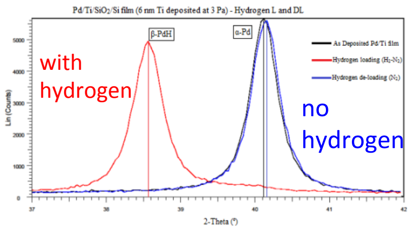
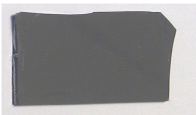
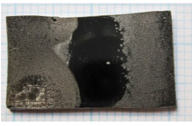



**Pd film**

5 cycles



20 cycles



**Pd on Ti film**

20 cycles

**Pd on PI film**

20 cycles

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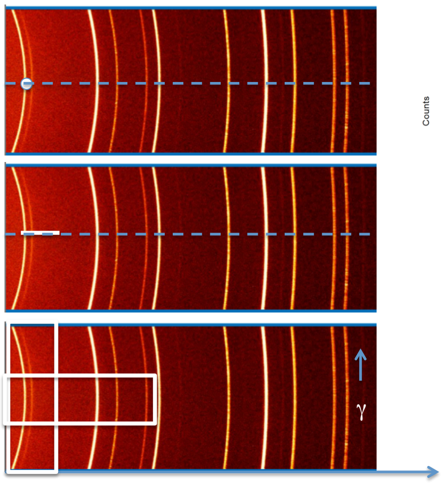
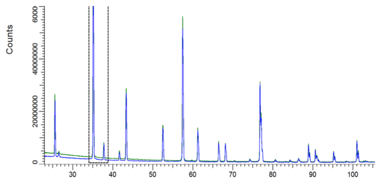
# Diffraction from 0D to 2D detector

Example: Corundum, SDD 225 mm




0D: point detector




1D: line detector typically 5-10°

2D: area detector typically 10° x 20°

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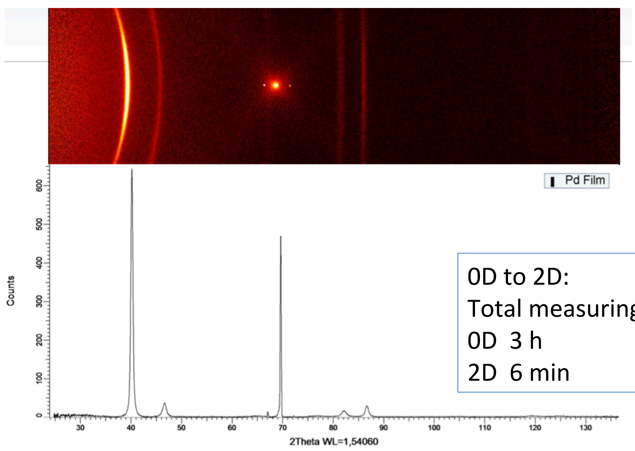
# Pd film 2D detector




0D to 2D:
Total measuring time:
0D 3 h
2D 6 min

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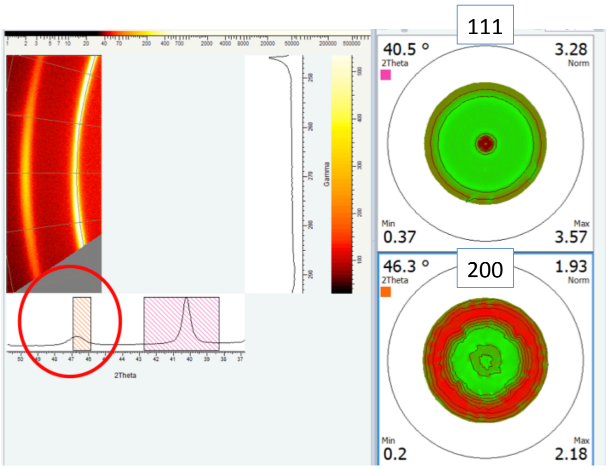
# Pd on Ti film 2D detector

Texture:
(111) fiber texture

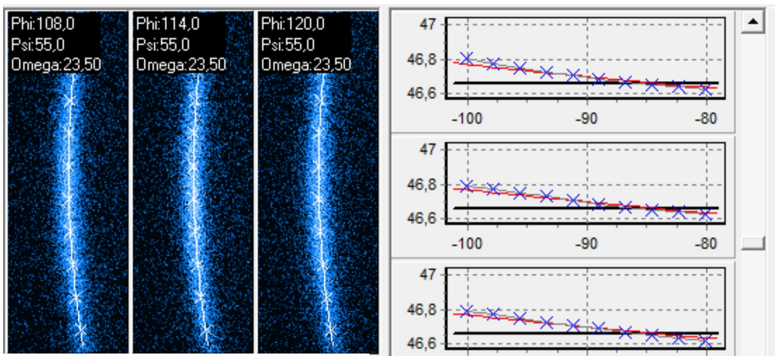
0D to 2D:
Total measuring time:
0D 6 h
2D 14 min






# Simultaneous Texture & Stress : Pd on Ti

Pd with Ti adhesion layer



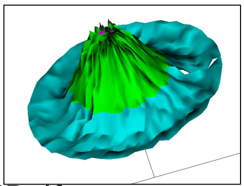
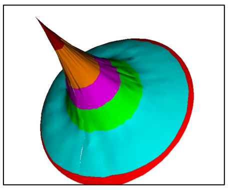

Biaxial state of stress : 870 ± 7 MPa

Simultaneous characterisation of texture and stress

# Optimal film performance

Pd on PI : good adhesion – no delamination
high performance – solubility and kinetics

- Resulting optimal film characteristics:
  - Open nano-structure
  - Texture: ‘weak’ texture 1,5 x random
  - Stress: ~ 100 MPa

Pd on PI film

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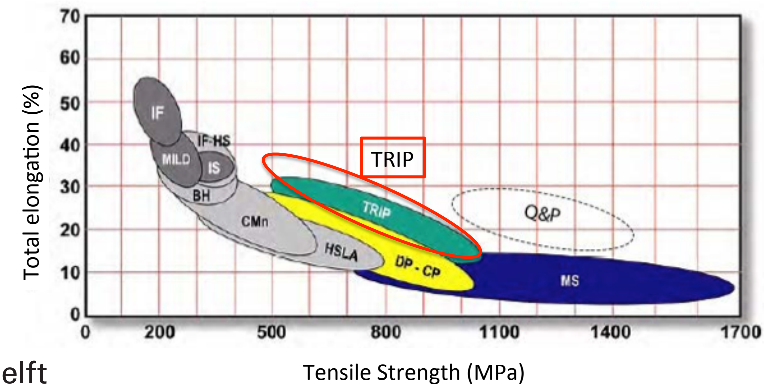
# TRIP steel

- Automotive :
  - maximal elongation at maximal strength
  - austenite – martensite transformation

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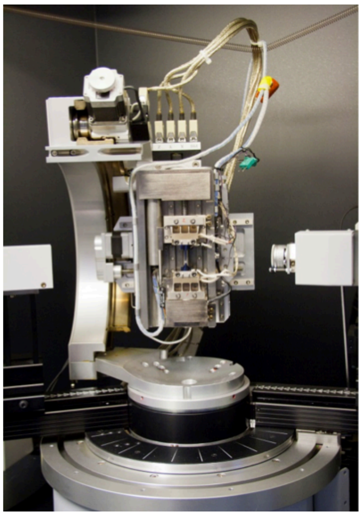
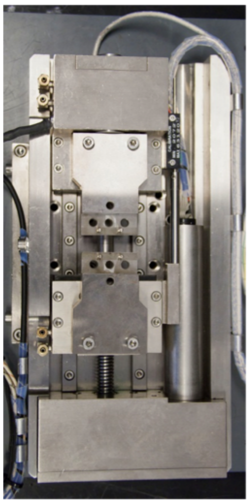
# In-situ tensile testing





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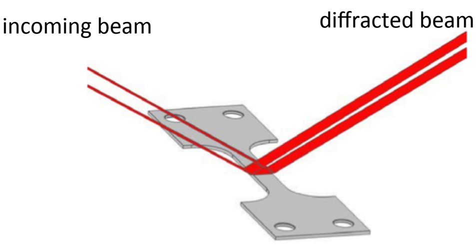
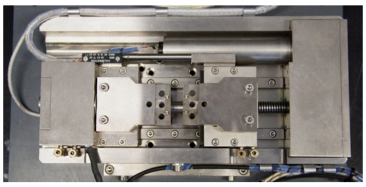
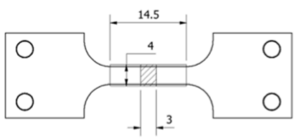
# In-situ tensile testing






Maximum force : 5 kN, RT, specimen size: L: 54 mm

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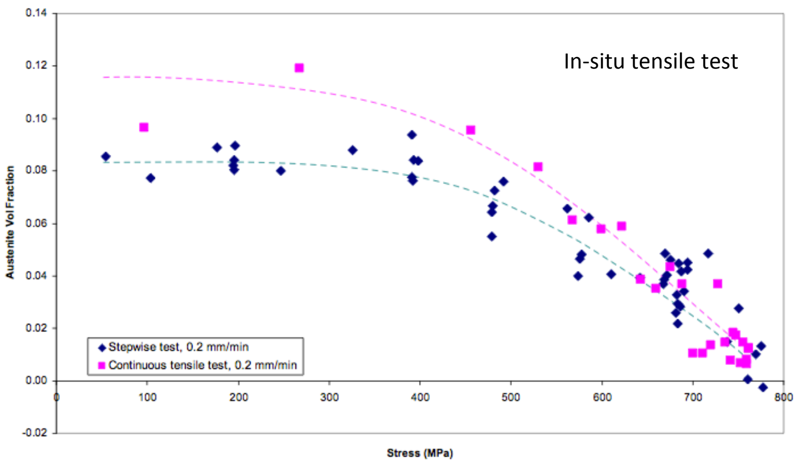
# Austenite – Martensite Transformation




*Ma Thesis T. Citrawati (2008)*

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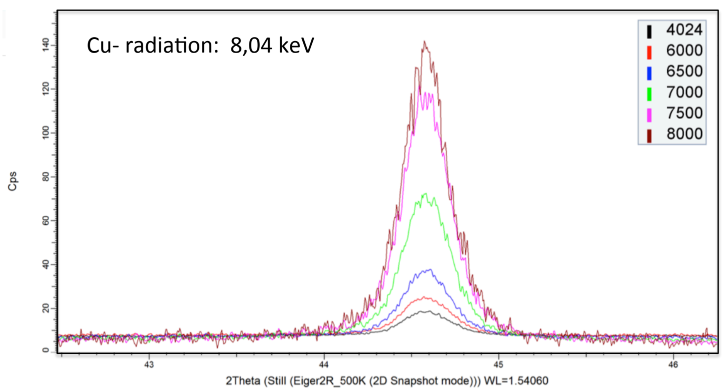
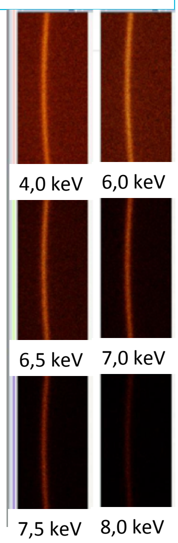
# Suppression of Fluorescence




Fluorescence of Fe (7,11 keV), Mn (6,5 keV)

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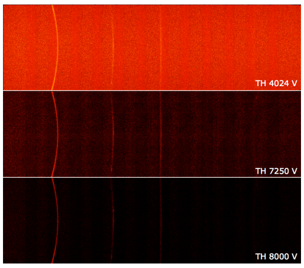
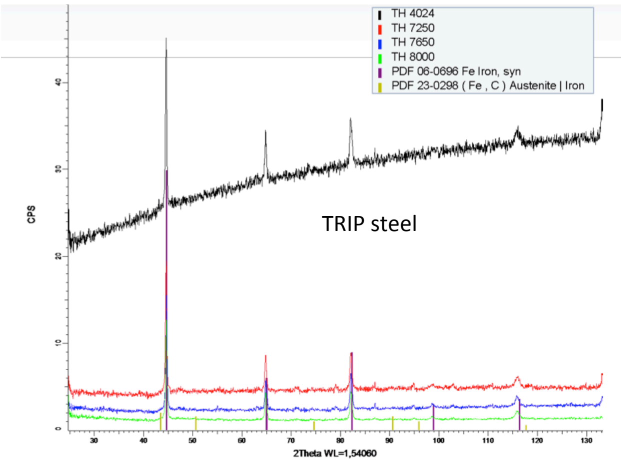
# Suppression of Fluorescence




Detector threshold settings




Parallel beam; Cu radiation;
2 min collection time;
detector 130 mm distance

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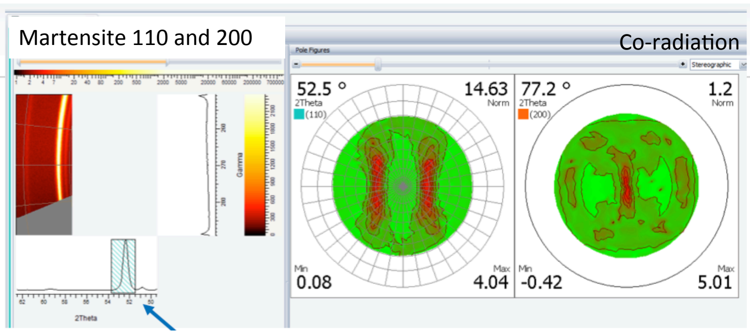
# Texture: martensite – austenite

TRIP steel




Data collection 5s per frame 144 frames per PF.
Total measuring time: 24 min

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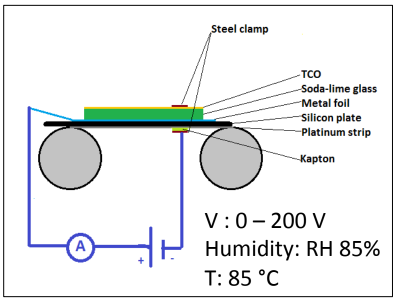
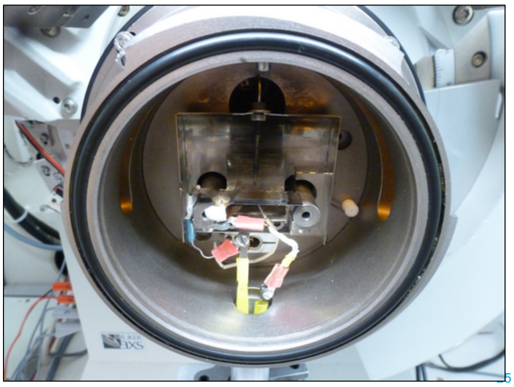
# In-situ degradation of TCO

- Transparent conductive oxide:
  - degradation under operation conditions:
  - damp - heat – illumination (applied voltage)



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