

Introduction to scratch testing: Adhesion and scratch resistance

EPFL SUMMER SCHOOL 2025

VIRGILE FAVRE AND XAVIER GUTHMANN



Established in 1922 in Graz, Austria

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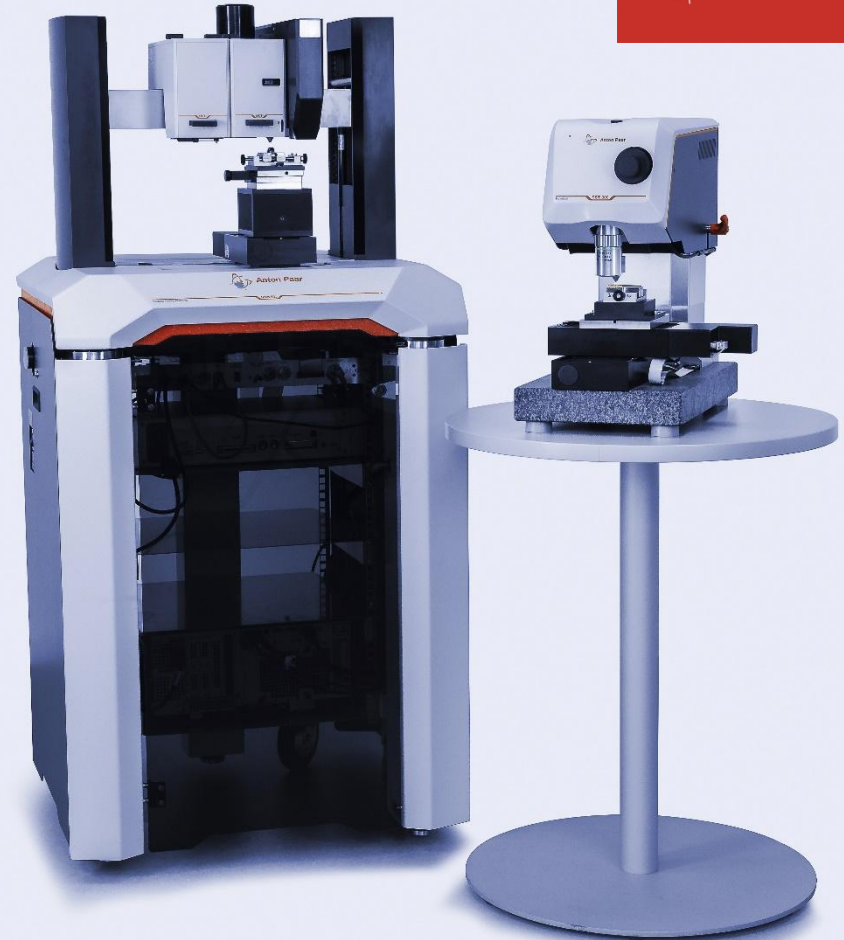
14.5 % INVESTMENT
IN R&D



- › **MECHANICAL SURFACE CHARACTERIZATION UNIT**
- › BASED IN CORCELLES (NEUCHATEL)
- › DEVELOPMENT AND PRODUCTION OF SCRATCH TESTERS, NANOINDENTERS AND TRIBOMETERS

Outline

- › Basic industrial methods for adhesion measurement
- › Principles of scratch test
- › Adhesion and scratch resistance by a scratch tester
- › Features of a typical scratch tester
- › Applications of scratch test
- › Summary



Basic methods for adhesion estimation

Knife test

- Two cuts are made through the film to the substrate with intersecting angle of 30° to 45° , using an utility knife and a cutting guide.
- The knife is used to peel the coating from the intersection point.
- A standardized method is given in ASTM D6677 or ISO 16276-2.

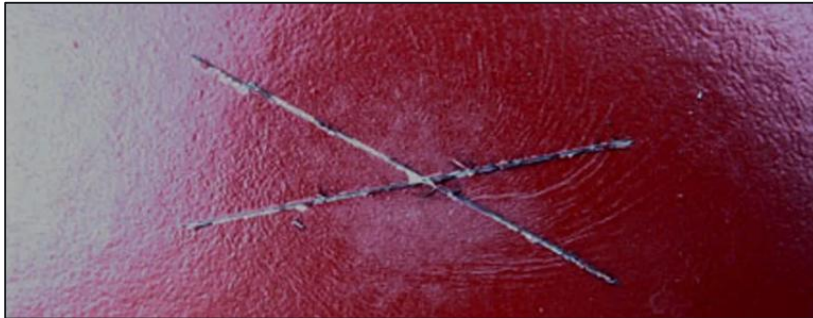


Image source: www.fitz-coatings.com

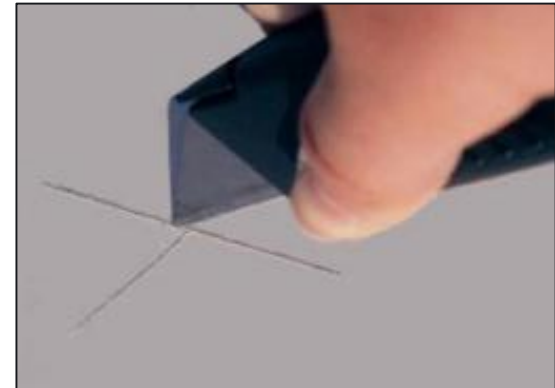


Image source: www.preservationproducts.com

Basic methods for adhesion estimation

Tape tests

- › X-cuts with intersecting angle of 30° to 45° or cross hatch cuts are made through the coating to the substrate using a knife and cutting guide
- › Self-adhesive tape is glued on the cuts and then pulled off
- › The coating adhesion is rated from 0 to 5 in compliance with ASTM D3359
- › Cross-hatch test: similar principle but for less adherent coatings

X-cuts

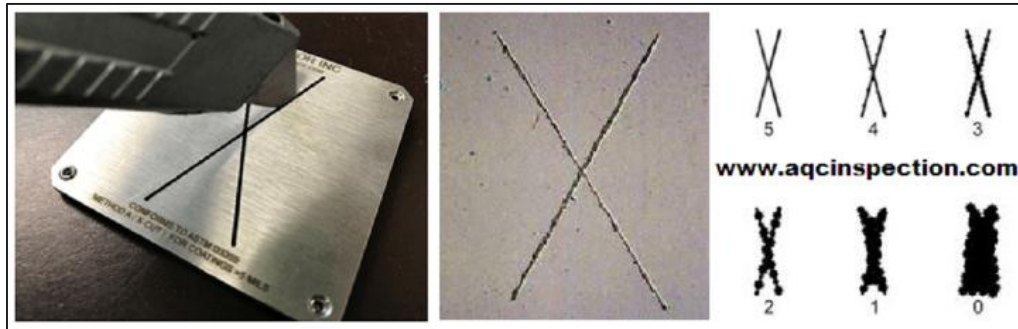


Image source: www.aqcinspection.com

Cross-hatch test

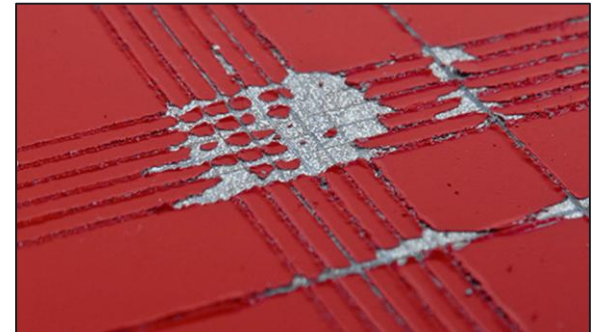


Image source: www.laboratuar.com

BASIC INDUSTRIAL methods

Pencil test

- › A standard wooden black lead pencil of known hardness is pushed over the coating at an angle of 45° .
- › The hardness of the pencil is increased from 6B (very soft) to 6H (very hard) until permanent deformation or rupture of the coating is observed.
- › The hardness of the hardest pencil which does not mark the coating is considered as final result.
- › The standardized test method is described in ISO 15184.



Basic methods for adhesion estimation

Drawbacks

- › Test protocol and analysis are often **dependent on the operator's skills**
- › Results are **not quantitative (result is not a number, value)**
 - › **Test results can vary** among production batch and manufacturers:
 - Type and shape of cutting blade, tape, glue, steel wool, pencil...
 - › Testing method are often **limited only to specific samples**:
 - › Flat samples for scrape test, Taber Abraser® test or pencil test
 - › Ceramic coatings not suitable for Scotchbrite® abrasion test...



What about hard and thin ($\sim\mu\text{m}$) ceramic coatings?

What about paints?

What about very thin coatings in semiconductor and electronics?

Basic industrial methods not sufficient

Cross-hatch test

- > Lacquered screw cap for cosmetics bottle
- > Cross-hatch test for adhesion – not able to determine differences in adhesion



Cross-hatch cuts

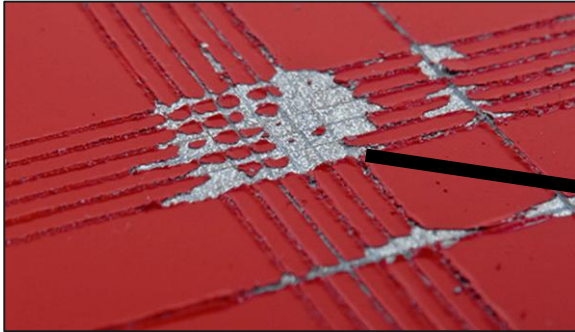
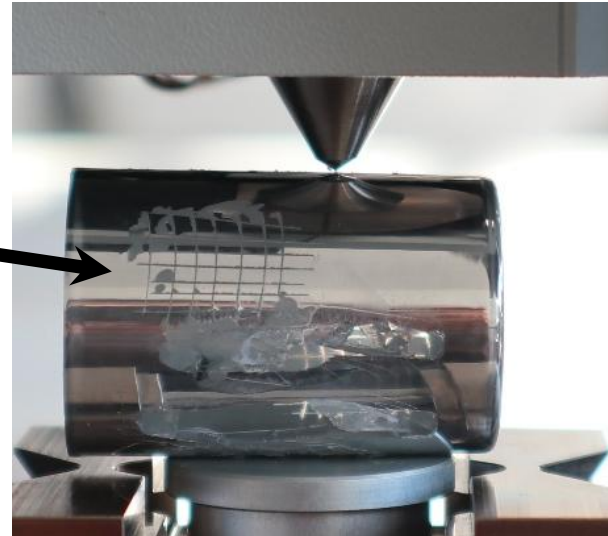


Image source: www.laboratuar.com



More sensitive scratch test method required!

New requests for adhesion testing

- New materials, new surface treatments, thin layers and coatings ($\sim\mu\text{m}$ range)
- More precisely defined test conditions
- Quantitative results: not qualitative

→ **Basic industrial methods are not sufficient!**



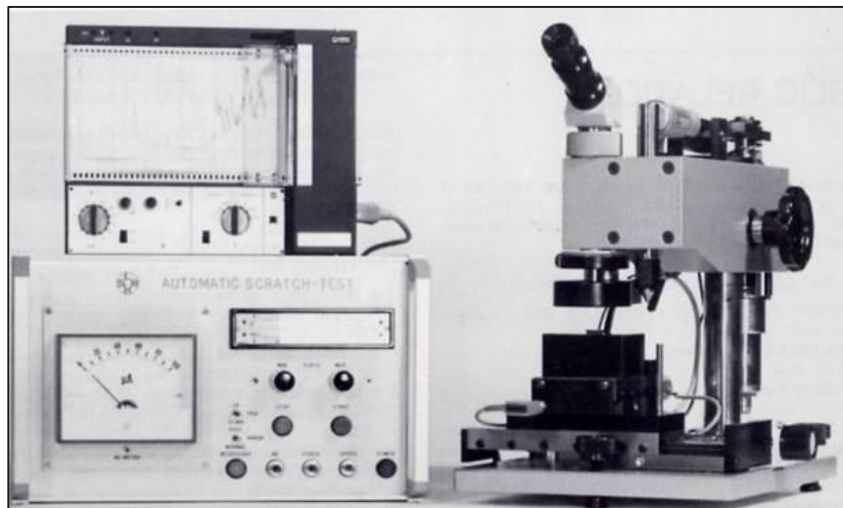
Scratch test with a diamond indenter – method suitable for much broader group of applications!

Principle of scratch test for adhesion and scratch resistance

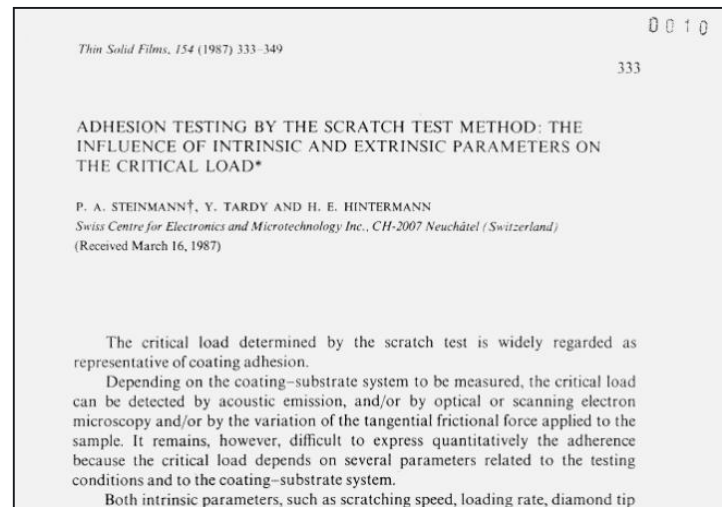
The first scratch tester

- The first industrial scratch tester, the REVETEST[®], has been developed in the 1980's by LSRH company in Neuchâtel (CH) which was acquired by Anton Paar in 2013
- It was a logical answer to the need of adhesion measurement of hard coatings used for cutting tools and watch industry

First Revetest[®] scratch tester

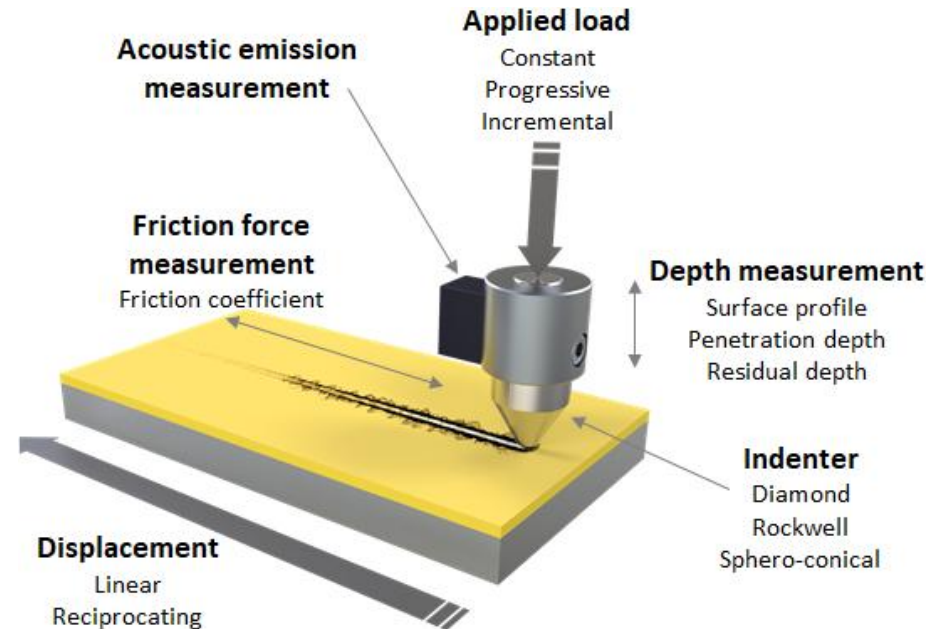


First publications about automated scratch method

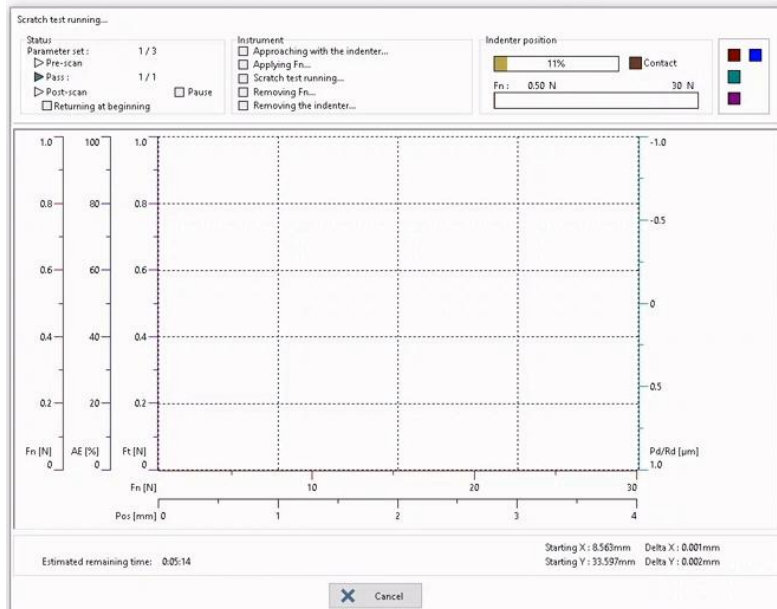


Principle of scratch test

A scratch is done by a **diamond indenter** of known geometry which is pressed with **increasing force** to the material surface while the sample moves at a constant speed over a specific **distance**.



Principle of scratch test

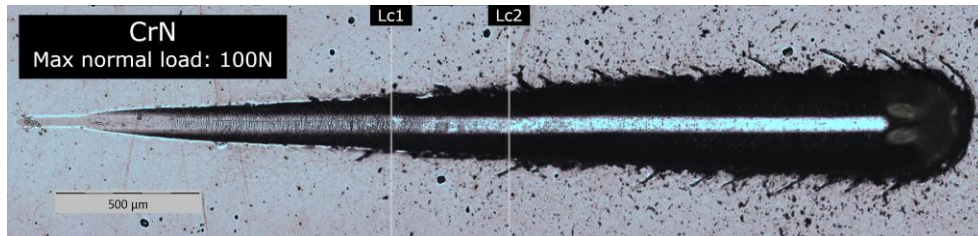
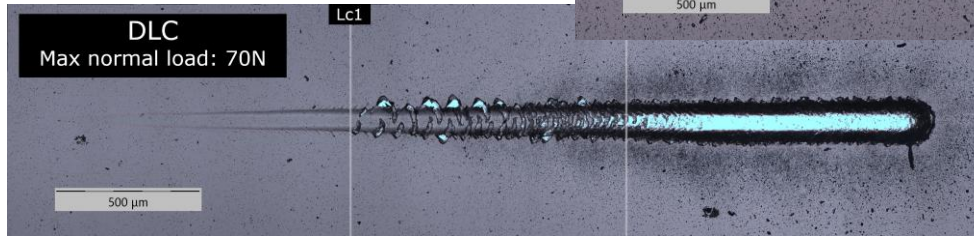


Speed: 2x

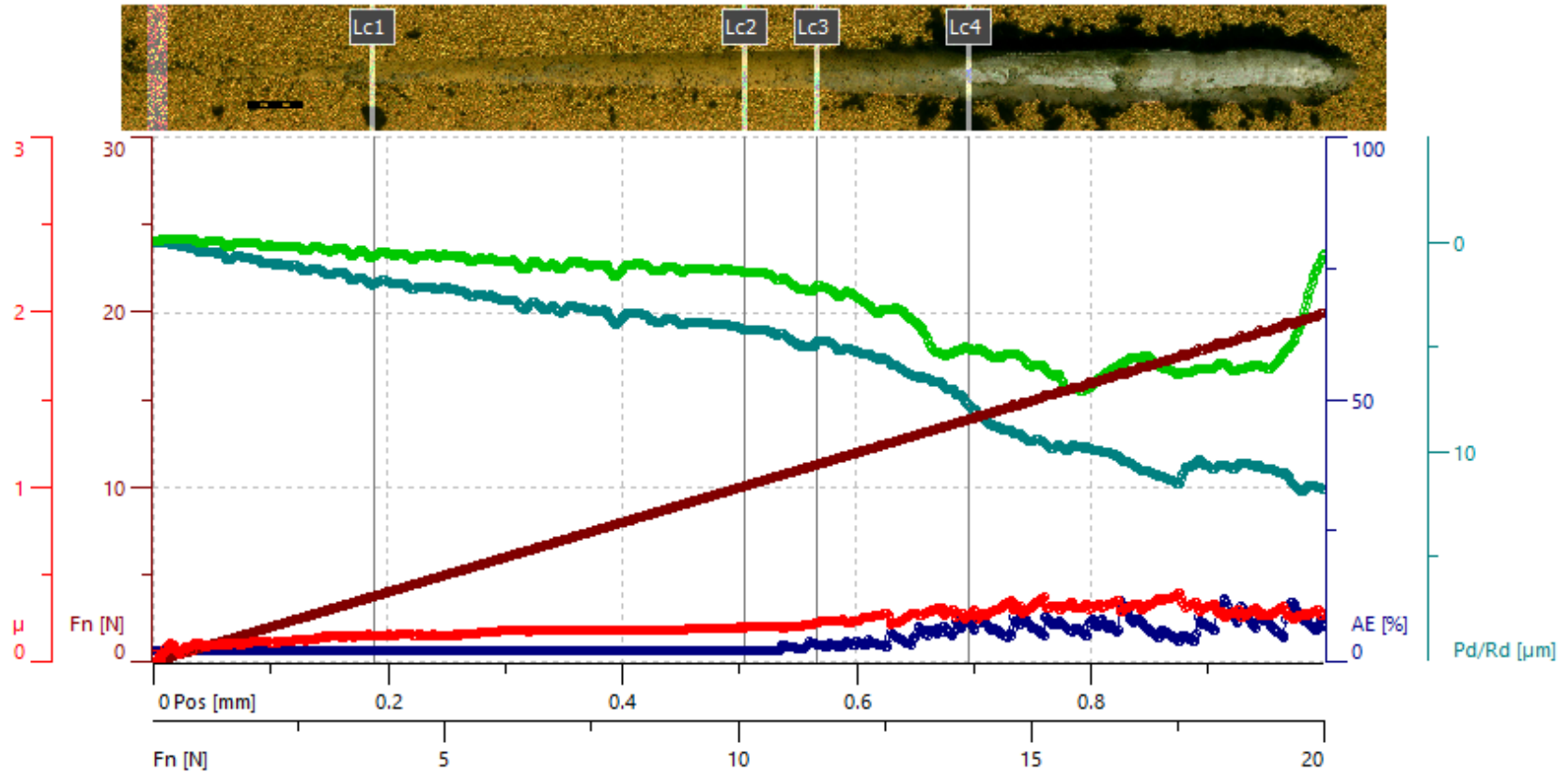


Most important part of scratch test – optical analysis!

Progressive load scratch test results

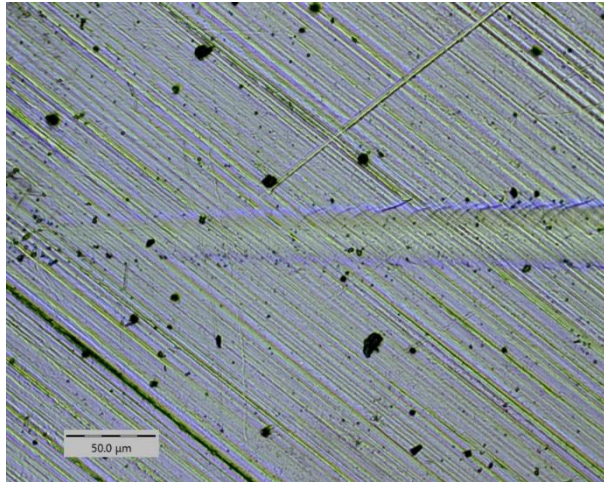


Scratch results: image and signals



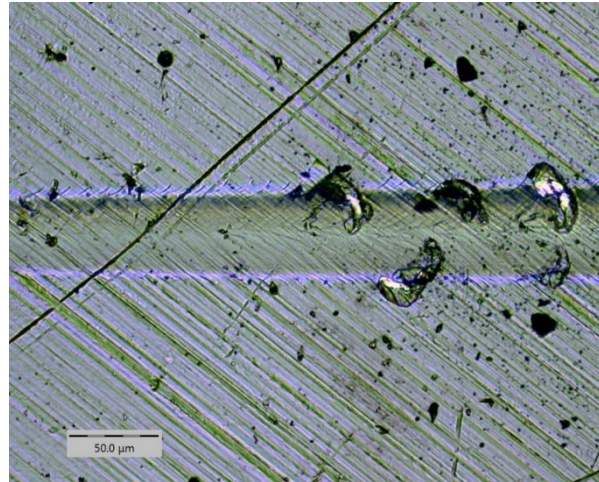
Adhesion? Critical loads!

After a scratch test, the user defines **critical loads** (Lc), which are loads at which different types of failures occur



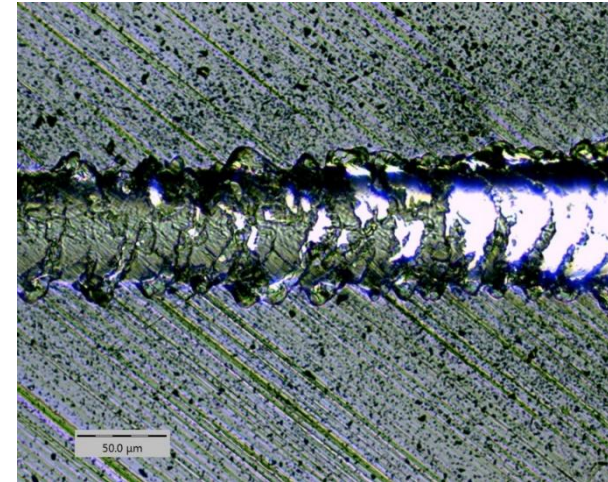
Usually considered as Lc1

- Cracks in the coating
- Cohesive failure



Usually considered as Lc2

- Chipping, partial delamination, side cracks
- Adhesive failure
- Usually the most relevant for determination of coating adhesion



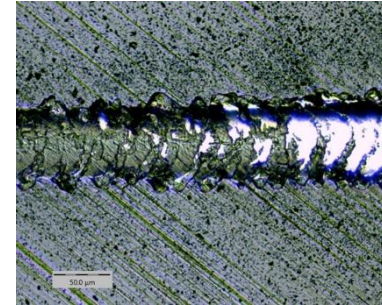
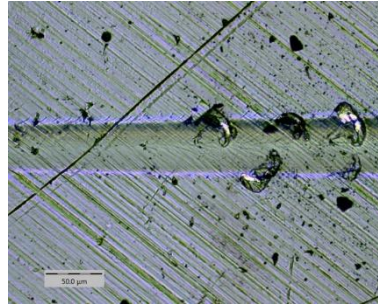
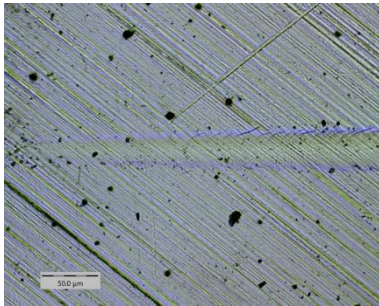
Usually considered as Lc3

- Total delamination, catastrophic failure
- Adhesive failure, sometimes also due to large deformation of the substrate

Interpretation of a scratch test

Critical load – load at which some damage start

Cracks, chipping, delamination



How are critical loads related to adhesion?

8 N critical load: is it a good adhesion?



Or is it a bad adhesion?



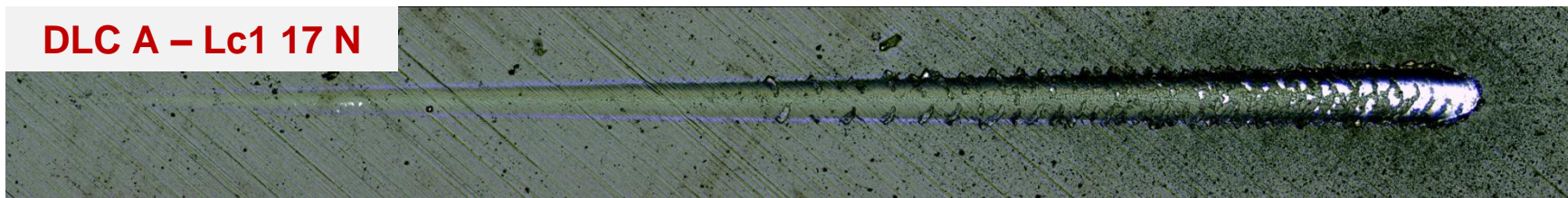
Interpretation of a scratch test

Two diamond like carbon (DLC) coatings

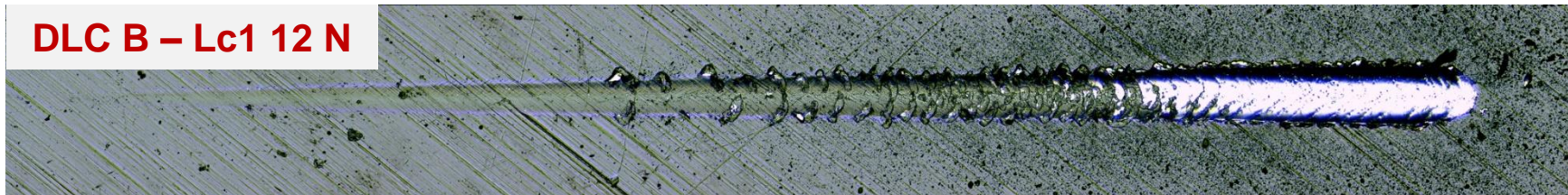
Same thickness (3 μm)

Same scratch parameters (R 100 μm indenter, 3 mm, max load 30 N)

DLC A – Lc1 17 N



DLC B – Lc1 12 N

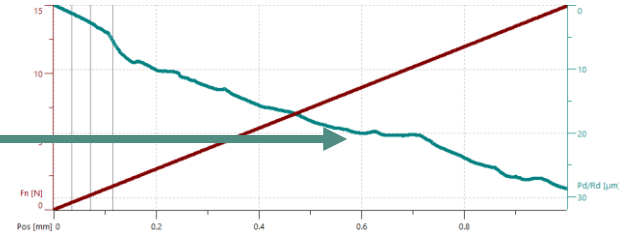


Lc1 DLC A > Lc1 DLC B
Higher Lc → Better adhesion!

True depth during scratch

We can measure depth during a scratch

With a depth sensor



BUT Do we measure **real** depth during a scratch?

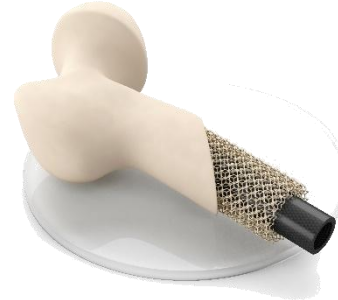
What about sample or surface inclination or surface profile (cylinder, sphere, curved)?



Depth measured during scratch is influenced by the inclination or surface profile!

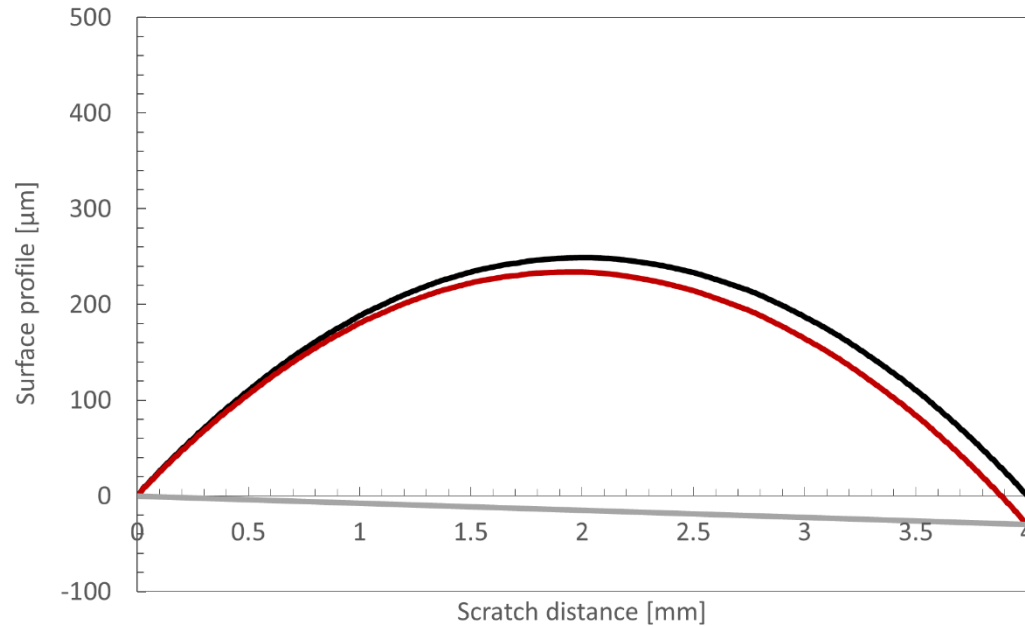


Surface profile must be measured and corrected before the measurement!



True depth during scratch

We can measure depth during a scratch



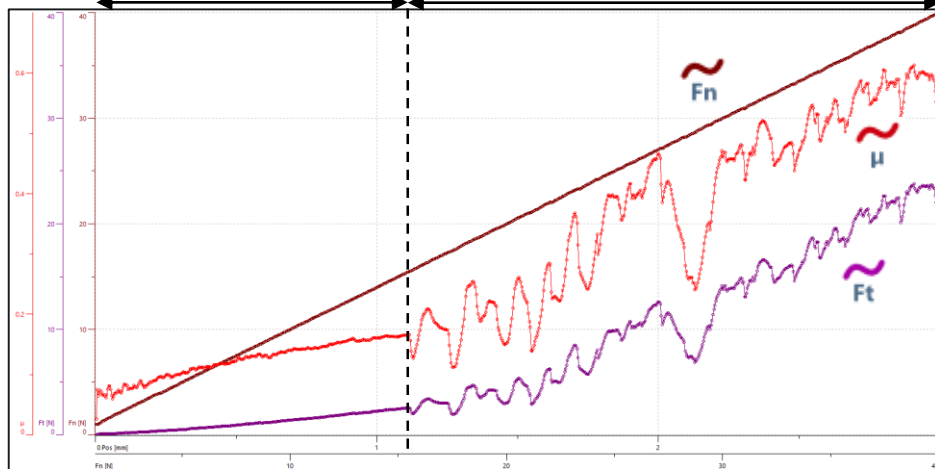
Friction force (coefficient of friction)

- **Coefficient of friction (CoF)** during the scratch can be using optional friction force sensor
- Fluctuations or change of the friction coefficient curve are often linked to coating delamination:
- Sliding on the coating → Delamination → Sliding on the substrate: change of CoF!
- Variations of CoF indicate delamination

Scratch results 1: brittle material

Scratch in the coating,
no damage

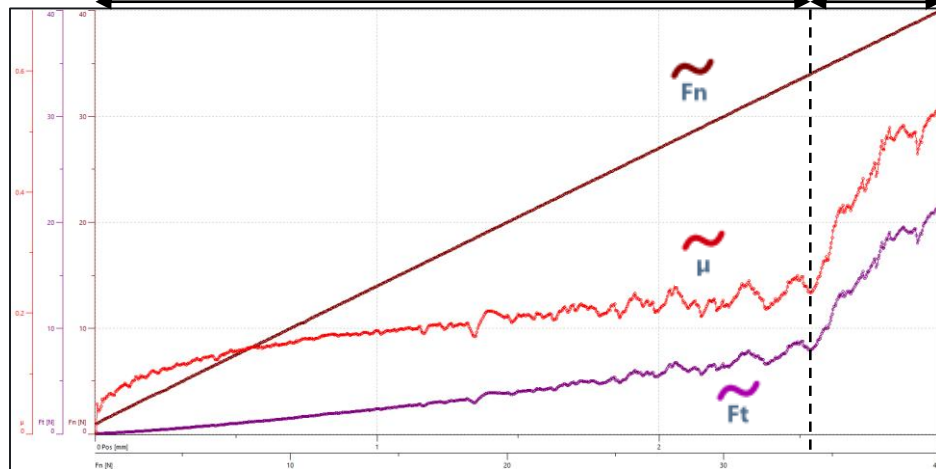
Chipping of the coating,
beginning of delamination



Scratch results 2: soft material

Scratch in the coating, no damage

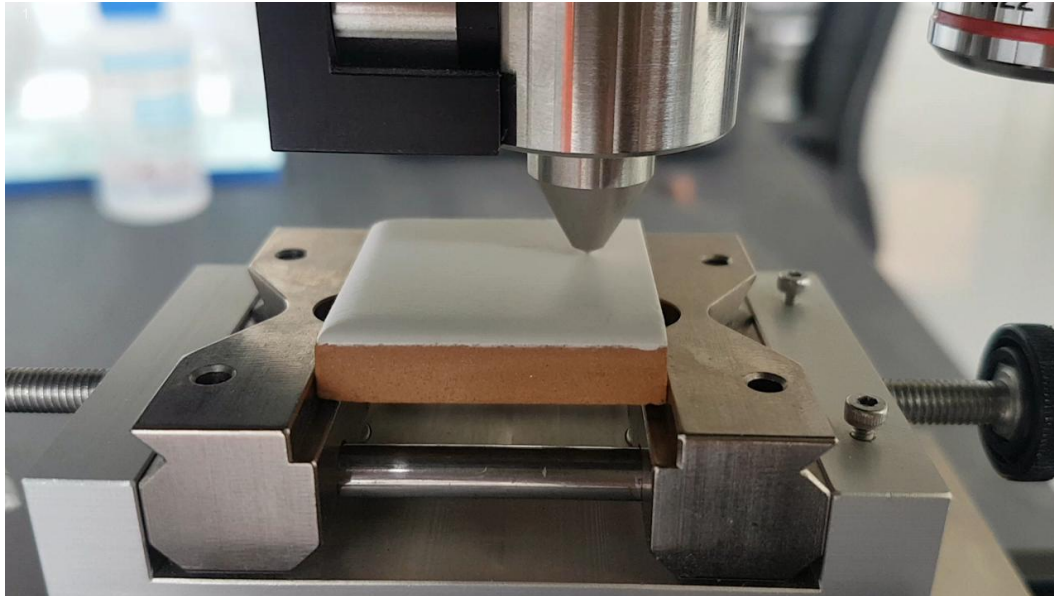
Full coating delamination,
scratch in the substrate



Acoustic emission

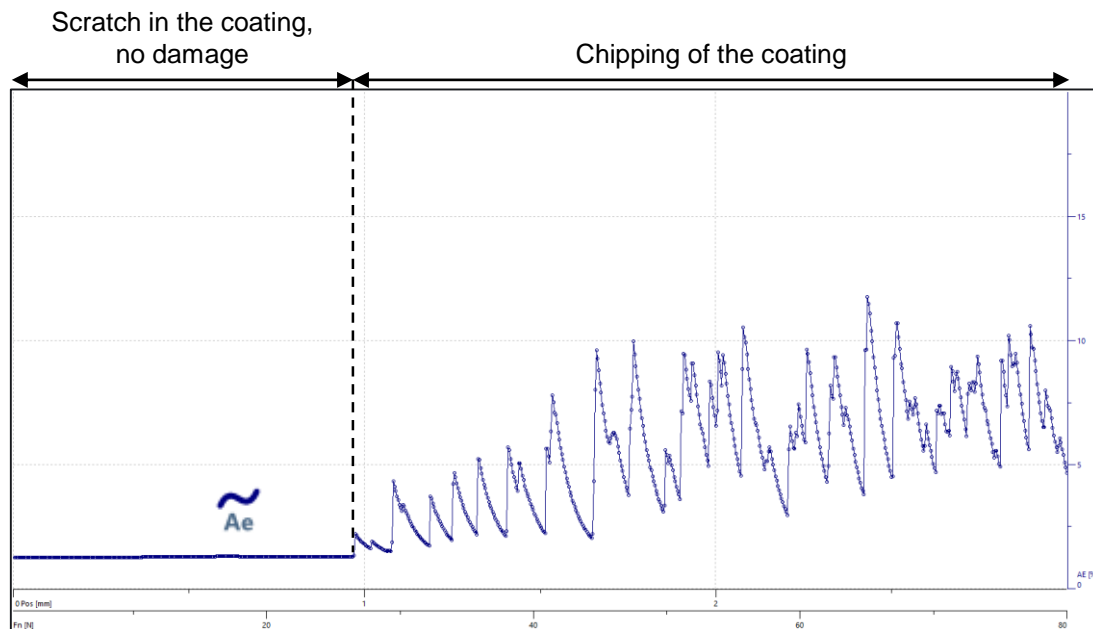
Damage – cracks, chipping or delamination is often producing acoustic waves

- Audible (less often on hard coatings)
- Non-audible: acoustic emission sensor

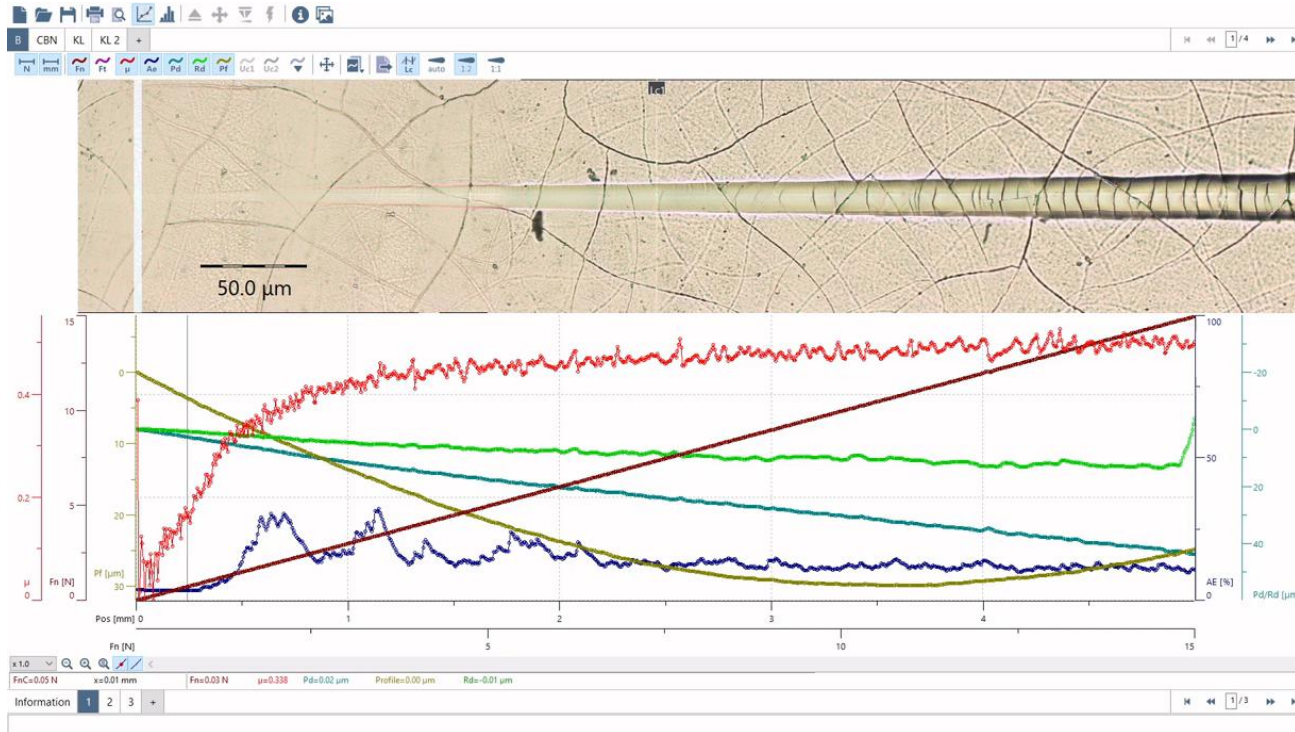


Acoustic emission

- **Acoustic emission** (AE) can also be recorded thanks to a dedicated sensor fixed next to the indenter.
- Detection of any high-frequency elastic waves generated in the coating-substrate system by brittle damage events (cracking, chipping, delamination...).



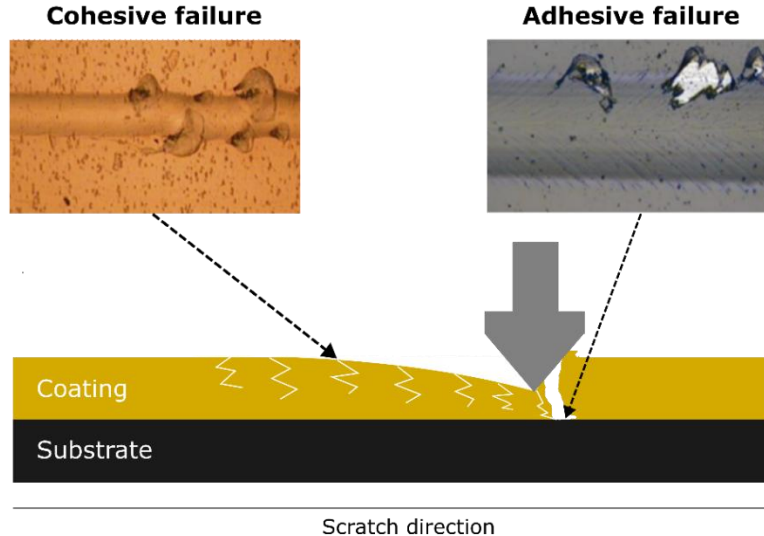
Panorama & Scratch signals



Optical analysis of a scratch test – typical types of scratch failure

Cohesive versus adhesive failure

Failure events during scratch can be divided in two main categories



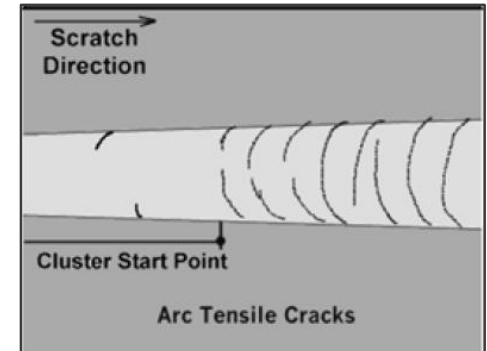
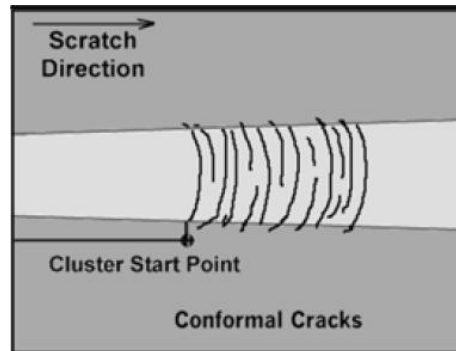
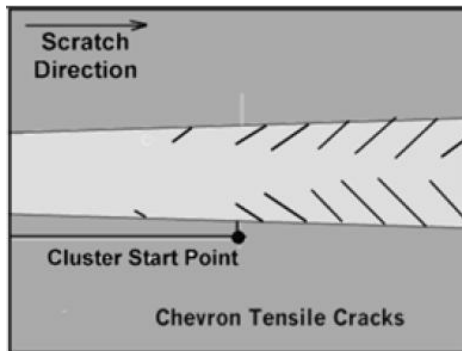
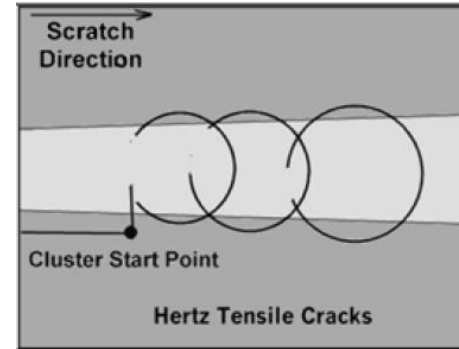
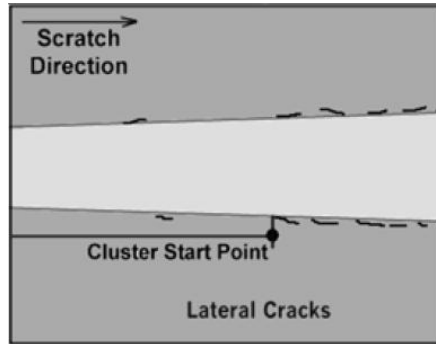
A **cohesive failure** indicates a failure in the coating (or in the substrate in case of an uncoated material). It reflects the cohesive strength of the coating or bulk material.

An **adhesive failure** is a failure at the coating/substrate interface. It reflects the adhesion of the coating to the substrate.

Adhesive failure does not occur on bulk materials (without coating).

Cohesive cracking I

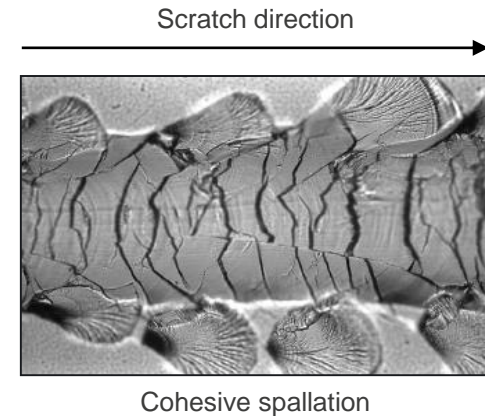
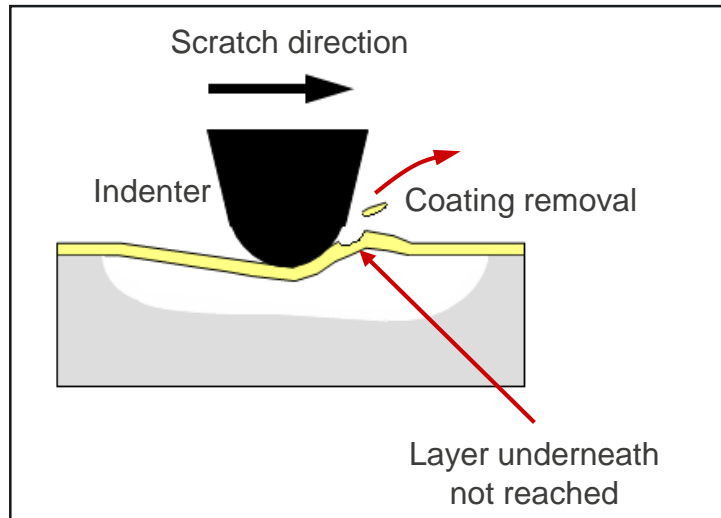
Cohesive cracks – in the coating, substrate is not visible



Cohesive cracking II

Cohesive failures: cohesive spallation

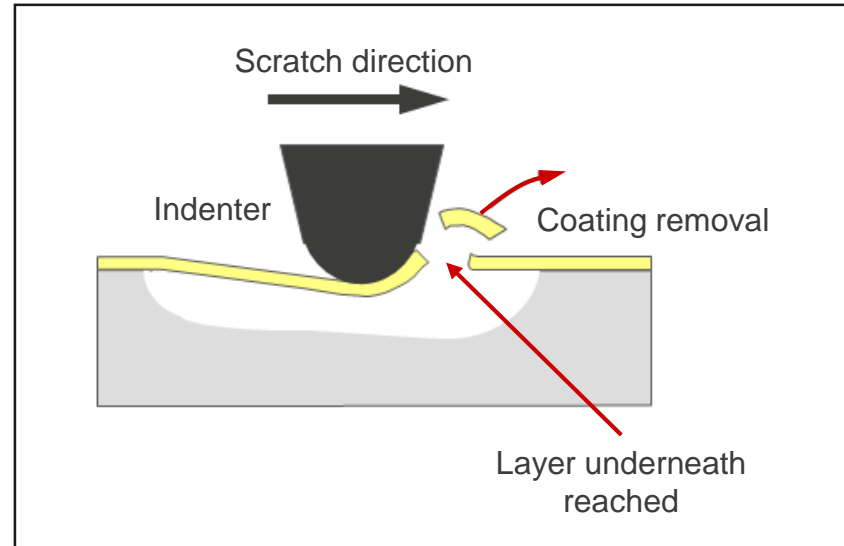
- It is also possible to observe spallation, defined as regions of detached coating along one or both sides of the groove (ASTM C1624-05).
- When spallation does not reach the layer underneath, it is considered as cohesive spallation.



Adhesive failure

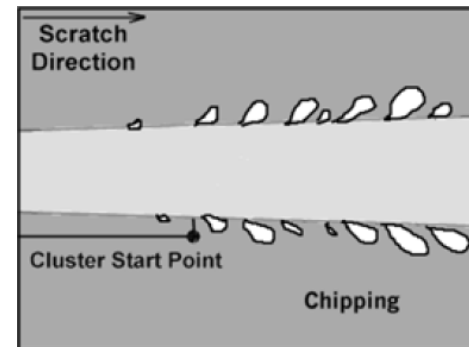
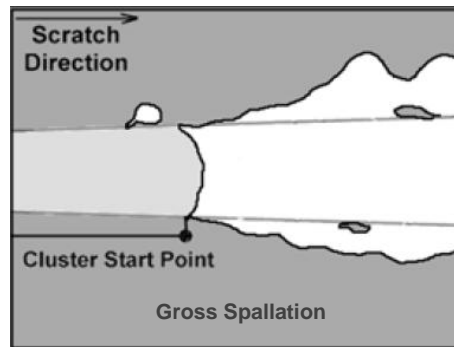
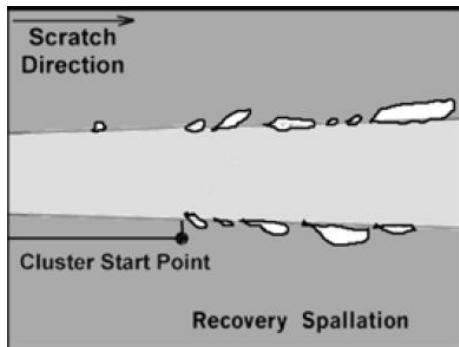
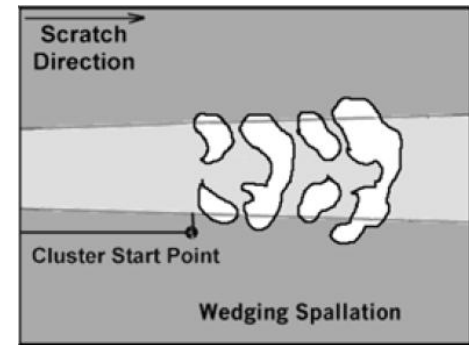
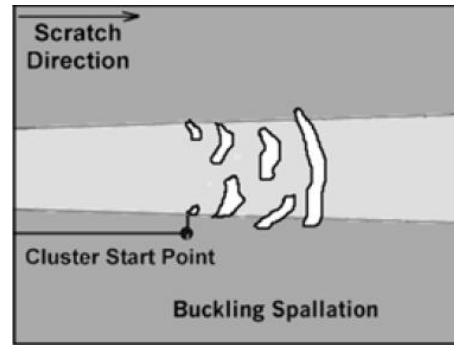
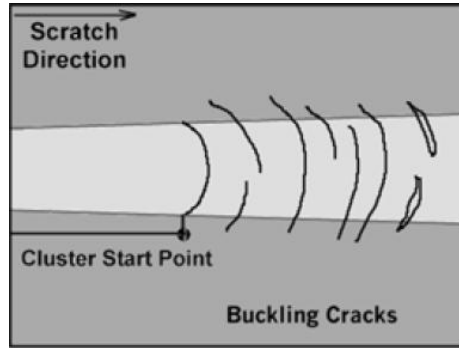
Adhesive failures

- > An adhesive failure occurs when a part of the coating is removed and the layer underneath can be observed.



Adhesive failure

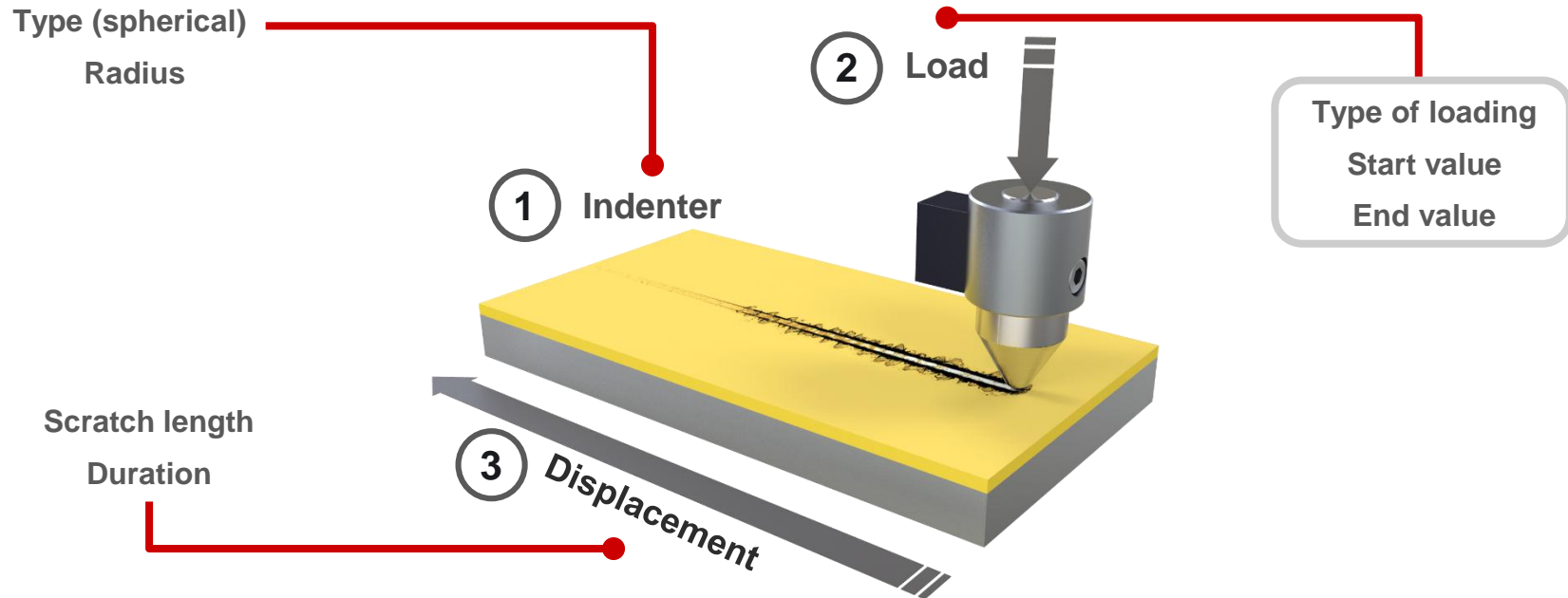
Adhesive failures: buckling, spallation and chipping



Scratch parameters

Scratch parameters

What are the testing parameters to take into account?



Main indenter shapes

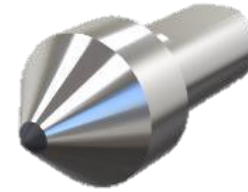
Two main indenter types with various radii.

Rockwell (spherical) diamond indenter



- Radius: from 10 to 800 μm
- For scratch testing at micro range (RST³ and MST³)

Sphero-conical diamond indenter



- Radius: from 1 to 20 μm
- For scratch testing at nano range (NST³)

– Use indenter with radius approximately 10 to 20 times the thickness of the coating.

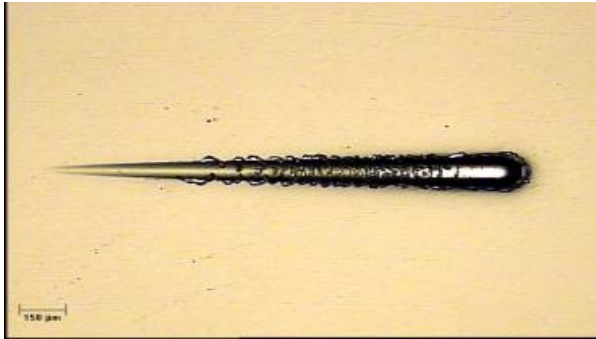
– For very hard and thick ceramic coatings: choose an indenter with larger radii.

– For coatings with thickness in the range of tens or hundreds of nanometers: select an indenter with small radii of 1 to 5 μm .



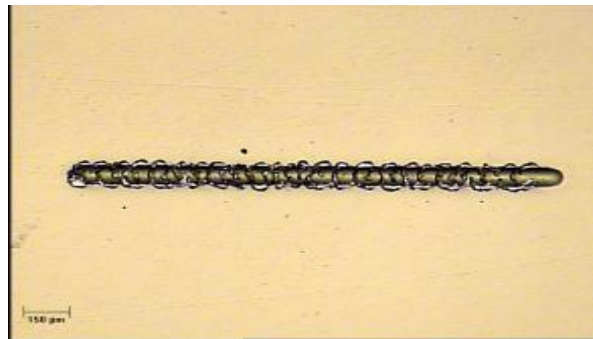
Type of loading during a scratch test

Progressive loading



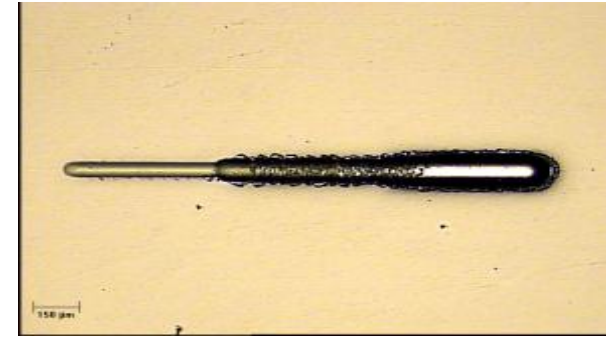
- Most common
- Adhesion of coatings
- Hard coatings
- Metallic coatings
- Thin coatings

Constant load



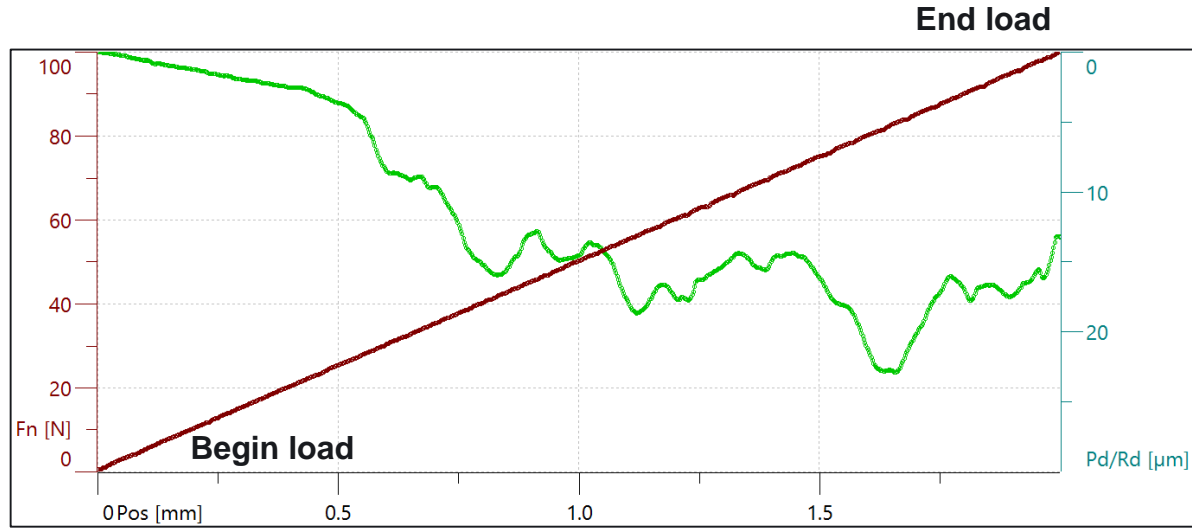
- For scratch resistance (no delamination of coating, no adhesive failure)
- Bulk materials, paints, thick coatings (>50 µm)
- Quality control of adhesion: cracks/no cracks at $F_{\text{threshold}}$

Incremental loading



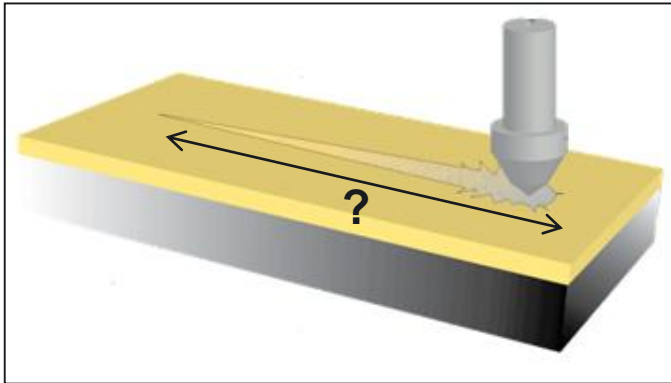
- Scratch resistance (no delamination of coating, no adhesive failure) at various loads during single scratch
- Bulk materials, paints, thick coatings (>50 µm)
- More precise quality control: delamination within load range

Influencing parameters – Load



- Begin load: Usually minimum load of the instrument.
- End load: set it so that maximal Residual depth (Rd) > coating thickness. No delamination → increase End load.
- Hard coatings - indenter damage prevention: start with low End load. Increase if no delamination observed.

Influencing parameters – Length



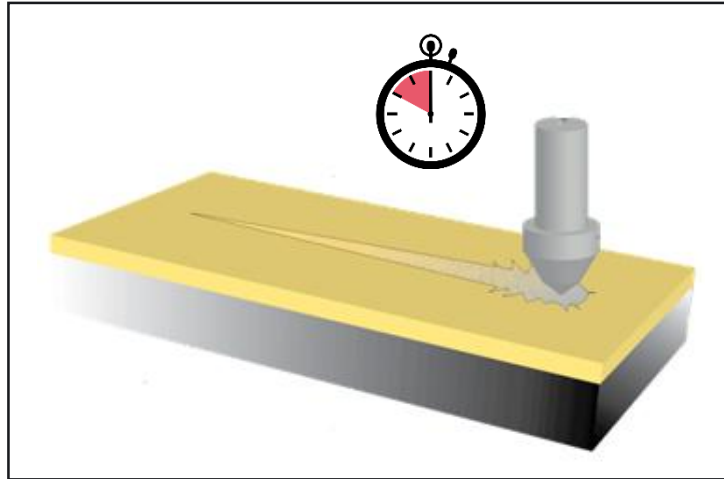
Coating thickness	Usual scratch length
Below $\sim 1 \mu\text{m}$	500 μm
From $\sim 1 \mu\text{m}$ to $\sim 20 \mu\text{m}$	1 mm to 3 mm
Above $\sim 20 \mu\text{m}$	$\sim 20 \text{ mm}$



- Scratch length: at least 5 to 10 times the indenter radius.
- Too long scratch – Panorama takes long!

Influencing parameters – Duration

Displacement

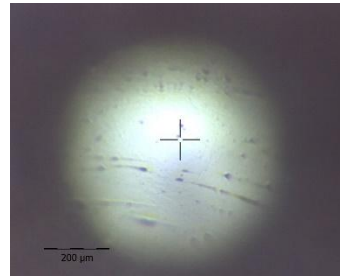
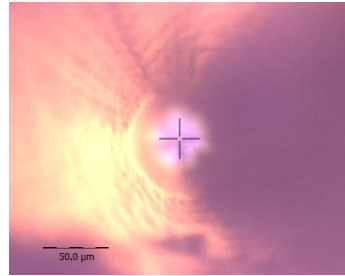


- We recommend scratch duration of 30 seconds.
- Hard materials: scratch duration can be decreased to 10 seconds.
- Polymers: scratch speed can affect the results due to their viscoelastic behavior.

Influencing parameters – Indenter shape

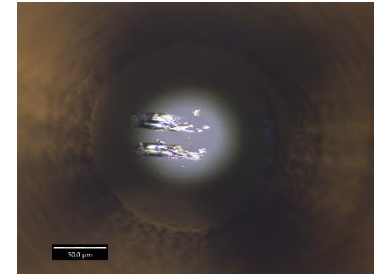
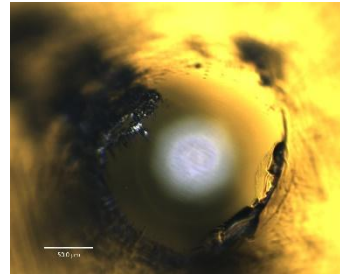
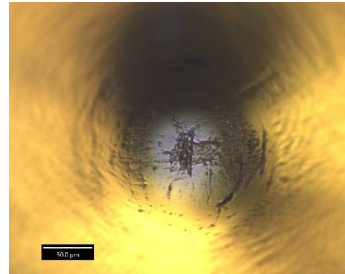
Indenters in good shape

Rockwell 200 μm radius



Damaged indenters

Rockwell 200 μm radius



- Verify Rockwell indenter in optical microscope before starting the tests.
- Clean the indenter with a Q-tip and laboratory alcohol. Sandpaper with superfine grain size can also be used.
- If non-removable scratches are present, the indenter should be changed.

Tooling industry – Hard coatings

Hard coatings for cutting tools applications:

- Machining process: turning, drilling, milling, threading, reaming, broaching, gear cutting...
- Substrate material: High Speed Steel (HSS) and cemented carbide
- Machined material: polymers, wood, metals, alloys, composites, graphite, ceramics...
- Coating thickness: 1 to 4 μm



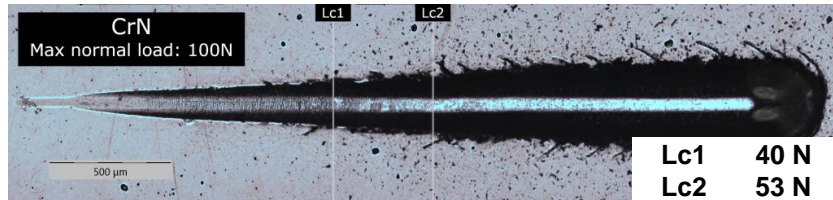
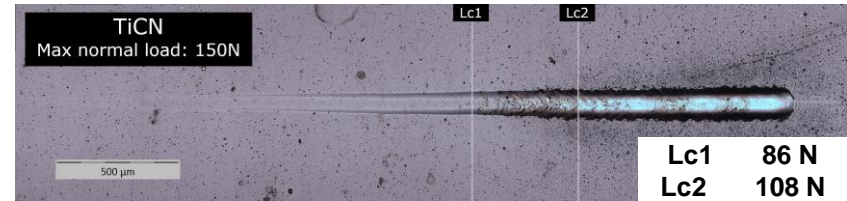
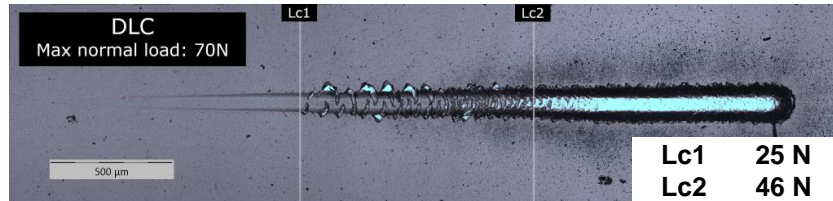
The classics: scratch on hard coatings

Thickness 3 to 5 μm

Lowest Lc1 to highest Lc1

High speed steel (HSS) substrate

→ Worst to best adhesion

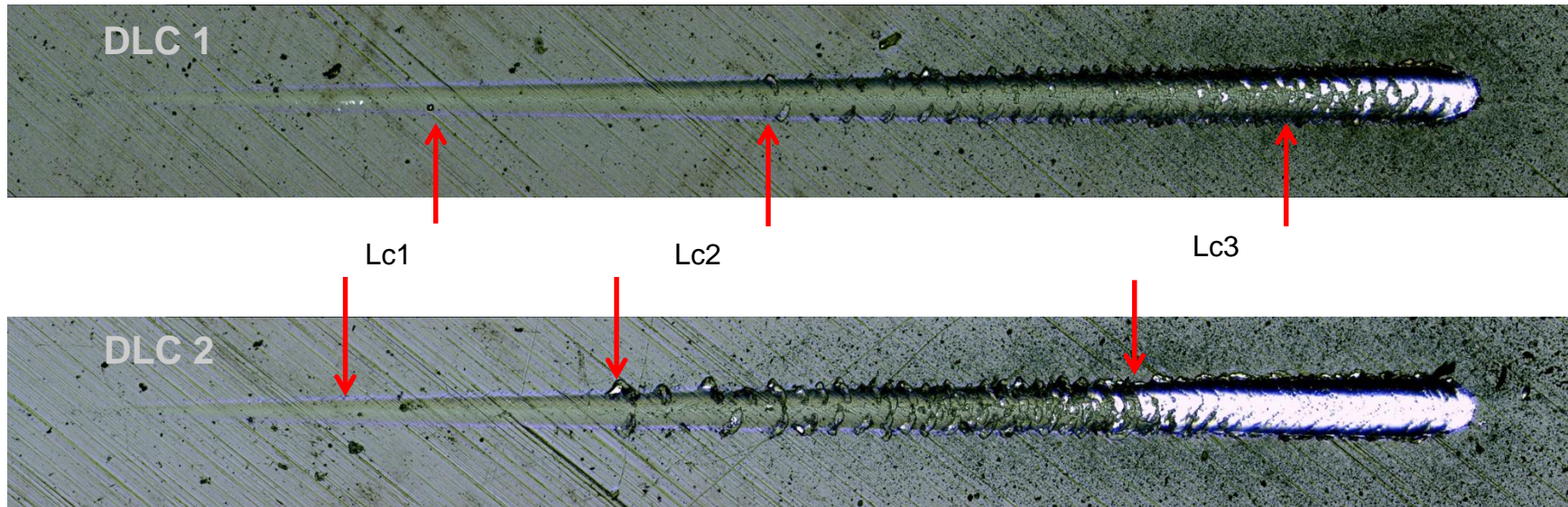


Adhesion of two DLC coatings

DLC coatings from two different providers

→ Which coating has better adhesion?

- Easy comparison of several scratches using Panorama images
- Higher Lc – better adhesion!



Paints and varnishes

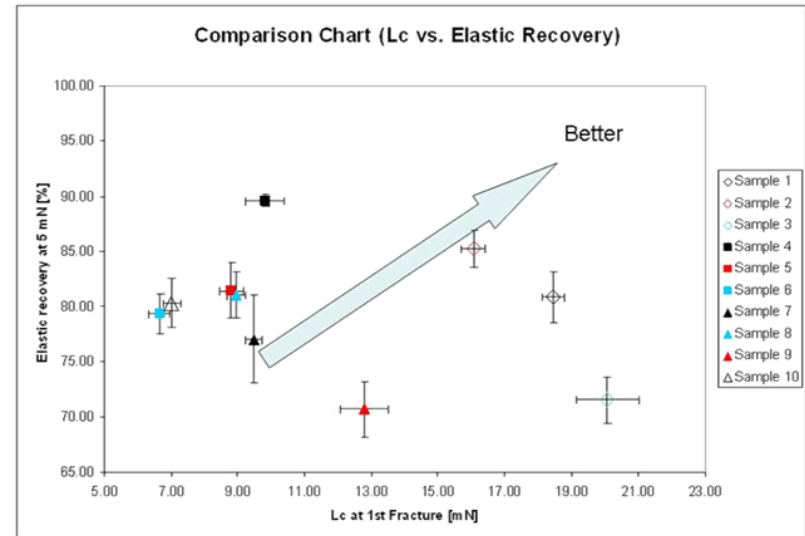
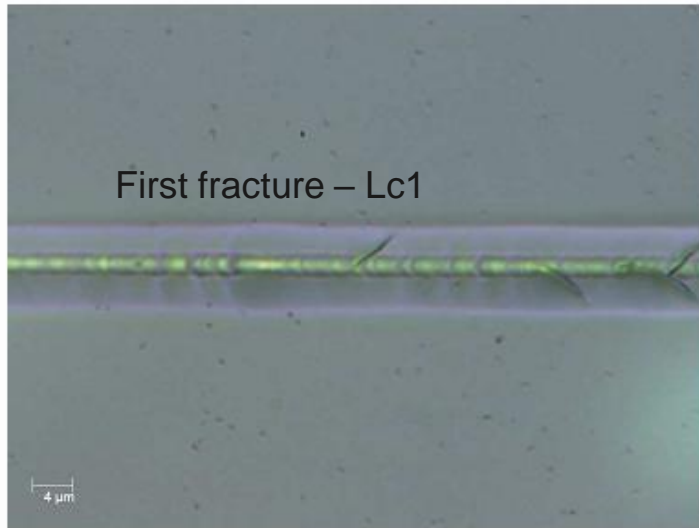
Automotive industry: example of problem

- › Car paint should resist **external forces** (car washing, blowing sand, branches, keys, fingernails...) and remain **shiny** as long as possible.



APPLICATIONS OF SCRATCH TESTING

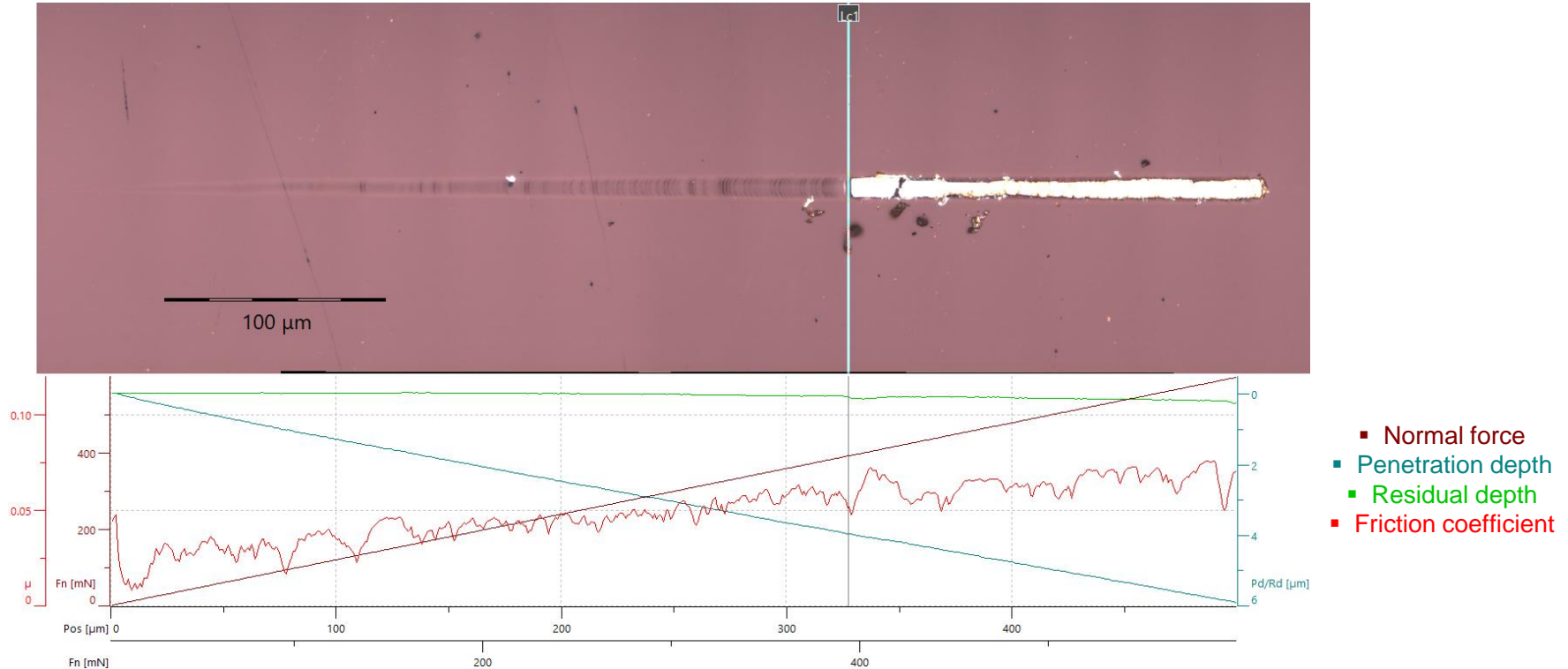
- › Characterization of mar resistance (crack formation and elastic recovery) with a nanoscratch
- › Check for the first crack or fracture (Lc1) and compare it with elastic recovery at some specific load



Mar resistance chart

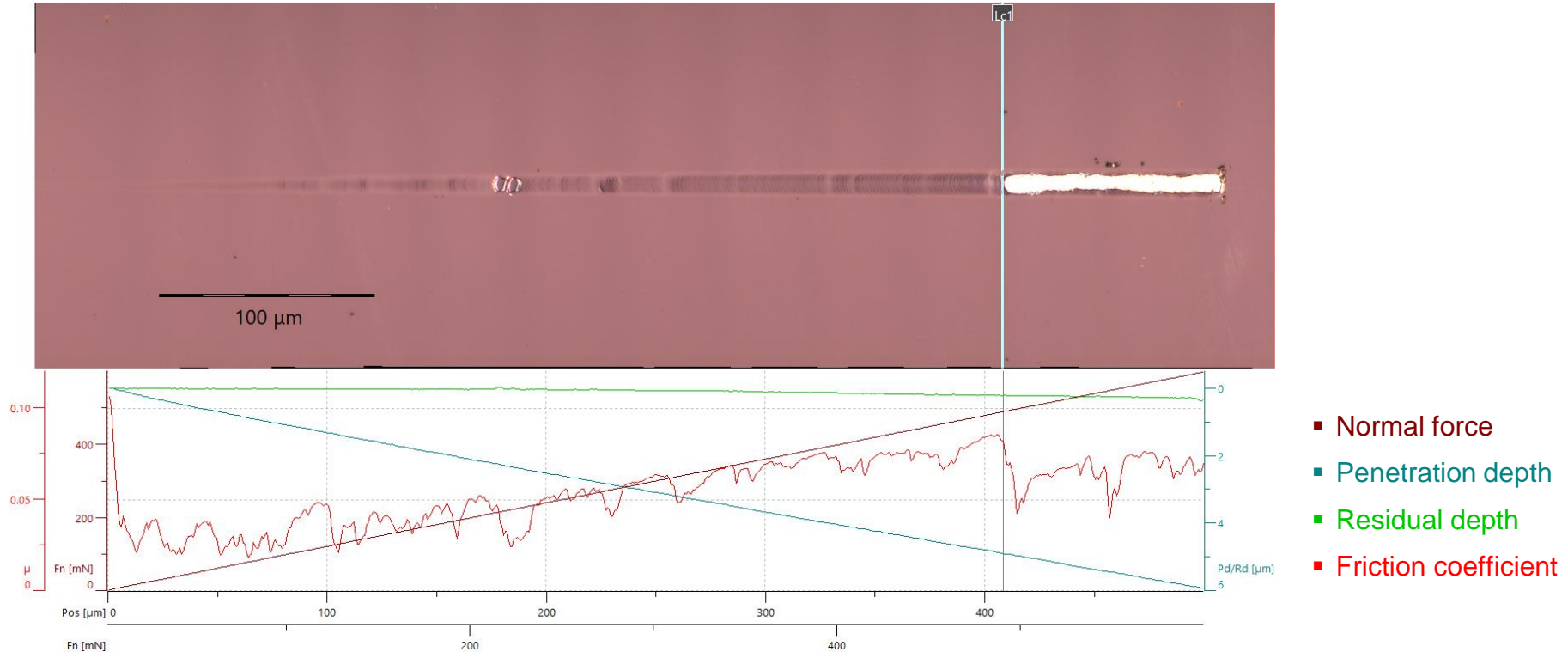
Anti-refeective layers - Nanoscratch

› Vertical line indicates the critical load corresponding to delamination of the layer



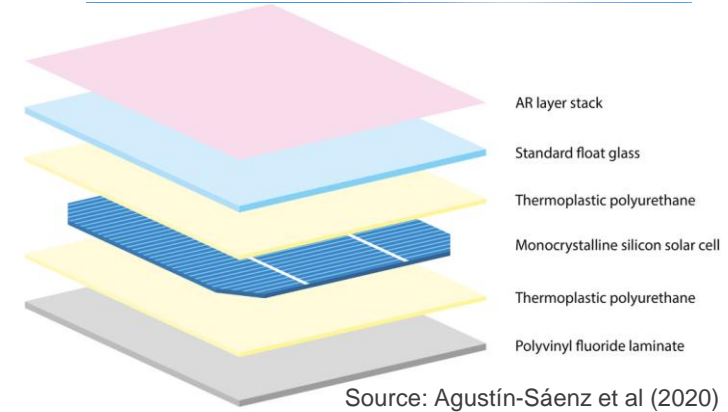
Higher LC – better adhesion!

- Typical panorama picture of the scratch on sample 21337
- Vertical line indicates the critical load corresponding to delamination of the layer



Adhesion of AR layers in photovoltaics

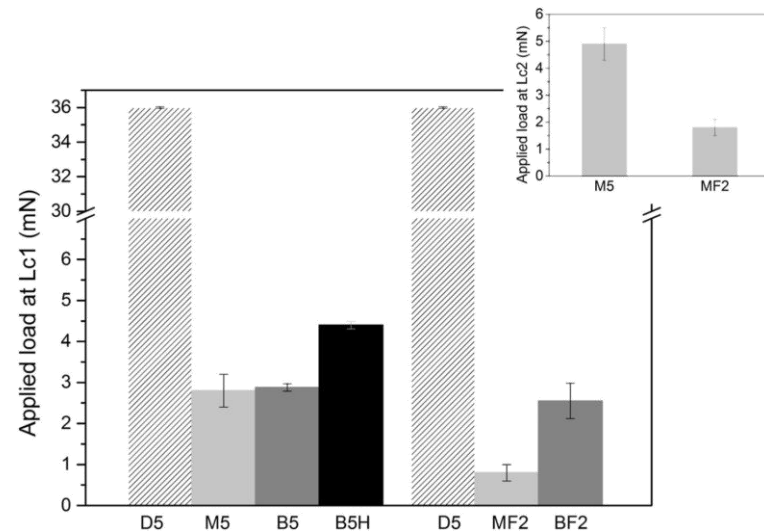
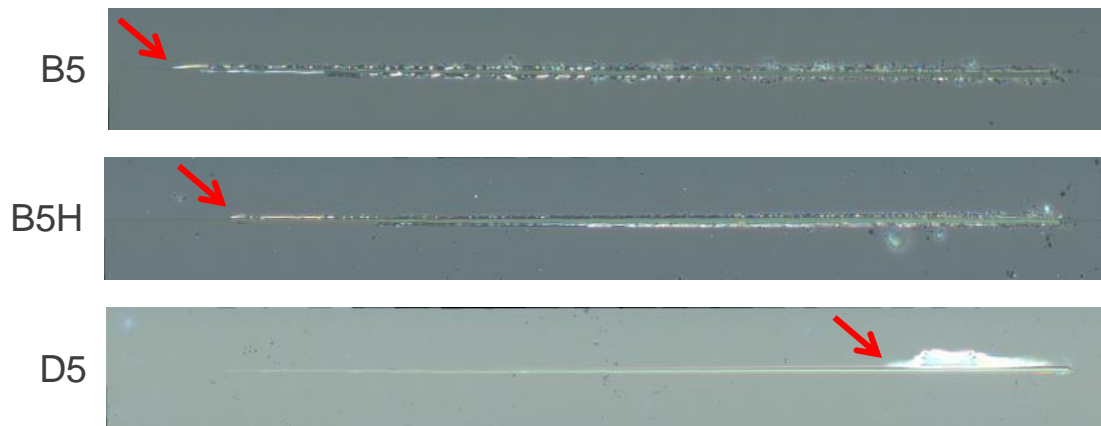
- Antireflective (AR) layers on photovoltaic (PV) panels increase the amount of light transmitted to the PV cells
- Thickness of the AR layers is ~120 nm
- To resist external damage, the AR layers must have good adhesion and scratch resistance



- AR layers were deposited by Tecnalía (ES) on glass slides by sol-gel technique using different deposition parameters (porous silica, methyl-silylation post-treatment, thermal treatment, etc.)
- Determination of scratch resistance: scratch tests with NST³
- Progressive load scratch tests on several different anti-reflective coatings (dense, heat treated, porous, etc.)

Adhesion of AR layers in photovoltaics

- NST³ with small indenter (radius 1 μm)
- Scratch load 0.1 mN to 50 mN
- Examples of Panorama on three coatings



Source: Agustín-Sáenz et al (2020)

- Optical analysis allows for determination of critical loads
- Critical loads different for different deposition parameters
- Large differences in adhesion (Lc1): D5 (dense coating) the highest, MF2 (porous coating) the lowest

Li-ion battery electrodes

For the best performance the electrode coating must:

- Adhere well to the aluminum or copper foil
- Be homogeneous

Bad adhesion of electrode coating can lead to:

- Loss of capacity
- Thermal runaway (short circuit)



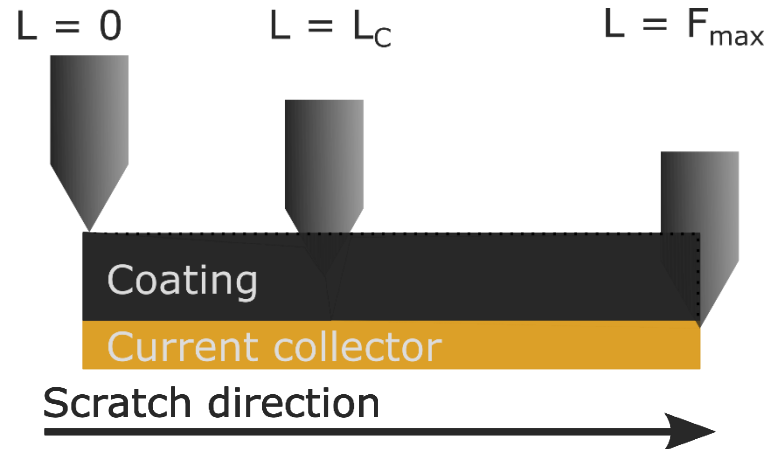
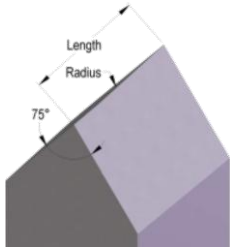
Laptop battery pressurized by thermal runaway

New approach to scratch – wedge blade

- **Spherical indenter** – typical shape for adhesion of ceramic coatings
- **Drawback:** not ‘sharp’ enough, very progressive coating removal – difficult to define L_c precisely
- **Optimization:** wedge blade indenter

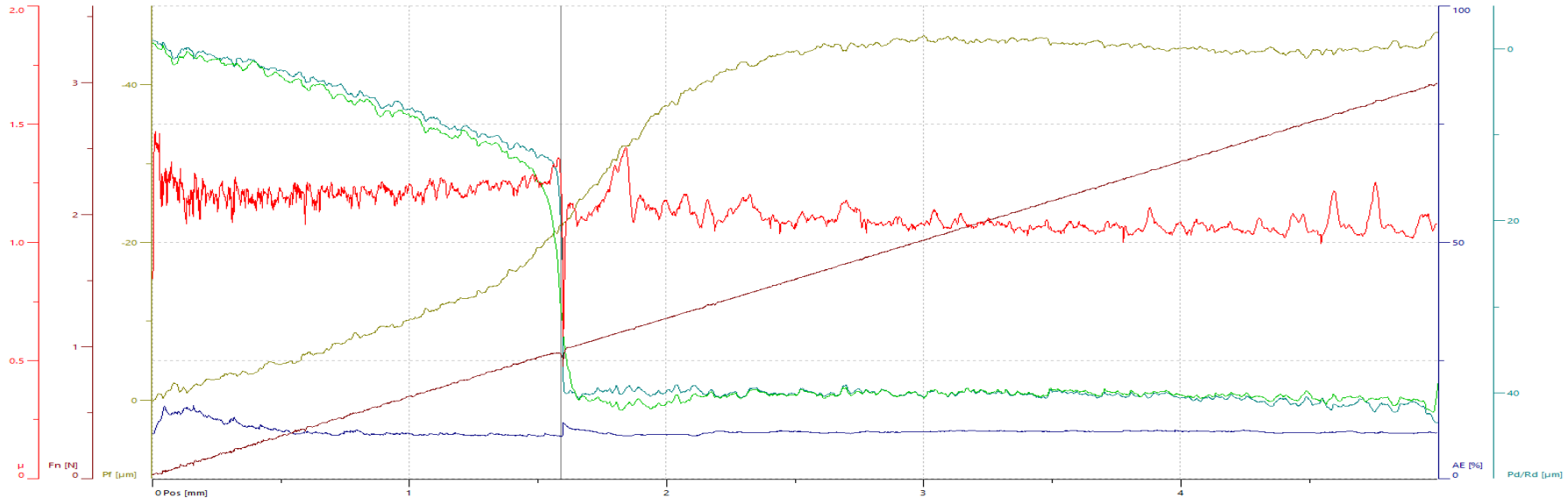
Sharp edge → Fast and more obvious delamination

Wedge blade (knife) indenter

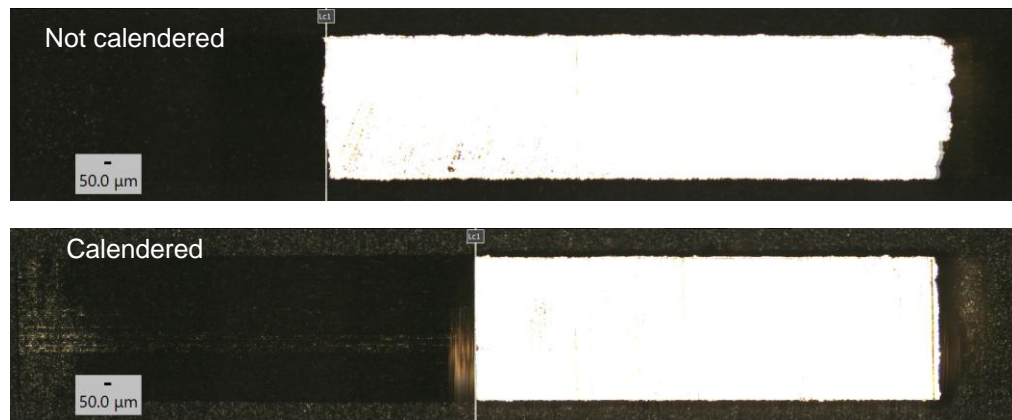


Results of wedge blade tests

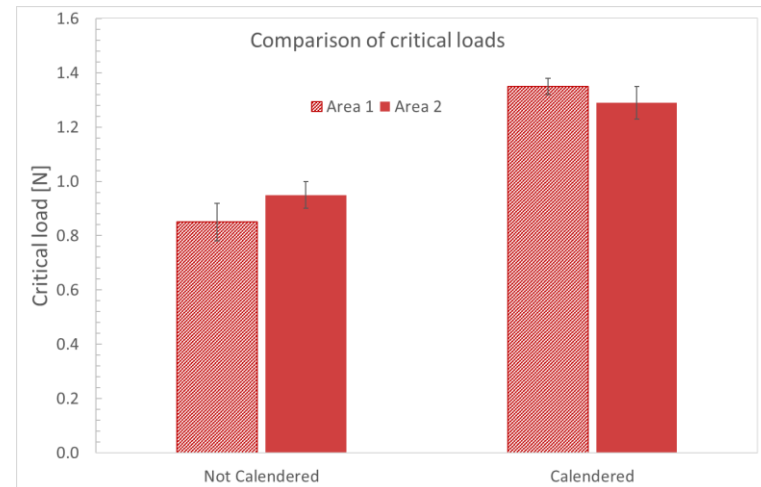
Scratch test with wedge blade



Calendered vs Not calendered



- Critical load (L_c) – easy to identify
- Not-calendered: $L_c \sim 0.9$ N, Calendered: $L_c \sim 1.4$ N
- Very similar L_c in different areas: homogeneous coating
- Low standard deviation

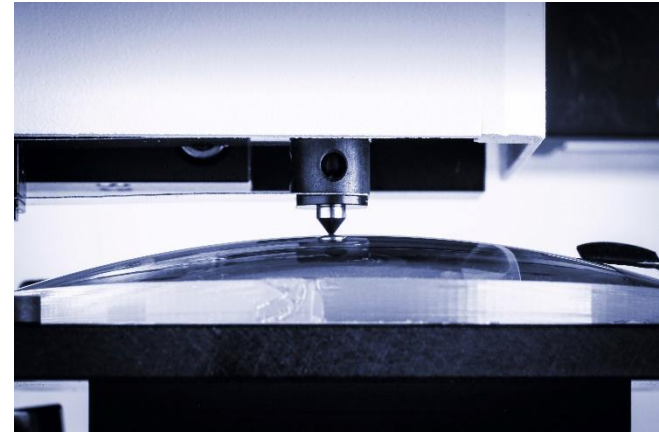


Adhesion of passivation layers on Si wafer

Different insulating layers can be used as passivation coatings on Si wafers to offer protection against the environment (humidity, electrical insulation) or mechanical damage.

- > Scratch test with NST³ nano scratch tester
- > Spheroconical indenter, radius 2 μm or 5 μm

Goal: Compare adhesion of different passivation coatings



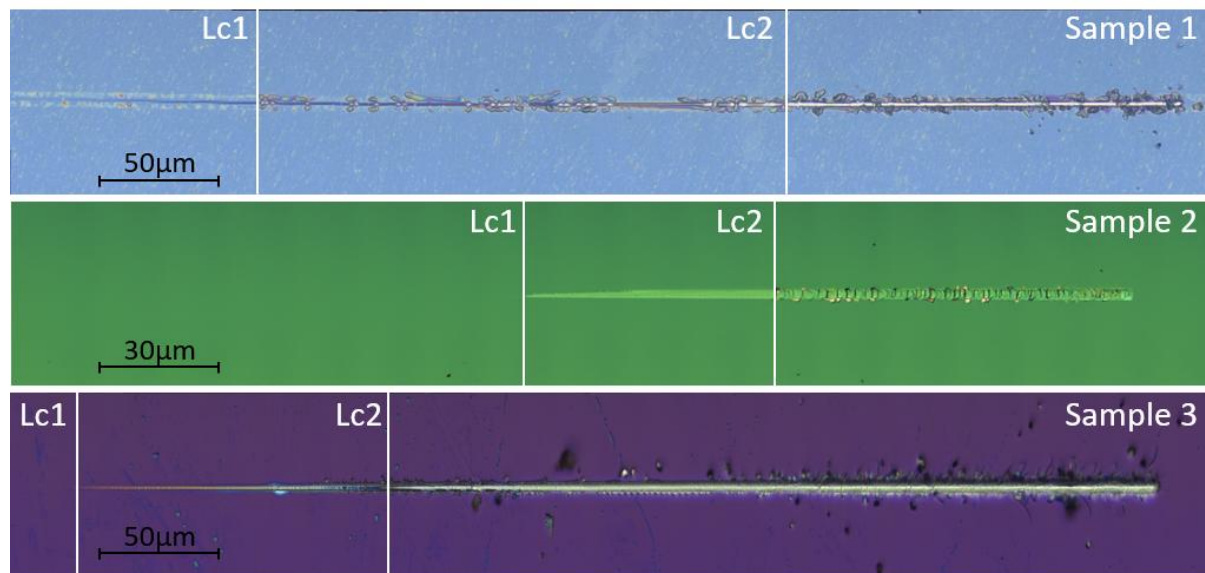
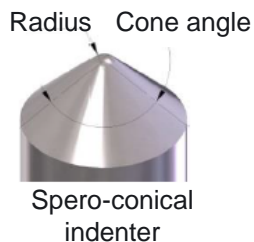
Nanoscratch Tester (NST³)

Coatings and test parameters:

Coating	Coating thickness [nm]	Max load [mN]	Scratch length [μm]
SiO_2	400	20	500
Si_3N_4	300	100	300
Al_2O_3	500	75	500

Adhesion of passivation layers on Si wafer

- › Lc1: first adhesion failure (partial delamination) -> change of color and cracks in the coating
- › Lc2: full delamination of the coating -> distinct color change due to appearance of the substrate

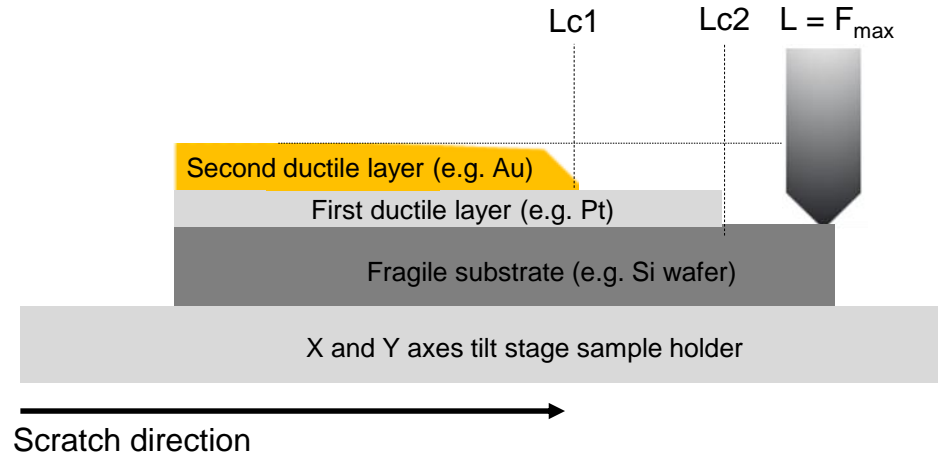
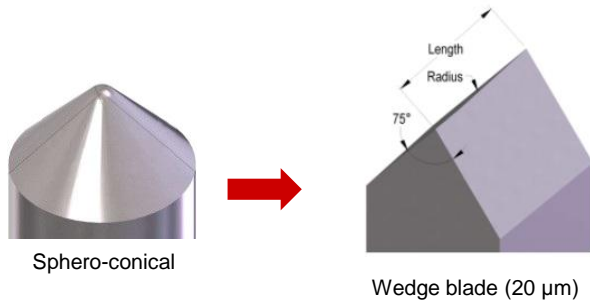


Sample 2: highest Lc → best adhesion!

Wedge blade indenter for semiconductors

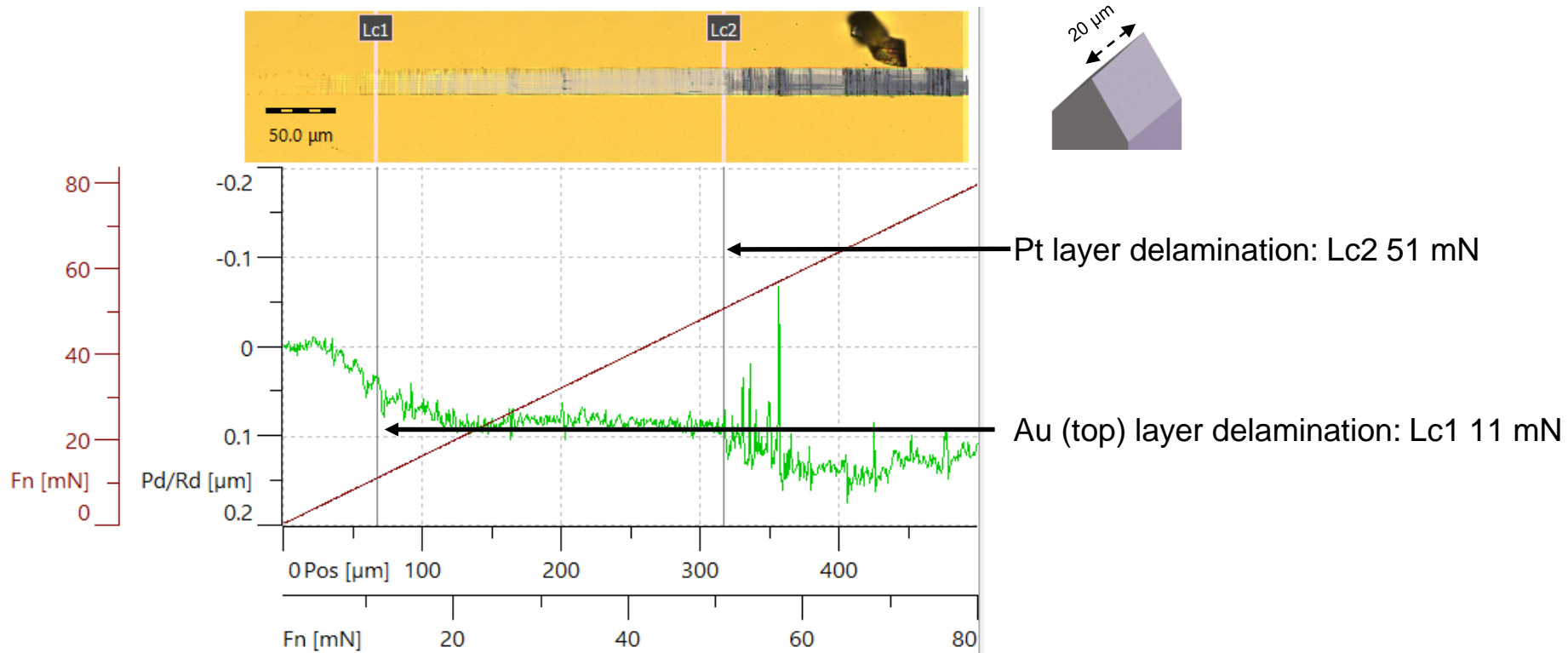
Scratch test using wedge blade indenter and two axes tilt stage

- Originally for thin soft films instead of sphero-conical indenter
- But in many cases it yields better results also for thin hard films



XY Tilt stage for precise alignment of the surface of the sample with the edge of wedge blade

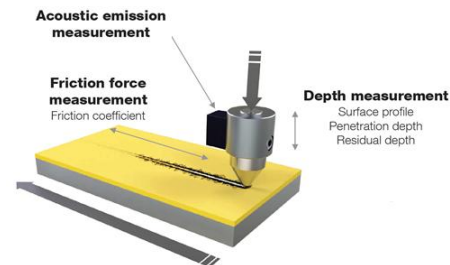
Adhesion of Au/Pt bilayer on GaAs wafer



Summary

Industry and research have improved coating deposition techniques

- > Current adhesion and scratch resistance test methods are not suitable
- > We need a better quality control test and quantitative results



This contributed to the development of controlled **scratch testing**

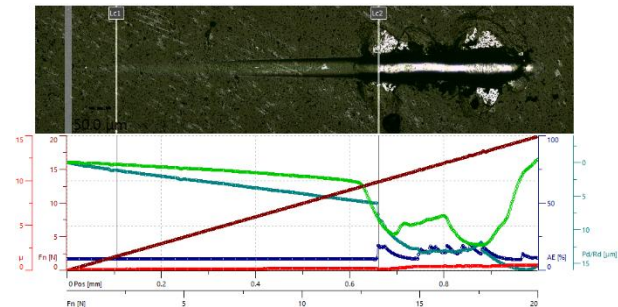
- > Typical scratch testers cover each a typical load and application range (macro, micro, nano)

Most common applications for scratch testing

- > Hard coatings, automotive, electronics, biomedical...

Many new applications

- > Glass, pharmaceutical, lithium-ion batteries...



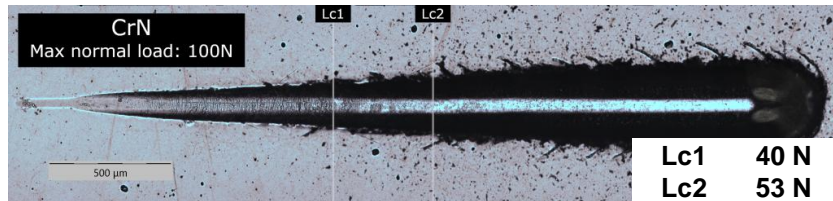
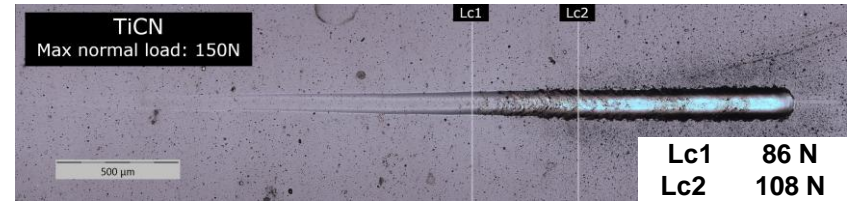
Get in touch

www.anton-paar.com

Comparison of adhesion

Ceramic coatings on high speed steel substrate

Thickness ~3 μm



- Comparison of critical load (adhesion) possible only for same type of coatings, same substrate and similar thickness
- Other standards (Rockwell) have similar limitations!