



Materials characterisation and equipment

January 2019

Equipment Overview

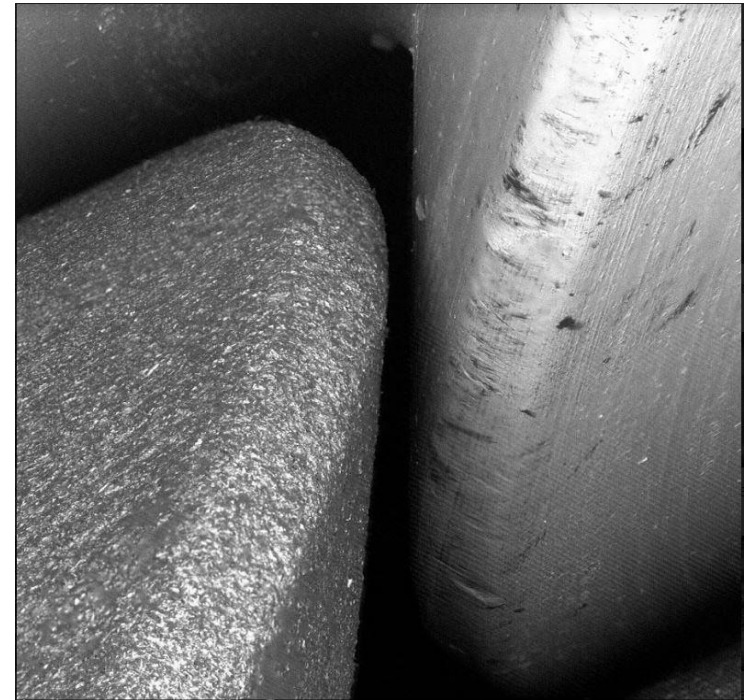
The following table gives an overview of the tests and equipment available to the customer. Also available is metallographic specimen preparation equipment. This and each of the sections in the table will be described in more detail throughout the document.

Visual	Powder	Chemical	Mechanical	Environmental
Scanning electron microscope	Particle size distribution	Surface energy / tension measurement	Tensile tester	Humidity ageing
Optical profilometry	Moisture analysis and precision weighing	Energy dispersion X-ray analysis	Stud adhesion tester and pin-on-disc tester	Salt-fog exposure testing
Optical microscopes	Pycnometry: density/porosity	Fourier transform infrared spectroscopy	Microhardness tester	Electrical properties
		UV-Vis-NIR spectroscopy		

Visual: Scanning Electron Microscope

Phenom XL SEM

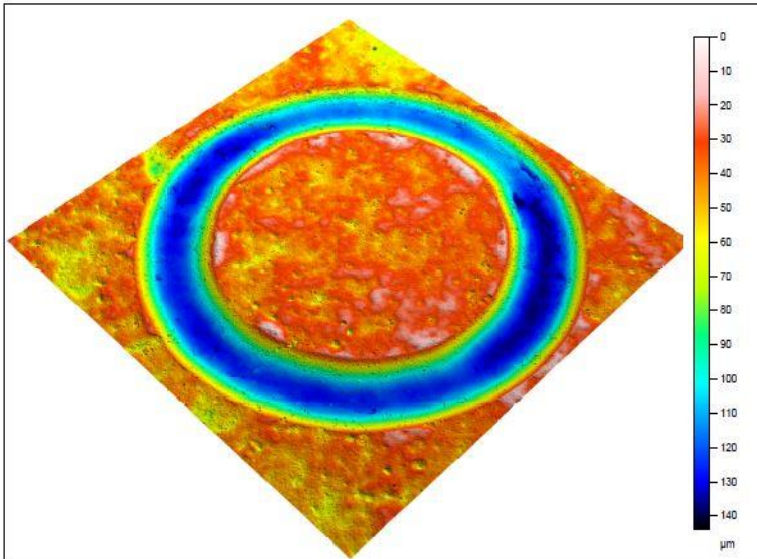
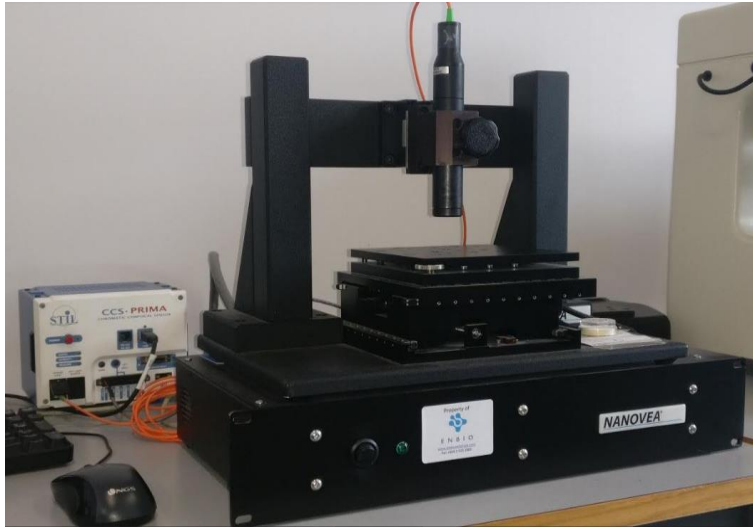
- A small yet powerful table top device capable of 100,000x magnification
- A large sample stage can cater for a single sample of up to 100 mm x 100 mm or multiple smaller samples
- Stage is height adjustable to a maximum of 65 mm
- A live camera allows for accurate tracking of position on a sample at all times
- Fast pumping and venting of analysis chamber increases sample throughput
- Default 5, 10 and 15 kV accelerating voltages and low (charge reduction mode), medium and high vacuum settings
- The system can also perform powder analysis and 2D and 3D roughness scans



SEM image of a coated (l) and uncoated (r) bolt thread at 250x magnification

Visual: Optical Profilometry

Nanovea PS50 Optical profilometer



Area scan generated by the Mountains™ software.

- Unlike stylus profilometry, there is nothing other than a beam of light in contact with the surface of the sample meaning no damage is done to the sample
- 2 optical pens are available – 300 μm for smoother surfaces and 3 mm for rougher surfaces
- The pens can analyse both highly reflective and dull surfaces or a combination of both
- The stage X and Y axes are motorised with 50mm of travel, the manual Z axis has 30 mm of travel
- Curved samples (up to 28°) can be measured
- Several templates are available based on Mountains™ software which perform any post processing required and report all the common roughness parameters including Ra, Rq, Rz and Rpc in .pdf format
- Line and area scan data can also be saved in .txt format for further processing

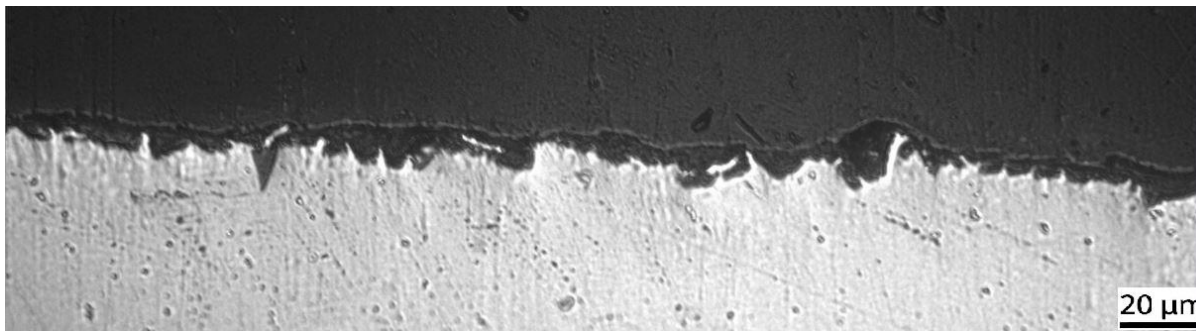
Reference standards: Rank Taylor Hobson® 0.035 μm , 0.35 μm , 0.38 μm , 0.39 μm , 0.82 μm , 0.87 μm , 2.11 μm , 2.36 μm and 2.57 μm calibration standards.

Visual: Inverted Microscope

Optical microscopes Leica DM IRB inverted microscope



- Often used in conjunction with the metallographic equipment to analyse surfaces, coating thickness layers etc
- Adjustable light intensity to cater for different material types
- Full x and y axis control through adjusting levers
- Several lenses available including 4x, 5x, 10x, 20x, 50x and 100x magnification
- There are a wide selection of adjustment options available to the user to improve the displayed image
- The Leica software records the onscreen image in various image formats
- These can then be imported into an image stacking software package to produce a clearer, better final image



Cross section of CoBlast treated metal surface indicating coated layer and texture. Image obtained using the Leica DM IRB microscope and stacked in Helicon focus.

Visual: Light Microscope

Optical microscopes

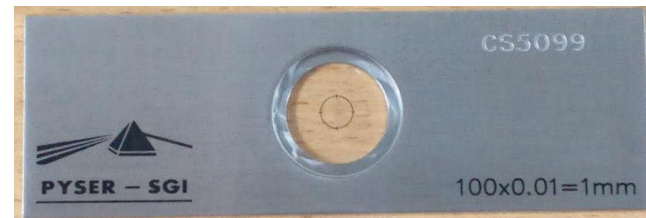
Brunel stereo inspection microscope



- Used to examine surfaces at between 10x and 40x magnification
- A light ring with adjustable light intensity can be used to provide illumination on the surface
- A camera mounted on top of the microscope transfers the image seen in the eyepiece to the monitor
- The software contains a wide range of adjustment options to provide an enhanced image to the user
- Images are saved in many of the regular image formats and if required, can be imported into an image stacking software package to improve image clarity

Both the inverted and light microscopes are calibrated using reference standard:

Pyser-SGI® Ltd 1 mm graticule in 100 x 10 µm divisions



Powder Analysis: Particle Size Distribution

Horiba LA-920 laser scattering particle size distribution analyser

- The Horiba relies on light diffraction of grains to measure the particle size distribution (PSD)
- Background measurements are gathered before readings
- Particle size limitations are between 0.02 to 2,000 μm , and the measurement time is about 20 seconds although this is adjustable
- Stirring and ultrasonic agitation are used to help with agglomeration breakdown
- DI water or other suspending fluids are acceptable and the measured background baseline eliminates error from the carrier suspending fluid
- 0.1 mg to 5 g of powder is required. The amount varies depending on the sample material
- Powder can be placed in a solution to assist with the dispersion of material that tends to adhere
- Data is saved in .txt format to allow for further processing and use with many software packages.



Powder Analysis: Moisture Analysis and Precision Weighing

AZI Computrac 4000XL Moisture analyser



- Samples can be in either liquid or powder form
- The device contains a small 700W heater to dry samples
- A precision balance weighing mechanism with a resolution of 0.0001g weighs the sample
- The user has the ability to set up to 250 programs using various parameters that can all be set using the keypad on the front panel
- The device heats a sample (0.2 g to 39.9 g) from a standby temperature of 25°C to a maximum of 275°C and records the mass loss of the sample throughout the program
- Moisture content and program duration are displayed on completion of the program

Calibration: Calibrated using standards whose accuracy is traceable to the National Institute of Standards and Technology (NIST).

Ohaus Discovery semi micro and Analytical balance



- Can measure powder and other materials up to a maximum of 210 g accurately to five decimal places
- Applications include
 - statistics mode
 - density mode
 - pipette calibration mode
 - percent weighing mode
 - parts counting mode
 - dynamic weighing mode
 - totalisation and high point modes
- Adjustable environmental settings to compensate for vibrations and other disturbances
- Self calibration: the device features Advanced AutoCal™ Automatic internal calibration

Image credit: Ohaus Corporation

Powder Analysis: Pycnometry – Density/Porosity

Micromeritics GeoPyc™ 1360 Envelope Density analyser

- Uses displacement of a quasi-fluid powder media to calculate envelope density of parts with irregular size and shape
- Syringes available include a 50.8 mm, a 25.4 mm and two 12.7 mm.
- These are capable of measuring powder tap densities using compression forces of 3 N – 180 N.
- This is equivalent to 1 N/cm² up to 140 N/cm²



Micromeritics AccuPyc™ 1330 Helium Pycnometer



- Precision measurement of absolute volume by gas displacement. Helium fills even the smallest surface features and pores for a better measurement of absolute volume
- By subtraction from the envelope density results of GeoPyc, the precise volume of these pores can be deduced
- Returns results based on the average of five or more consecutive runs

Chemical: Surface Energy/Tension Measurement

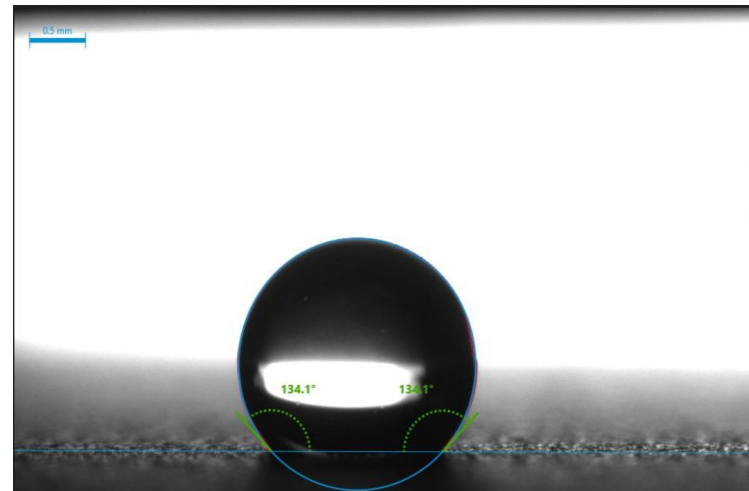
Krüss Mobile Surface Analyser



Image credit: KRÜSS GmbH

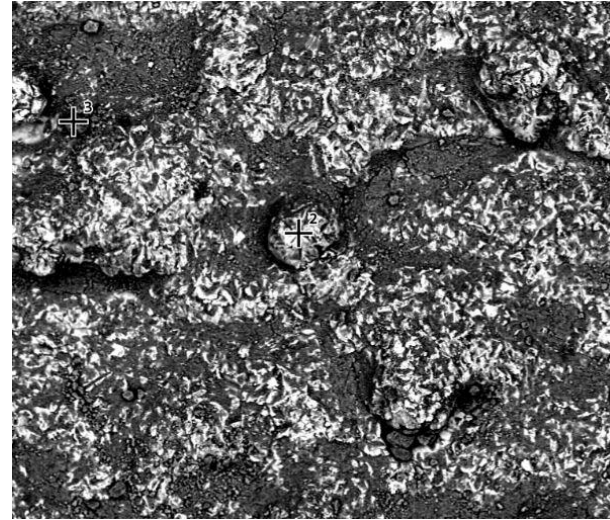
- Lightweight and portable device
- Uses a combination of liquids (usually water and diiodo-methane) to deposit two droplets onto the surface in specific amounts (usually 2, 1 or 0.5 μl)
- Liquid deposition amount will depend on the hydrophobicity of the surface
- The handheld device analyses the droplets and fits a contact angle line to either side of the droplet
- The user can reposition the angle lines should the software place them incorrectly
- The software derives the surface free energy (a combination of polar and disperse) from the contact angles and reports to the user in .xlsx and .pdf format

An image of a droplet on a surface recorded by the camera on the underside of the device

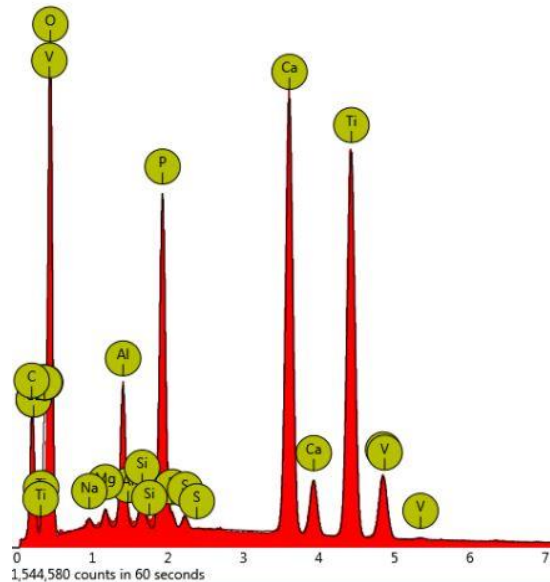


Chemical: Energy Dispersant X-ray Analysis (EDX)

- EDX is performed in conjunction with the SEM described in slide 3
- With the SEM set to 15 kV and in either point or map mode, the chemical composition of a region at the user's choice of magnification can be tested
- EDX duration is normally 60 seconds per point or map
- A line scan or an area scan can be performed through a region to display the change in chemical composition; these scans can take longer than 60 seconds depending on parameters chosen



An EDX map and 2 point scans (crosshairs centre and upper left) were performed on this surface



An elemental spectrum of the upper right image

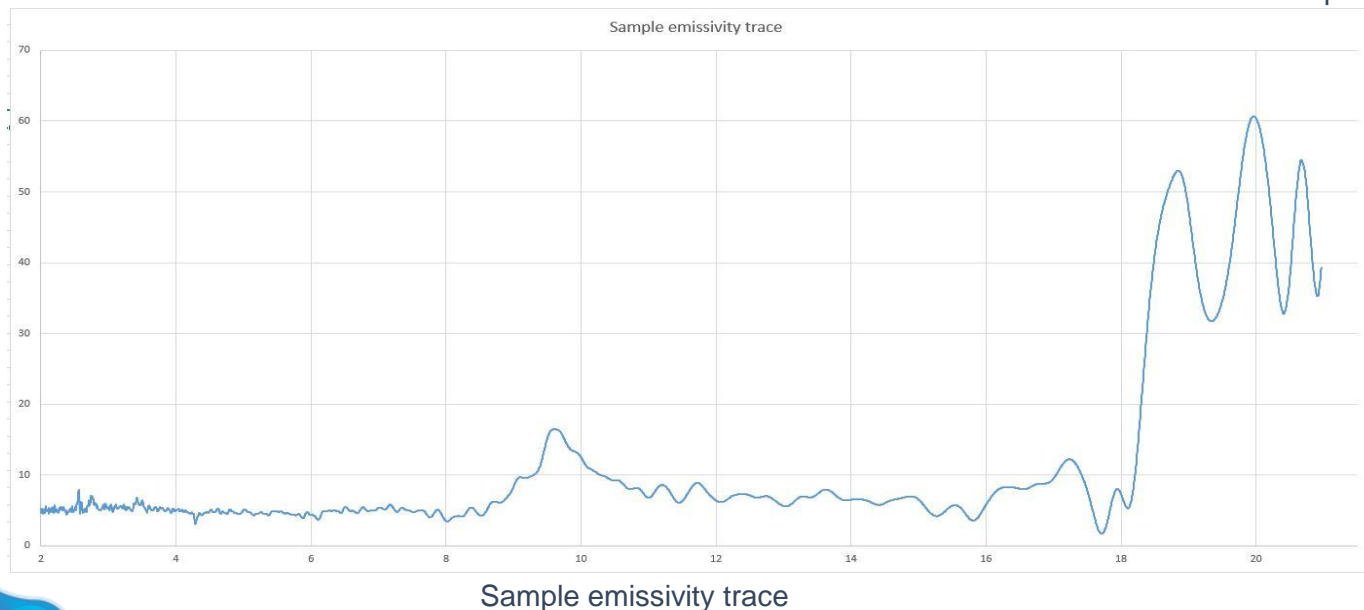
- Results are broken down by element, atomic concentration, weight concentration and stoichiometric weight concentration
- EDX can identify elements between Boron (Z = 5) and Americium (Z = 95)
- In a situation where the software incorrectly identifies a peak, the user can manually assign the element
- The software generates a report in pdf format of the results

Chemical: Fourier Transform Infrared (FTIR) Spectroscopy

Perkin Elmer Frontier MIR/FIR spectrophotometer



- A Pike LN2 cooled Mercury-Cadmium-Telluride (MCT) gold coated integrating sphere attachment is available for emissivity measurements (see image on the left)
- 3D printed specimen holders ensure correct sample placement at the base of the sphere
- An Attenuated Total Reflection (ATR) attachment is available which allows for direct examination of a solid, liquid or powder without further preparation
- A slide holder for transmission analysis using KBr slides is also available
- For emissivity measurements, samples should be at least 25 mm x 25 mm to avoid stray light entering the integrating sphere which could skew results
- Emissivity scanning range is normally 5000 cm^{-1} - 476 cm^{-1} (2000 nm – 21000 nm)
- Emissivity data is returned in .csv format can be combined with data from the UV-Vis-NIR to form a trace spanning 250 nm – 21000 nm



Reference standard:
Diffuse gold coated
reference coupon used to
baseline the system prior
to measurements being
taken.

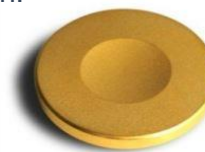
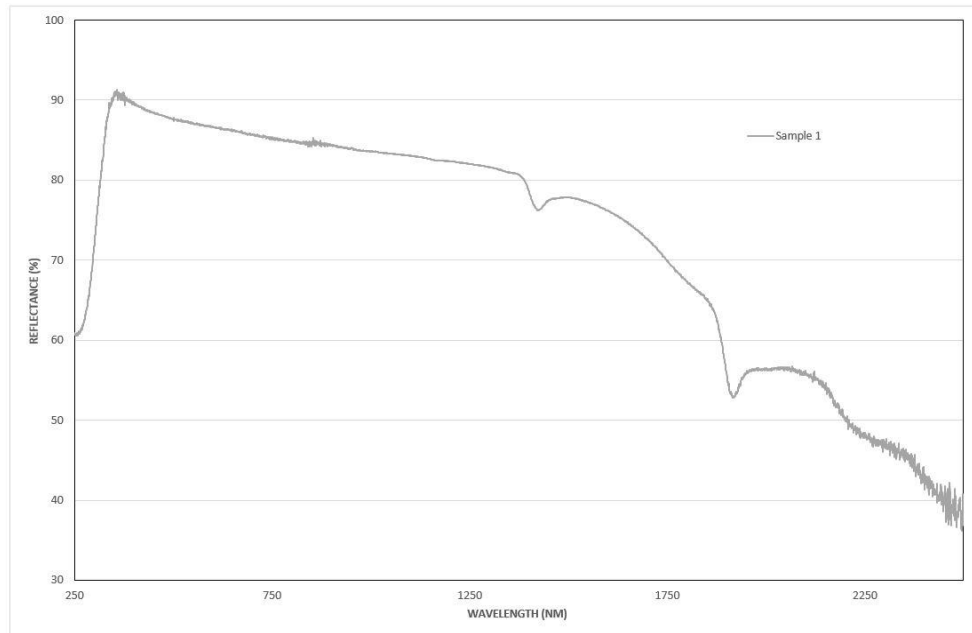


Image: Pike Technologies

Chemical: UV-Vis-NIR Spectroscopy

Perkin Elmer Lambda 900 spectrophotometer

- Double beam, double monochromator ratio recording optical system
- Spectroscopy can be performed across a wavelength range of 185 nm – 3300 nm
- A Deuterium lamp and a Tungsten Halogen lamp cover the UV and visible wavelength range respectively
- Flat samples of at least 25mm x 25mm and larger are required to eliminate stray light reaching the detector
- The device has a 150 mm Spectralon™ coated integrating sphere attachment for uniform scattering or diffusing of light
- Data is returned in .csv format to allow for further processing



Sample absorption trace

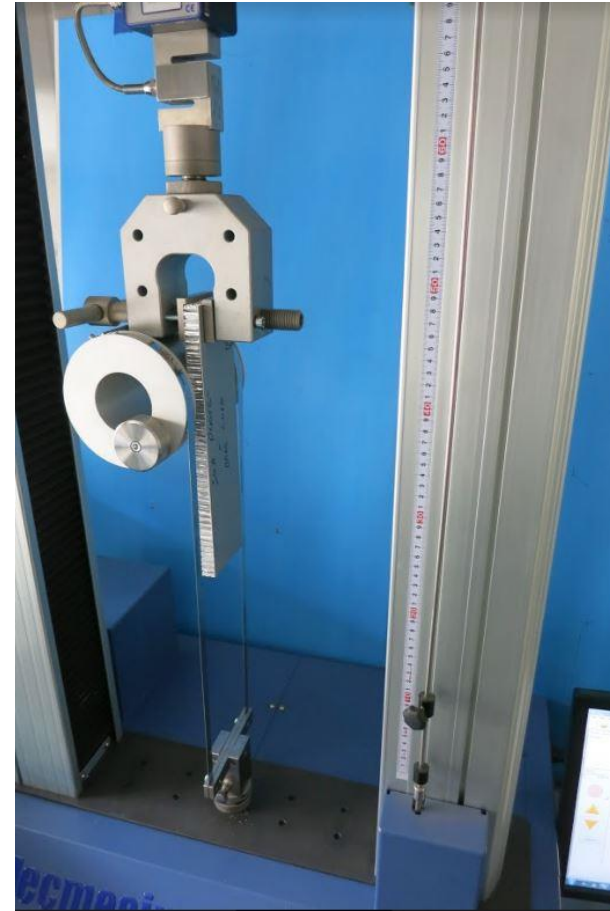
Reference standards:
2%, 50%, 75% and 99% reflective
Labsphere® certified
reference standards
(ISO 9001:2008).

Mecmesin MultiTest 50-i tensile tester



Image credit: Mecmesin Ltd

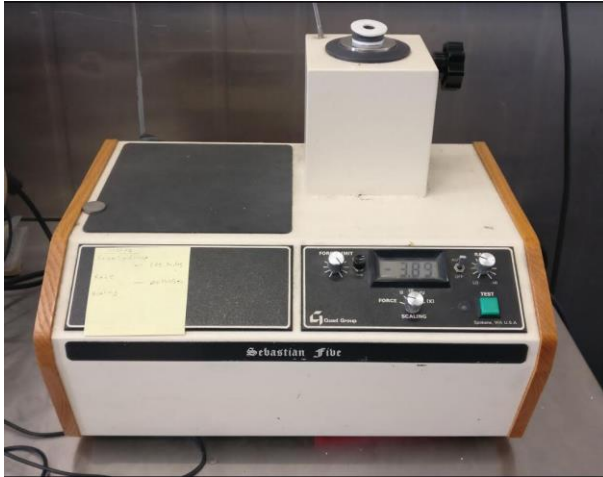
- A powerful 50 kN twin column tensile and compression system for force measurements
- System has a range of precise and accurate load cells available depending on the type of testing required
- These include 50 kN, 10 kN, 5 kN, 1 kN and 100 N load cells
- Software auto-detects the load cell and will run and auto-stop when a certain load is reached, an extension is achieved or when the sample breaks
- Accessories include a climbing drum peel tester per ASTM D 1781, for testing of adhesives and laminate assemblies
- A range of light and heavy-duty grips allows for the handling and testing of various metals and materials ranging from 25 or 50 μm thick foils, to Aluminium and Titanium coupons 1 mm thick and above



Climbing drum peel test rig attached to a 10kN load cell

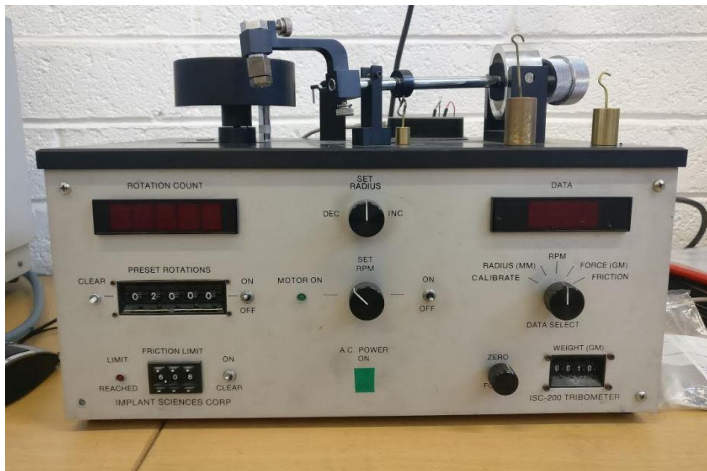
Mechanical: Stud Adhesion Tester and Pin-on-disc Wear Tester

Quad Group Sebastian Five pull tester



- This machine is used to test the force required to remove an epoxy (or other adhesive) coated stud from a surface
- Rated up to 76 N/mm² pull force
- The stud is placed in the test port at the top of the machine and is fixed in place using the black handle at the top right of the image
- The machine uses a grip locking mechanism to hold the stud in place while it pulls it down into the machine
- 3D printed washers ensure the stud is level and pulled perpendicular to the machine while the run is ongoing
- Pull force increases until separation is confirmed
- Digital display shows the changing force throughout the test

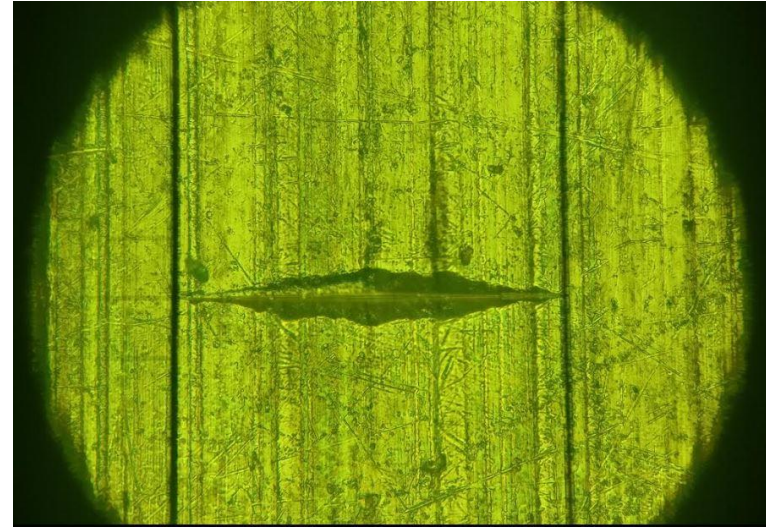
Implant Sciences model 200 pin on disc wear tester



- Measures the wear on a material's surface due to continuous friction between a ball bearing and the surface
- Rotation speed and force applied are set prior to the beginning of the run
- Rotation creates a wear groove in the surface
- This can then be analysed using a profilometer (see image on slide 4)
- The machine can be used to test the wear rate of a coating on a material's surface or the wear on orthopaedic devices, just two of many potential uses
- If required, the pin and sample can be submerged in fluid to change the friction or wear rate
- Results can be exported to a computer for further processing

Mechanical: Microhardness Tester

Buehler Micromet® 2100 series
microhardness tester



- Digital device which automatically calculates the hardness of a material
- It uses the Knoop hardness (HK) test corresponding to ASTM E384 – 17 : Standard Test Method for Micro-indentation Hardness of Materials
- The device uses a diamond indenter to indent the surface
- A 40x scanning objective allows for sharp focussing giving the user a clear view of the indentation
- The user adjusts the black vertical lines until they line up with the edges of the indent (see the image above of an indent in an Aluminium coupon)
- The user then presses 'Read' on the touchscreen. The instrument calculates and displays the HK result

Reference standards: Zwick Roell Indentec 30.3 HRC, 44.3 HRC, 46.4 HR30T, 48.5 HRB, 50.8 HR30N, 59.9 HRB, 60.1 HRC, 61.8 HR30T, 61.8 HRC, 63.7 HR30N, 76.3 HR30N and 91.9 HRB Hardness test blocks. Blocks were calibrated in the Indentec NAMAS calibration laboratory and meet the requirements of BS891 or 4175.

Binder KBF720 constant climate chamber

- Chamber operating conditions 0 °C to +70 °C, and humidity ranges from 10 to 90%rH
- Microprocessor controlled humidifying and dehumidifying systems
- Operates in manual or automatic mode
- Stores multiple programs (30+). Each program multiple cycle, ramp, dwell times, internal and external loops
- Internal Dimensions: 973 * 1250 * 576 mm
- Internal volume 700 l
- Stainless steel inner chamber, preheating chamber and outside doors
- Inner glass sealing doors



Image credit: Binder Inc.

Memmert HCP108 constant climate chamber

- Offers precisely controller atmospheres with a humidity working range of 20 to 90% rH
- Stainless Steel easily cleanable internal chamber
- Programmable profiles including rates times and dwells
- Chamber dimensions 560 * 480 * 400 mm
- Humidity chamber operates in humidity mode up to 90 °C, with the operating window range listed below:
 - @ 30 °C Humidity limits: 30 -95 %rH;
 - @ 40 °C Humidity limits: 20 -95 %rH
 - @ 55 °C Humidity limits: 20 -95 %rH
 - @ 90 °C Humidity limits: 30 -70 %rH
- Operates in non-humidity mode to 160 °C



Image credit: Memmert GmbH + Co. KG

Environmental: Salt-fog Exposure Testing

C+W 450L Cyclic corrosion chamber with extended humidity capabilities:



Image credit : C+W specialist equipment Ltd

- Salt spray - All Corporate, National and International Standards.
- Humidity - Constant or cyclic temperatures between ambient and 70°C with 98% Relative humidity.
- Dry heat - Dry Heat conditions typically ambient to 70°C
- Air drying - Hot and cold air introduction.
- Humidity - Cyclic temperature testing
- Dwell and ramp functions - variable humidity option 10% to 98% Rh, from ambient to 70°C.

Capable Standards include:

ASTM B117 (preprogrammed)
SAE J 2334 (preprogrammed)
DIN 50.021
BS 3900 F2

HONDA
NISSAN M0007 C.C.T. 1,11 & 1V
FORD EU B153-2
TOYOTA TSH 1555G
ROVER GROUP RES 30 CT 107
V.W CYCLING TEST
UNICHEM
GENERAL Motors GM 9540P

Other standards or custom testing programmable through Eurotherm controller interface.

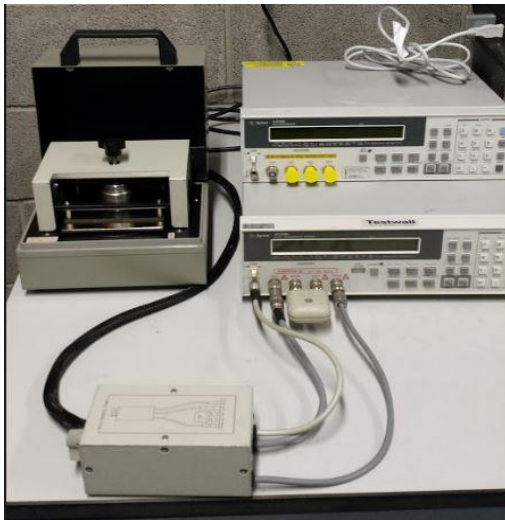
Sheet resistance: RR Mechatronics Sherescan 9000



Image credit : R&R mechatronics international BV

- Uses a Tungsten Carbide 4 point probe to determine the type of base material (P or N type)
- X, Y and Z traverse axes
- Vacuum to hold a sample in position
- Data exported in .csv format for ease of processing

Concentric ring resistance meter (HP4339B + HP16008B)



- The 4339B resistance meter measures very high resistance of insulation materials
- Measurement parameters are
 - Resistance (R)
 - Volume resistivity (QV)
 - Surface resistivity (QS)
 - Current (I)
- R measurement range is $10^3 - 1.6 \times 10^{16} \Omega$
- Test voltage ranges from 0.1 – 1000 V dc
- The 16008B resistivity cell is designed for safe and stable measurements of materials and components
- Surface resistivity (ρ_S) and volume resistivity (ρ_V) are calculated and are shown on the device's display

Metallographic Sample Preparation

Cut off wheel



- Performs precise and clean sectioning of samples in preparation for mounting in resin
- Silicon Carbide and Aluminium Oxide blades are available to section non-ferrous and ferrous materials respectively
- A non-corrosive coolant can be used to protect the samples and the blade if required
- Adjustable blade RPM and jog wheel speed allow for the sectioning of materials of various thicknesses

Buehler Phoenix 4000 automatic grinder/polisher



- Can operate in single or central force application mode
- The single force specimen holder can cater for up to six 50 mm samples. The central force specimen holder can cater for up to six 30 mm samples
- Adjustable head pressure depending on the hardness of the material being grinded and/or polished
- 250 mm diameter platen
- 150 and 300 platen rpm speeds available
- SiC grinding papers available including P80, P120, P240, P320, P600, P1200 and P2500 can grind the roughest up to the smoothest surfaces (the lower the grade, the rougher the paper)
- 3 μm and sub μm polishing pads when used with diamond suspension and colloidal silica respectively result in a mirror finish on metal surfaces