DETERMINATION OF ADHESION AND MECHANICAL PROPERTIES OF OPTICAL COATINGS BY NANOSCRATCH AND NANOINDENTATION

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Mechanical properties of optical film: external contact

Optical coatings are <u>not</u> primarily wear and scratch resistant

- Typical optical coatings (Mo, Si, TiO_2, SiO_x, MgF_2) have rather low hardness and wear resistance

But optical components are in contact with hard materials

- Mechanical damage will deteriorate the optical properties[3]
- The coating can delaminate

The hard layer or the protection must have[4]

- High hardness
- High scratch resistance
- Do not interfere with optical properties

[3] Price, James et al. "Nanoindentation Hardness and Practical Scratch Resistance in Mechanically Tunable Anti-Reflection Coatings." *Coatings* 11 (February 2021).
[4] Paulson, Charles A. et al. "Industrial-Grade Anti-Reflection Coatings with Extreme Scratch Resistance." *Optics Letters* 44, no. 24 (December 15, 2019): 5977–80.

Challenge: optical coatings are thin

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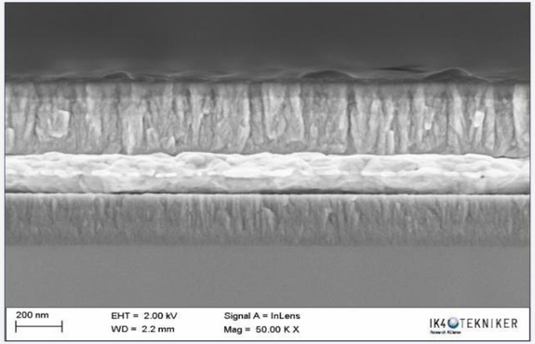
Typical thickness of optical coatings – related to wavelength

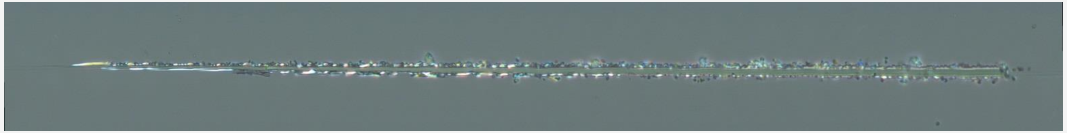
- Tens and hundreds of nanometers
- Often multilayers

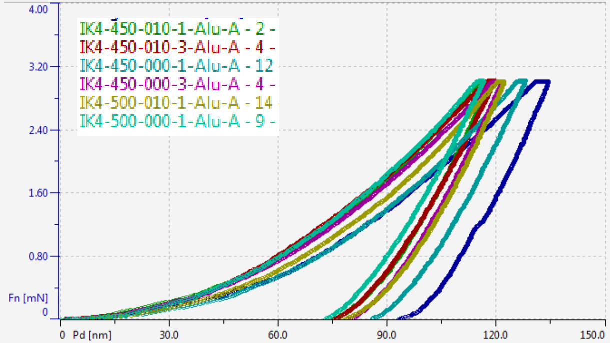
How can we measure their hardness and scratch resistance – or even adhesion?

- Nanoindentation: using low forces and depth profiling
- Nanoscratch: scratch at low forces with optical or SEM analysis

Multilayer absorber coating (Tekniker)







Nanoindentation

Hardness and elastic modulus of thin films

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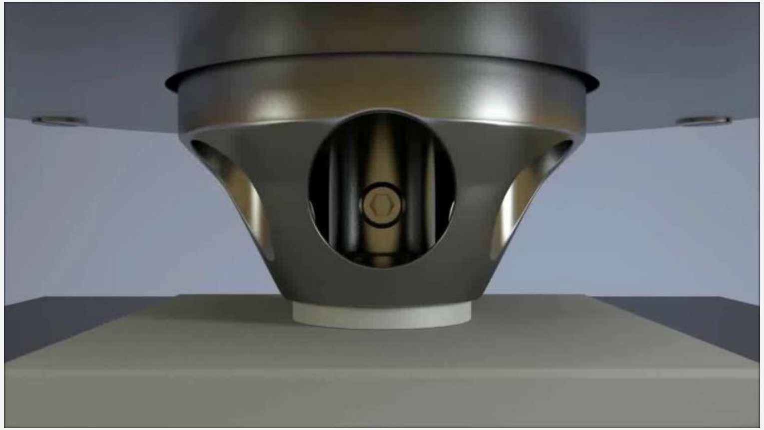
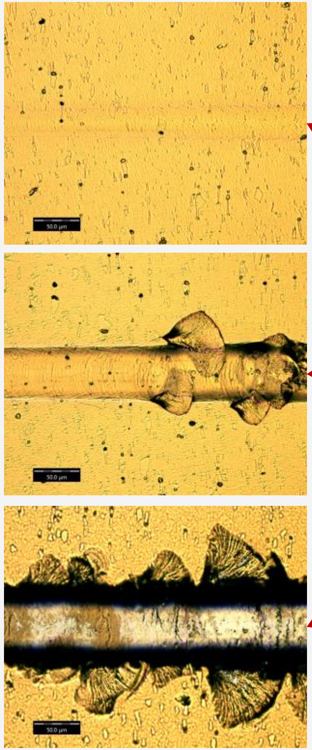
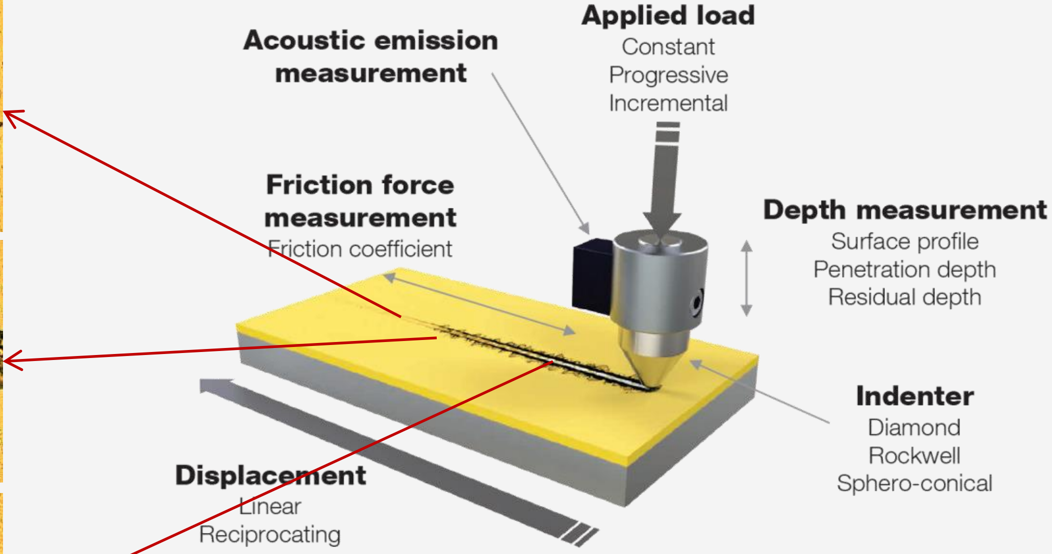


- We applied force and we measure indentation depth
- Hardness and Elastic Modulus are calculated from the force-displacement curve
- Anton Paar: surface referencing for increased thermal stability and elimination of frame compliance
- Lowest indentation depths down to ~5 nm
- Standardized hardness and elastic modulus calculations (ISO 14577)

Nanoscratch testing

For determination of scratch resistance and adhesion





Cohesion/adhesion by critical load events from optical observation

Case study: AR coating on glass cover for photovoltaics

- Glass increases stiffness of the PV cells
- Protects the cells against mechanical damage
- Wear and damage from impacts of dust, rain and sand
- Scratching by hard particles but relatively low force

  - Glass is coated with antireflective coating
  - Large panels: sol-gel deposition method
  - Thickness of the coating: ~120 nm

[1]Agustín-Sáenz, C. et al: *Solar Energy Materials and Solar Cells* 216 (October 2020)

Characterization – nanoscratch & nanoindentation

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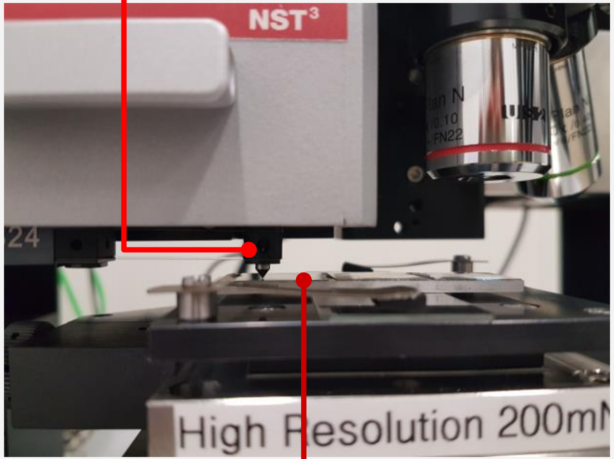
Scratch tests by Nanoscratch Tester (NST)

– Low load – thin films
– 2 µm radius spherical diamond indenter

Spherical diamond indenter



Glass with AR coating

Berkovich diamond indenter

Active surface reference

Hardness and elastic modulus by UNHT

– Berkovich indenter (diamond)
– Force 10 µN up to 50 µN

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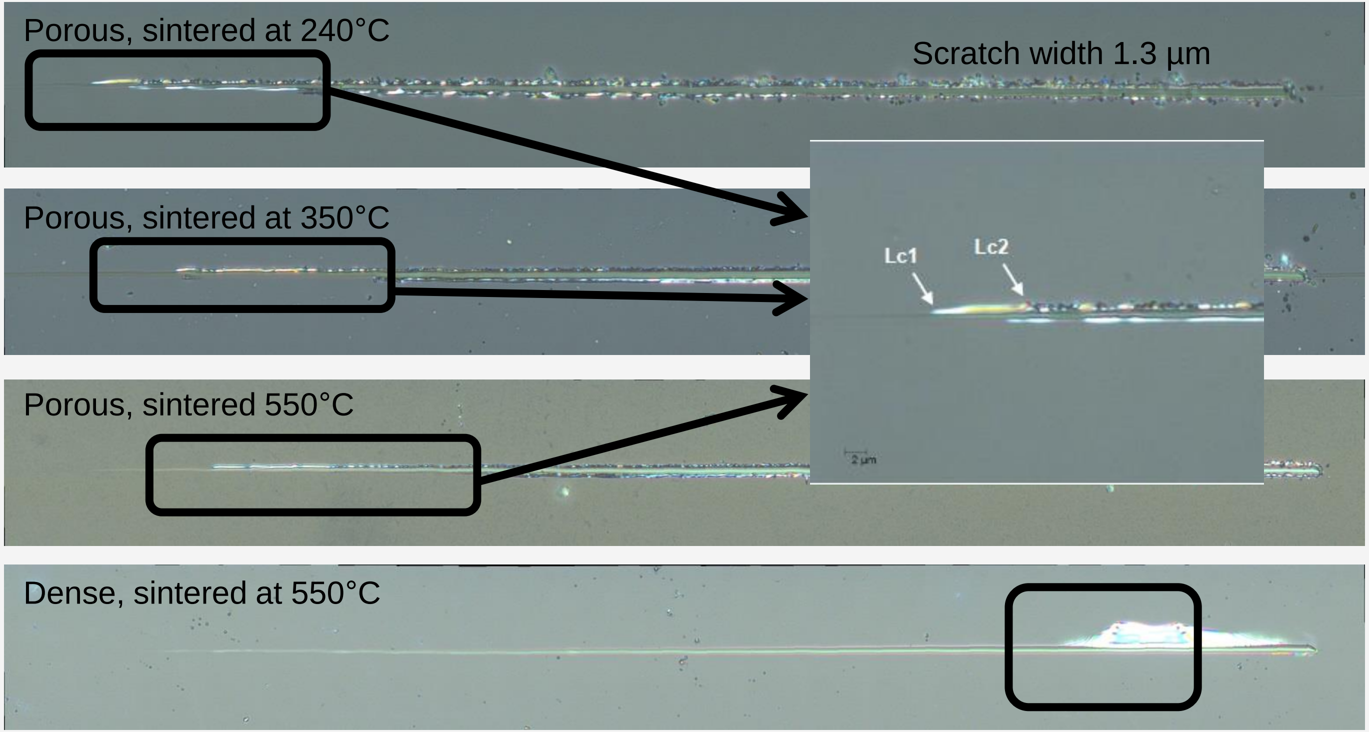
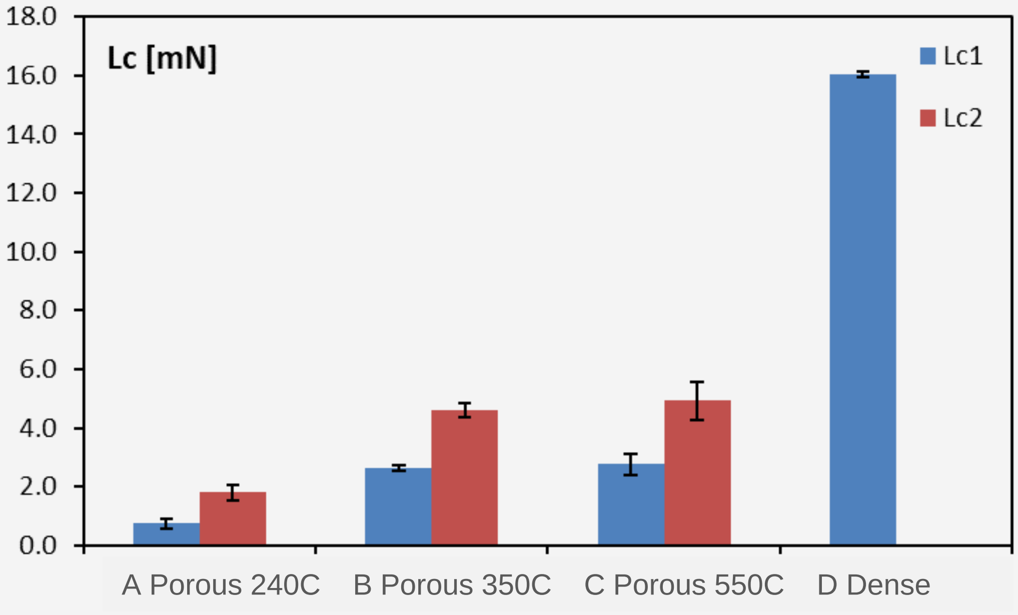
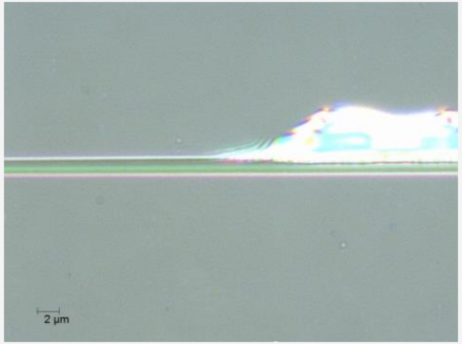
Results – scratch test

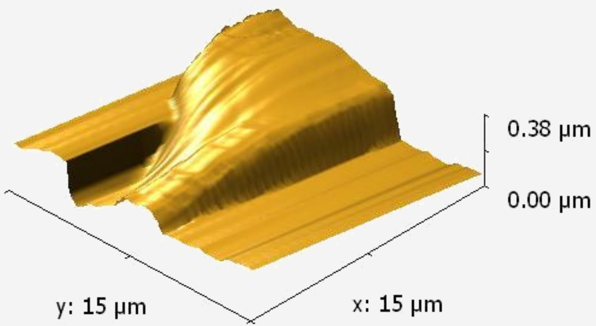
Panorama image of the scratch track



Scratch: comparison of critical loads







Atomic force microscope (AFM)

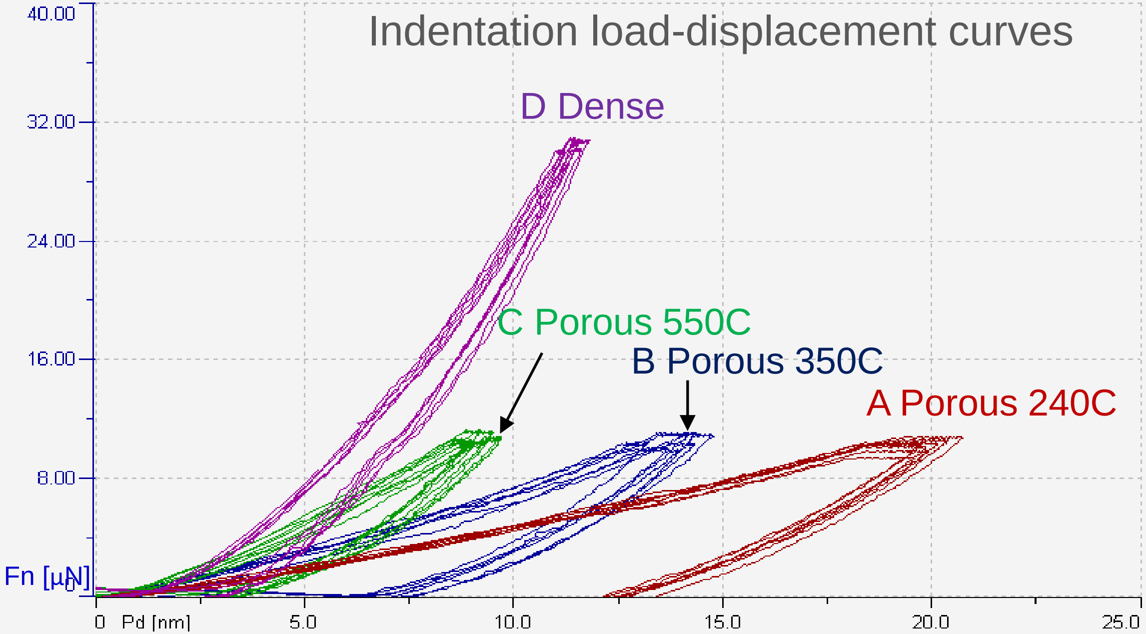
Conclusion – adhesion

– Nanoscratch can determine quickly the adhesion of thin AR films

Nanoindentation

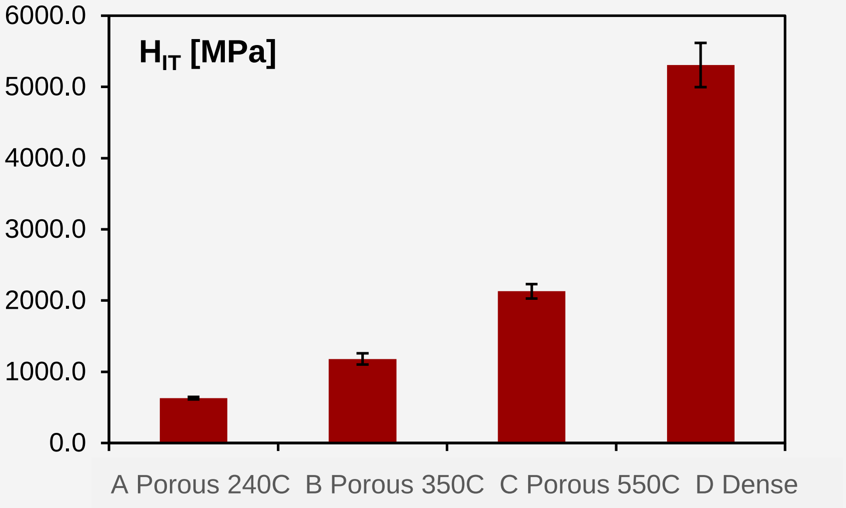
Thin films (~100 nm) ⇒ low indentation depths & loads

- F_{max} 20 µN,
- Maximum depth 10 nm to 20 nm

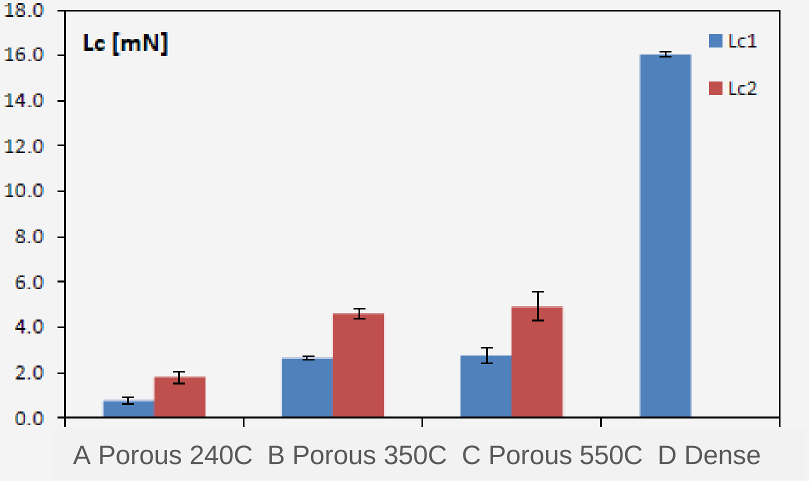


Indentation & scratch test results: comparison

Indentation



Scratch



Correlation – harder films have better adhesion

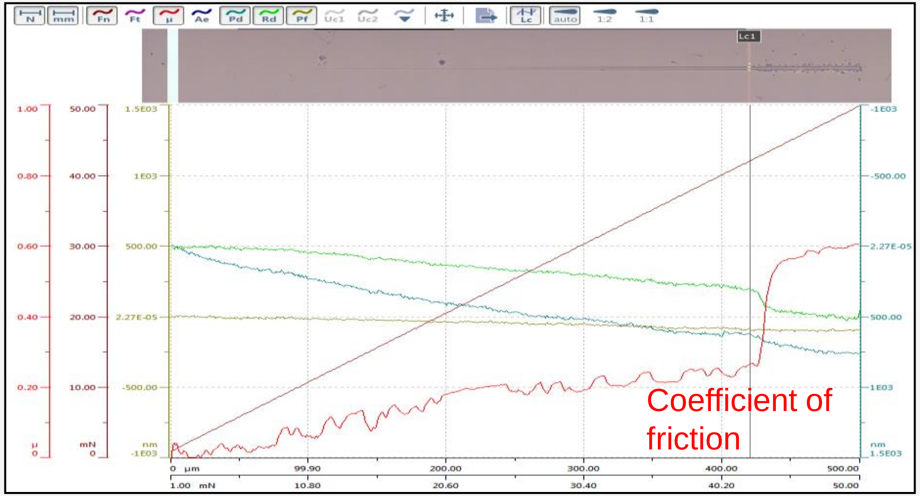
- Not a rule - but this is why we measure it

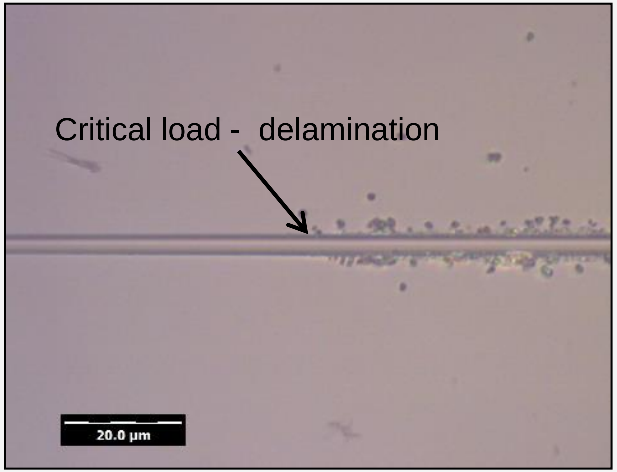
Sapphire coated Gorilla glass: nanoscratch

– Thin wear resistant film ~120 nm

– Critical load determined by integrated optical microscope

– Analysis of other signals (coefficient of friction) ‘support’ the optical analysis





Typical data acquired during a progressive scratch with panorama image

Red curve: Coefficient of friction (CoF)

Nanoscratch is just the beginning

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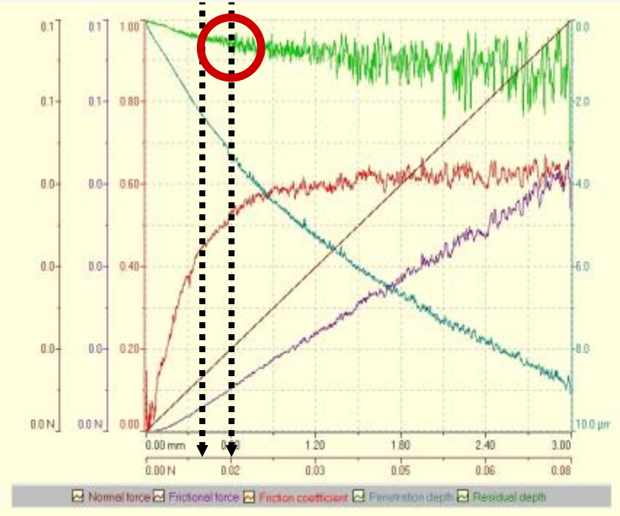
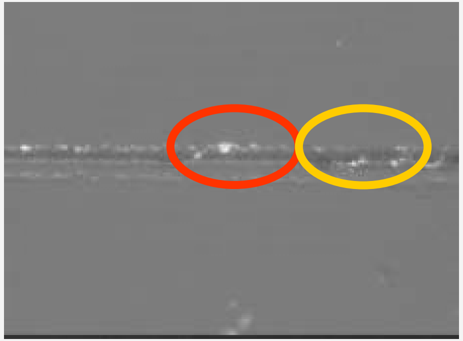
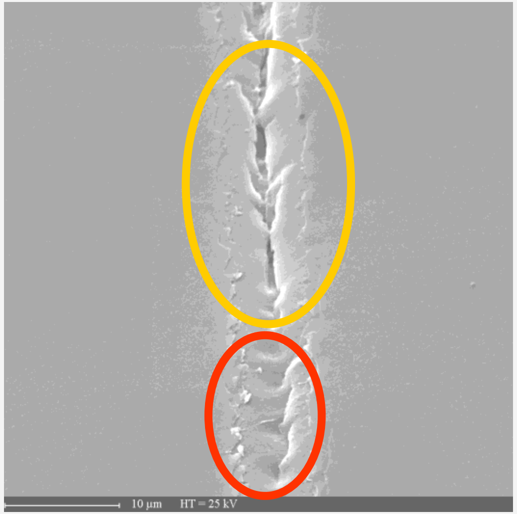




Integrated optical microscope



Scanning Electron Microscope (SEM)

SEM Observations – more detailed info about the film failure

- Critical load Lc1: Transition from AR layer → hard layer
- Critical load Lc2: Transition from hard layer → substrate

Nanoscratch & nanoindentation for optical layers

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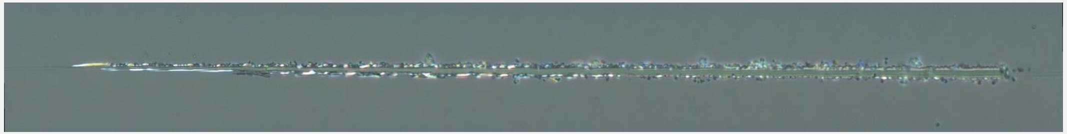
Nanoscratch: scratch resistance and adhesion by defined scratching

- Applicable also on very thin coatings (~50 nm)
- SEM for more details



Nanoindentation: hardness and elastic modulus of thin layers

- Indentation depths down to ~10 nm
- Automatic measurements