

Mechanical Surface Characterization



Instrumented Indentation Testers Scratch Testers Nano Tribometers Coating Thickness Testers

## A new era of precision

The third generation of Anton Paar's mechanical surface testers measure a wide range of mechanical properties of materials ranging from the hardest diamond-like carbon (DLC) coating to the softest hydrogel. The high-precision instruments measure what others estimate.

Anton Paar covers four of the most important test methods for mechanical surface characterization. Instrumented indentation testers provide mechanical properties of thin films, coatings, or substrates such as hardness and elastic modulus, creep, fatigue, stress-strain, elastic and plastic energies. Scratch testers are used to characterize film-substrate systems and to quantify parameters such as adhesive strength and friction force for determining coating adhesion, scratch resistance, and mar resistance for research and quality control. With tribometers you can study friction, wear, lubrication, and abrasion. Additionally, abrasion testers like Calotest provide quick, simple, and inexpensive determination of coating thicknesses.

## We measure what others estimate

Anton Paar is the only company to provide high-resolution nanoindentation and nano scratch testers with a real force sensor. This means that the force is really measured continuously with a direct sensor and not estimated from a derivative coming from an actuator.

## Instrument portfolio

The state-of-the-art design of Anton Paar's mechanical surface testers enables accurate and efficient testing solutions in a compact and modular format. In fact, different modules (scratch, indentation, microtribology) can be easily combined on one testing platform. Depending on your requirements and application you can combine different testing modules on one platform or you can use a single test method with a stand-alone device – just choose whatever you need.

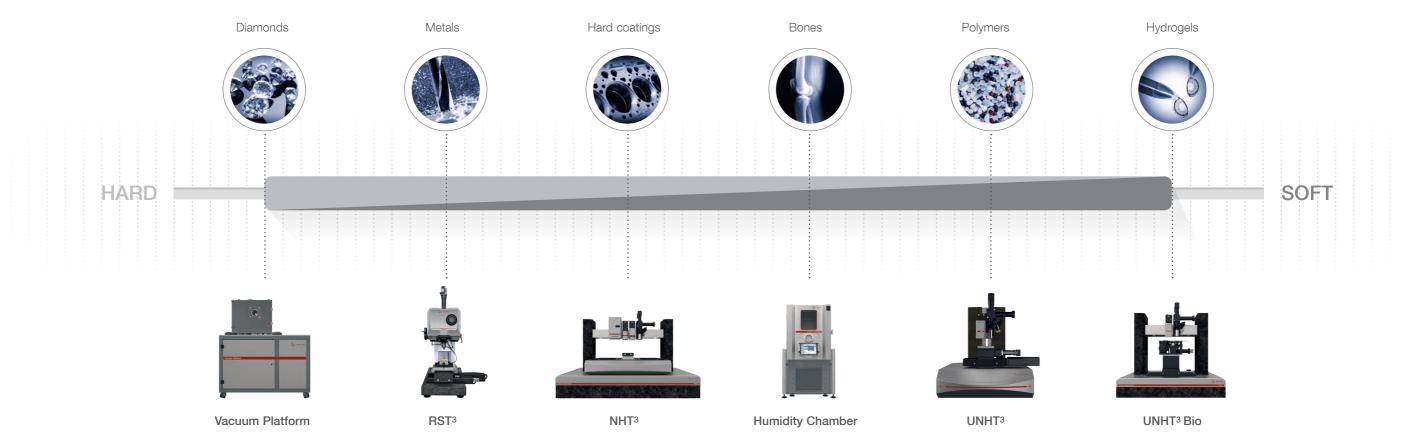
## **Applications & industries**

High productivity and high throughput for both academic and industrial applications.

The materials tested come from a very wide range of industrial applications, e.g. the cutting tools, automotive, electronics, biomedical, semiconductor, polymer, optics, civil nuclear, MEMS, and watch industries.

These instruments are also used in various research areas:

- Viscoelastic properties
- Stress-strain curves
- Strain hardening
- Fracture toughness
- Surface mapping
- Depth profiling



# Measuring parameters & standards

## **Scratch testing**

#### Measuring parameters

Adhesive strength, friction force, coating adhesion, scratch, and mar resistance

#### Standards

#### ISO 20502

Fine ceramics – determination of adhesion of ceramic coatings by scratch testing

#### ISO 1518

Paints and varnishes - scratch test

#### **DIN EN 1071-3**

Advanced technical ceramics

Determination of adhesion and other mechanical failure modes by a scratch test

### **ASTM C1624**

Standard Test Method for Adhesion Strength and Mechanical Failure Modes of Ceramic Coatings by Quantitative Single Point Scratch Testing

#### **ASTM D7027**

Evaluation of Scratch Resistance of Polymeric Coatings and Plastics Using an Instrumented Scratch Machine

#### **ASTM D7187**

Standard Test Method for Measuring Mechanistic Aspects of Scratch/Mar Behavior of Paint Coatings by Nanoscratching

#### **ASTM G171**

Standard Test Method for Scratch Hardness of Materials Using a Diamond Stylus

## **Indentation testing**

#### Measuring parameters

Hardness and elastic modulus, creep compliance, relaxation, Hertz analysis, dynamic mechanical analysis (E', E'', tan delta), stress-strain curve, fatigue

#### Standards

#### ISO 14577

Metallic materials – Instrumented indentation test for hardness and material parameters

#### ISO 6508

Metallic materials - Rockwell hardness test

#### ISO 6507

Metallic materials - Vickers hardness test

#### ISO 4516

Metallic and related coatings – Vickers and Knoop microhardness tests

#### **ASTM E2546**

New Standard Practice for Instrumented Indentation Testing

## ASTM B933

Standard Test Method for Micro Indentation Hardness of Powder Metallurgy Materials

#### **ASTM D785**

Standard Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials

#### ASTM E140

Standard Hardness Conversion Tables for Metals

## **ASTM E384**

Standard Test Method for Microindentation Hardness of Materials

### ASTM B578

Standard Test Method for Microhardness of Electroplated Coatings

## **Coating thickness**

## Measuring parameters

Coating thickness

#### Standards

#### ISO 26423:2009

Fine ceramics (advanced ceramics, advanced technical ceramics) – determination of coating thickness by crater-grinding method

#### ISO 1071-2

Methods of test for ceramic coatings – determination of coating thickness by the crater-grinding method

#### VDI 3198

Coating (CVD, PVD) of cold forging tools

## **Tribology**

#### **Measuring parameters**

Friction coefficient, wear rate

#### Standards

#### ISO/TR 11811

Nanotechnologies – guidance on methods for nanoand microtribology measurements



## Scratch testing: Features

# Unique, patented synchronized panorama to be analyzed whenever, wherever

Anton Paar is the exclusive holder of the patents US 8261600, and EP 2065695. The scratch tester's panorama mode is the most important feature of the software. After the scratch, you have the option of recording the full panorama. When your panorama is recorded, you can re-analyze your results at any time.

# Patented (US 6520004 and EP 1092142) true penetration depth measurements for advanced elastic recovery studies

The displacement sensor  $D_z$  monitors the surface profile of your sample before, during, and after a scratch. This means you can evaluate the penetration depth of the indenter during and after the scratch, for even more reliable insights into scratch and mar resistance. A unique feature of multi-post-scan with time is available for advanced studies on viscoelastic properties.

## Active force feedback for full reproducibility

The system's active force feedback ensures reproducible scratch testing, even when you investigate more complex surface geometries like non-parallel, rough, or curved samples. Anton Paar's testers are the only commercially available systems that have active force feedback.

# Automatic detection of critical loads to optimize the results

The scratch testers have auto-detection for the critical failures. Using the signal of friction force, penetration depth, or acoustic emission, an algorithm based on our experience is now able to fully automatically analyze the difference of signals and therefore to define the critical loads without any human factor.



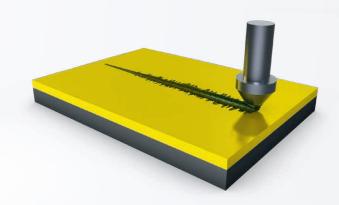
# Measuring principles

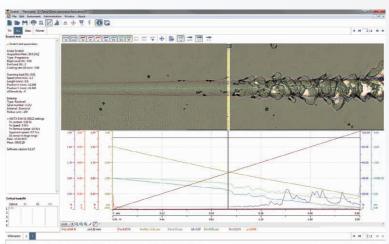
## Scratch test principles

Anton Paar scratch testers (previously from CSM Instruments) are ideal instruments for characterizing the surface mechanical properties of thin films and coatings, e.g. the adhesion, fracture, and deformation.

The ability of the scratch tester to characterize the film-substrate system and to quantify parameters such as friction force and adhesive strength, using a variety of complementary methods, makes it an invaluable tool for research, development, and quality control. The technique involves generating a controlled scratch with a diamond tip on the sample under test.

The tip is drawn across the coated surface under constant, incremental, or progressive load. At a certain load the coating will start to fail. Critical loads are very precisely detected by means of the tangential force, the penetration depth, and the acoustic emission sensors together with observations from a built-in optical microscope. The critical load data is used to quantify the adhesive properties of different film-substrate combinations by using different sensors (acoustic emission, penetration depth, friction force) and video microscope observations.





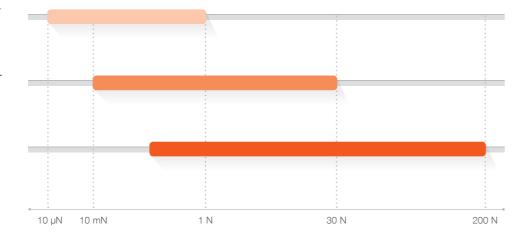
## Scratch testing: Instruments

## Measuring range of Anton Paar scratch testers

Nano Scratch Tester (NST<sup>3</sup>)

Micro Scratch Tester (MST<sup>3</sup>)

Revetest® Scratch Tester (**RST**<sup>3</sup>)





## NST<sup>3</sup> Nano Scratch Tester

The most accurate nano scratch tester on the market

The Nano Scratch Tester is particularly suited for the characterization of the practical adhesion failure of thin films and coatings with a typical thickness below 1000 nm. The Nano Scratch Tester can be used to analyze organic and inorganic coatings as well as soft and hard coatings. The unique design of the nano-scratch measurement head includes two sensors for force and depth measurements associated with a state-of-the-art piezoelectric actuator. These unique features provide a fast response time (down to milliseconds), great accuracy, and great flexibility for all kinds of scratch measurements.



## MST<sup>3</sup> Micro Scratch Tester

#### The wide range tester for demanding users

The Micro Scratch Tester is widely used to characterize the practical adhesion failure of thin films and coatings with a typical thickness below 5  $\mu$ m. The Micro Scratch Tester is also used in the analysis of organic and inorganic coatings as well as soft and hard coatings.

Applications include thin and multilayer CVD, PVD, PECVD, photoresist, lacquers, paints, and several other types of films. Research areas and industries utilize this instrument for microelectronics, optical coatings, protective and decorative surface coatings. Substrates may be soft or hard, including glass, semiconductors, refractive and organic materials.



## RST<sup>3</sup> Revetest® Scratch Tester

## The industry reference

The Revetest® scratch tester is a typical system designed for evaluating hard-coated material having a coating thickness of several microns. The coatings can be either organic or inorganic, which can be used for magnetic and decorative applications including CVD, PECVD, PVD, metallization and passivation layers, or friction-and-wear protective coatings. The substrates used can be refractive and organic materials, minerals, glass, semiconductors, alloys, and metals.

The Revetest® scratch tester comes with an external data acquisition unit and acoustic emission detection. It also meets the requirements of ASTM C1624 and EN 1071 standards. Anton Paar has sold over 1500 Revetest® scratch testers all around the world.

## Indentation testing: Features

A wide range of testing possibilities: Hardness, elastic modulus, viscoelastic properties, creep compliance, and stressstrain curve

The wide ranges of load and penetration depth enable you to measure the mechanical properties of a large variety of materials. Soft and hard materials as well as thin and thick coatings can be tested. Hardness, elastic modulus, and other properties such as viscoelasticity, creep compliance, and stress-strain curve can be determined in a single measurement.

# Maximum stability due to unique top surface referencing

Top surface referencing protects the tip from collision, provides a high thermal stability and a high frame stiffness. The reference probes the surface position while the indentation tip is testing the material. As a result, our instruments do not require any thermal drift correction.

# "Quick Matrix" indentation mode for highly precise results within a few minutes

Anton Paar's indentation testers achieve a high sample throughput by performing up to 600 measurements per hour with full instrumented indentation curves.

# Accurate motorized tables for precise positioning

Motorized tables move the sample in every direction with an accuracy of 1  $\mu$ m. The sample moves from the microscope to the indentation tester with just one click. Automated matrices and multisampling testing are also available.

# Multi-objective video microscope for a clear view of the sample

A high-quality multi-objective microscope provides a surface visualization before and after the indentation measurements. The turret holds up to 4 objectives. Visual matrices can additionally be defined under the microscope to run indentation measurements on areas of interest.

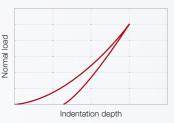


## Measuring principles

## Measuring principles

The instrumented indentation technique (IIT) involves pressing an indenter of known geometry into the surface while both penetration depth and normal load are monitored. The indentation hardness ( $H_{\rm IT}$ ), elastic modulus ( $E_{\rm IT}$ ), and other mechanical properties are obtained from the force-displacement curve. The analysis of this curve is done automatically according to the ISO 14577 standard. This is a great advantage compared to classical hardness measurements in which each imprint has to be precisely measured separately with an optical microscope.

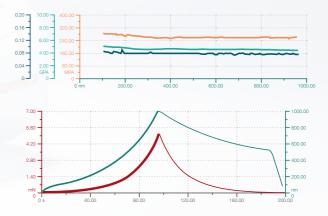






## Dynamic mechanical analysis (sinus mode)

Dynamic mechanical analysis (DMA) uses sine wave loading curves to obtain a more complete analysis of the mechanical properties of viscoelastic or coated materials. This method allows for a continuous acquisition of hardness, elastic modulus, storage modulus, and loss modulus data as a function of indentation depth and the results can be advantageously used for the characterization of coated materials or polymers.



An example of sinus mode (dynamic mechanical analysis, DMA) performed by a nanoindentation tester.

## Indentation testing: Instruments



### **UNHT**<sup>3</sup> Ultra Nanoindentation Tester

The ultimate high-resolution, high-stability nanoindenter

The Ultra Nanoindentation Tester with real force sensors examines the mechanical properties of a material at the nanoscale. The UNHT³ virtually eliminates the effect of thermal drift and compliance due to its unique and patented active surface referencing system (EP 1828744 and US 7685868). Therefore, it is perfectly suited for long-term measurements on all types of materials, from polymers to hard coatings.

The UNHT³ includes advanced indentation modes such as sinus mode, load/depth control, constant strain rate, advanced matrix, and more. Its "Quick Matrix" indentation mode allows up to 600 measurements per hour with full nanoindentation curves. Its unique and active top referencing system is patented (EP 1828744 and US 7685868) and provides the highest thermal stability (raw drift rate down to 10 fm/s) on the market.



### NHT<sup>3</sup> Nanoindentation Tester

The most versatile and user-friendly nanoindenter on the market

The nanoindentation tester has a range from low loads (0.1 mN) to high loads (500 mN) and from shallow depths (less than 20 nm) to greater depths (up to 200  $\mu$ m). It is not only robust but also fast and easy to use for multiple advanced indentation modes: Continuous multi cycles (CMC), user-defined sequences, sinus mode (optional), advanced matrix, and multi-sample protocols.

NHT³ is compatible with liquid testing. Its "Quick Matrix" indentation mode allows up to 600 measurements per hour with full nanoindentation curves. Its high load frame stiffness (107 N/m) and high thermal stability (raw drift rate <0.05 nm/s) result in high accuracy.



## MHT<sup>3</sup> Microindentation Tester

The higher force range of instrumented indentation testing (IIT)

The Microindentation Tester is ideally suited to the measurement of mechanical properties such as hardness and elastic modulus based on instrumented indentation testing (IIT). It is applicable to bulk samples and films, from soft to hard materials (metals, ceramics, polymers) with a high depth of measurements (up to 1 mm).

Optical Vickers hardness (HV) is possible to a maximum of 30 N and instrumented indentation testing (IIT) to a maximum of 10 N. Visual matrices of indentations and continuous multi cycles (CMC) allow you to measure the mechanical properties as a function of position on the surface and depth in the sample.



### MCT<sup>3</sup> Micro Combi Tester

The only available high-quality combined microindentation and micro scratch tester

The Micro Combi Tester directly measures the hardness and elastic modulus with high loads (10 N with instrumented indentation testing, 30 N with Vickers hardness). It is therefore even more valuable than conventional microhardness testers which only provide hardness measurement. It is applicable to many materials from bulk samples with rough surfaces to thin coatings. It features two independent sensors: one for force and one for depth.

The scratch mode of MCT³ also offers unique scratch testing features, including a patented synchronized panorama mode (US 8261600, and EP 2065695) from low loads (10 mN) to high loads (30 N) and wear testing capabilities.

## Indentation testing: Instruments



## **UNHT**<sup>3</sup> Bio Bioindenter

The Bioindenter is a unique device for measuring local mechanical properties of soft and biological samples. It combines instrumented indentation with the requirements of testing of soft samples, samples immersed in liquid, and biological samples. The concept of UNHT³ Bio is based on the successful ultra nanoindentation technology with extended travel range, improved force resolution, and full compatibility of testing in fluids.

Easy characterization of time-dependent properties such as creep, flow properties, or poroelasticity are possible.

The integrated true force sensor is able to apply a maximum load of up to 20 mN adapted for soft materials. The displacement sensor allows a large travel range of 100  $\mu$ m.

The elastic modulus can also be calculated from the loading part of the indentation curve using the Hertz's model, which is more appropriate for biological materials.

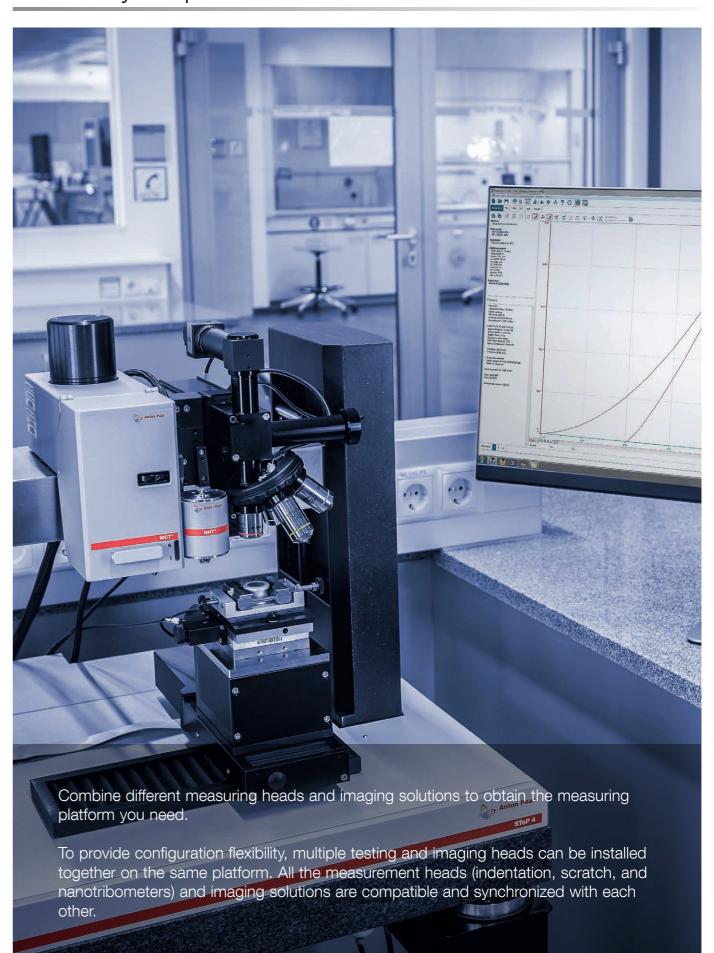
The Anton Paar Bioindenter™ works with different kinds of indenters.



# UNHT<sup>3</sup> HTV High-temperature Ultra Nanoindentation

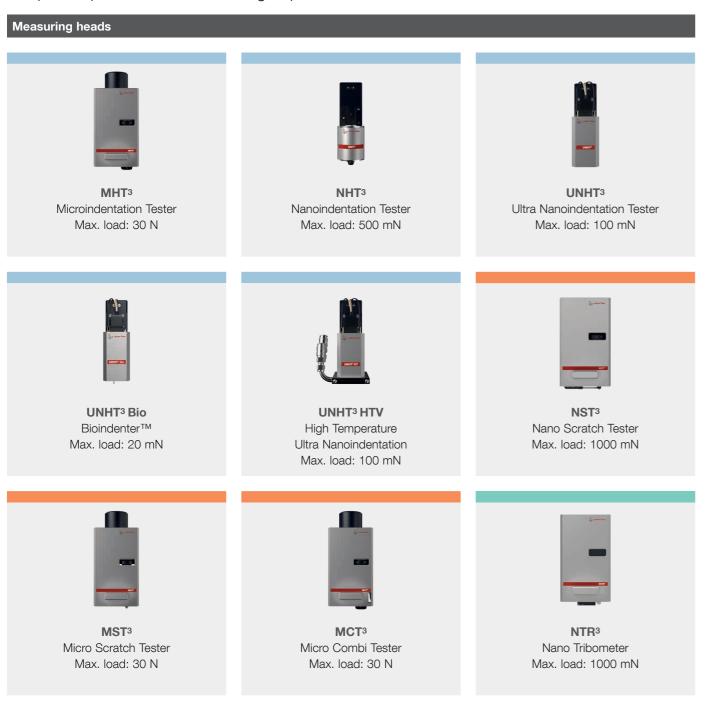
Anton Paar is a pioneer in environmentally controlled, high-temperature instrumented indentation with various solutions up to temperatures of 800 °C and down to -150 °C. The actuating system is based on the patented technology (patent US 7685868 and EP 1828744) of the Ultra Nanoindentation Tester (UNHT³) with two independent depth and load sensors combined with high-resolution capacitive sensors. Thermal barriers, water circulation and reflective mirrors prevent the head from heating, resulting in an unmatched stability. In addition, a high vacuum chamber minimizes oxidation as well as heat loss from convection. The lowest thermal drift at ambient conditions (<0.5 nm/min) and over the entire temperature range (<3 nm/min) ensures a high reliability for the measurements.

# Choose your perfect combination



# Choose your perfect combination

The perfect platform for all measuring requirements in mechanical surface characterization



## **Platform features**

- Fully automated 3-axis motion control
- High positioning accuracy over a great length with a table top Integrated anti-vibration table (TTX) and surface testing platforms (STeP 4 and STeP 6)
- One-click synchronization of position from video microscope to indenter tip
- High modularity with reversibly exchangeable measuring heads
- Customized synthetic granite for enhanced vibration damping
- Optional acoustic enclosure
- Customized platforms for glove box and vacuum chamber available

## Imaging solution

# Platforms



Optical Video Microscope





Atomic Force Microscope



Surface Testing Platform







STeP 6 Surface Testing Platform

# Coating thickness: Features

# Quick and simple determination of coating thicknesses

The Calotest instruments from Anton Paar provide quick, simple, and inexpensive determination of coating thicknesses. Employing the simple ball-cratering method, the thickness of any kind of single or multilayered coating stack is accurately checked in a short time, in full compliance with relevant international standards.

## Easy and accurate evaluation of results

The video module in the form of a USB color camera with two kinds of objectives (5x and 10x magnification) supplies the software with the crater picture. Based on the pictures and by performing line measurements and also taking the contact geometry into consideration, the software can calculate the coating thickness of the sample. In this way single and multilayer analysis in accordance with ISO 1071-4 can be performed. Automatically generated user-defined reports provide complete documentation.

# Measuring principles

## Spherical abrasion test method

A small crater is ground into a coating with a ball of known geometry, providing a tapered cross-section of the film when viewed under an optical microscope. In this way Calotest instruments measure the thickness of coatings in a very short time of just 1 to 2 minutes.



## **CATc**

The CATc is widely used for analyzing coatings with thicknesses between 0.1 µm and 50 µm. Typically measured materials include CVD, PVD, plasma spray coatings, anodic oxidation layers, chemical and galvanic deposits, polymers, paints, and lacquers. Flat, spherical, or cylindrical samples can be fixed in the sample holder.



## CATi

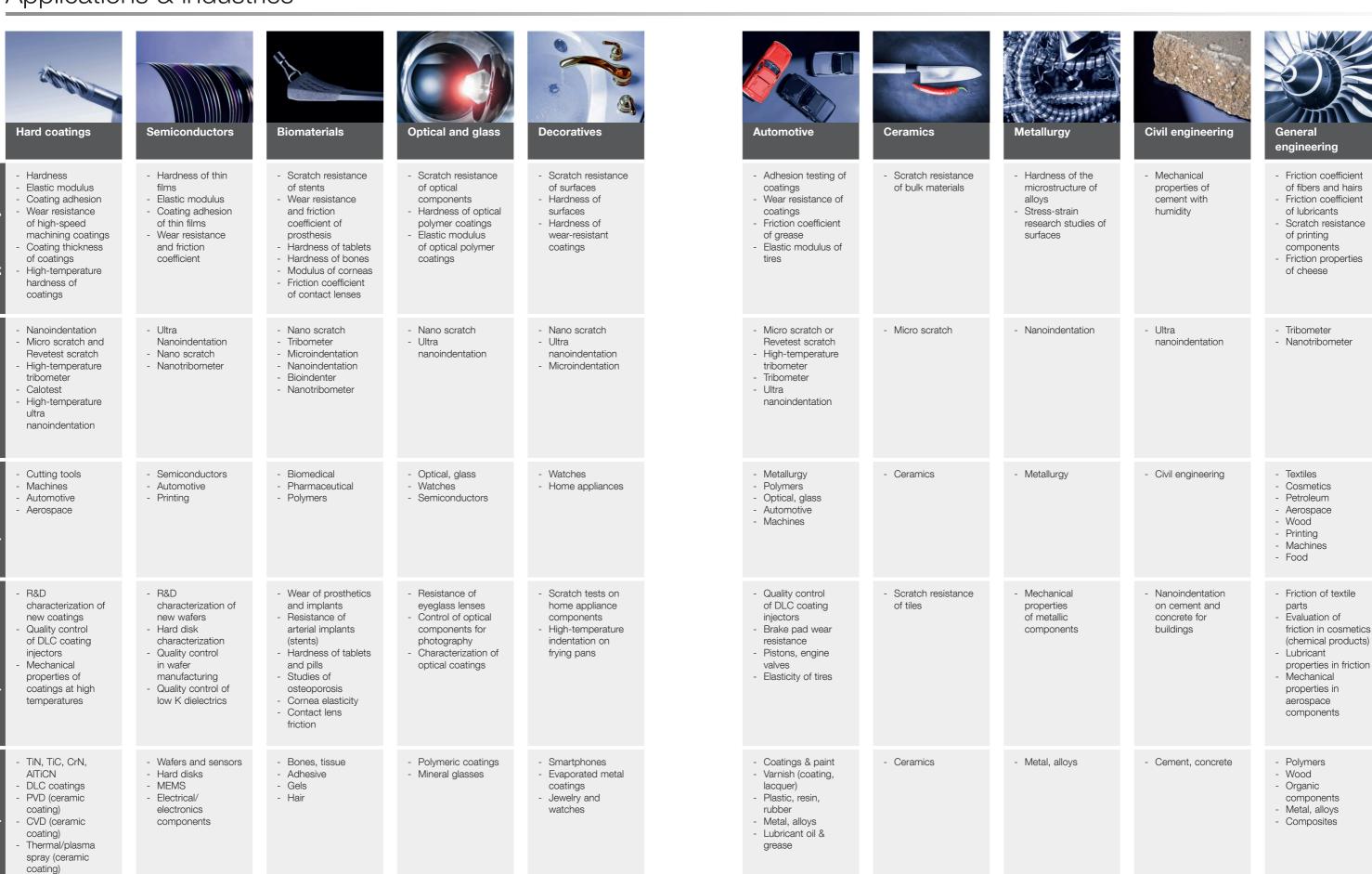
The CATi measures the thickness of coatings in a typical time of 2 to 5 minutes. In this industrial version the motor is fixed on a hydraulic arm, allowing you to target samples of unlimited size. It is the ideal instrument for quick and precise determination of coating thickness on common industrially coated components



### CATcombo

The CATcombo is the perfect combination for testing both small and relatively large samples with one instrument and output device.

# Applications & industries



# Specifications

Scratch testing			
	NST <sup>3</sup>	MST <sup>3</sup>	RST <sup>3</sup>
Maximum load [N]	1	30	200
Load resolution [µN]	0.01	10	100
Load noise floor [rms] $[\mu N]^*$	0.1	100	1000
Loading rate [N/min]	up to 100	up to 300	up to 300
Depth range [µm]	600	1000	1000
Depth resolution [nm]	0.1	0.05	0.05
Depth noise floor [rms] [nm]*	1.5	1.5	2.5
Data acquisition rate [kHz]	192	192	192
Scratch speed [mm/min]	0.1 to 600	0.1 to 600	0.4 to 600
Options			
Peltier heating up to 120 °C	✓	✓	✓
Heating stage up to 200 °C	✓	✓	✓
Heating stage up to 450 °C		✓	✓
Cooling down to -120 °C	✓	✓	✓
Electrical contact resistance (ECR)	✓	✓	✓
Liquid testing	✓	✓	✓

Indentation testing						
	UNHT <sup>3</sup>	NHT <sup>3</sup>	MHT <sup>3</sup>	MCT <sup>3</sup>	UNHT <sup>3</sup> Bio	UNHT3 HTV
Maximum indentation load [mN]	100	500	30,000	30,000	20	100
Load resolution [µN]	0.003	0.02	6	6	0.001	0.006
Load noise floor [rms] [µN]*	< 0.05	<0.5	<100	<100	0.1	0.5
Maximum indentation depth [µm]	100	200	1000	1000	100	100
Depth resolution [nm]	0.003	0.01	0.03	0.03	0.006	0.003
Depth noise floor [rms] [nm]*	<0.03	<0.15	<1.5	<1.5	0.25	0.15
Options						
Sinus mode	<b>✓</b>	<b>✓</b>				<b>~</b>
Liquid testing	<b>~</b>	✓	✓	<b>✓</b>	✓	
Heating stage up to 100 °C	<b>✓</b>		~	~	~	
Heating stage up to 200 °C	<b>✓</b>		~	~		
Heating stage up to 450 °C			<b>✓</b>	✓		
Heating stage up to 800 °C						✓
Petri dish holder					~	

The raw acquisition rate of indentation testers is 192 kHz, which is then filtered down to 400 Hz maximum for display.

Additional options and accessories are available on specific request: Electrical contact resistance (ECR), cooling for low temperatures (-150 °C in vacuum), wafer holder, multiple sample holder, ...

\*Noise floor value specified under ideal laboratory conditions and using an anti-vibration table.

✓ available option

## Coating thickness testing

Calotest Compact (CATc)   Calotest Industrial (CATi)   Calotest Combo (CATcombo)		
Shaft speed [rpm]	10 to 3,000	
Abrasion time ranges [seconds]	1 to 10,000	
Standard ball diameters [mm]	10, 15, 20, 25.4, 30	

## Tribology

	Nanotribometer (NTR³)
Normal force range [mN]*	1000
Normal force resolution [µN]*	0.003
Friction force range [mN]*	1000
Friction force resolution [µN]*	0.006

Rotating movement		
Speed [rpm]	1 to 200	
Radius [mm]	0.1 to 20	

Linear reciprocating movement <sup>1)</sup>		
Stroke length [mm]**	up to 5	
Speed [mm/s]**	up to 26.6	
Frequency [Hz]**	0.01 to 10	

Rotational reciprocating movement <sup>2)</sup>		
Speed [rpm]	1 to 200	
Angular amplitude [°]	±10 to ±150	
Angular resolution [°]	0.1	

Options	
Online wear depth [µm]	up to 250
Electrical contact resistance [Ohm]	up to 1000

<sup>\*</sup> Nanotribometer normal load and friction load specifications depend on the choices of different cantilevers (high-resolution, standard, and high-load cantilevers).

<sup>\*\*</sup> Nanotribometer linear reciprocating movement specifications depend on the choices of different linear stages.

<sup>1)</sup> Linear reciprocating movement specifications depend on the combination of stroke length, frequency, and mass on the stage.

<sup>2)</sup> Rotational reciprocating movement specifications depend on the combination of angular amplitude, frequency, and mass on the stage.