

# Laboratories & Instruments

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## MICROSCOPY

# Metallographic microscope

ZEISS Axio Observer 7m

The Axio Observer inverted microscope allows to investigate, develop and analyse the materials. The inverted construction promotes the study of a large number of samples in no time at all.

Using dedicated software, it is possible to analyse, for example, non-metallic inclusions, grain sizes and phases in automatic way.

Thanks to the inverted platform, the Axio Observer can analyze a large number and heavy samples. In fact, its inverted design facilitates parallel alignment to the objective lens: simply, it is possible to put the specimen on the stage, focus once and keep the focus for all further magnifications and samples.

The Axio Observer allows to correlate the light and electron microscopes thanks to the Shuttle & Find tool. It is a correlative micro copy interface useful in materials analysis.

A combined hardware and software solution allows transferring the specimen from one microscope system to another and analyse the same region of interest.



Brightfield





Darkfield



Polarization with Additional

I ambda Plate

Polarization Contrast

#### **Specifications**

ТҮРЕ	Inverted Reflected light
SAMPLE HOLDER	<30 mm diaphragm Motorized/Manual
SEM CORRELATIVE	Yes
SAMPLE GEOMETRY	<50Kg
ANALYSIS	Bright field/Dark field Polarized light (Interference contrast)
ELABORATION	Image analysis software module/particle analysis
OPTICS	2.5x, 5x, 10x, 20x, 50x, 100x





## Applications

Typical Applications, Typical Samples	Task	ZEISS Axio Observer Offers
Grain Size Analysis	Analyze grain size supporting ASTM E 112, ASTM E 1382, DIN EN ISO 643.	Perform a standard-supporting grain size analysis using variable methods: automatic reconstruction of grain boundaries and determination of individual grain sizes; semiautomatic linear intercept methods; comparison to reference series images; present the result of the analysis in a report.
NMI (Non-Metallic Inclusions)	Determine steel purity level supporting EN 10247, DIN 50602, ASTM E45, ISO 4967, JIS G 0555, GB/T 10561; determine the percentage of non-reflective inclusions and rate non-metallic inclusions.	Analyze steel purity in accordance with current international standards; overview of results in image and chart form; selection of various gallery views with corresponding analysis and classification data; storage and management of all analysis data such as charts, images, galleries, reports, testing procedures in the asset archive.
Birefringent Samples: Ores, Metals, Metal Alloys, Coals, Ceramic	Analyze anisotropic samples such as Barker etched aluminum alloys, zinc alloys, graphi- te, titanium alloys and magnetic materials.	Analyze anisotropic samples under polari- zation contrast with a range of polarization accessories such as analyzer and polarizer.
Analyze Layer Thickness	Measure layer thickness and geometric properties of e.g. electrodes	Analyze simple and complex layers; identify layers by color value or gray scale; precise, individual and automatic calculation of the course of measuring axis for each layer, regardless of the number of layers; presenta- tion of results in an easy-to-read report with sample data and findings such as maximum and minimum axis length, mean value and standard deviation.
Analyze Graphite Particles	Analyze of shape, size and distribution the graphite particles.	Analyze the shape, size and distribution of the graphite particles and classify them in line with standards using automated image analysis. Determine size and shape in accordance with EN ISO 943 or nodularity in accordance with SAE J 1887; present the results with all classifications in a report.
Multiphase Analysis	Measure phase distribution in multiphase samples.	Analyze your samples' phase distribution; measure up to 32 phases and determine phase percentages or other parameters like size, shape and orientation of particles; classify the detected particles and document the results in a report.



## MICROSCOPY

# Optical microscope

ZEISS Axio Zoom V16

Axio Zoom stereomicroscope delivers images of large components with low and medium magnification and high image quality.

The low magnifications of the stereomicroscope allow to have a wide depth of focus, then to obtain images on large areas of components appreciating the morphological irregularities. By this instrument, it is possible to carry out a preliminary analysis of the components or samples.

## Highlights

- Zoom between large object fields in minute detail
- Optimized zoom for all applications
- EpiRel produces a relief-like image contrast





## Applications

Typical Applications, Typical Samples	Task	ZEISS Axio Observer Offers
Scanner Component	Fast imaging and measurement of large components with high resolution to analyze structure details statistically	Imaging of large components with low and medium magnification and high image quality. Homogenous coaxial illumination for high quality capture of large MosaiX images.
Materialography	Materialographic routine analysis of standard materials, quantitative and qualitative structural analysis, resolution of fine structures	Highly detailed image information even with low-to-medium magnification of complex material structures owing to the large numeric aperture

## Specifications

Objective Eyepiece PL 10x/23		3	Eyepiece PL 16x/16			
	Factor	FWD 1) in mm	Magnification	Object field (mm)	Magnification	Object field (mm)
PlanNeoFluar Z**	1.0x	56	7x 112x	33 2.0	11x 179x	23 1.4



## MICROSCOPY

# Scanning electron microscope

#### ZEISS EVO MA10

The ZEISS EVO MA10 is a high-resolution scanning electron microscope (SEM). It is used in different fields of application especially in materials science.

This instrument allows to analyse samples of massive material or with coatings, fractures on small components (failure analysis).

Moreover, it provides the study of engineered materials, such as alloys, coatings and multi-layer nanostructures.

It is possible to obtain topographic information from the samples thanks to the high-vacuum secondary electron detector, as well as compositional information thanks to the high- and low-vacuum backscattered electron detector. The ZEISS EVO MA10 is able to work both at high vacuum and at variable pressure, thus allowing the analysis of all conductive or non-conductive materials.

It also features a Bruker QUANTAX 200, a microanalysis system based on energy dispersion spectroscopy (EDS) that provides the qualitative and semi-quantitative compositional data of the samples to be studied.

It is also possible to perform chemical analysis on profiles to determine compositional gradients, or mapping elements on two-dimensional images, useful for associating phases and composition.

#### **Materials**

- Metals
- Polymers
- Ceramics
- Composites
- Coatings
- Multilayers
- Conductive and non-conductive specimens

## Applications

- Morphological analysis
- Microstructural analysis
- Chemical composition analysis (qualitative and semi-quantitative)
- Failure analysis
- Structural analysis
- Analysis of coatings and multilayers
- Image analysis
- Identification of material defects
- Measure of particles size



- Electron source Tungsten filament
- Accelerating voltage of 0.2 – 30kV
- Current beam 0.5pA-5µA, continuous

EVO MARO

ENO MATO

- Detectors SE and BSE
- Vacuum condition High and low vacuum
- Sample size
  230x100mm
  (diameter x height)

#### Shuttle for correlative microscopy

Shuttle & Find from ZEISS is a correlative microscopy interface for light and electron microscopes, designed specifically for use in materials analysis. A combined hardware and software solution, it allows to transfer specimen from one microscope system to another in just minutes.

Shuttle & Find is an extremely flexible two-way system that allows to combine any number of ZEISS systems for correlative microscopy. It also supports intermediate preparation steps, ensuring the sample is optimally prepared for use when you switch from one system to the other.

Importantly for materials analysis, Shuttle & Find speeds up workflow by automating the process of searching the same region of interest. This reduces cycle times, allowing to process a considerable larger number of samples in a shorter period.

Stand Har

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## **PREPARATION SYSTEMS**

# Cut-off machine

Lapping machine

STRUERS SECOTOM-50

#### STRUERS TEGRAMIN-25

- Tabletop, precision cut-off machine with movable cutting table and variable speed.
- Automatic feeding with electronic control of feed speed.
- Motorized positioning of cutoff wheel and workpiece and digital read-out.
- 10 cutting methods and cutoff wheel database.
- Complete with recirculation cooling unit and flange set 50 mm (2") dia.

Tegramin-25 Automatic is a semi-automated system for lapping and polishing samples, with various types of abrasive both on disk and in suspension, complete with an automatic dosing system.

# Mounting press

STRUERS CITOPRESS-1

The Citopress-1 is an embedding press with hot reactive resin. By applying controlled pressure, the press ensures excellent adhesion to the sample of the support material.

Cold mounting systems are also available for heat sensitive or fragile materials.











## MECHANICAL CHARACTERIZATION

# Hardness and scratch tester

ANTON PAAR STEP 4 Surface Testing Platform

This instrument can be used for all kinds of industrial coatings, from the plasma-processed layers used in semiconductor and optical technology to the decorative and protective coatings used for consumer goods and automobile parts.

The Step 4 Surface Testing Platform is equipped with a nano/micro indenter and a scratch tester.

During the measurement, the instrument records the load and penetration depth, returning a complete loading and unloading curve where the force is plotted as a function of the indenter penetration depth. From the processing of these curves, it is possible to obtain the instrumented hardness, elastic modulus and a wide range of micromechanical characteristics. The micro and nano indentation testers cover a wide loading range ( $0.002 \div 30$  N). This allows to characterize the mechanical properties of different materials.

The scratch test allows the characterization of the scratch resistance of a wide range of materials (polymeric, metallic, ceramic) and provides a quantitative, comparative measure of the adhesion of thin films onto hard or soft substrates. The test consists of sliding a tip of known geometry onto the sample surface, under a constant or variable normal load.

#### Indentation

- Automatic hardness/elastic modulus calculation
- Different indentation modes: applied load control, penetration depth control, strain rate control
- Depth profiling with continuous multi cycles modes
- Creep and relaxation analysis
- Elastic and plastic energies
- Sinusoidal mode analysis (1-20Hz Nano)
- Statistical analysis functions (average & std deviation)

#### Scratching

- Full control of scratch tests: scratch length, scratch speed, loading rate
- Loading modes: progressive, constant, incremental
- Pre-scan and post-scan modes



#### **Materials**

- PVD/CVD coatings (TiN, TiCN, DLC)
- Thermal barrier coatings
- Metals and alloys
- Ceramics and composites
- Glasses
- Semiconductors
- Polymers (coatings, paints and bulk materials)
- Biomaterials (bones, cartilage, cornea, prostheses, stents, contact lenses, tissues and hydrogels)
- Pharmaceutical pills
- Cementitious materials (concrete, cement)

## **Specifications**

ELABORATION	Control and elaboration software for micro/nano indentation and scratching
STABILIZATION SYSTEM	Antivibration air table
SAMPLE HOLDER	Motorized
INDENTATOR	MICRO (MST3) Vickers Rockwell (scratch)
	NANO (NHT3) Berkovich
LOAD/PENETRATION	MICRO (MST3) 0.01 N - 30 N (50mN-10N Instrumented hardness, 10N-30N Vickers hardness) 0 µm - 1000 µm Scratch up to 30 N
	NANO (NHT3) 0 mN – 500mN 0 μm - 200 μm
OPTICS	Digital 5x, 20x, 50x, 100x









## MECHANICAL CHARACTERIZATION

# Dynamic Mechanical Analyser (DMA)

#### TA Instruments RSA-G2

RSA-G2 is an advanced platform for mechanical analysis of solids. It is capable of applying a variety of deformation types and collect material parameters, providing a wealth of information about material performance characteristics.

The RSA-G2 imposes a mechanical deformation to a specimen and measures the resulting stress response.

Thanks to its advanced design, characterized by separated motor and transducer, the instrument provides very accurate measurements of stress and strain.

The RSA-G2 features a variety of sample clamps that provide multiple modes of deformation.

The instrument is provided by a convection oven for precise and accurate temperature control that allows to perform:

- Test at room temperature
- Test at isothermal temperature
- Test in temperature ramp
- Test in air or in inert atmosphere, as argon or nitrogen gas

It is possible to characterize different materials, plastics or metals, performing tailor-made tests to investigate mechanical properties and simulate the exercise conditions.

#### **Materials**

#### Plastic and Rubber

- Rigid plastics
- Flexible plastics
- Elastomers
- Fibre
- Films

#### Metals

- Thin metallic samples
- Flexible samples
- Metallic foils



- Minimum force
- Maximum force 35 N
- Force resolution 0.00001 N
- **Dynamic displacement** ± 0.000005 to ± 1.5 mm
- Displacement resolution 1 nm
- Modulus range 10<sup>3</sup> to 3•10<sup>12</sup> Pa
- Modulus precision ±1%
- Frequency range 2.10<sup>-5</sup> to 100 Hz
- Temperature range Room temperature to 500° C
- Heating/Cooling rate 0.1 to 60°C/min
- Isothermal stability ± 0.1°C







COMPRESSION

TRACTION

#### **Investigated properties**

- Modulus of Elasticity
- Complex Modulus
- Storage and Loss Moduli
- Damping properties
- Compliance
- Frequency effects
- Creep and recovery
- Stress Relaxation
- Temperature-time superposition
- Glass transition
- Orientation effects
- Stress-Strain curves
- Dynamic Fatigue
- Toughness
- Viscoelasticity of materials
- Thermal transitions







## MECHANICAL CHARACTERIZATION

## Friction tester

#### THWING-ALBERT PF 2260

The Thwing-Albert FP-2260 Friction/Peel Tester is a versatile testing instrument for measuring the coefficient of friction, peel strength, seal strength and tensile strength of flexible plastic films, paper, labels, tapes, nonwovens, textiles and other sheet materials. This Coefficient of Friction Tester (COF) / Peel Tester can measure static and kinetic coefficient of friction as well as run seal strength tests, 180° peel, 90° peel, and T-peel tests.

The FP-2260 also offers a tensile test mode to perform lightweight tensile tests up to 10 kg.

The instrument allows to realize measures varying temperature, furthermore it is simple to use, thanks to a user interface with an immediate interpretation.

# Optional accessories and fixtures to perform a variety of peel, COF:

- Clip clamp, for thick or thin sample materials
- Sample clamp, ideal for thin-sheeted materials
- The 180 degrees peel arm for peel testing



#### Model name

FP-2260 Friction/Peel tester

#### Industry

Adhesives, corrugated, foils/metals, medical, nonwovens, packaging, paper, paperboard, plastic film, rigid plastics

#### Testing property

Adhesion, coefficient of friction, peel, tensile

#### List of standards

ASTM D1894, D4521, D3330, TAPPI T816, ISO 8295

Load cell 500 g

#### Force accuracy

10% to 100% load capacity: ±0.25% measuring value | less than 10% load capacity: ±0.025% of load cell capacity

- Force resolution 0.1g for all load cells
- Force units Grams, kilograms, ounces, pounds, newtons
- Travel distance 0.1 to 14.0 in (0.3 to 38 cm)
- Test times 0.1 to 99 seconds- variable for COF, and peel
- Standard travelspeed 1 to 20 in/min (25.4 to 508 mm/min)
- High travel speed 10 to 110 in/min(254 to 2,794 mm/min)
- Standard COF sleds 200 gm
- Power requirements 110-230 Volts, 50-60 Hz





## MECHANICAL CHARACTERIZATION

# Modular tribometer

#### NANOVEA T 50

The tribometer Nanovea T50 allows to perform tribological tests, to evaluate the performance of a material coupling in relative motion with applied load, determining the coefficient of friction generated and the wear of materials.

In this instrument the load on the coupling to be tested is obtained with a passive system of calibrated masses (1-40N), applied on the static part of the tribological pair by means of a balanced arm. The same arm is moved with a load cell that records the developed reaction force (up to 10kHz acquisition), allowing to calculate the friction coefficient.

The second part of the tribological pair is moved by a motor with position, speed and acceleration control, to which various modules can be connected to perform pin-on-disk (rotative), semi-linear, or linear tribological tests. Implementation of a block-on-ring system is possible.

Other modules allow the modification of environmental variables, such as the furnace for tests up to 1000°C, the liquid-cup for tests with liquids or lubricants and the cell for tribocorrosive evaluations.

Wear rates of the tested parts are obtained by post-analysis of the components, such as microscopy or profilometry, which also allow for the evaluation of wear mechanisms.

	CHARACTERISTICS	NOTES
Load	1÷40 N	Expandable up to 60N
Motor speed	0,01÷5000 RPM	0-1000 RPM in 0,15 s
Position control	0,01°	Possible fretting tests
Temperature control	Static thermocouple Furnace thermocouple Temperature and humidity recording	
Data-logger	1÷10000 Hz	On all channels
High temperature module	25÷1000° C	1000° C on rotary tests 500° C on linear tests
Tribocorrosion module		Reciprocating movement
Liquid cup		For rotary tests Compatible with HT module



## **Test modules**



## **Environmental modules**



Tribo corrosion



High temperature



Cryogenic\*





Atmosphere / humidity controlled\*

0





## THERMAL ANALYSIS

Thermal diffusivity and conductivity analyser

NETZSCH LFA 447 NanoFlash

The NETZSCH LFA 447 NanoFlash is used to measure thermal diffusivity and thermal conductivity on a variety of materials, such as ceramics, metals, composites, and multilayer systems.

The advantages of this technology are the variety of materials that can be analysed, the easy preparation of samples, the testing speed and the high accuracy of the results.

This instrument can determine abovementioned properties at room temperature and their evolution up to 300°C, due to embedded heating system.

The NETZSCH LFA 447 NanoFlash is based on laser flash technology. During a measurement, the lower surface of a sample with planar and parallel faces is initially heated by laser pulse with known energy and duration (flash) The change in temperature resulting on the upper face of the sample is then measured by an infra-red detector.

From the recorded temperature-time curve, which describes the thermal evolution of the sample, characteristic time intervals are calculated to determine the thermal diffusivity.

The thermal conductivity of the material to be analysed can be determined if its specific heat capacity and density are known.

#### **Applications**

- Measure of thermal properties in function of temperature
- Change of thermal properties in function of reaction time
- Evaluation of thermal barrier effect of a material or coating
- Evaluation of thermal conductivity ability of a material or coating



- Temperature range RT to 300°C
- Thermal diffusivity range 0.01 mm<sup>2</sup>/s to 1000 mm<sup>2</sup>/s
- Thermal conductivity range 0.1 W/(m·K) to 2000 W/(m·K)
- Repeatibility Thermal diffusivity: ± 2%

Specific heat: ± 3%

### Accuracy

Thermal diffusivity:  $\pm$  36% Specific heat:  $\pm$  5%

#### Flash source

Xenon Flash Lamp, wavelength: 150nm to 2000 nm Pulse Energy: up to ≈10 Joules

#### Sensor type

IR detector (InSb) with integrated dewar for LN2 cooling

### Samples

Standard sample size: disk up to 25.4 mm diameter, or 6 mm / 8 mm / 10 mm / 12.7 mm square, up to 3 mm thick.

In addition, several sample holders are available to characterize different material forms:

- Bulk material
- Thin sheet for in-plane measurements
- Liquid with low viscosity
- Multilayers samples
- Powder





## THERMAL ANALYSIS

# Differential scanning calorimetry tester

#### NETZSCH DSC 214 Polyma

The DSC 214 Polyma is an instrument used to identify and characterize materials, by analysing their thermal properties. Its working is based on a specific thermal scanning program that highlights the temperature ranges at which the tested material undergoes a thermal event i.e. melting, crystallization, glass transition, or chemical reactions. Morover it allows to determine the specific heat of the material.

These information are of utmost importance in predicting the materials' behaviour during technological processes, and on this base determine compliance of materials a process that is sensible to thermal characteristics.

The DSC analysis is mainly used for polymeric materials, and polymer based composites, but it's also suitable for inorganic materials.

The analysis consists in subject a sample and a reference to a specific thermal program in a specific atmosphere. The instrument measures the difference in heat flow needed to maintain sample and reference at nearly the same temperature, indicating the occurrence of exothermic or endothermic events.



#### Thermal Characteristics Which Can Typically Be Detected by Using DSC

- Melting temperatures and enthalpies (heats of fusion)
- Crystallization temperatures and enthalpies
- Glass transition temperatures
- Oxidative-induction time (OIT) and oxidative-onset temperature (OOT)
- Degree of crystallinity
- Reaction temperatures and enthalpies
- Cross-linking reactions (curing)
- Degree of curing
- Specific heat capacity
- Distribution of crystal molecular weight (qualitative, via peak shape)

#### DSC 214 Polyma

TEMPERATURE RANGE	-170°C to 600°C
HEATING/COOLING RATE	0.001 K/min to 500 K/min*
INDIUM RESPONSE RATIO	> 100 mW/K**
RESOLUTION (TECHNICAL)	0.1 μW
ENTHALPY PRECISION	± 0.1% for indium ± 0.05% to ± 0.2% for most samples
SPECIFIC HEAT DETERMINATION	Equipped
TEMPERATURE MODULATION	Equipped
COOLING DEVICE OPTIONS	Compressed air cooling (RT to 600°C) IC40 (-40°C to 600°C) IC70 (-70°C to 600°C) LN2, automatically controlled (-170°C to 600°C)
GAS ATMOSPHERES	Inert, oxidizing, static and dynamic operation
GAS CONTROLLER	Switches for 3 gases included MFC for 3 gases, optional





# Compact profilometer

#### NANOVEA PS50

Nanovea PS50 is a contact-less profilometer, suitable to scan profiles and areas of a surface by correlation of a x-y position of a moving sample table, with a z reading of a chromatic confocal sensor.

Chromatic confocal technique uses white light filtered through a series of lenses with high degree of chromatic aberration.

As a result, different wavelengths focus at different distances, creating a vertical measurement range.

When the surface of interest is within measurement range, a single wavelength is in focus, and is returned through the pinhole accessing the CCD detector, and determining a height reading in the instrument.

Resulting surface reconstruction is suitable for a variety of uses, spanning geometric control, roughness and texture measurement, morphology evaluation.





#### PS3 OPTICAL SENSOR

- Vertical range: 1100µm
- Can be extended up to 25mm by optional sensors
- Working Distance: 12mm
- Lateral accuracy: 2µm
- Height repeatability: < 5nm</p>

#### X-Y MOTORIZED TABLE

- Scan range: 50 x 50mm movement
- Resolution: 0,1µm
- Max sample weight: 8 kg

ROUGHNESS & FINISH	
TEXTURE & GRAIN	
GEOMETRY & SHAPE	
STEP HEIGHT & THICKNESS	
VOLUME & AREA	
PLATNESS & WARPAGE	



X - Y Scan Area 50 x 50 mm Motorized



Height Range 110 µm to 25 mm





Desktop Dimensions 38 x 33 x 43 cm Scan Speed 20 mm/s



Multisensor coordinate measuring machine

#### ZEISS O-INSPECT

The O-Inspect multisensory measuring machine enables to optimally measure each characteristic, optically or through contact measurement.

The O-Inspect delivers reliable 3D accuracy, compliant with ISO standards at a temperature range of 18-30°C.

ZEISS CALYPSO, the dedicated software, not only delivers results easily, but also makes detecting and identifying defects straightforward.

#### **Specifications**

## OPTICAL SENSOR

- HD Camera
- 12x Zoom Zeiss Discovery
- Bicolor light (red/blue) for optimal contrast
- White/Black analysis of image
- White light distance sensor

#### **CONTACT SENSOR**

- Zeiss VAST XXT
- Point to point measure
- Continuos scan measure

#### SOFTWARE CALYPSO

- Camera image
- CAD model
- Results view



#### **Specifications**

Traverse 50mm

Measuring Speed

0.05; 0.1; 0.5; 1 mm/s

Detector Measuring Force 0.75 mN

Stylus Tip Angle 60°

**Stylus Tip Radius** 2 μm

Maximum number of channels

**Range** 800; 80; 8 μm

Positioning ±1.5° (tilting) and 10 mm (up/down)

**Profiles** Primary Profile (P), Roughness Profile (R), Waviness (W), MOTIF (R, W)

Standard EN ISO, VDA, JIS, ANSI and customized settings

Analysis graphs AC and ADC

Digital filter Gauss, 2CR75, PC75

No. of sampling length (L) x 1, x 3, x 5, x L\* (\* = or any other value)

**Cut-off length** λc : 0.08 mm; 0.25 mm; 0.8 mm; 2.5 mm; 8 mm λs : 2.5 μm; 8 μm; 25 μm Surface roughness tester

MITUTOYO SURFTEST SJ-412

MITUTOYO SURFTEST SJ-412 is a contact-type surface roughness tester that acquires surface morphology of a specimen fixed on a planar surface by a stylus detector that is drag linearly over the specimen itself.

The contact technique is based on the conversion of the vertical movement of the tip sensor into an electrical signal, which will be quantified according to various international standards (EN ISO, VDA, ANSI, JIS) and customized settings.

The skidless detector allows to measure the primary profile (P), roughness profile (R), waviness profile (W) and more.

The software can perform a mathematic compensation of curved, radiused and tilted surfaces. It allows two different evaluation conditions within one adjustable measurement.

Resulting profilometry is suitable for a variety of uses, as: geometric control, roughness and texture measurement, morphology evaluation.

Mitutovo



# Electrochemical analyser

#### PGSTAT 302N

The potentiostat/galvanostat PGSTAT 302N is used for the study of the electrochemical properties of metallic materials and coatings: it allows to test the corrosion resistance of the samples in different environmental conditions.

In potentiostatic mode, a potentiostat/galvanostat (PGSTAT) will accurately control the potential of the Counter Electrode (CE) against the Working Electrode (WE, tested material) so that the potential difference between the working electrode and the Reference Electrode is well defined, and corresponds to the value specified by the user. In galvanostatic mode, the current flow between the WE and the CE is controlled. The potential difference between the RE and WE and the current flowing between the CE and WE are continuously monitored.

The value specified by the user is accurately controlled anytime during the measurement by using a negative feedback mechanism.

Thanks to the additional module (FRA32M), it is furthermore possible to perform electrochemical impedance spectroscopy (EIS).



- Compliance voltage range in volt 30
- Current resolution 0.0003 % (of current range)
- Input impedance in Ohm 1 TOhm
- Maximum bandwidth in Hz 1 MHz
- Maximum current in ampère 2
- Maximum number of channels 1
- Maximum number of modules 8

- Modular instrument Up to 4 connections - EIS Module FRA32M
- Number of current ranges 9.0
- Number of current ranges remarks 10 nA to 1 A
- Potential and current accuracy 0.2 % or 0.2 % of current range
- Potential range in volt 10
- Potential resolution 0.3 µV (gain 1000)





### CHEMICAL AND PHYSICAL CHARACTERIZATION

# Tribocorrosion machine

#### TRIBOCORR

Thanks to the industrial research activity and the competences acquired in the Reliability and Surface Engineering fields, II Sentiero has designed and built TriboCorr. This machine is able to carry out tribocorrosion tests by means of the simulation of specific tribological conditions in a corrosive environment.

TriboCorr is able to reproduce not only operating conditions of materials to be tested, but also

the most difficult working conditions accelerating degenerative phenomena that generally occur in the long term.

TriboCorr is both a versatile and modular tool: thanks to the interchangeable modules that compose it, it is possible to reproduce different operating conditions.

Wear is caused by a counterpart (often hard material) that puts pressure on the test-piece of material or on the treatment to be analyzed; this sample can have different shapes, according to customers' needs and the physical phenomenon to be studied. On the other side, corrosion is reproduced recreating an environment that simulates or reproduces aggressive conditions of the real working system.

Therefore, in TriboCorr we can find:

- Aqueous solution of chlorides
- Aqueous solution of hydrogen peroxide
- Lubricants
- Aggressive substances
- Acids and bases

The degradation of the test-piece can be monitored in real time through electrochemical analyses, thanks to the integrated potentiostat and the set of electrodes. It is possible also to control the friction coefficient and carry out other specific analyses.

The plant can also be used for static corrosion experiments.





## **Tribological aspect variables**

#### Input data in the system

INPUT	GENERATED BY	MEASURED BY
Rotational speed/Linear speed	Electric motor + adapter	Servomotor
Normal load	Pneumatic actuator	Load cell
Test duration	-	Interface software
Environment	VHP-Circuit	Concentration detector
Temperature	Resistances	Thermocouple

#### Output data from the system

OUTPUT	GENERATED BY	MEASURED BY
Friction force (COF)	Test-piece sliding contact	Load cell
Volume of removed material	-	Profilometer, optical/electron microscope
Track depth	Test-piece sliding contact	Profilometer, optical/electron microscope
Wear rate	Test-piece sliding contact	Profilometer, optical/electron microscope

VHP plant, Vaporized Hydrogen Peroxide, allows recreating environmental conditions required by the customer.

## **Corrosive aspect variables**

#### Input data in the system

INPUT	GENERATED BY	MEASURED BY
Electrolyte composition	Circuit	Circuit
Electrolyte PH	VPH	PH-metro
% of chlorides	VPH	Conductivity meter
Polarization potential	Potentiostat	Potentiometer
Environment	VPH	Concentration detector
Temperature	Resistances	Thermocouple

#### Output data from the system

OUTPUT	GENERATED BY	MEASURED BY
Corrosion potential	-	Potentiostat voltmeter
Polarization density (potentiostatic tests)	Potentiostat	Potentiostat ammeter
Potentiodynamic polarization plot	Potentiostat	Potentiostat ammeter



Potentiodynamic curve



## CHEMICAL AND PHYSICAL CHARACTERIZATION

# Corrosion simulator

#### SIMUCORR

This instrument, designed and built by II Sentiero, provides useful information on the corrosion behaviour of different materials according to the environment they are used in.

The test consists of three tanks which can all be used independently. Each tank can house a maximum of 48 samples with a standard size of 150 X 150 mm and 2 mm thick. It can also house samples with the actual shape of the component to be analysed.

The system is able to automatically recreate the environments used by the customer in terms of electrolyte liquid concentration and temperature. The circuit is designed to use acid substances, basic substances, hydrogen peroxide and other substances, from room temperature to a maximum of 90°C.

There are 3 simulation zones: in the lower section the samples are completely immersed in the liquid; in the centre section there are two sprinklers to reproduce the external wash cycle and, in the upper section, there is a tank with calibrated holes that regulates the dripping of the liquid to simulate the drops of condensate that forms inside the machine.

#### **Monitored corrosion:**

- Uniform corrosion
- Pitting corrosion
- Crevice corrosion
- Inter-granular attack
- Galvanic corrosion
- Hydrogen damage (can be implemented)
- Microbiologically inducted corrosion (can be implemented)
- Stray currents (can be implemented)



## The tests are based on:

- Visual inspection for a general analysis
- Level of free corrosion potential
- Level of free corrosion potential in the vicinity of crevice washers
- Visual and stereoscopic inspections to detect the presence of pitting
- Loss of weight after the removal of the oxides with an ultrasonic wash cycle





## ADDITIVE MANUFACTURING

# 3D printer

#### SISMA MySint 300

The Additive Manufacturing technology is based on a process of accretion of the component layer on layer and allows the creation of very complex geometries, difficult or not achievable with traditional technologies.

Thanks to the advantages offered by design freedom and the continuous expansion of the variety of materials that can be processed, this technology is now beginning to take hold in the field of industrial manufacturing of metal components.

In the context of metallic AM technologies, Selective Laser Melting (SLM) technique has been in particular adopted: after an initial distribution of a layer of metal powder on the building platform, a laser beam melts the powder following a calculated path.

The platform is then lowered in order to permit the subsequent distribution of a new layer of powder. At the end of the process, the manufactured component can be extracted by removing excess unmelted powder. In such a scenario, Il Sentiero decided to adopt Sisma MySint 100 and MySint 300 machines, which differ only in terms of production capacity (volume and printing time).



## MySint 300

- Simple and robust design
- Large printing volume
- High flexibility
- Speed of execution
- Automatic sieving



Building volume	ø 300x400 mm
Laser Source	Fiber Laser 500W
Laser spot diameter	From 100 to 500µm (variable)
Layer thickness	30-60 micron
Production rate (Stainless Steel)	Up to 25 cm³/h
Inert gas supply	Nitrogen, Argon
0 <sub>2</sub> concentration	< 100ppm
Compressed air requirement	Min 4 - max 10 atm
Power supply	400V – 3ph – 50/60Hz – 32A
Max power absorbed	15kW
Machine dimensions*	∟3400 x w1400 x н1970 mm *
(Filtration unit included)	

## **Materials**

Bronze	
Cobalt Chrome	
Stainless Steel AISI 316 - 1,4404	•
Maraging Steel M300 - 1,2709	•
Nickel Alloys	•
Aluminium Alloys AlSi 12 - AlSi 10 Mg	•
Titanium Ti6Al-4V gr 5	•

The features and performances are indicative and may change.





## ADDITIVE MANUFACTURING

# 3D printer

### SISMA MySint 100

## **Specifications**

ø100x100 mm
Fiber Laser 200 W
Quartz F-Theta Lens
55 µm
20-40 µm
Nitrogen, Argon
ø 6 mm / 2.5 ÷ 5 bar @ 35 L/min
<0,3 L/min @ 0,5% 02
<100 ppm
Glove Box
Removable
220-240 V 1ph - 50/60 Hz
1,53 kW
1390x777x1600 mm (LxWxH)



## **Materials**

Precious Metals	•
Bronze	•
Cobalt Chrome	•
Stainless Steel AISI 316 - 1,4404	•
Maraging Steel M300 - 1,2709	•
Nickel Alloys	•
Aluminium Alloys AlSi 12 - AlSi 10 Mg	•
Titanium Ti6Al-4V gr 5	•



## MySint 100

- Simple and robust design
- High resolution
- Reduced gas consumption
- High flexibility



3D printer

MARKFORGED X7

## ADDITIVE MANUFACTURING



#### **Specifications**

Print technology	Fused Filament Fabrication (FFF) with Continuous Fiber Fabrication (CFF)
Build volume	320 mm X 250 mm X 200 mm
Used materials	Base material: Nylon, Onyx, Onyx FR Reinforcement fiber: Carbon, Kevlar, Glass, High Strength/ High Temp glass
Layer resolution	50 -100 - 125 - 200 μm
Extrusion - nozzles	Double, quick-change
Software	"Eiger", cloud-based, offline

Markforged X7 3D printing is a kind of additive manufacturing that can be used to rapidly fabricate components of composite material with highly customizable geometries, using a laye bylayer fabrication process.

The base material used to print the components is called Onyx: a polyamide loaded with short carbon fibers. The base material can be reinforced by adding continuous reinforcing fibers: carbon, Kevlar, Glass, High Strength/ High Temp glass.

These composite materials can match the strength of many metals, but at much lighter weight. In particular, the addition of carbon fibres to polymeric materials is becoming a widely used strategy to enhance the mechanical properties of 3D printed parts.

The double extrusion, Continuous Fibre Fabrication (CFF) and Fused Filament Fabrication (FFF), allows to create composite parts incredibly strong and versatile.





## ADDITIVE MANUFACTURING

## 3D printer

#### Kentstrapper MAVIS

Kentstrapper MAVIS is a professional 3D printer that uses Fused Filament Fabricaton (FFF) technology.

The combinaton of great build volume (400 x 400 x 700 mm XYZ) and direct drive extrusion allows users to print a vast range of thermoplastc flaments with great precision. In additon, the printer is equipped with fully automated calibraton system that minimize the risk of print failure. Moreover, since the printer works with standard slicer sofware as Cura or PrusaSlicer, all printng parameters can be set at will, allowing users to fully personalize every print.

Real size, high precision, reliability and mult-materials are essental characteristcs for the industrial sector.







Print technology	Fused Filament Fabrication (FFF)
Build Volume	400mm X 400mm X 700mm (XYZ)
Layer Resolution	0.1-0.4 mm
Extrusion Nozzle	0.4-0.6-0.8 mm
Software	Simplify 3D, Cura, PrusaSlicer (offline)
Materials	PLA, PETG, ABS, FLEX, ASA, PC, NYLON and reinforced versions



## **COATING PROCESS**

Physical Vapor Deposition (PVD) Plasma Enhanced Chemical Vapor Deposition (PECVD)

DURALAR CENTURION

The Duralar Centurion deposition system is a versatile hybrid design for advanced coatings incorporating both PVD and PECVD technologies.

Thanks to this system it is possible to deposit thin films (metallic and ceramic) with a thickness from hundreds nanometers to tenth microns.

This system is tailored to meet industrial manufacturing needs, with high flexibility of use and configuration required by research. The deposited coatings can be used in different application fields with different features: hard protective coatings, self cleaning coatings, self lubricant coatings, functional surfaces and decorative films. The system configuration allows to coat a wide range of substrates such as metals, ceramics and heat – sensitive materials (polymers, brass, etc.).

The main features of the plant are the cathodic arc evaporator, the magnetron sputtering source and the plasma beam source driven by a RF power supply.

The Cathodic Arc Evaporator (CAE) is a well-known technology of deposition in vacuum, with the biggest advantages of high metal and plasma ionization and "simple" use in hard coating applications. Its typical defects in films, the droplets of metal, are reduced in the proposed source because of the presence of two electromagnetic coils that can "steer" the arc along the target surface, improving the quality of the deposited films. The Magnetron Sputtering (MS) source can be used to evaporate a wide range of materials, from the more standard metals like Titanium, Chromium or Zirconium, to "softer" metals that normally cannot be evaporated by the Cathodic Arc technology, like Aluminum, Nickel, Silver, Gold. The presence of Two Magnetron Units driven by a Bipolar Pulsed Power Supply can be used to deposit multilayer films with a wide range of materials, creating alloys from different targets; moreover, the flexibility of that generator permits the creation of graded films (gradually passage between a compound to another), changing not only the chemistry of the plasma but also the waveform of the generator, choosing how much material we want to evaporate from each target during the various phases.

The Plasma Beam Source driven by a RF power supply is a versatile tool because it can be used as a support in Reactive Sputtering depositions, increasing the plasma ionization, in pretreatment phases, to etch the samples with high energy ions and as a PECVD source, efficiently cracking the desired precursor that is introduced in the chamber. Typical films that can be deposit with this source are, for example, DLC (at higher deposition rate and energy respect to standard ion sources), or optical films (oxides). The PECVD is equipped with the liquid delivery system because not only gas precursor, but also liquid precursors can be introduced in the deposition chamber. Typical liquid precursors for PECVD process are compounds that contain metal-oxides or silicon-oxides (like HMDSO, TEOS).

Parts being coated have independent bias control and are rotated in a planetary motion for optimized uniformity. The chamber is fully equipped with heaters, turbo pumps, a dry roughing pump, and mass flow controllers, as well as a delivery system for the liquid molecular diamond precursor. All hardware is controlled through an advanced programmable logic controller (PLC) and an intuitive human machine interface (HMI). Processes are automated through recipes.



## **PVD-PECVD** Centurion plant features

CAE source	Current out: 0-200A Tension out: 80V Cooling: air
Magnetron sputtering source	Power out: 20kW Tension out: 1000V Cooling: water
PECVD source	Power out: 6kW Cooling: water Frequency: RF
Bias	Power out: 10kW Tension out: 1000V Current out: 33A Cooling: water
Temperature range	Room temperature up to 400°C
Maximum vacuum level	5*10-6 mbar
Speed of rotating table	1-5 rpm
Useful plasma volume	1000 mm Ø, 900 mm h
Thickness of the coatings	from hundreds of nanometers to tenth microns
Application field	Hard protective coatings, self cleaning coatings, self lubricant coatings, functional surfaces, decorative films.





## **COATING PROCESS**

High-Velocity Oxygen-Fuel thermal spray system (HVOF)

HVOF is a technique used to apply a metallic or composite metal/ceramic coating. It is based on the use of a gas flow with high temperature (over 2000°C) and high speed (over 2000m/s) to project molten particles on a target surface.

The system is composed by a spray-gun, containing a combustion chamber and expansion nozzles responsible for the combustion reaction and acceleration of the gas flow, that is mounted on a robot that moves the gun along the target component at precise distance and speed. These are enclosed in a soundproof spraying booth, with some ancillary devices that provide process gases, electric power, cooling air and water, and feedstock powder.

Coatings obtained with this technology are optimized for wear resistance, often in combination with corrosion-resistant material formulations that allows use in harsh environmental conditions and extreme stressing applications. Hard-facing metallic alloys as Stellites, or composite systems of carbides in metallic matrix, as WC-Co or Cr3C2-NiCr are commonly used compositions. Given high density and low flight time of particles, it is also possible to obtain metallic coatings with low oxidisation for corrosion resistance at low and high temperatures, based on Inconel, Me-CrAIY alloys, stainless steels, copper-alloys, and other. Typical thickness range for these coatings is 10µm up to over 1mm. Industrial application of HVOF ranges from wear resistant components in manufacturing (glassmould plungers, steel mill guides and rolls), heavy equipment (hydraulic rods and pistons), petrochemical (valve assemblies and pump components), aviation (compressor and turbine blade coating, landing gears, actuators, flaps), power generation (industrial gas turbines and hydroelectric turbine coating, nozzles).

Our system is designed for research purpose, with a 2-axis manipulator combined to a rotating headstock with horizontal axis, suitable for coating plane samples and cylinders, and could be used for manufacturing of small batches. A coating designed and realized with this equipment is easily scalable on industrial production, since industrialization relies in a simple upgrade in manipulation of workpiece and spray gun.



## Thermal spraying plant features

<b>HVOF System Oerlic</b>	on Metco:
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Spray gun HVOF DJ-M	Fuel gas: Hydrogen-fed Water/Air-cooled Diamond Jet Gun
Control unit DJCE-H	Gas speed: 2140 m/s (water-cooled) - 1373 m/s (air-cooled);
Handling controller DJCE-2600	Gas temperature: 2600-3000 °C; Total heat output of 113 kW (water and air-cooled);
Powder feeder 9MPE-DJ	Gravimetric powder feed operation system; Closed-loop feed rate monitoring and control system; Hopper capacity of 5.5 liters; Rotameter flow carrier gas metering; Feed rate: 5-300 g/min Particle size: 2-200 µm

#### Manipulator and handler - Ticienne:

Gun handler with X-Z translation translation	PLC controlled cartesian manipulator; X axis: 1100 mm travel and 0-200 mm/s speed Z axis: 500 mm travel and 0-120 mm/s speed Manual regulation of spray distance
Workpiece headstock with horizontal axis	Horizontal axis rotating headstock with Manually operated tailstock Rotation speed: 0-300 RPM Supported weight: 150 Kg max. (with tailstock) Ø = 500 mm max., L = 900 mm max.
Soundproof booth	
SITA Impianti	400x300x250 H cm
Air filter and dust collector	
Donaldson Torit DF03-18-R	Residual emission ≤ 2 mg/m3 Processed air 10.000 m3/h





## COATING PROCESS

# Calotest Cat<sup>2</sup>c compact

The calotest, also called ball crater method, is a simple, though destructive technique, to measure the thickness of a single or several layers on a substrate in the 100 nm - 50  $\mu$ m range, with a ± 5% tolerance. The technique can be applied to planar, spherical and cylindrical samples.

A steel ball of known diameter is pressed by a fixed load against the coating surface and is rotated while a diamond suspension flows in the contact point in order to generate abrasion.

A spherical cap-shaped abrasion mark is produced on the coating, until the substrate is reached. The width of the various coating layers is measured by an optical microscope and the thickness values are obtained by applying known geometrical relations.

Dimensions of the abrasion mark allow calculation of coating thickness through easy geometrical relations implemented in the software.





Shaft Speed and Rotation Direction	Motorised Speed: 10 – 30000 rpm Rotation direction: clockwise/counter - clockwise
Abrasion Time Range	1 - 10000 sec
Nominal Motor Torque (Max. Continuous)	28 mNm
Typical Thickness of Coating Measurement	0.1 – 50 μm
Measurement Precision	1 - 5%
Water Based Diamond Suspensions (Slurry)	Superfine slurry with diamond particles size < 0.2 µm → for thin or soft coatings (e.g. metallic, polymeric)
	Hi – quality slurry with diamond particles size from 0.5 to 1 $\mu$ m $\rightarrow$ for thick and hard coatings (e.g. ceramic)
Ball Material and Diameters	Material: AISI 420 steel Diameters: 10, 15, 20, 25.4, 30 mm
Sample Holder Movement	For translation adjustment: 0 – 55 mm For tilt adjustment: 0° - 60°
Sample dimensions	Width: max. 47 mm Diameter: min. 2 mm, max. 55 mm

## **Materials**

- PVD and CVD coatings
- Plasma spray coatings
- Anodic oxidation layers
- Surfaces treated by ion sputtering or ion plating
- Chemical and galvanic deposits
- Polymers

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Paints and lacquers

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