

## **MATERIALS SCIENCE AND TECHNOLOGY GROUP**

Lead by Prof. FABIO CARASSITI and Prof. EDOARDO BEMPORAD

### **Research activities carried out by the Materials Science and Technology group, University of Rome "Roma Tre"**

The Materials Science and Technology group (STM), is composed by five staff unities (two full tenured professors, three researchers and one tenured technician) and a team, financed mainly through research projects, composed of about 6 unities with the following profiles: two fellowship researchers, three doctoral students and one person with technical-administrative role; the staff comprises also other master graduate in Mechanical Engineering involved in training activities concerned with research projects supported by industrial enterprises and undergraduate students in mechanical engineering which are carrying out their final master thesis work.

Research activities can be summarized in two principal themes:

- Study of surface properties of systems for advanced mechanical engineering and micro-devices applications, through numerical and analytical planning, advanced characterization methods, technological, mechanical, tribological and chemical tests.
- Development of advanced multi-scale methodology related with morphological, micro-structural and micro-analytical characterization of materials through optical, ionic, electronic and contact probes.

The aims of the two themes are:

- Development of techniques for application of the nano-microstructure-process-property-performance correlations to the control and the optimization of materials processing especially for the development of anti-wear and corrosion resistant coatings.
- Development of integrated methodologies for nano and micro structural characterization of materials and analysis of the nanostructure-microstructure-process-property-performance correlations, aimed to the prediction of the materials behavior in real-use conditions, of reliability and of degradation (failure analysis).

The group carries out its research activities in three structures, the Interdepartmental Laboratory of Electronic Microscopy (LIME), constituted by the Engineering Department (DIMI) and Departments of Biology, Applied Electronic, Physics, Geology; the Materials Technology laboratory (MATEC) that constitutes the Technology and Materials Tests section of the

experimental laboratory of DIMI and the Data and Image Processing laboratory (MATEDP), located in the "Calculation for the Research" area of the DIMI.

#### The LIME

It was qualified from the National Inter University Consortium as referring centre: "Laboratory of Superficial Treatment Engineering"; it extends on a total surface of about 330 m<sup>2</sup> and it is coordinated by the STM group. It hosts devices assigned to the group members (completely or in part) mainly dedicated to the materials characterization.

#### The MATEC

(50 m<sup>2</sup>) hosts the devices for the technological and mechanical surface tests. In the new area recently assigned to the group a device/structure for the production of thin coatings by the PVD techniques is scheduled to be realized.

#### The MADEP

(30mq) hosts a laboratory for the thin nano-structured film deposition (PVD) and a prototypal instrument for tribological tests.

#### The MatEDP

(25 m<sup>2</sup>) hosts the calculation's station (continuously updated) and the servers.

### **Main International Scientific Cooperations (2005-2012)**

- University of Tennessee (UTK), Department of Materials Science, prof. George Pharr
- University of Oxford Department of Engineering Science, prof. Alexander Korsunsky;
- Karlsruhe Institute of Technology (KIT), Institut für Zuverlässigkeit von Bauteilen und Systemen (IZBS) Karlsruhe, (Germany), Dr. Christoph Eberl
- Central University of Venezuela, School of Metallurgy and Materials Science, prof. Mariana Staia and Eli Saúl Puchi-Cabrera
- Lille University Laboratoire de Mécanique, prof. Jacky Lesage and Didier Chicot
- Karlsruhe Forschungszentrum, Dr. Wilhelm Pfleging
- University of Rosario (Argentina), prof. J. N. Feugeas
- SIOMECA (Saxonian Institute of Surface Mechanics) and Technische Universität Chemnitz, dr. Norbert Schwarzer

### Most relevant projects in the last four years

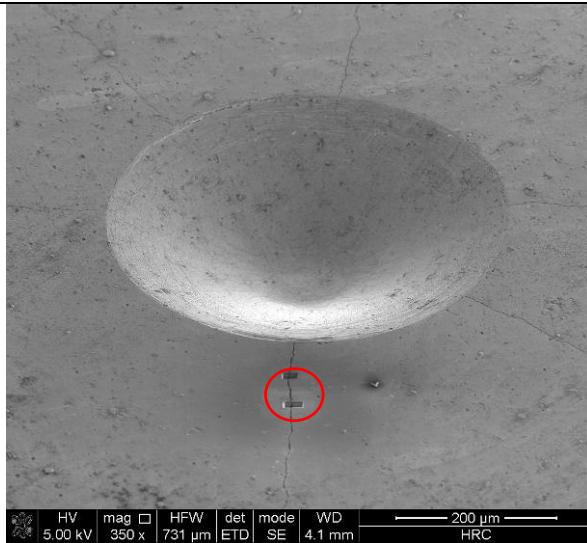
- FIRB-Italnnotet: National Research Network on Nanosciences, regional hub, gov funds, 150k€
- PRIN – New materials for made in Italy products, gov funds, 150k€
- MSE-ENEA, New materials for electrical power conservation for transport engineering, gov funds, 50 k€
- MSE-CNR, Innovative coatings for energetic applications, gov. funds, 50k€
- CIRA, Innovative anti-icing surfaces, private company, private funds, 30k€
- PVD coatings on polymer substrate, private company, regional funds, 100k€
- Advanced Characterization of tribological system, private company, regional funds, 100k€
- Development of an innovative techniques for abrasive wear rate evaluation of bulk materials and coatings, private/public consortium, private and UE funds, 140k€
- FEM analysis and optimization of mechanical components for food, private company, gov funds, 28k€
- Mechanical and sub-microstructural characterization of MEMS, private company, private funds, 350k€
- Development of multiscale integrated technologies for material characterization, gov funds, 50k€
- Study on innovative NiAl-based bondcoats for turbine's blades, private/public consortium, gov funds, 160k€
- Modelling, production and mechanical and morphological of Zirconia ceramic foams, private/public consortium, gov funds, 22.5k€
- Corrosion analysis for stainless HT piping, private/public consortium, gov funds, 350k€
- Characterization of nanostructured systems for biotechnology, private company, regional funds, 50k€
- Development of an innovative techniques for abrasive wear rate evaluation of bulk materials and coatings, private company, regional funds, 140k€
- Mechanical and microstructural characterization of wear and corrosion resistant coatings, private company, regional funds, 60k€
- Development of nanostructured Al/Ti alloys for aerospace applications, private/public consortium, gov funds, 100k€.

**Investigation capabilities, some examples**

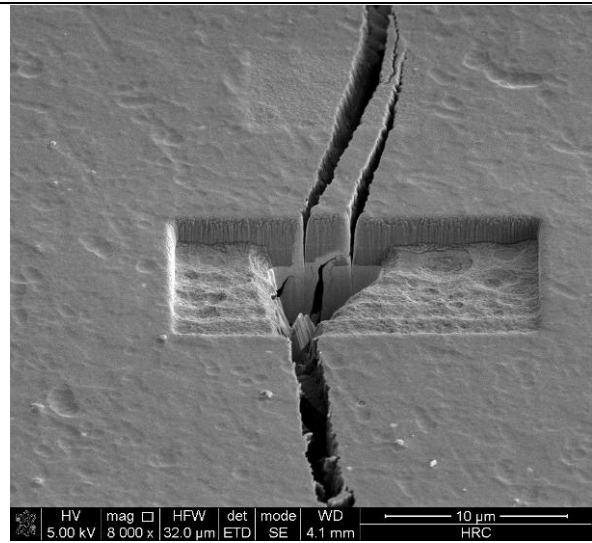
**FEG-FIB Micrographs - FEI Dual beam Helios Nanolab:**



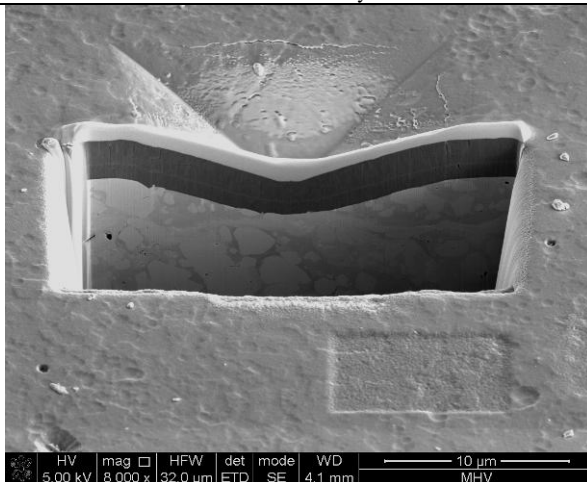
What puts the Helios NanoLab in a class of its own is its ability to offer the highest imaging, contrast, stability and speed performance together with the largest range of SEM / FIB applications. It ensures best resolution, reproducible metrology and best control of the beam for writing purposes. The outstanding imaging capabilities of the Helios NanoLab start with its novel FESEM technology, featuring resolution in the sub-nanometer at 15kV and better than 1.5nm at 1kV without beam deceleration. Stunning image quality and contrast are achieved, especially when using the new Helios NanoLab through-the-lens detector. Its innovative design allows for superb imaging in SE and BSE modes over the entire energy range. While pushing the limits of 2D and 3D nanocharacterization, through integrated Slice and View™ tomography or FEI's automated 3D EBSD collection package called EBS3™, Helios NanoLab also delivers the most advanced integrated solutions for nanoprototyping. Its 16-bit digital patterning engine teams up with FEI-developed FIB milling protocols, which are readily available from the user-friendly software interface. A wide range of patterning strategies are available to optimize electron and ion beam writing and processes; including FEI's proprietary gas injection processes to deposit the largest number of different materials in 3D. For electron beam lithography, Helios Helios NanoLab excels in preparing the highest quality samples. Using FEI's AutoTEM™ G<sup>2</sup> software, thin samples can be prepared rapidly and automatically, with high reliability and extreme ease of use.



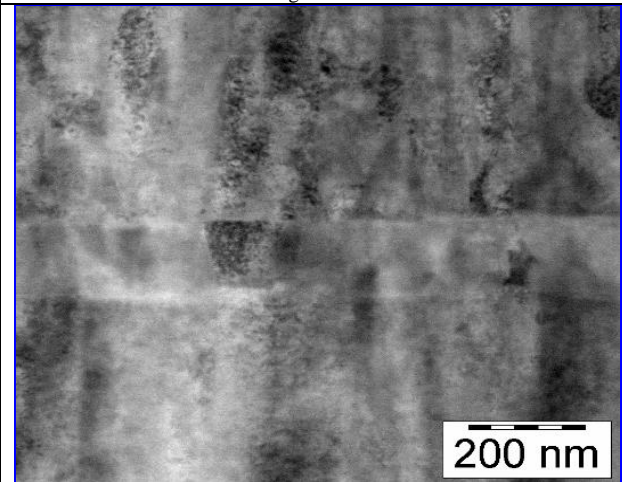
Cross section on crack created by HRC indentation



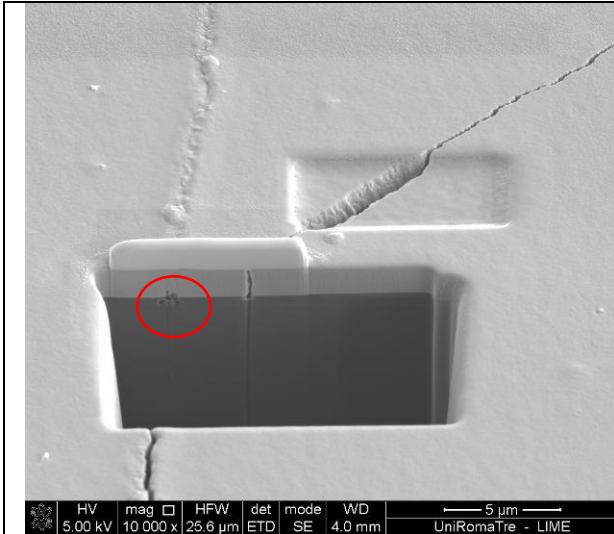
Zoom of image shown in the left



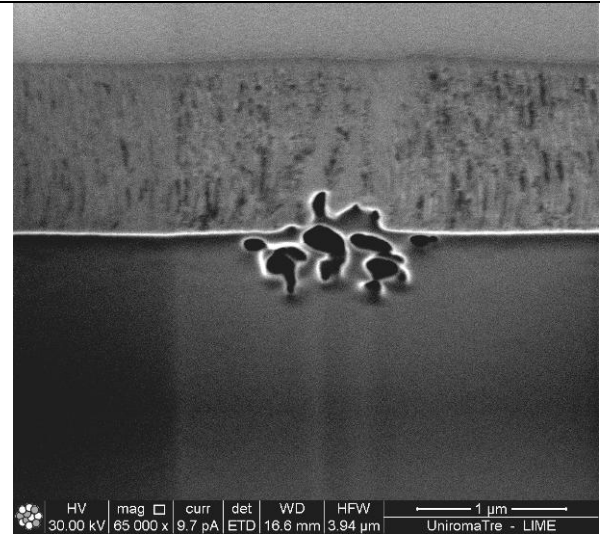
Cross section on micro indentation MHV



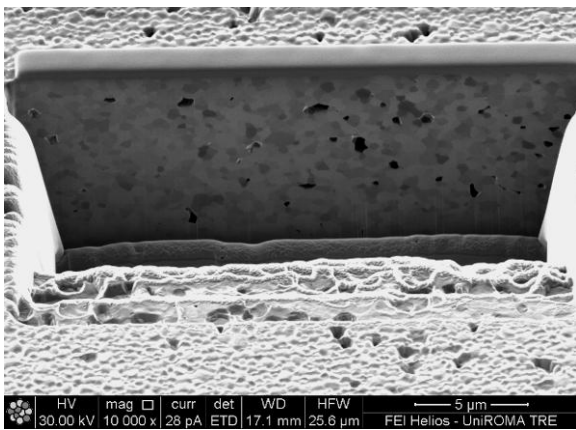
in-situ STEM, after FIB thinning, 30kV 250,000X



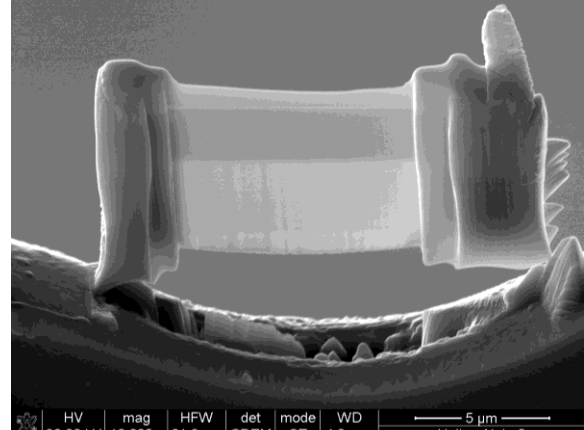
Failure analysis of Chrome coating on polymeric substrate



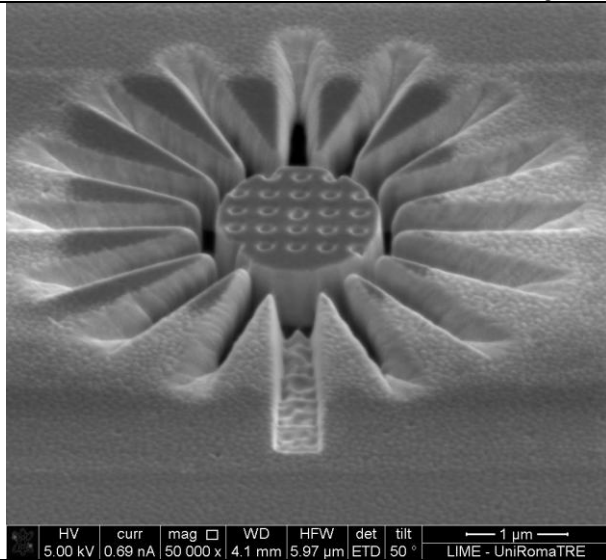
Zoom of defect shown in the left image



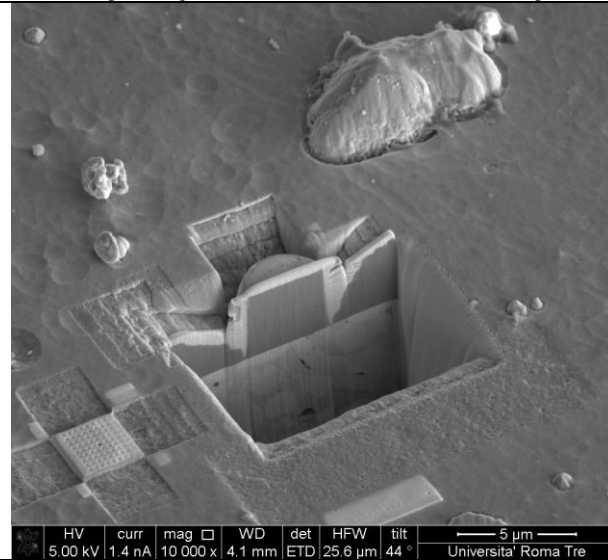
Cross section of ceramic foam in Zirconia (ion source image)



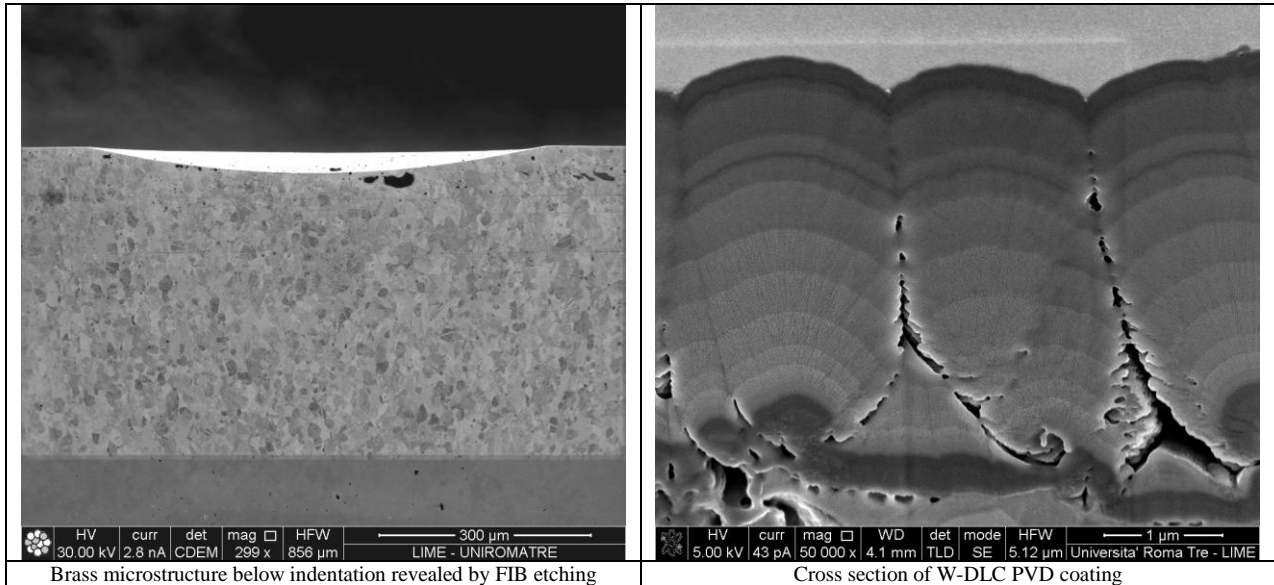
Final polishing of a TEM lamella (in situ lift out technique)



Residual stress analysis on a suspended membrane (MEMS) by focused ion beam (FIB) ring-core milling



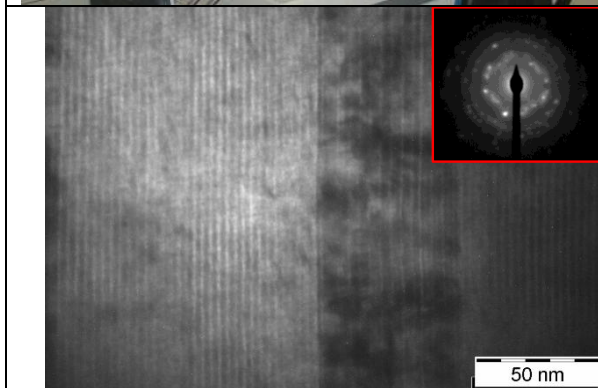
FIB section of a micro-pillar



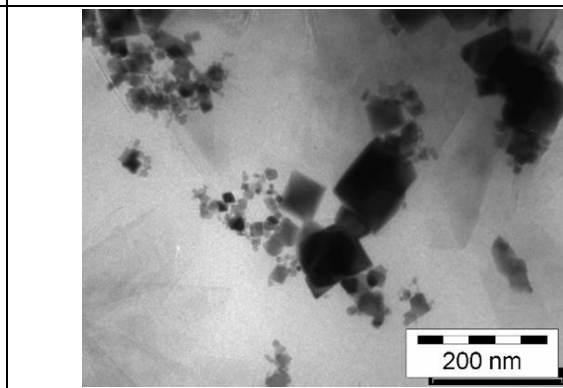
### TEM Micrographs - Philips CM120 (LaB6 analytical):



FEI CM 120 EDS Edax DX-4  
 120kV, analytical with LaB6 filament, sample holder with 5 axes motorized. Linear resolution of 0.2nm.  
 EDS probe with range of detectable elements since boron, resolution MnKa 134eV, digital acquisition of rX compositional profile and maps.  
 Software for quantitative-semiquantitative analysis using ZAF and PhiRhoZ models.  
 TEM CCD:  
 Effective pixels [pixel] 1392 x 1040  
 Digitization [bit] 12  
 Frame rate [fps] at full resolution > 10  
 Binning 2x, 4x and 8x  
 PC interface FireWire (IEEE 1394)



Multilayer coatings TiAlN-TiN, 120kV, 660.000x

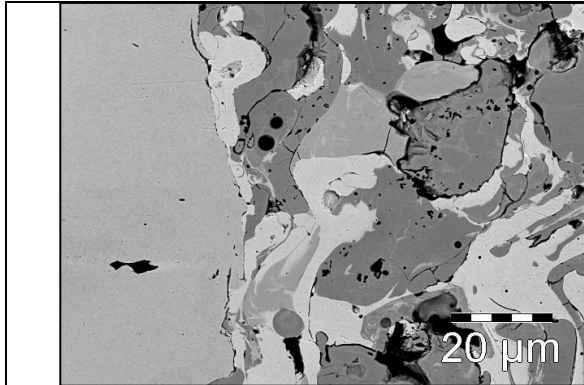


ITO powder, 120kV, 110.000x

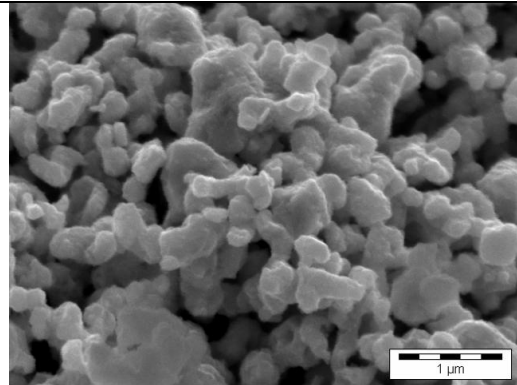
<p>Plasma spray coatings ZrB<sub>2</sub>-SiC (eutectic) 120kV, 110.000x</p>	<p>Electron diffraction (SAD) of eutectic, 120kV, L=1100mm</p>
<p>yttrium Nanopowder , the SAD rings evidenced, correspond to crystallographic planes (10 2 0); (6 5 3); (6 4 2); 120kV, 660.000x</p>	<p>Ceria Nanopowder, 120kV, 380.000x</p>

### SEM Micrographs - Philips XL30 (LaB6 analytical):

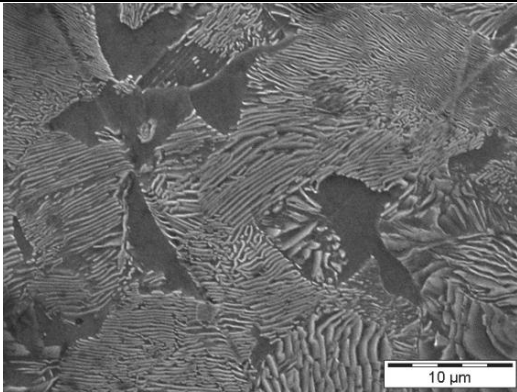
	<p>SEM stands for scanning electron microscope. The SEM is a microscope that uses electrons instead of light to form an image. The scanning electron microscope has many advantages over traditional microscopes. The SEM has a large depth of field, which allows more of a specimen to be in focus at one time. The SEM also has much higher resolution, so closely spaced specimens can be magnified at much higher levels. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, which focus the beam down toward the sample. Once the beam hits the sample, electrons and X-rays are ejected from the sample. Detectors collect these X-rays, backscattered electrons, and secondary electrons and convert them into a signal that is sent to the screen.</p>
<p>PVD Ti-TiN coatings a 9 layer, BSE, 25kV, 5000x</p>	<p>PVD CrN-Cr-CrN coatings, BSE, 15kV, 15000x</p>



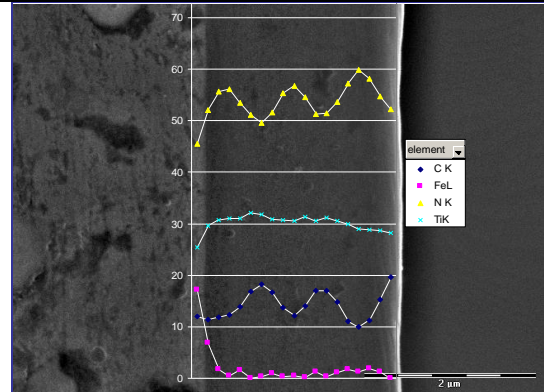
Plasma spray  $\text{Cr}_3\text{C}_2\text{-NiCr}$ : BSE, 15kV, 1500x



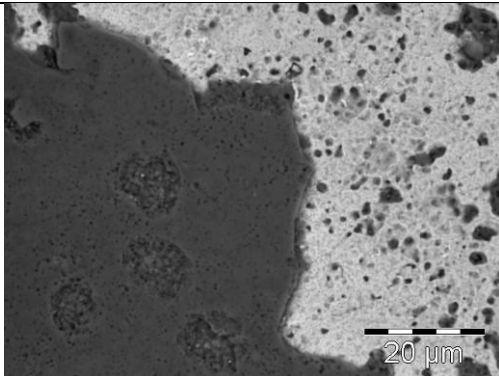
Nanostructured powder  $\text{Al}_2\text{O}_3\text{-TiO}_2$ , CS, SE, 15kV, 50000x



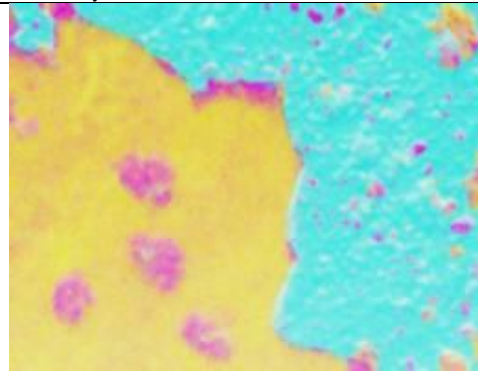
Microstructure of spring steel Si, SE, 25kV, 3000x



Semiquantitative EDS analysis on -cathodic arc PVD coatings of 6 layer TiN-TiCN shown in cross section



Film of aluminum e magnesium phosphate ( $\text{Al}_2\text{H}_2\text{PO}_4$ ) on Si steel



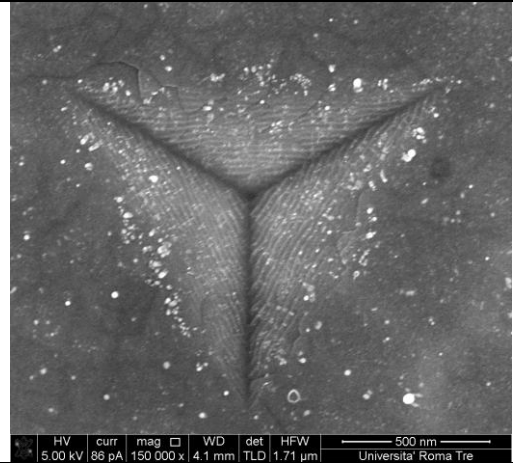
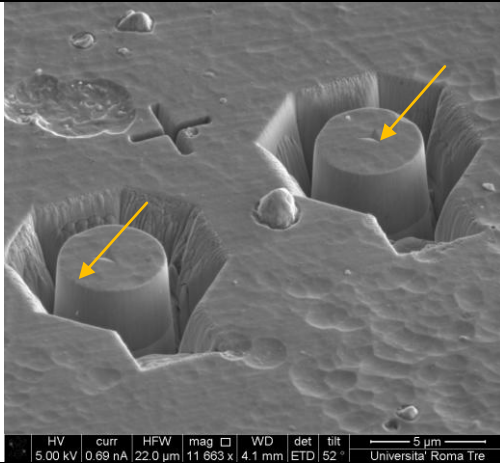
EDS map of figure shown at left of: Fe, Mg, P



## Nano Indenter® Agilent G200 (equipped with Continuous Stiffness Measurement module, nano-positioning stage and lateral force measurement)

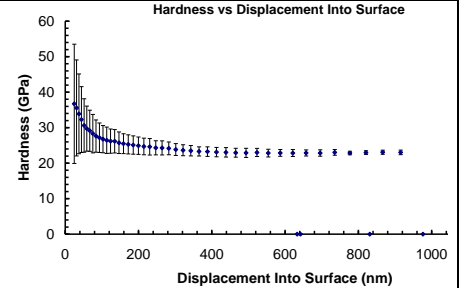
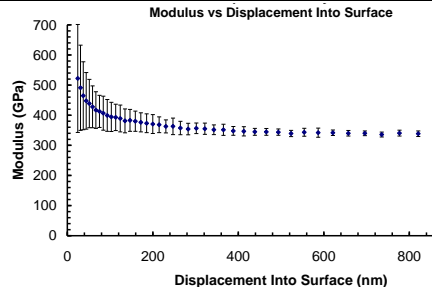
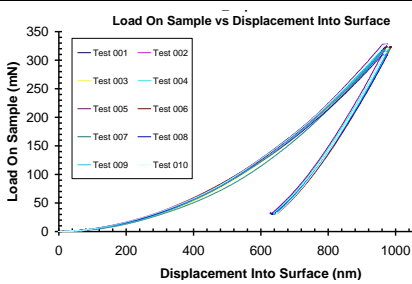


Nano Indenter G200 system represents the market's most advanced platform for exploring small-scale material behavior. While the Nano Indenter G200 system is a flagship instrument for performing nanoindentation experiments, its capabilities extend to other modes of testing; such as mechanical probing, scratch testing and nanomechanical microscopy. With a Nano Indenter G200 system you can perform a variety of different tests; all with unparalleled levels of control. All measurements made in instrumented indentation testing are derived from the fundamental force and displacement data. Incorporating electromagnetic actuation-based force transducers, the Nano Indenter G200 system offers outstanding precision in force application. Current passing through the coil drives the indenter shaft downward while a capacitance gauge measures displacement. Dual leaf springs, separate from the capacitance gauge, hold the indenter column stable and eliminate the possibility of lateral excursions. When configured with two force transducers and a high load device, the Nano Indenter G200 system is capable of applying forces that range from a few microNewtons up to 500 mN. The Nano Indenter G200 system may also be used in either quasistatic or dynamic mode. Quasi-static mode calculates properties at the maximum penetration depth, delivering a single value for stiffness. In dynamic mode, the patented Continuous Stiffness Measurement (CSM) technique is applied to record stiffness data along with load and displacement data as a continuous function of depth. With the CSM technique, hardness and Young's modulus may be calculated at every data point acquired during the experiment. Such capabilities offer valuable information for test samples such as thin films, coatings and other surface treatments. Nano Indenter systems from Agilent are the only testing platforms to offer a patented technique for dynamic testing at this scale, and they make the process easy. Users need to make only two decisions: where to place the tests and what experiments to perform at those positions.



Residual stress measurement by nanoindentation on FIB prepared micropillars

SEM FEG 5kV BSE 150000X, Berkovich Indentation on substrate

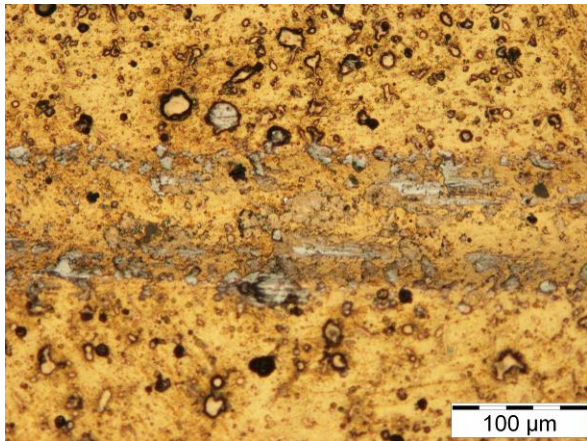


The graphs describe the curve load-unload of indenter, Young modulus and the hardness vs penetration depth

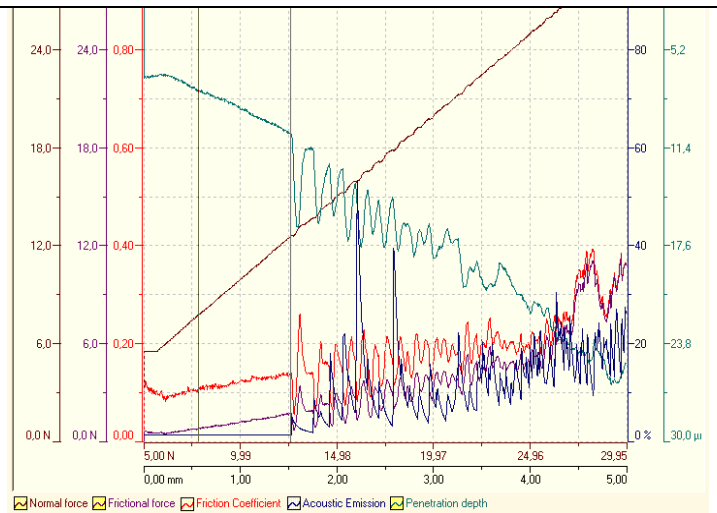
## Scratch test (CSM Revetest)



The REVETEST® Scratch Testing instrument is globally regarded as the ideal system for characterizing hard-coated materials, with a typical coating thickness of various microns. Coatings may be organic or inorganic, covering Tribological, magnetic and decorative applications, such as PVD, CVD, PECVD, metallization and passivation layers, wear and friction protective coatings. Substrates comprise metals, semiconductors, alloys, minerals, glass, refractive and organic materials. CSM Instruments is the world leader in Scratch testing as there are, as a matter of fact, more than 1000 Revetests sold worldwide.



Optic 200x, delamination due to scratch test



Performance of frictional force, Acoustic emission, penetration depth vs normal force applied

## Confocal/interferometric 3D profilometer (LEICA DCM 3D)



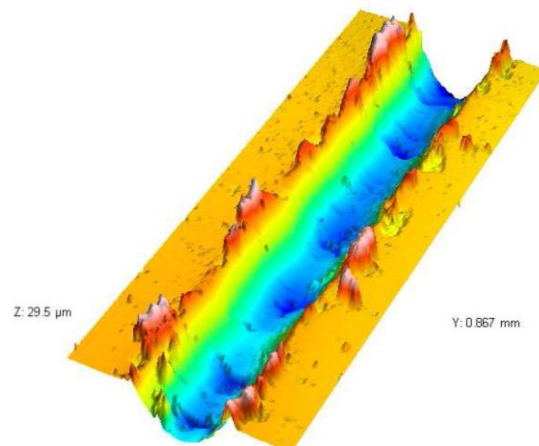
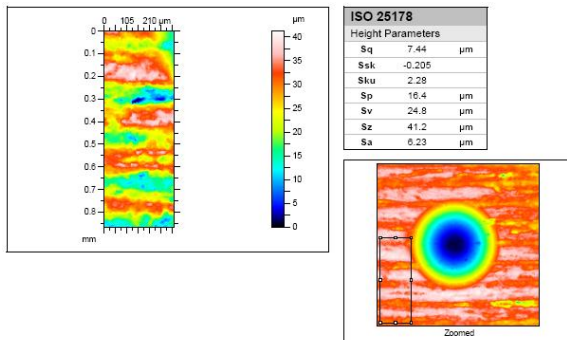
Micro optical measurement technology fulfils two important requirements of metrology: non-destructive measurement combined with high accuracy. The measuring capabilities of the Leica DCM 3D range from a few nanometers to several millimeters and therefore serving a wide variety of different applications. Besides the capabilities of adapting to the requirements of the application from super smooth to very rough surfaces, the Leica DCM 3D is specifically designed to carry out measurements at extremely high speed. This does not only save valuable time, but also significantly improves the return of investment. The integrated technologies of the Leica DCM 3D overcome the physical limits of conventional Profiling systems. With a single system it is possible to analyze rough (confocal) as well as smooth (Vertical Scanning Interferometry or VSI) and super smooth (Phase Shift Interferometry or PSI) surfaces. Sub-micron lateral resolution and a vertical resolution in the nm range is obtained in confocal mode, while large fields of view in combination with sub-nanometer Z resolution are acquired in the Interferometry mode.

3 Systems in one:

Brightfield and darkfield color digital microscope;

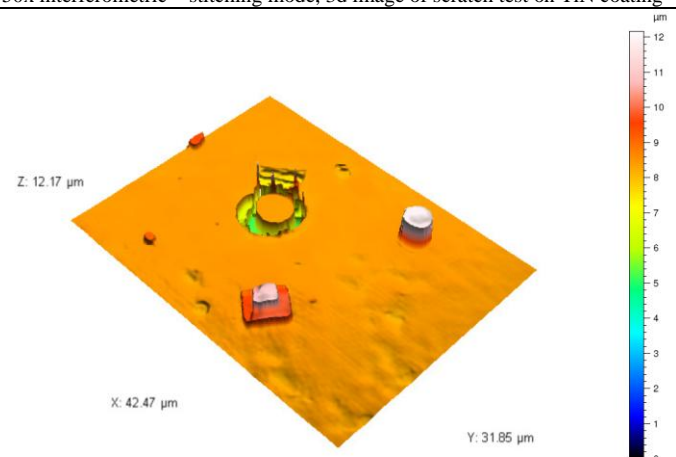
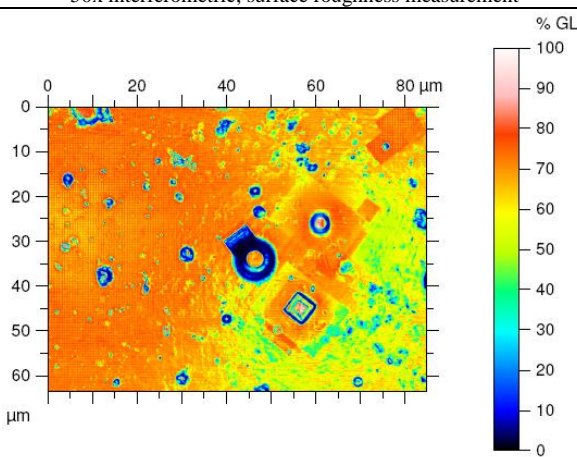
High Resolution Confocal imaging and measuring system;

Dual Optical Interferometric Profiler



50x interferometric, surface roughness measurement


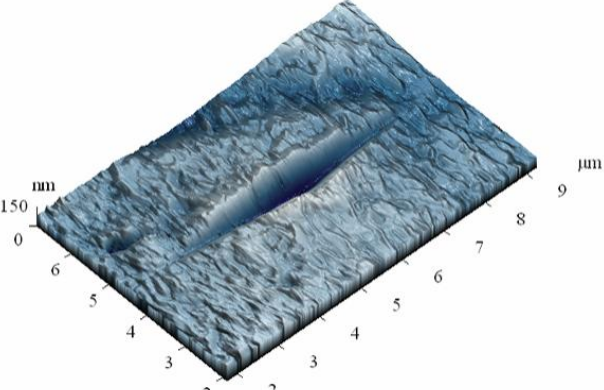
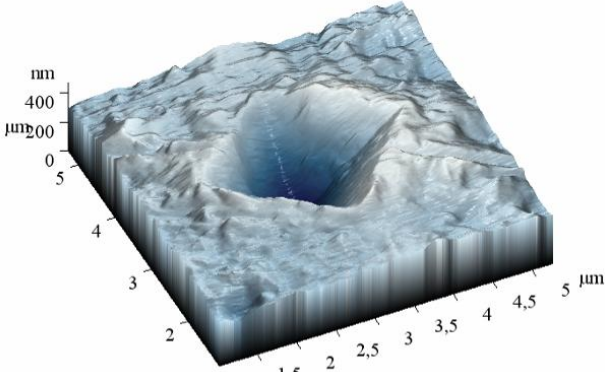
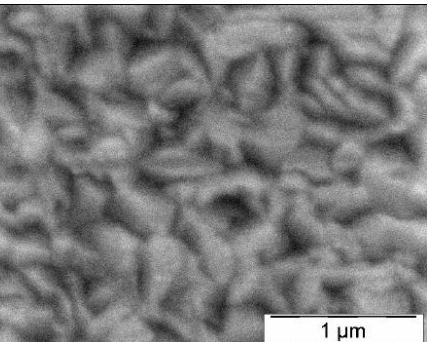
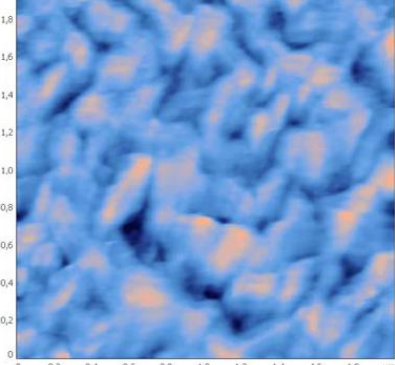
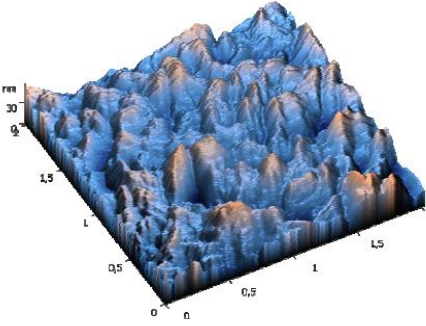
50x interferometric – stitching mode, 3d image of scratch test on TiN coating



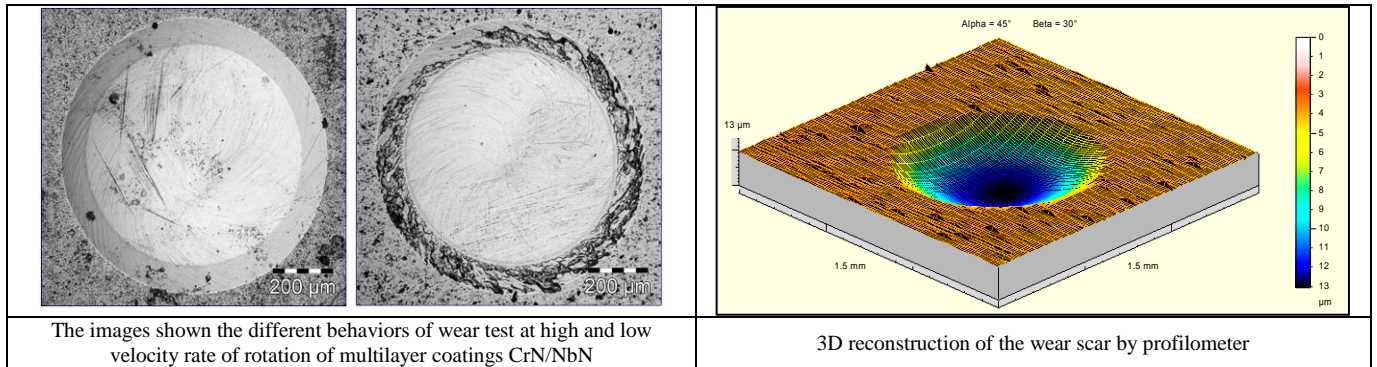
150x confocal Plane view of small structures

150x confocal 3D image of small structures

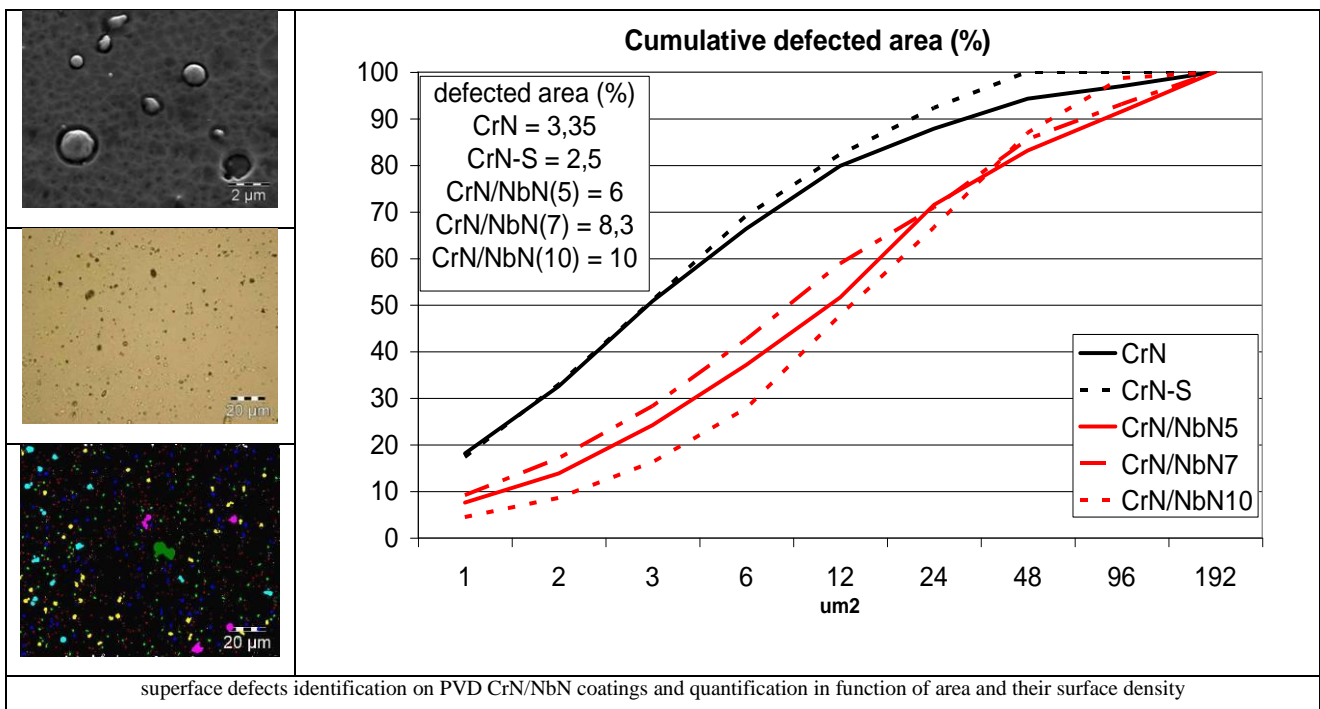
## AFM NT-MDT Smena

	<p>Atomic Force Microscope (NT-MDT, contact, semi-contact, lateral force and other modes of measure), scanning area 100x100 <math>\mu\text{m}^2</math>. Atomic force microscopy (AFM) or scanning force microscopy (SFM) is a very high-resolution type of scanning probe microscopy, with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit. The AFM consists of a cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers. When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law. Depending on the situation, forces that are measured in AFM include mechanical contact force, van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces.</p>	
		
<p>3D Reconstruction of Knoop indentation</p>	<p>3D Reconstruction of MHV</p>	
		
<p>Nb Coating on copper substrate, SEM-SE, 20kV, 40.000x</p>	<p>Morphological 2D Reconstruction of surface</p>	<p>Morphological 3D reconstruction of surface</p>

### Rotating wheel test (calculation of wear coefficient and friction)



### Statistical analysis of surface defects of PVD coatings



## List of the principal available equipments

Full name	Features	Year
workshop for big sample resizing		2001
SEM sample preparation line	3 lapping machines, 2 precision cutting saws, Carbon evaporator coaters & Gold sputter coaters, Automatic Hot Mounting Press: Sample preparation line (TEM) Ion milling Dimpling grinder Ultrasonic Drill Electro-polisher	1999
TEM sample preparation line	Dimpling grinder: Fischione model 2000 Ultrasonic Drill : Fischione model 120 Electro polisher: Fischione model 330	1999
Confocal/interferometric 3D profilometer	Dual Core Optical Imaging Profilometry (Confocal and Interferometry) non-contact, 3 dimensional Confocal, Interferometry (PSI, ePSI, VSI), Brightfield Color, Brightfield Greyscale (high resolution), Darkfield. Objectives from 20x up to 150x in confocal, 50x in interferometry 6-fold objective revolver manual Stage travel range (x,y) stitching available Motorized: From 114 × 75 mm Illumination: High power white LED 530 nm for coaxial light, controllable Vertical Scanning Range 40 mm High power blue LED 460 nm for coaxial light, controllable	2011
Colorimeter	to obtain the CieLab coordinates of non reflective media	2009
FEI Helios NanoLab™ 600	Electron (FEG) and ion beam (GaLMIS) scanning microscope equipped with SE, SI, TLD (SE+BSE) , STEM detectors and Omniprobe "lift out" option.	2007
Nikon Digital Sight DS-Fi1 Digital Camera	A digital camera for the image acquisition from optical systems (microscops and enlargers) with CCD 2/3", 5.2MPx small pixel size and a field of view 2,560 x 1,920, enhanced color reproduction, expanded range and superior reduction technology.	2007
CAM Contact Angle Measurement Instrument - Goniometer	Sample stage on 3 axis (X, Y, Z), 1 µm sensibility Objective (enlargement) 6 x Possibility of use in dual mode: Pendant DropSessile Drop	2007
TEM CCD Camera Olympus MegaView III	Telecamera TEM: Effective pixels [pixel] 1392 x 1040 Digitization [bit] 12 Frame rate [fps] at full resolution > 10 Binning 2x, 4x and 8x PC interface FireWire (IEEE 1394)	2007
Atomic Force Microscope	NT-MDT SMENA	2003
Optical Microscopy	NIKON Eclipse ME600, SMZ 800, EPIPHOT 300	2000
Scanning electron Microscope	FEI SEM XL30	1996
Transmission electron Microscope	FEI CM 120 EDS Edax DX-4Sample holder with double tilt for SAD analysis Nanoprobe for nano EDS analysis	1996

MS-PVD	<ul style="list-style-type: none"> <li>-Cylindrical deposition chamber 90l, 525x410 mm (Dxh)</li> <li>-RF generator PR 600 RF Hüttinger with quartz oscillator ( 13.56 MHz ±0.05% ), providing a power of 600W</li> <li>-DC generator PFG 2500 DC Hüttinger providing 2.5 kW of power</li> <li>-Mass-flow control 1179A MKS provided of two flow-meter (Full scale: 10 to 30000 sccm, Accuracy: ± % F.S., Repeatability: ±0.2 F.S., Resolution: 0.1% F.S., Maximum Inlet Pressure: 150 psig)</li> <li>-Roundabout substrate-carry speed-control machine</li> <li>- Two-stage rotary vane pump Pfeiffer with pumping speed of 20 m3/h @ 50Hz and minimal value of pressure reachable of 5•10<sup>-3</sup> mbar</li> <li>- Turbomolecular drag pump Pfeiffer with minimal value of pressure reachable of 5•10<sup>-10</sup> mbar</li> </ul>	2011
Durometro Shore D	Hardness scale: shore D & A. diameter of the indenter 1,25 mm. Tip radius: 0.1 mm. Tip angle 30°. Maximum contact profile 3 mm. penetration depth 2.5 mm.	2010
Agilent NANO Indenter® G200	<p>Pre-mounted Berkovich diamond indenter tip, Electromagnetic actuation-based force transducer  Automated motion system with mouse control  Complete optics assembly with 10X and 40X objectives and color CCD camera  NanoSwift™ Controller for fully automated data acquisition and control  Vibration isolation table and environmental enclosure cabinet  Complete TestWorks operating and data analysis software  Analyst™ data reduction utility  200 mm of travel  maximum load applied 500 mN  Microscope with position feedback  ISO 14577 compatible with temperature measurement  Nano-vision stage for nano-positioning and 3D indent reconstruction  Lateral force measurement for friction analysis during scratch  Continuous stiffness measurement (CSM)</p>	2007
Industrial Durometer - Universal Hardness Tester Galileo	Standard Durometer Rockwell (all the scales) HB and HV (load from 30 kg to 185 kg)	2004
Hardness Tester	Mitutoyo HM124	2003
CSM REVETEST® Scratch Testing instrument	<ul style="list-style-type: none"> <li>Diamond-stylus</li> <li>Feedback-controlled force actuator</li> <li>Acoustic emission detection</li> <li>Optical microscope with video camera</li> <li>PC controlled operation</li> <li>Long term stability of calibration</li> <li>Compatible to ASTM C1624 and EN 1071</li> </ul>	2007
Complex wear system tribometer		2007
Dry corrosion Cabinet		2003
Fretting machine		1995

**PUBLICATIONS IN THE LAST FOUR YEARS (full list at [www.stm.uniroma3.it](http://www.stm.uniroma3.it))**

Pub year	Title	Authors	Published in
2012	Optimized coating procedure for the protection of TiAl intermetallic alloy against high temperature oxidation	Varlese F. A., Tului M., Sabbadini S., Pellissero F., Sebastiani M., Bemporad E.	Intermetallics
2012	Kircherite, a new mineral of the cancrinite - sodalite group with a 36-layer stacking sequence: occurrence and crystal structure	Cámara F, Bellatreccia F, Della Ventura G, Gunter M E, Sebastiani M, Cavallo A	American Mineralogist
2012	Effects of nanosilica addition on workability and compressive strength of Portland cement pastes	Berra M, Carassiti F., Mangialardi T., Paolini A. E., Sebastiani M.	Construction and Building Materials
2012	Influence of mechanical properties of tungsten carbide-cobalt thermal spray coatings on their solid particle erosion behaviour	Santana Y Y, La Barbera-Sosa J G, Bencomo A, Lesage J, Chicot D, Bemporad E, Puchi-Cabrera E-S, Staia H. M.	Surface Engineering
2012	High resolution residual stress measurement on amorphous and crystalline plasma-sprayed single-splats	Sebastiani M., Bolelli G., Lusvarghi L., Bandyopadhyay P.P., Bemporad E.	Surface and Coatings Technology
2012	Effects of intra-crystalline microcracks on the mechanical behavior under indentation of a marble	Bandini A., Sebastiani M., Bemporad E.	International Journal of Rock Mechanics and Mining Sciences
2012	FIB/SEM and SEM/EDS microstructural analysis of metal-ceramic and zirconia-ceramic interfaces	MASSIMI F., MERLATI G., SEBASTIANI M., BATTAINI P., MENGHINI P., BEMPORAD E.	Bulletin du Groupement International pour la Recherche Scientifique en Stomatologie et Odontologie
2012	X-ray diffraction study of microstructural changes during fatigue damage initiation in steel pipes	Pinheiroa B., Lesage J., Pasqualino I., Benseddiq N., Bemporad E.	Materials Science and Engineering A
2012	Residual stress measurement in thin films at sub-micron scale using Focused Ion Beam milling and imaging	Xu Song; Kong Boon Yeap; Jing Zhu; Jonathan Belnoue; Marco Sebastiani; Edoardo Bemporad; Kalyang Zeng; Alexander Korsunsky	Thin Solid Films
2012	Wear mechanisms and in-service surface modifications of a Stellite 6B Co-Cr alloy	M. Sebastiani, V. Mangione, D. De Felicis, E. Bemporad, F. Carassiti	Wear
2012	An easy way to measure surface free energy by drop shape analysis	Mazzola L., Bemporad E., Carassiti F.	Measurement
2012	An innovative non-contact method to evaluate Surface Free Energy on micro-areas	Mazzola L., Sebastiani M., Bemporad E., Carassiti F.	Journal of Adhesion Science and Technology
2011	Ricoprimenti CAE-PVD: Difetti e Corrosione	Bemporad E., Sebastiani M., Mangione V., Carassiti F.	Trattamenti e Finiture
2011	Growth and Characterisation of LaZr2O7 Buffer Layers Deposited by Chemical Solution Deposition (Superconductivity Centennial Conference)	Angrisani A.A., Augieri A., Fabbri F., Freda R., Galluzzi V., Mancini A., Rizzo F., Rufoloni A., Vannozzi A., Sotgiu G., Pompeo N., Torokhtii K., Silva E., Bemporad E., Contini G., Celentano G.	Physics Procedia
2011	Residual stress measurement in thin films using the semi-destructive ring-core drilling method using Focused Ion Beam	X. Song, K.B. Yeap, J. Zhu, J. Belnoue, M. Sebastiani, E. Bemporad, K.Y. Zeng, A.M. Korsunsky	Procedia Engineering
2011	Depth-resolved residual stress analysis of thin coatings by a new FIB-DIC method	M. Sebastiani, C. Eberl, E. Bemporad, G. M. Pharr	Materials Science and Engineering: A
2011	Focused Ion Beam and Transmission Electron Microscopy as a powerful tool to understand localized corrosion phenomena	E. Bemporad, M. Sebastiani, D. De Felicis, V. Mangione, F. Carassiti	Corrosion Reviews
2011	Influence of Mechanical Properties of Tungsten Carbide-Cobalt Thermal Spray Coatings on their Solid Particle Erosion Behavior	Yucelys Y Santana, José G La Barbera-Sosa, Alfonso Bencomo, Jacky Lesage, Didier Chicot, Edoardo Bemporad, Eli S Puchi-Cabrera, Mariana H Staia	Surface Engineering
2011	Effect of composition on mechanical behaviour of diamond-like carbon coatings modified with titanium	Caschera, F. Federici, L. Pandolfi, S. Kaciulis, M. Sebastiani, E. Bemporad, G. Padeletti	Thin Solid Films
2011	Use of nanosilica in cement-based materials. Part II. Leaching behaviour of cement pastes	M. Berra, F. Carassiti, T. Mangialardi, A.E. Paolini, M. Sebastiani	Cement and Concrete Composites
2011	Filler-matrix interaction in solid-state foaming of composite foams	L.Mazzola, E. Bemporad, E.A. Squeo, F. Trovalusci and V. Tagliaferri	Journal of Cellular Plastic
2011	Flame treatment on plastic: a new surface free energy statistical prediction model and characterization of treated surfaces	L.Mazzola, E. Bemporad, F. Carassiti	Applied Surface Science
2011	On the Influence of Residual Stress on Nano-Mechanical Characterization of Thin Coatings	Sebastiani M., Bemporad E., Carassiti F.	Journal of Nanoscience and Nanotechnology.
2011	Surface Analysis and Osteoblasts Response of a Titanium Oxide Film Deposited on Titanium by Ion Plating Plasma assisted (IPPA)	Mazzola L., Bemporad E., Misiano C., Pepe F., Santini P. and Scandurra R.	Journal of Nanoscience and Nanotechnology
2011	Residual stress measurement at the micrometer scale: focused ion beam (FIB) milling and nanoindentation testing	Sebastiani M. and Bemporad E. and Schwarzer N. and Carassiti F.	Philosophical Magazine: Structure and Properties of Condensed Matter
2010	Nell'ottica delle fibre	A. Calabrò, L.Mazzola	Trasferimento Tecnologico - rivista delle innovazioni tra scienza e lavoro
2010	Residual Stress Evaluation at the Micrometer Scale: Analysis of Thin Coatings by FIB milling and Digital Image Correlation	A. M. Korsunsky, M. Sebastiani, E. Bemporad	Surface and Coatings Technology
2010	Characterization of expanded austenite developed on AISI 316L stainless steel by plasma carburization	Molleja J. G. and Nosei L. and Ferrón J. and Bemporad E. and Lesage J. and Chicot D. and Feugeas J.	Surface & Coatings Technology
2010	Structural characterisation of High Velocity Suspension Flame Sprayed (HVSFS)	Bemporad E. and Bolelli G. and Cannillo V. and De Felicis D. and Gadwo R. and Killinger A. and Lusvarghi L. and Rauch J. and Sebastiani M.	Surface & Coatings Technology
2010	Depth-sensing indentation modeling for determination of Elastic modulus of thin films	Tricoteaux A. and Duarte G. and Chicot D. and Le Bourhis E. and Bemporad E. and Lesage J.	Mechanics of Materials



2010	Fantappièite, a new mineral of the cancrinite-sodalite group with a 33-layer stacking sequence: Occurrence and crystal structure	Cámara F. and Bellatreccia F. and Della Ventura G. and Mottana A. and Bindi L. and Gunter M.E. and Sebastiani M.	American Mineralogist
2010	Effect of Titanium Carbide Coating by Ion Plating Plasma-Assisted Deposition on Osteoblast	Longo G., Girasole M., Pompeo G., Cricenti A., Misiano C., Acclavio A., Tizzoni A. C., Mazzola L., Santini P., Politi L., Scandurra R.	Surface and Coatings Technology
2010	On the measurement and interpretation of residual stress at the micro-scale	Korsunsky A.M. and Bemporad E. and Sebastiani M. and Hofmann F. and Dave S.	International Journal Of Modern Physics B (IJMPB)
2010	Austenite Modification of AISI 316L ss by Pulsed Nitrogen Ion Beams Generated in Dense Plasma Focus Discharges	Feugeas J. and Rico L. and Nosei L. and Gomez B. and Bemporad E. and Lesage J.	Surface and Coatings Technology
2010	F-substituted hydroxyapatite nanopowders: thermal stability, sintering behaviour and mechanical properties	Bianco A. and Cacciotti I. and Lombardi M. and Montanaro L. and Bemporad E. and Sebastiani M.	Ceramics International
2009	Hydrothermal N-doped TiO <sub>2</sub> : Explaining photocatalytic properties by electronic	D'Arienzo M. and Scotti R. and Wahba L. and Battocchio C. and Bemporad E. b,	Applied Catalysis - B: Environmental
2009	COMPLEX WEAR MEASUREMENT ON THIN COATINGS BY THE CRATERING METHOD	Bemporad E. and Comis E. and Sebastiani M. and Carassiti F. and Palumbo B.	LUBRICATION SCIENCE
2009	Preparation and mechanical characterization of dense and porous zirconia produced by gel casting with gelatin as a gelling agent	Tulliani J-M and Bartuli C. and Bemporad E. and Naglieri V. and Sebastiani M.	Ceramics International
2009	Mechanical properties of cellular ceramics obtained by gel casting: Characterization and modeling	Bartuli C. and Bemporad E. and Tulliani J-M and Tirillò J. and Pulci G. and Sebastiani M.	Journal of the European Ceramic Society
2009	Focused ion beam ring-drilling for residual stress evaluation	Korsunsky A.M. and Sebastiani M. and Bemporad E.	Materials Letters
2009	L'evoluzione del tradizionale	Bemporad E.	Roma Tre News
2009	Graded selective coatings based on zirconium and titanium oxynitride	RIZZO A and SIGNORE MA and TAPPER L and PISCOPIELLO E and CAPPELLO A and BEMPORAD E and SEBASTIANI M	Journal of physics d: applied physics

Aziende o Privati  
possono richiedere al Dipartimento di Ingegneria  
le seguenti prestazioni relative a competenze,  
disponibili presso il gruppo di Scienza e Tecnologia dei Materiali (STM)

**MISURE DI DENSITA' BULK:**

- Misura di densità bulk

**DUROMETRIA SU SCALA MACRO:**

- Misura di durezza Rockwell (tutte le scale), Brinell e Vickers con carichi variabili su materiali omogenei piani e curvi secondo normativa ISO 6506, 6507, 6508 e ASTM E18-02
- Misura qualitativa della adesione e tenacità di rivestimenti sottili ceramici tramite durezza Rockwell-C secondo normativa UNI EN 1071-8
- Misura di durezza Shore D su gomma dura o plastiche piane secondo normativa UNI ISO 868

**DUROMETRIA SU SCALA MICRO:**

- Misure di microdurezza tipo MHV e MHK su campione disomogeneo con carichi variabili secondo normativa ASTM E384
- Realizzazione di profili di durezza (cuciture) in sezione
- Calcolo della durezza superficiale di sistemi rivestiti con film sottili mediante l'applicazione di modelli semi-empirici (Jonhson-Hogmark, Chicot-Lesage o altri)

**DUROMETRIA SU SCALA NANO:**

- Misure di nanodurezza Berkovich su campioni massivi o rivestiti a comportamento non viscoso per valutazione delle curve di variazione di durezza e modulo elastico. Prove secondo normative UNI EN 14577-1-2-3.
- Misure di nanodurezza su campioni a comportamento viscoelastico (indentatore Flat-Punch) per la valutazione delle curve di variazione di durezza, modulo elastico e modulo dissipativo. Prove secondo normative UNI EN 14577-1-2-3.
- Misure di nanodurezza con l'utilizzo di indentatori non convenzionali (indentatore wedge, indentatori sfero-conici) per la valutazione delle curve di variazione delle proprietà di interesse al variare della profondità di indentazione. Prove secondo normative UNI EN 14577-1-2-3.

**ADESIONE SU SCALA MICRO:**

- Misura dell'adesione e resistenza al graffio con Macro/micro- scratch tester su rivestimenti sottili. Prove secondo normativa UNI EN 1071-3

**ADESIONE SU SCALA NANO:**

- Misura dell'adesione e resistenza al graffio mediante Nano-scratch tester su rivestimenti ultra-sottili o soffici.

**PROFILOMETRIA**

- Misura di rugosità superficiale mediante profilometria "non contact" in modalità interferometrica o confocale. Prove eseguite secondo norme UNI EN ISO 4288, UNI ISO 25178
- Ricostruzione di topografie in 3D di superfici e calcolo di volumi
- Misure di spessore per rivestimenti

#### **PROVE DI CORROSIONE:**

- Test per la misura della corrosione in camera a nebbia salina a temperatura costante e soluzione di NaCl secondo normative ASTM: B112-02, G1-90, G85-02, G46-94, G33-99

#### **PROVE TRIBOLOGICHE:**

- Misura del coefficiente di usura (modello di Archard) tramite Implemented Rotating Wheel
- Misura degli spessori di rivestimenti mono e multistrato tramite Calotest

#### **ANALISI CHIMICO-FISICHE DI SUPERFICI:**

- Misura della bagnabilità delle superfici secondo il metodo "sessile drop"
- Misura della bagnabilità ed energia superficiale tramite l'applicazione di 4 famiglie di solventi
- Misura della tensione superficiale di liquidi tramite metodo "pendant drop"

**Inoltre, Aziende e industrie possono ottenere dal LIME, tramite il gruppo STM del DIMI che ne è socio, le seguenti prestazioni con e senza certificazione:**

#### PREPARATIVA METALLOGRAFICA CLASSICA (norma ASTM E3)

- Taglio
- Inglobamento
- Assottigliamento e lucidatura manuale ed automatica
- Attacco chimico metallografico in soluzione o elettrolitico
- Rivestimento conduttivo tramite Sputter Coater per l'osservazione al SEM di campioni non conduttivi (oro o grafite)

#### PREPARATIVA METALLOGRAFICA AVANZATA

- Realizzazione lamella TEM tramite Focused Ion Beam
- Assottigliamento e lucidatura tramite Tripod
- Assottigliamento chimico in bagno elettrolitico con o senza criostato (secondo normativa ASTM E1558)
- Assottigliamento a conca fino a circa 5  $\mu\text{m}$  di spessore tramite Dimpling Grinder
- Trasparenza elettronica tramite Electropolisher con e senza criostato

#### ANALISI IN MICROSCOPIA OTTICA

- Osservazione in riflessione ed in trasmissione fino a 1.000x con possibilità di impiegare filtri speciali (DIC, polarizzatore, contrasto di fase, ecc.)

#### ANALISI IN MICROSCOPIA ELETTRONICA A SCANSIONE (SEM)

- Osservazione tramite rivelatore di elettroni secondari, retrodifusi, catodoluminescenza e correnti indotte
- Metrologia elementare secondo normativa ASTM B748

#### ANALISI COMPOSITIVA rX (via SEM)

- Analisi qualitative puntuali e di area per l'individuazione degli elementi presenti
- Analisi qualitative di linea con l'individuazione dei profili di concentrazione

- Mappe elementali della distribuzione di concentrazione degli elementi
- Analisi semi-quantitative standardless con calibrazione interna
- Analisi quantitative con calibrazione da campioni standard

#### ANALISI IN MICROSCOPIA A SCANSIONE FEG E IONICA A FASCIO FOCALIZZATO (FIB)

- Osservazione tramite rivelatore SE o BSE e tramite rivelatori speciali (CDEM-SI, correnti indotte) fino ad ingrandimenti 1.000.000x
- Metrologia elementare secondo normativa ASTM B748
- Realizzazione di cross section su campioni conduttivi e non conduttivi, analisi di spessore film nanometrici
- Analisi degli stress residui di rivestimenti nanostrutturati e microsistemi (MEMS), tramite tecnica (FIB-DIC)
- Realizzazione di pattern utilizzando i modelli predefiniti dello strumento o tramite script personalizzati

#### ANALISI COMPOSITIVA rX (via FEG)

- Analisi qualitative puntuali e di area per l'individuazione degli elementi presenti
- Analisi qualitative di linea con l'individuazione dei profili di concentrazione
- Mappe elementali della distribuzione di concentrazione degli elementi
- Analisi semi-quantitative standardless con calibrazione interna
- Analisi quantitative con calibrazione da campioni standard

#### ANALISI IN MICROSCOPIA ELETTRONICA A TRASMISSIONE (TEM)

- Osservazione in campo chiaro e scuro con ingrandimenti fino a 660.000x
- Osservazione in diffrazione elettronica (SADP) per analisi cristallografiche

#### ANALISI COMPOSITIVA rX (via TEM)

- Analisi qualitative puntuali tramite NanoProbe e portacampioni low noise per l'individuazione degli elementi presenti
- Analisi qualitative su una linea con l'individuazione dei profili di concentrazione degli elementi

#### ANALISI IN MICROSCOPIA A FORZA ATOMICA

- Esecuzione di immagini in 2D e 3D in modalità contact o tapping con area di scansione massima di 100×100µm fino a scale submicrometriche (minori di 5×5 µm) e una altezza massima (Z) di 5µm.
- Segnali acquisibili: height (topografia), lateral force, phase, magnetic, ecc.
- Misura di rugosità (R<sub>a</sub>) complessiva e intradifetti
- Ricostruzione tridimensionale di microindentazioni Vickers e Knoop

#### ELABORAZIONE DATI

- Elaborazione delle immagini per evidenziare particolari caratteristiche morfologiche (ottimizzazione dei parametri di contrasto/luminosità locale, orientazione preferenziale, periodicità, fattori di forma, ...);
- Indagini metrologiche quantitative su immagini SEM calibrate per numerosità, dimensioni (poligoni, aree,...) e classificazione di oggetti comunque definiti;
- Interpretazione cristallografica approfondita con elaborazione software e ricerca su database PDF

## PRESENTAZIONE DEI DATI

- Servizio di deposito dei risultati delle analisi sul sistema informativo interno con la possibilità di accesso tramite FTP riservato e controllato o in modalità extranet estesa.
- Generazione di rapporto o di una relazione tecnico-scientifica sulla campagna di prove, comprensiva di tutti i dati raccolti (immagini, grafici, tabelle) in formato PDF.