



Diagnostic and analytical techniques for advanced materials and nanostructures

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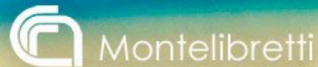
Outline

- ❑ Glimpse on CNR Research Area - Roma 1 research infrastructure

- ❑ Ex situ characterization & diagnostic techniques
 - SEM-EDS
 - Auger & X-ray Photoelectron Spectroscopy
 - Combined use of SEM, EDS, XPS
 - Raman and IR Spectroscopy
 - SPM
 - Laser Scanning Spectroscopy

- ❑ Potential and perspectives

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Biology, Agriculture and Food Sciences

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Functional Materials

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- INSTITUTE OF AGRICULTURAL BIOLOGY AND BIOTECHNOLOGY (IBBA)
- INSTITUTE OF CELL BIOLOGY AND NEUROBIOLOGY (IBCN)
- INSTITUTE FOR THE CONSERVATION AND VALORIZATION OF CULTURAL HERITAGE (ICVBC)
- INSTITUTE OF CRYSTALLOGRAPHY (IC)
- INSTITUTE OF ENVIRONMENTAL GEOLOGY AND GEOENGINEERING (IGAG)
- INSTITUTE OF ATMOSPHERIC POLLUTION RESEARCH (IIA)
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- INSTITUTE OF WATER RESEARCH INSTITUTE (IRSA)
- INSTITUTE FOR THE STUDY ON ANCIENT MEDITERRANEAN (ISMA)
- INSTITUTE OF STRUCTURE OF MATTER (ISM)
- INSTITUTE OF NANOSTRUCTURED MATERIALS (ISMN)
- INSTITUTE FOR TECHNOLOGIES APPLIED TO CULTURAL HERITAGE (ITABC)

HIGHLIGHTS

RESTYLING DEL SITO DELL'AREA DI RICERCA DI ROMA 1 MONTELIBRETTI
 8 Settembre 2017 RESTYLING DEL SITO DELL'AREA DI RICERCA DI ROMA 1 MONTELIBRETTI Grazie alle risorse interne all'Area di Ricerca [...]

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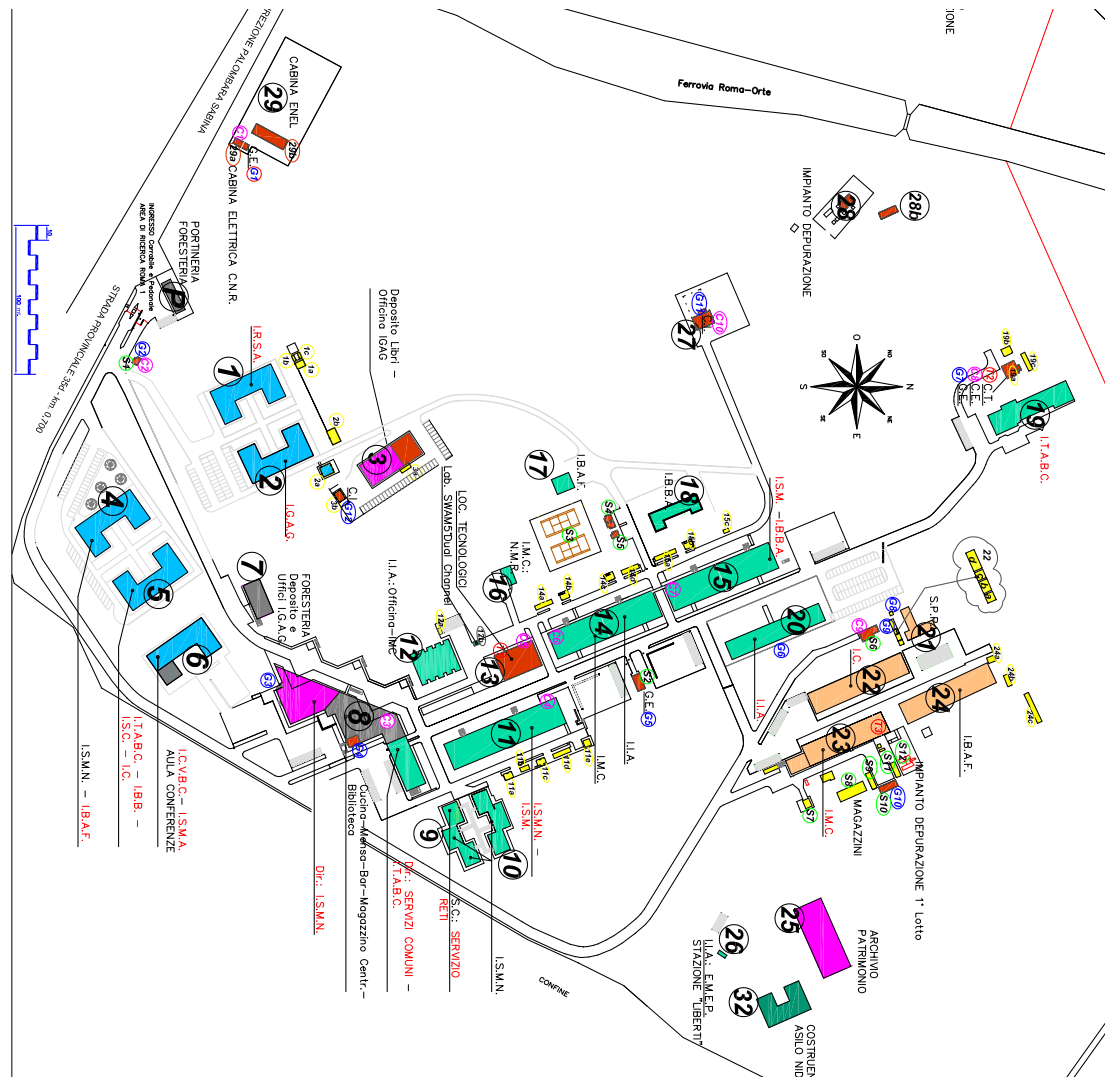


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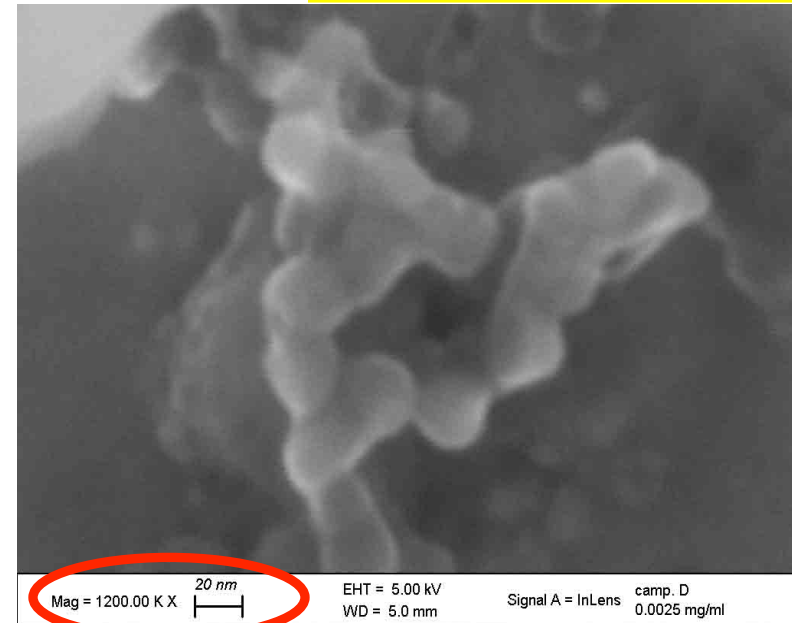
700 employees
37,000 sqm
technological infrastructure's area.



Scanning Electron Microscopy (SEM)

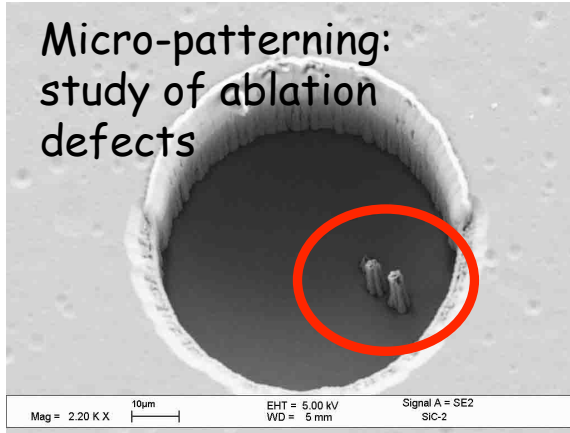
Due to its wide versatility, SEM is largely used to investigate the micro and nano-morphological-structural-chemical features and behaviour of a wide panorama of materials with a large industrial use or high technological interest.

Morphological investigation of organic nanostructured materials: lipid nanoparticles for drug delivery (Bondi)

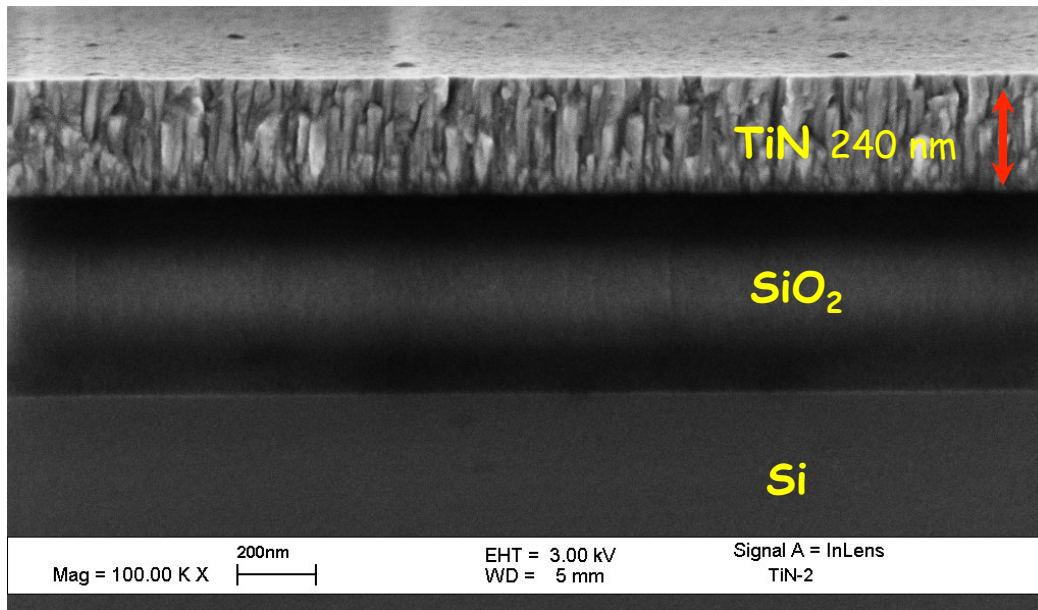
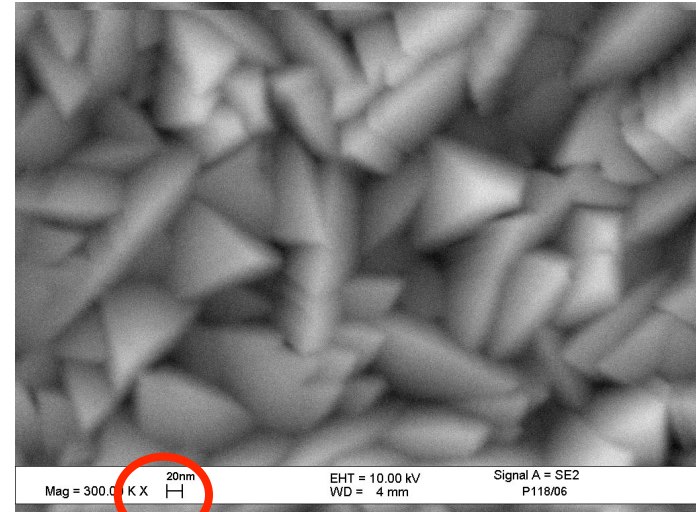


Process evaluation of microelectronic components production

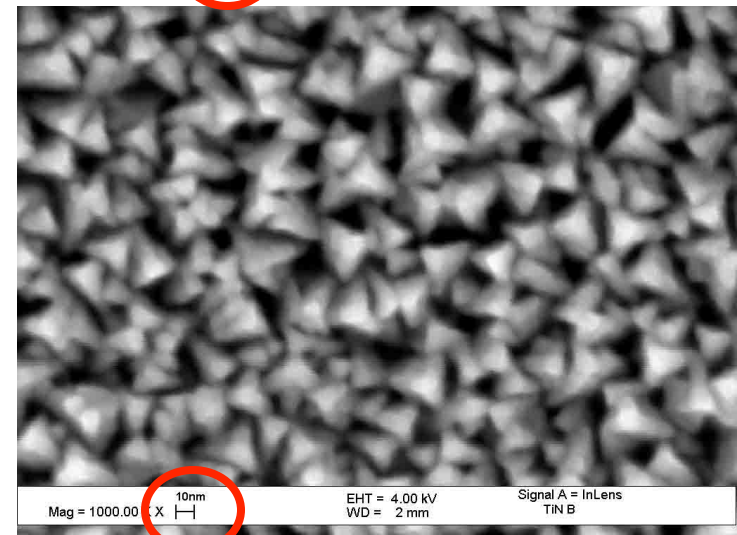
Micro-patterning:
study of ablation
defects



Optimisation of the
ITO deposition
parameters

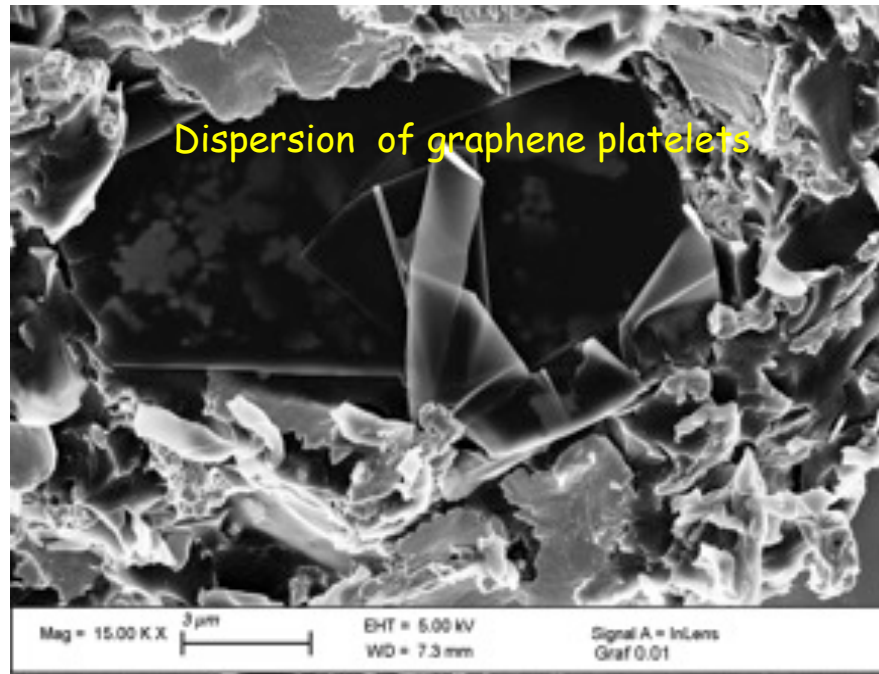
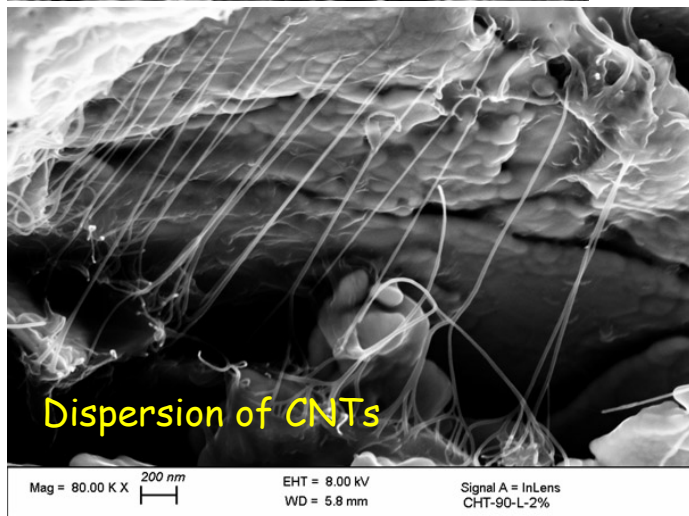
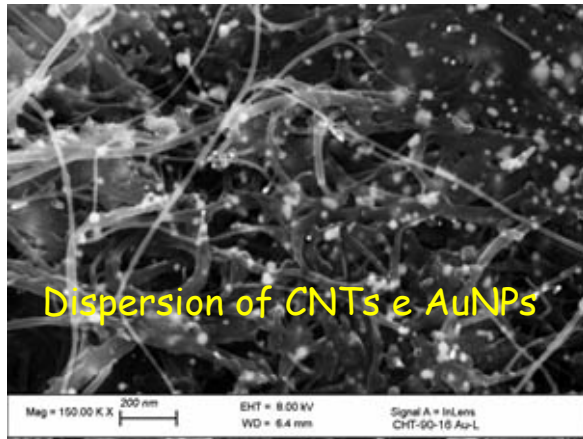


Structural and morphological study of thin films
multi-layered structures for electronic devices



Investigation of the deposition
processes of TiN protective films

Production and analysis of epoxy resins for the packaging of avionic system components

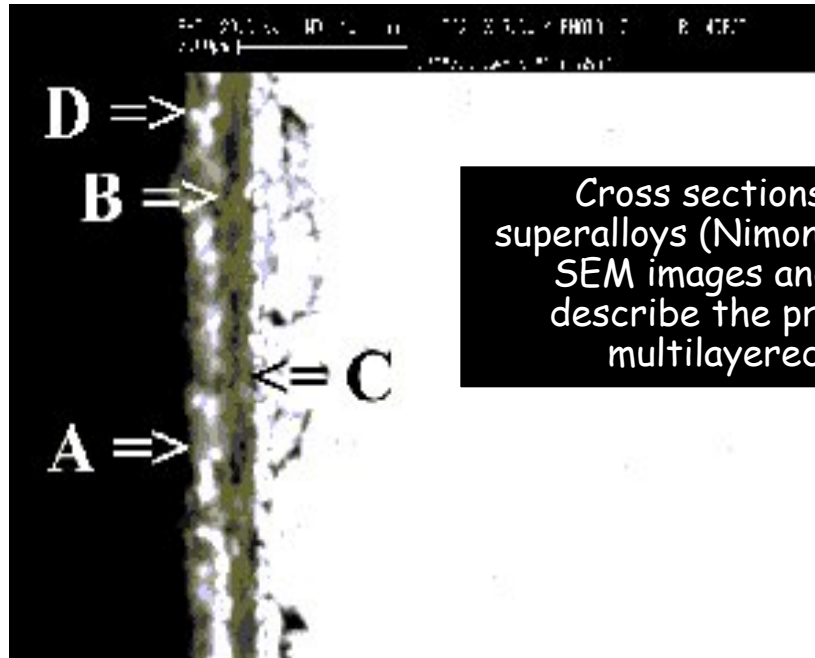


The morphological investigation has guided the synthesis and the dispersion procedures of nanostructures improving:

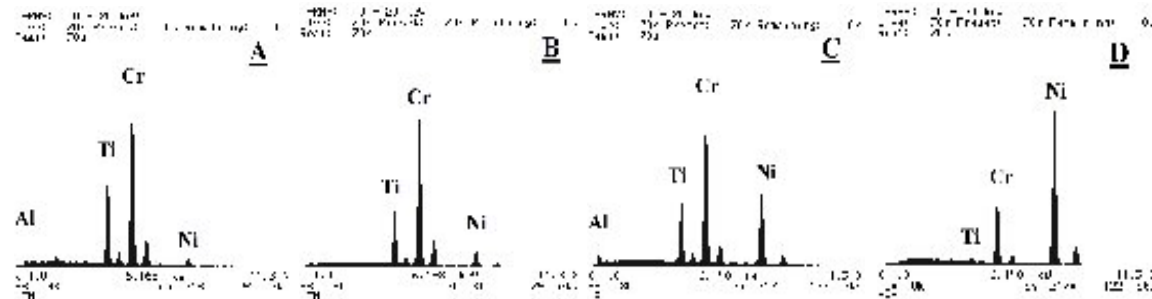
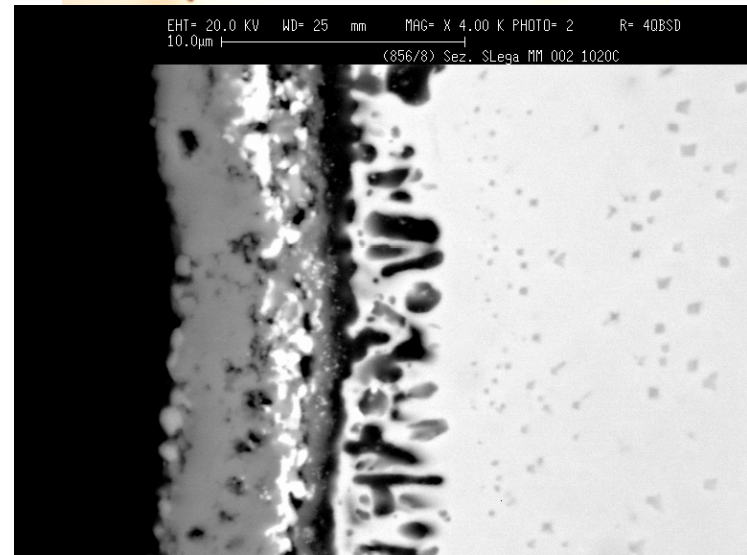
- the electrical conductivity of 1100 %;
- the thermal conductivity of 470 %.

SEM-EDS morphological and chemical analysis

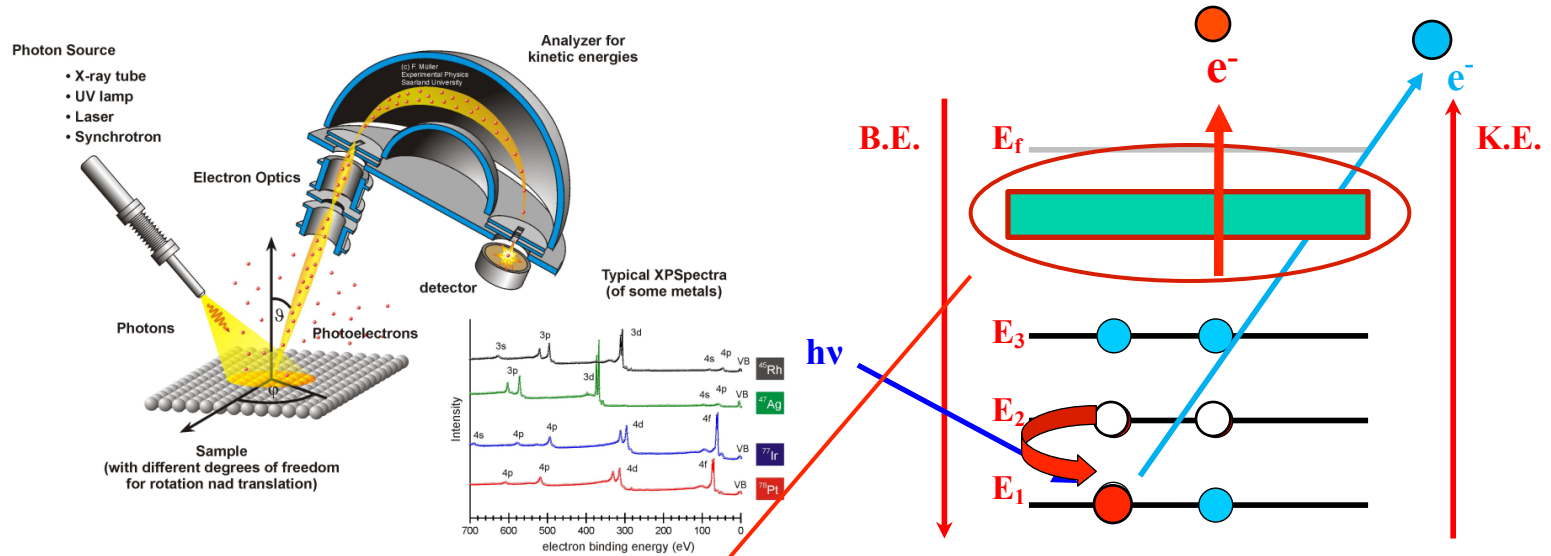
Failure analysis of jet engine turbine blades
Degradation mechanisms identified



Cross sections of Ni-based superalloys (Nimonic 80 A, MM002). SEM images and EDS spectra describe the protective oxide multilayered structure.



Auger & X-ray Photoelectron Spectroscopy



CORE LEVEL SPECTRUM (XPS)

$$BE = h\nu - KE - \phi_{spec}$$

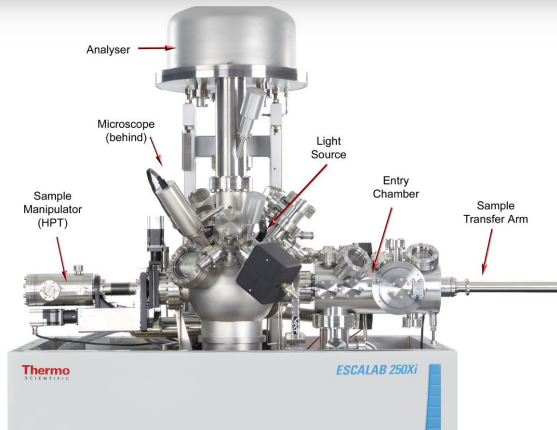
AUGER SPECTRUM (AES)

$$KE_{123} = KE_1 - KE_2 - KE_3^*$$

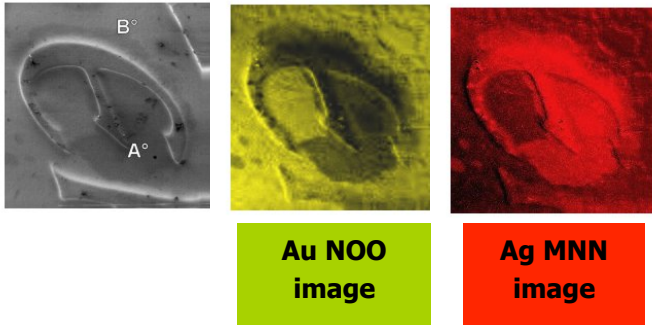
VB SPECTRUM

➤ SURFACE CHEMICAL COMPOSITION (1-10 nm)

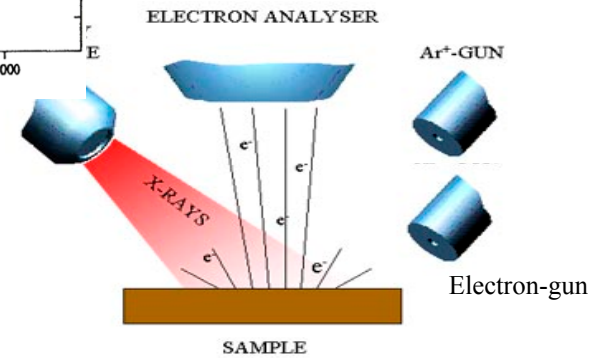
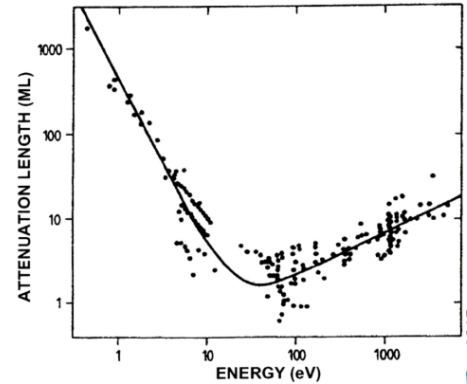
➤ CHEMICAL IMAGING



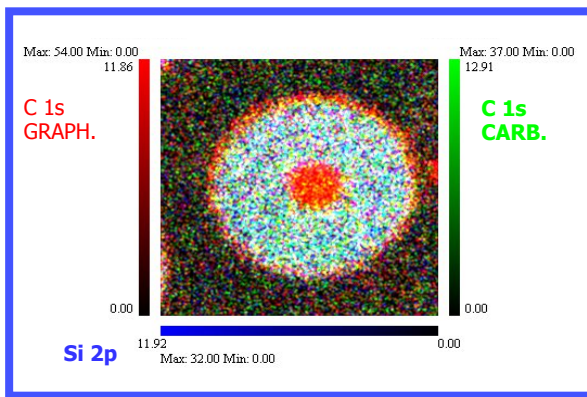
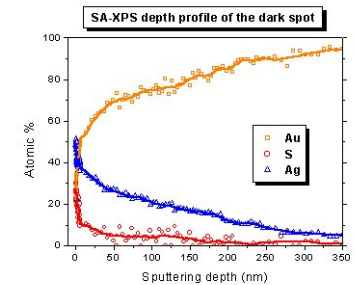
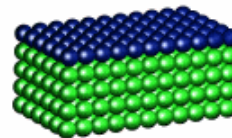
➤ CHEMICAL IMAGING



➤ SURFACE CHEMICAL COMPOSITION (1-10 nm)

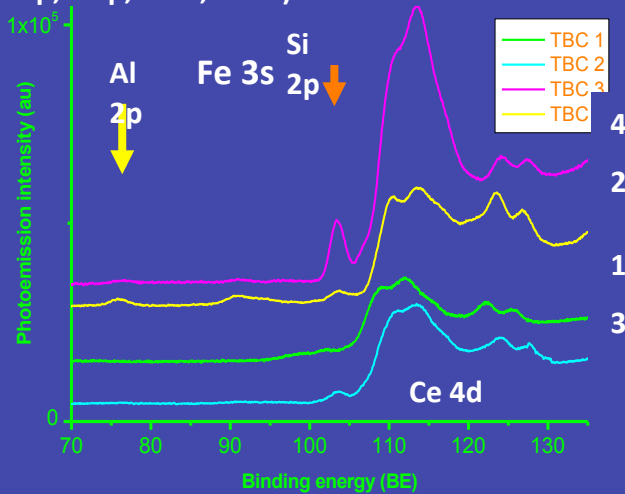


➤ ION SPUTTERING



Coatings of turbine blades and combustion chamber in jet engines

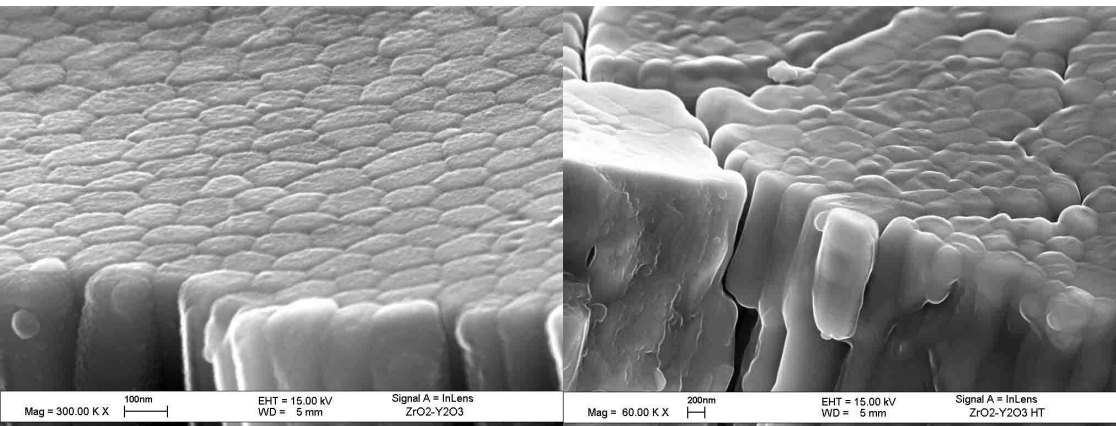
XPS spectra of TBCs produced by adopting different deposition parameters (energy region Al 2p, Si 2p, Fe 3s, Ce 4d)



Combined XPS and SEM analysis allows:

- to optimise the production processes;
- to evaluate the durability and reliability
- to prevent catastrophic failure

TBC deposited on a jet engine turbine blade (25.5 wt% $CeO_2-2.5 Y_2O_3 - ZrO_2$) after a thermal cycling test. The coating fracture has been caused by impurities (Si, Al, Na) segregation phenomena inducing also a columnar growth.



μ-Raman spectroscopy



Renishaw 2000 μ-Raman with a Peltier cooled CCD camera in conjunction with a Leica optical microscope and laser excitation

Raman spectroscopy is a scattering technique based on the inelastic scattering of incident radiation through its interaction with vibrating molecules (Raman effect).

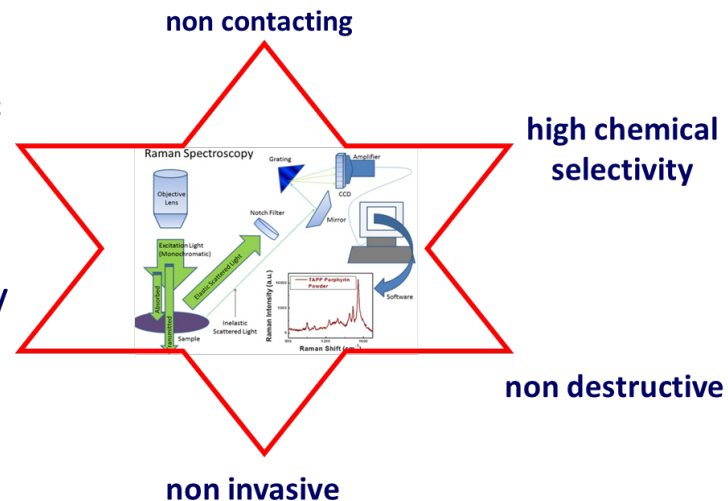
The vibration modes of a substance depend on:

- mass of the atoms
- bonding forces
- symmetries of the molecules

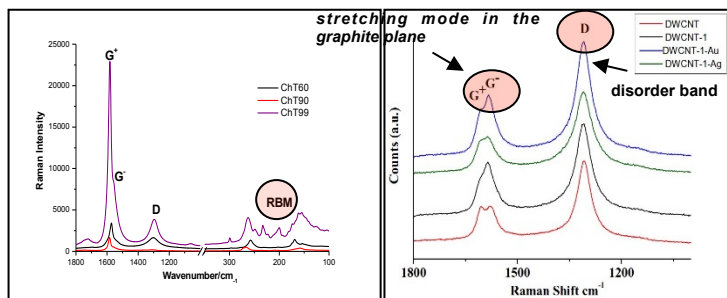
Therefore, the **Raman spectrum** of a molecule corresponds to a "fingerprint" with which to identify it.

organic /inorganic
solid/liquid

flexible
laser wavelength/
power



- **Not all** the molecules are Raman actives!
- The **fluorescence emission bands** can cover the Raman signal.
- Some compounds may suffer of a **thermal degradation** due to the laser irradiation.

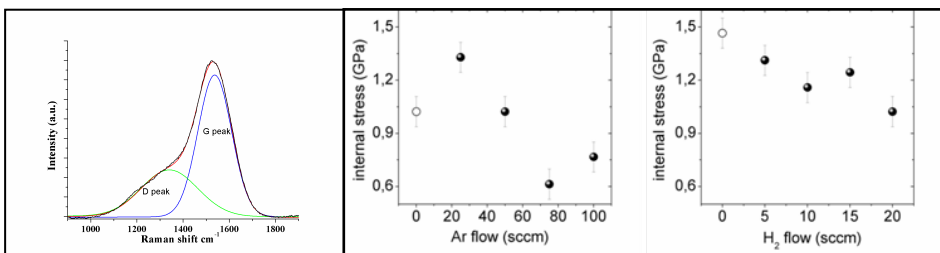


carbon nanotubes

G/D and **G/G⁺** ratios are indicators of **sample quality** and **conductivity** of the sample

Radial Breathing Mode (RBM) is directly related to the **diameter of nanotubes**

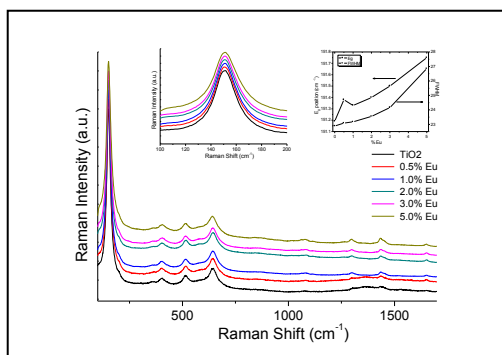
diamond like carbon DLC



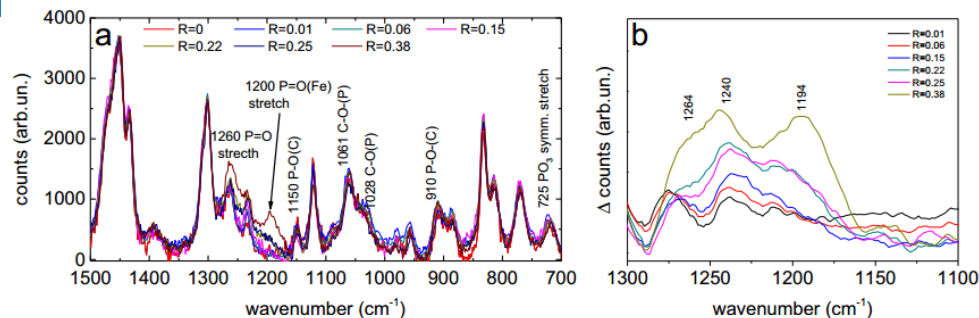
Evaluation of internal **stress** of a DLC film on a Si substrate:

$$\sigma = 2G \left(\frac{1+\nu}{1-\nu} \right) \cdot \left(\frac{\Delta\omega}{\omega_0} \right)$$

Eu-TiO₂ NCs functionalized cotton



Molecular self assembly



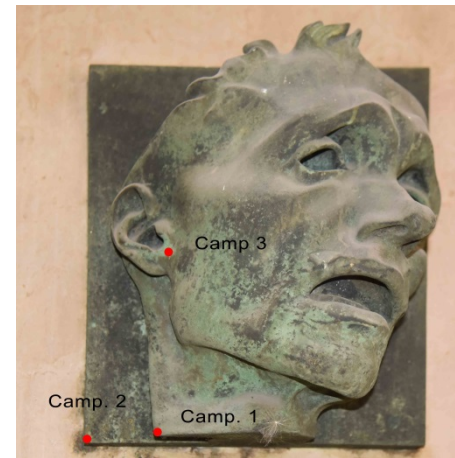
ATR-FTIR spectroscopy for Cultural Heritage

Investigation of the composition of degradation products on copper-based works of art.



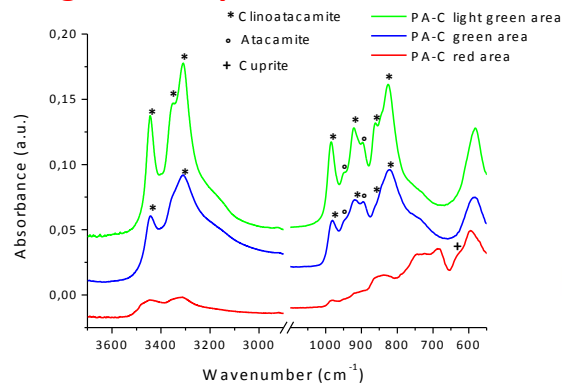
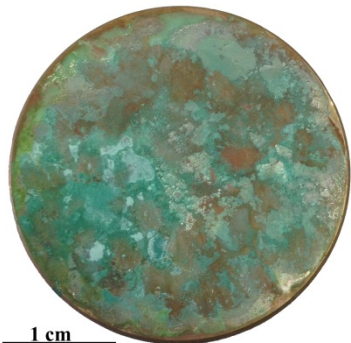
Analysis of patina composition:

- ✓ Identification of degradation products on copper-based alloys, as copper hydroxychlorides and hydroxysulphates
- ✓ Distinguish degradation products polymorphs with different chemical reactivity (as clinoatacamite and atacamite)

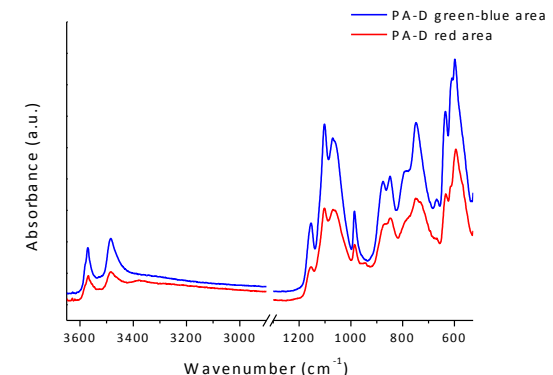
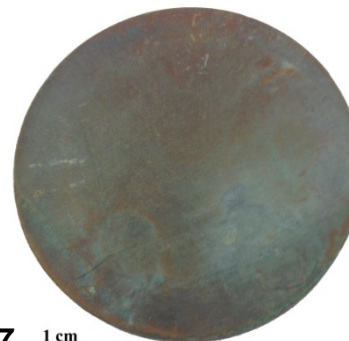


Adolfo Wildt

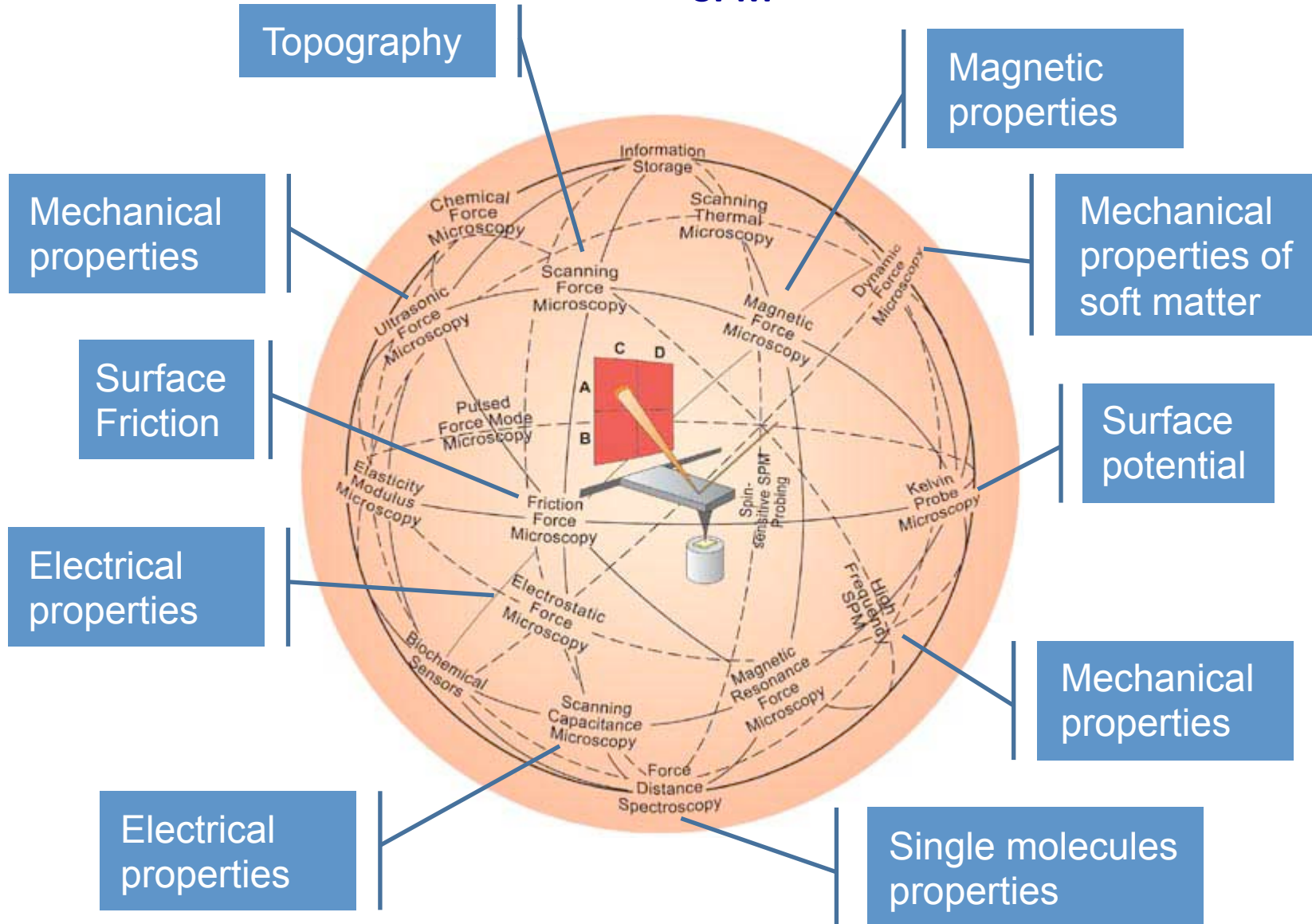
Copper hydroxychloride degradation products



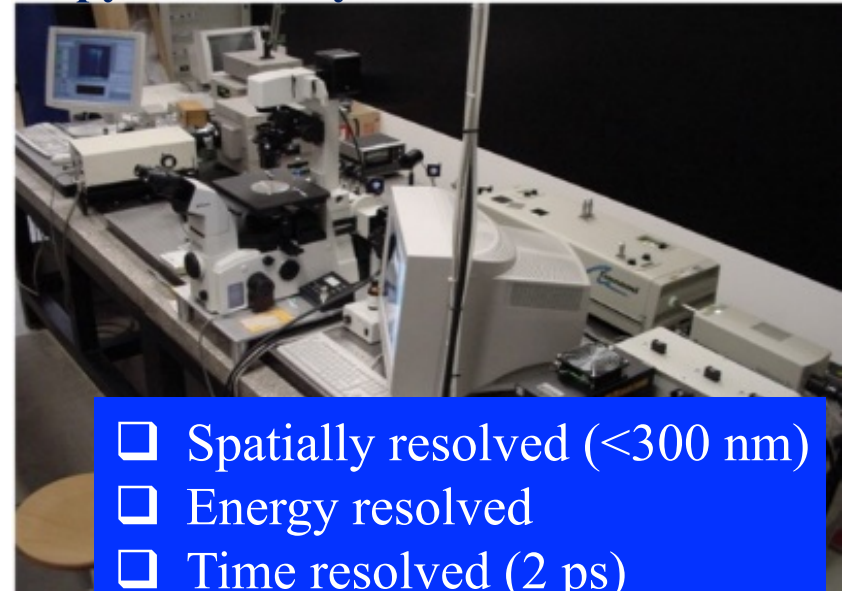
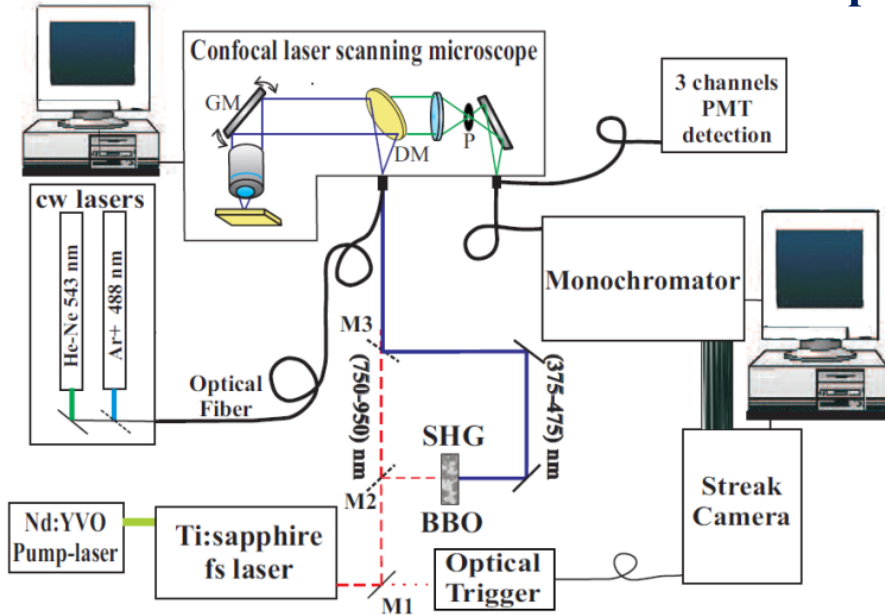
Copper hydroxysulphate degradation products



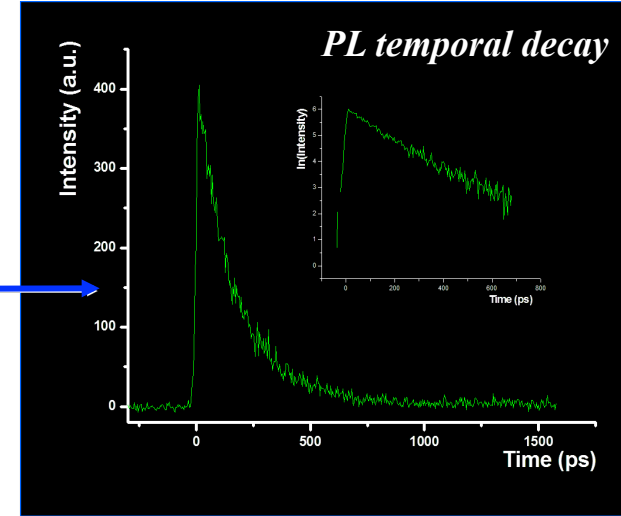
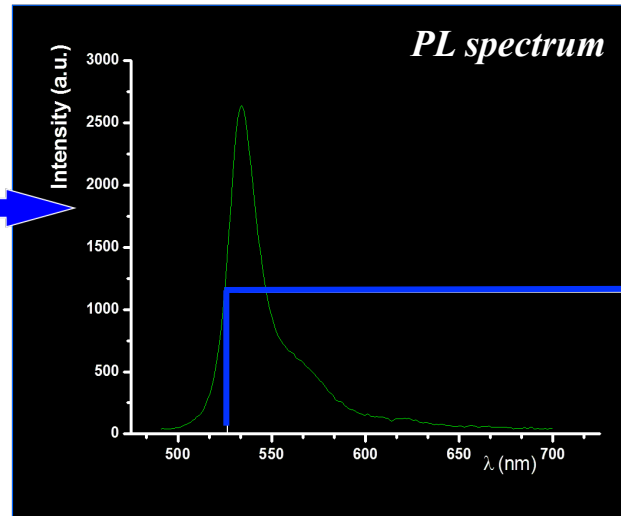
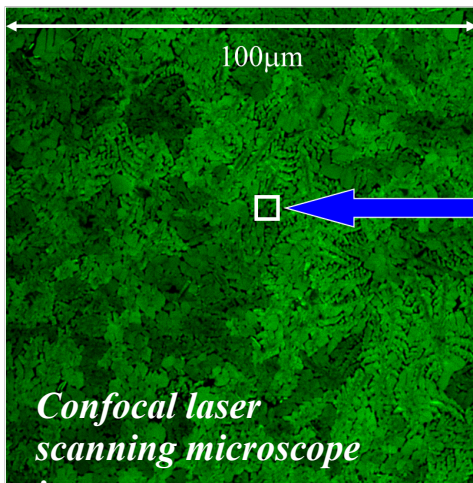
SPM



Time-resolved micro-spectroscopy laboratory

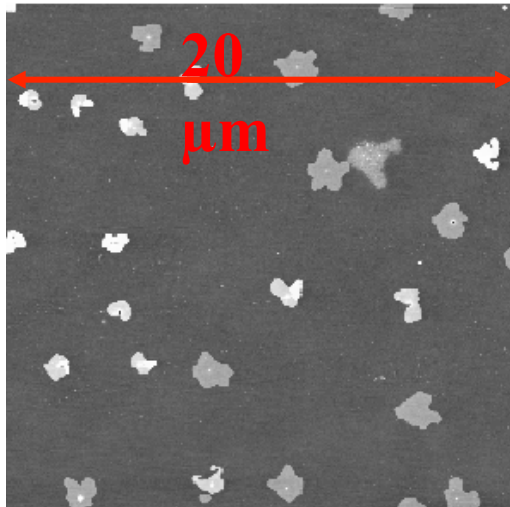


- ❑ Spatially resolved (<300 nm)
- ❑ Energy resolved
- ❑ Time resolved (2 ps)
- ❑ Temperature 450 – 4,2 K

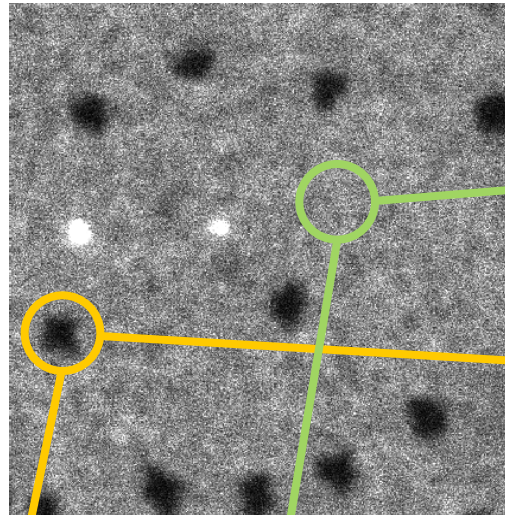


Correlation morphology-structure-function in composite systems

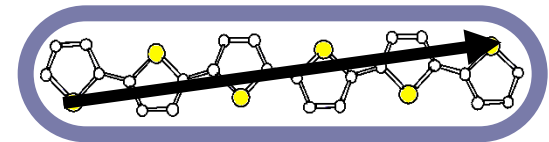
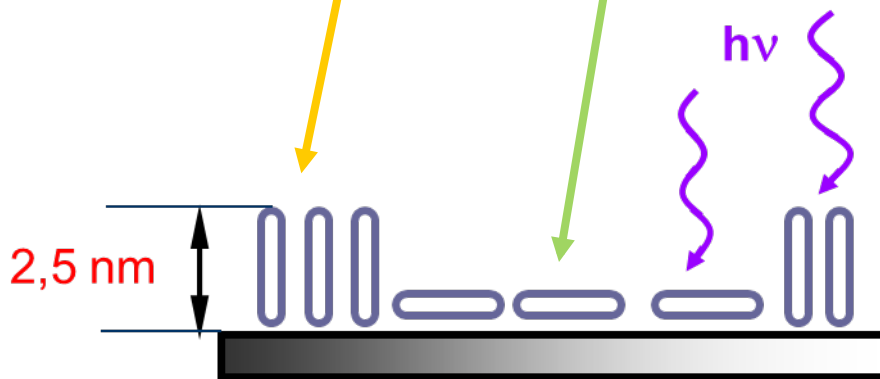
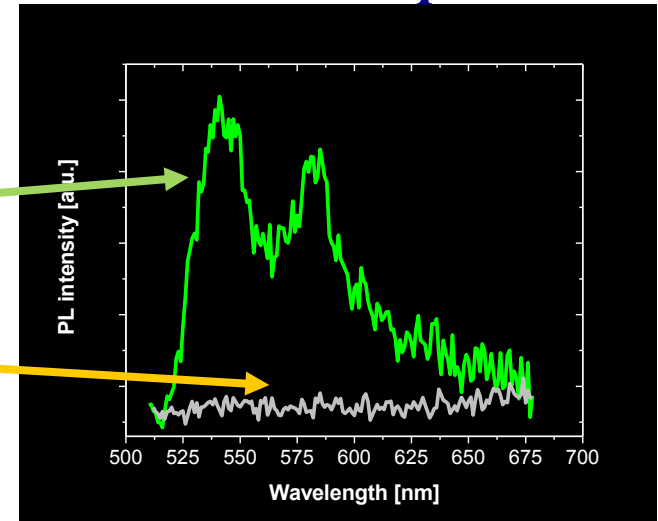
AFM



CLSM



Localized PL spectrum



Molecular orientation

In summary

- ❑ The combination of different analysis techniques is essential for the development of ALM Technology
- ❑ SEM, XPS, XRD, SPM, RAMAN, FTIR, DTA-TG-DSC, LASER SPECTROSCOPY and OM, allows the thorough
 - investigation of degradation phenomena (failure analysis) of structures and components
 - optimization of production processes
 - tailoring of the materials final properties



Aim at in-situ process monitoring and metrology

Acknowledgments

Electron & Optical Microscopy Lab

Ingo
Ricucci
Messina
Pascucci

Surface Analysis Lab

Kaciulis
Mezzi

PECVD – MOCVD Lab

Caschera
Toro
Federici

XRD Lab

Veroli

Raman Spectroscopy Lab

De Caro
Ruani

Laser Scanning

Micro-Spectroscopy Lab

Toffanin
Bolognesi

FTIR Spectroscopy Lab

Di Carlo
Giuliani
Ingo

SPM Lab

Leo
Cerri
Padeletti
Valle
Brucale
Albonetti

Some of the projects @ISMN Mlib

MIUR-PON PANREX
EU-NANORESTART
EU-HERACLES
EU-MOSTOPHOS
Min Difesa – FABRISAFE
EU-INNOVACONCRETE

Thank you!