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# State of The Art of Atomic Force Microscopy in Material Sciences

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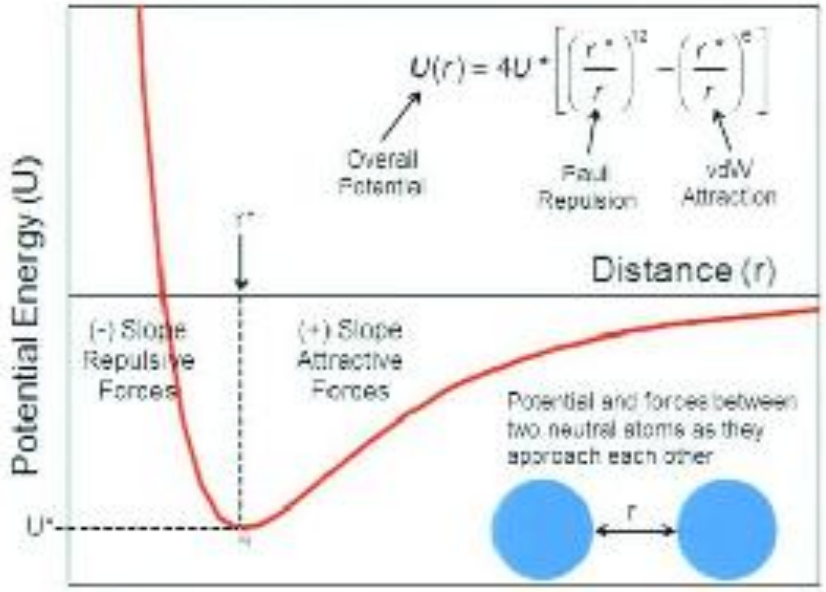
# SUMMARY

1. How AFM works (in short)
2. AFM modes and variants
3. Material science specific studies and examples.

# 1. How AFM works

## Lennard-Jones Potential




## Tip-Sample Interactions

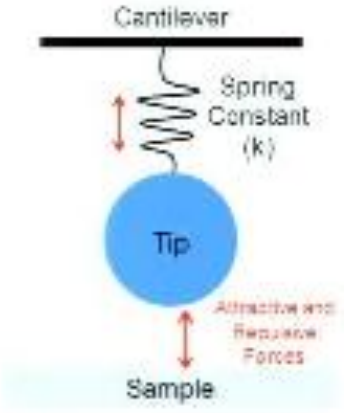
The tip is mounted at the end of a cantilever.

The cantilever is flexible and behaves like a spring.

As the tip approaches the surface, it will experience forces due to tip-sample interactions.

The spring-like action of the cantilever also acts on the tip.

A force balance can be set up that models all forces acting on the tip as it approaches the sample.



# 1. How AFM works

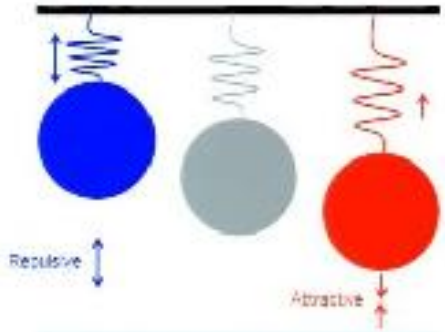
## Tip-Sample Interactions

Examples of Cantilever Deflection

Repulsive force between tip and sample

Attractive force between tip and sample

Note: If the tip is not moving, the forces must be balanced.



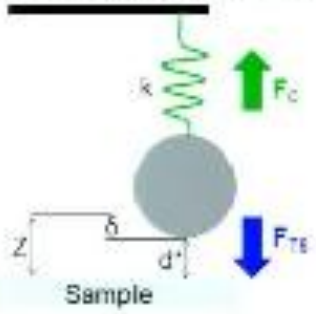

## Force-Distance Curves

The data collected as the tip approaches, contacts, and retracts from the surface can be used to construct Force-Distance curves.

The collected data is a series of (Z, δ) data points.

These curves give information about the interaction forces between the tip and the surface.

The data points can be converted to (d, $F_{TS}$) data as follows:




At equilibrium

$F_{TS} = F_C$

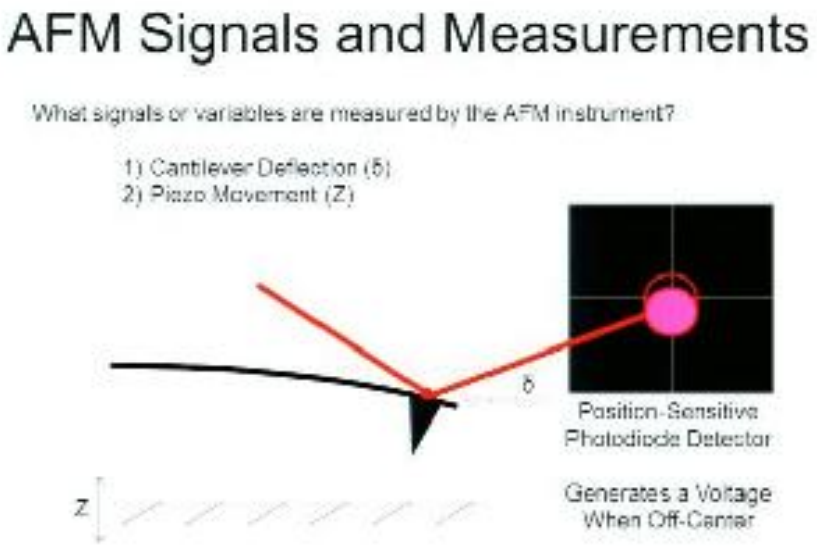
$F_{TS} = F_C = k\delta$

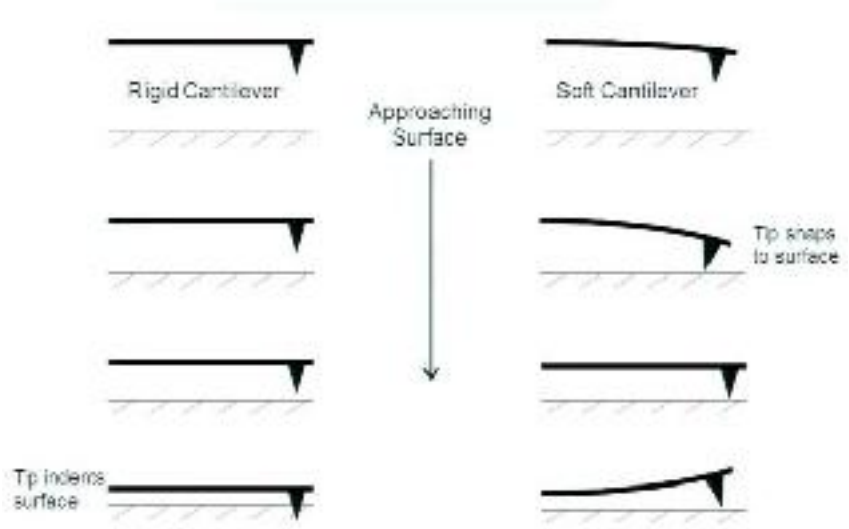
$d^* = d = \delta + Z$

Plug each (Z, δ) into these equations to convert to (d, $F_{TS}$).

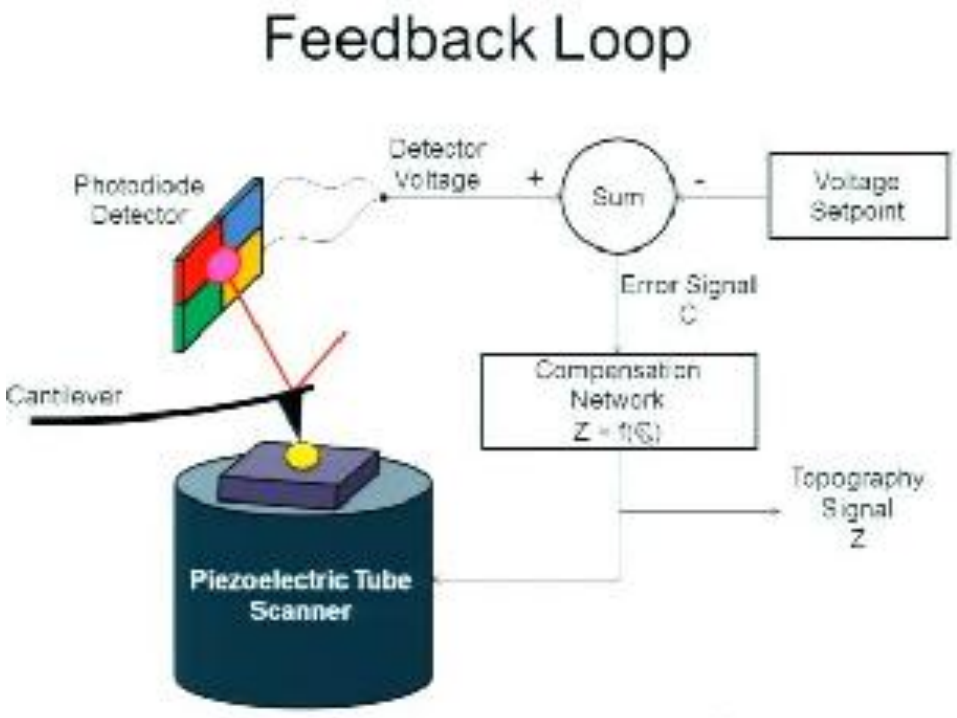
This generates a Force-Distance curve.

# 1. How AFM works



# 1. How AFM works

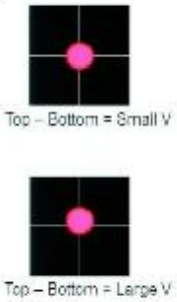


# 1. AFM modes and variants

## Modes:

### Contact Mode

- Detector: DC signal
- Voltage Setpoint: how hard/soft the tip touches surface
- Higher Setpoint = Harder Contact (greater cantilever deflection)
- Must watch for damage to sample (horizontal streaks)




### Noncontact Mode

- In noncontact mode, the instrument senses the van der Waals attractive forces between the surface and the probe that is held above the surface.
- Eliminates damage to the surface that could occur in contact mode.
- Best results are obtained in UHV since moisture on surfaces creates capillary forces in ambient conditions.

### Tapping Mode

- Also called intermittent contact mode.
- In this mode the cantilever is excited close to its resonant frequency by an external piezoelectric ceramic attachment.
- Resonant frequencies are typically from 15 kHz to 500 kHz. (Compare to rate of scanning.)
- The amplitude of cantilever vibration is usually in the 10 – 100 nm range.

## Variants of Scanning Probe Microscopy:

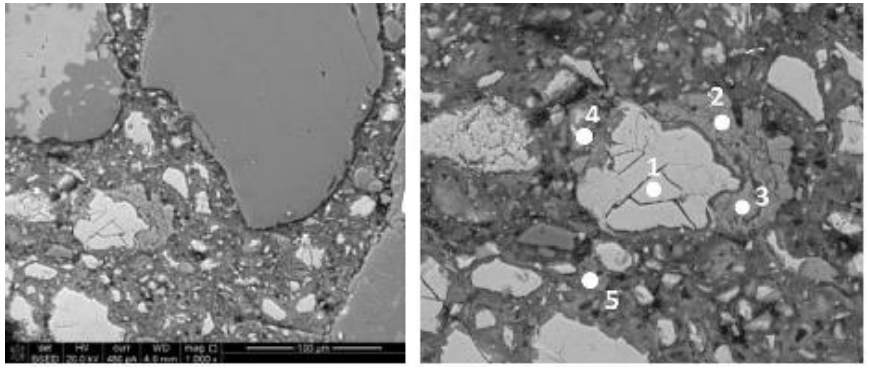
- STM – Scanning tunneling microscopy
- EFM – Electrostatic force microscopy
- LFM – Lateral force microscopy
- KPFM – Kelvin probe force microscopy
- MFM – Magnetic force microscopy
- CSAFM – Current sensing force microscopy

## Environment:

- Gases
- Liquids

# 3. Material science specific studies and examples.

Peled A et al. Atomic force and lateral force microscopy (AFM and LFM) examinations of cement and cement hydration products: Calcium-silicate-hydrate (CSH); Calcium hydroxide crystals (CH); unhydrated cement particles.

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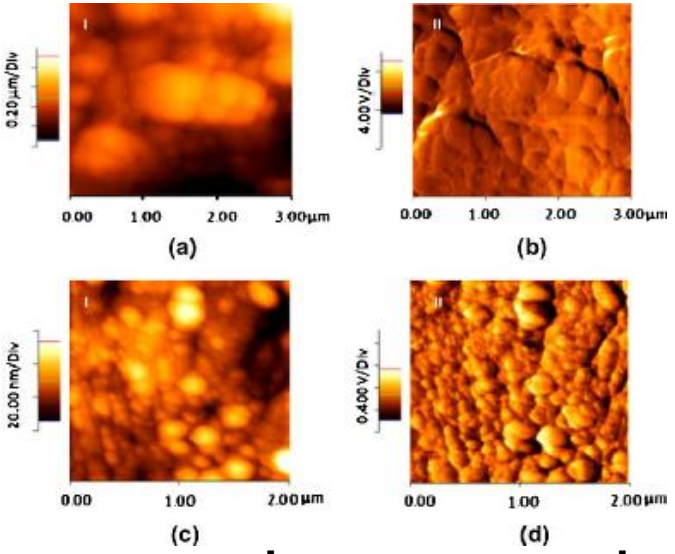



**2.**

1. *SEM image and zoomed area;*
2. *(a,c) AFM; (b,d) LFM topography*

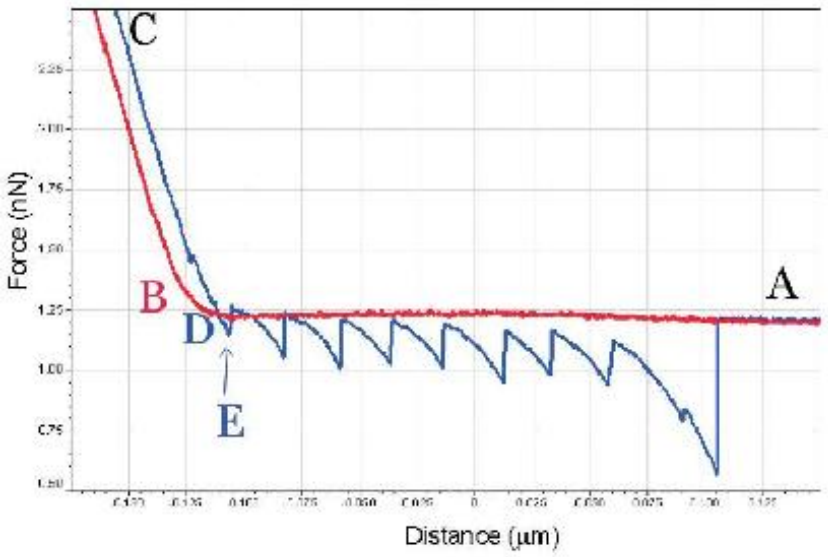
# 3. Material science specific studies and examples.

- Wenhai Han, F. Michael Serry, Force Spectroscopy with the Atomic Force Microscope, Application Note, Agilent Technologies

Atomic Force Microscope (AFM) Spectroscopy is an AFM based technique to measure, and/or control the polarity and strength of the interaction between the AFM tip and the sample.

The quantity that is measured first is always the tip-sample *force*.

In force spectroscopy, the cantilever-tip assembly acts as a force sensor.



# REFERENCES


- Peled A et al. Atomic force and lateral force microscopy (AFM and LFM) examinations of cement and cement hydration products. Cement Concrete Comp (2012)
- Wenhai Han, F. Michael Serry, Force Spectroscopy with the Atomic Force Microscope, Application Note, Agilent Technologies
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# *Opening Conference*

# SMARTMAT project

## University of Debrecen & Univerity of Oredea

***Hungarian-Romanian Research Platform for Smart Materials Research Projects Support***

*HURO/1101/191/2.2.1*

***Debrecen 15. 02. 2013***

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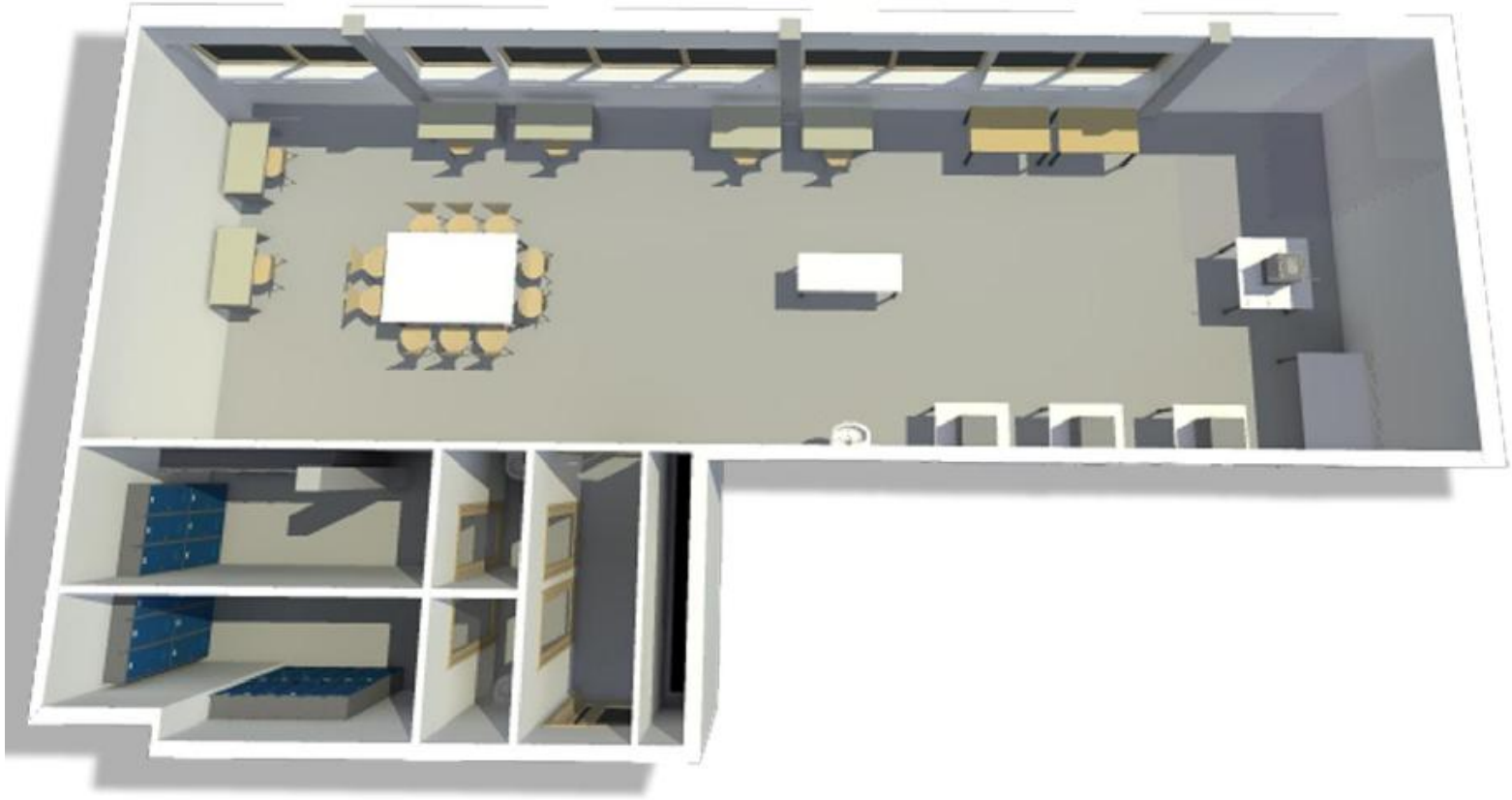

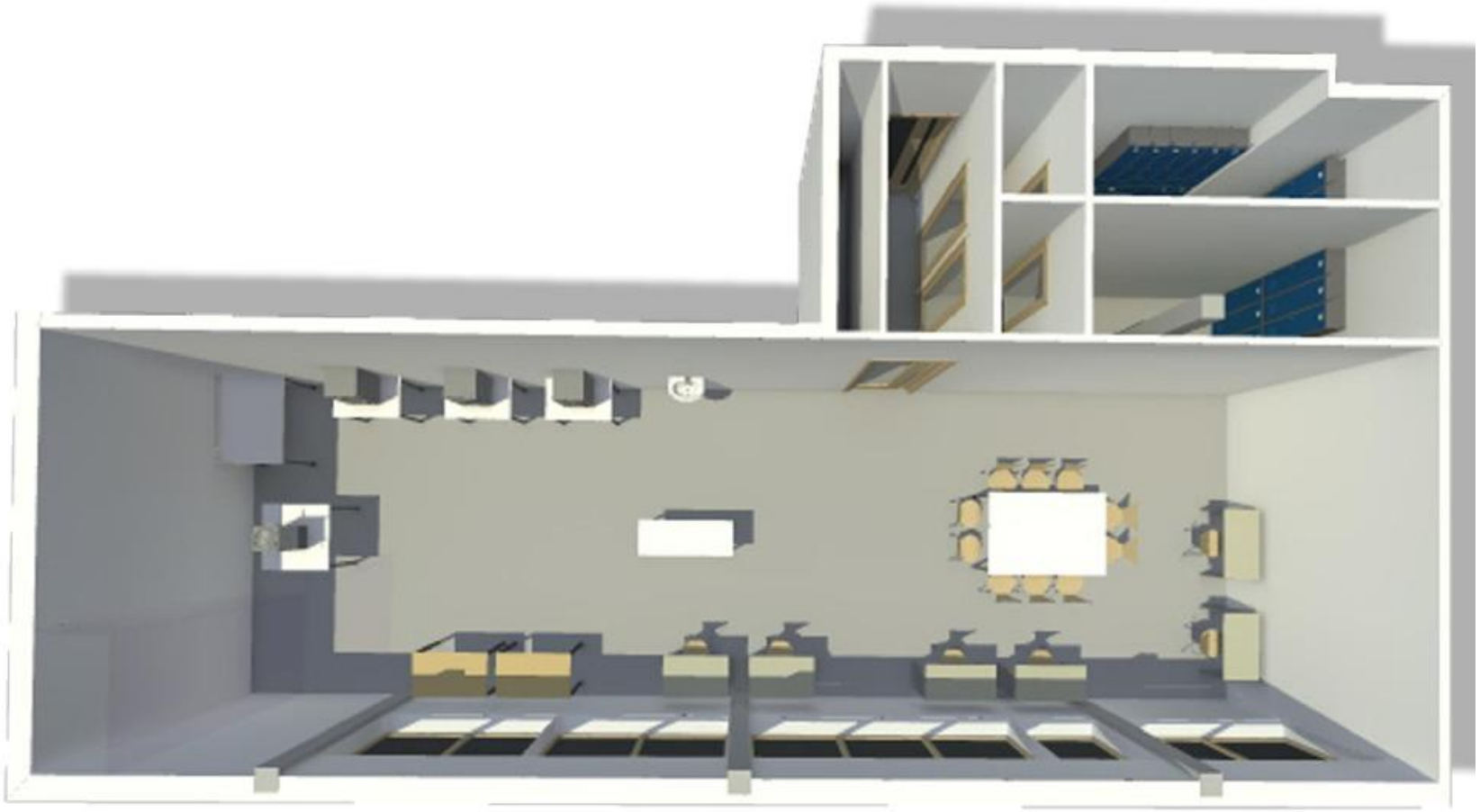

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# GO SMARTMAT

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# *Fiber Optic Sensor Technologies –*

*Prof.dr.eng. Ţarcă Radu Cătălin,*
*University of Oradea, (Romania)*

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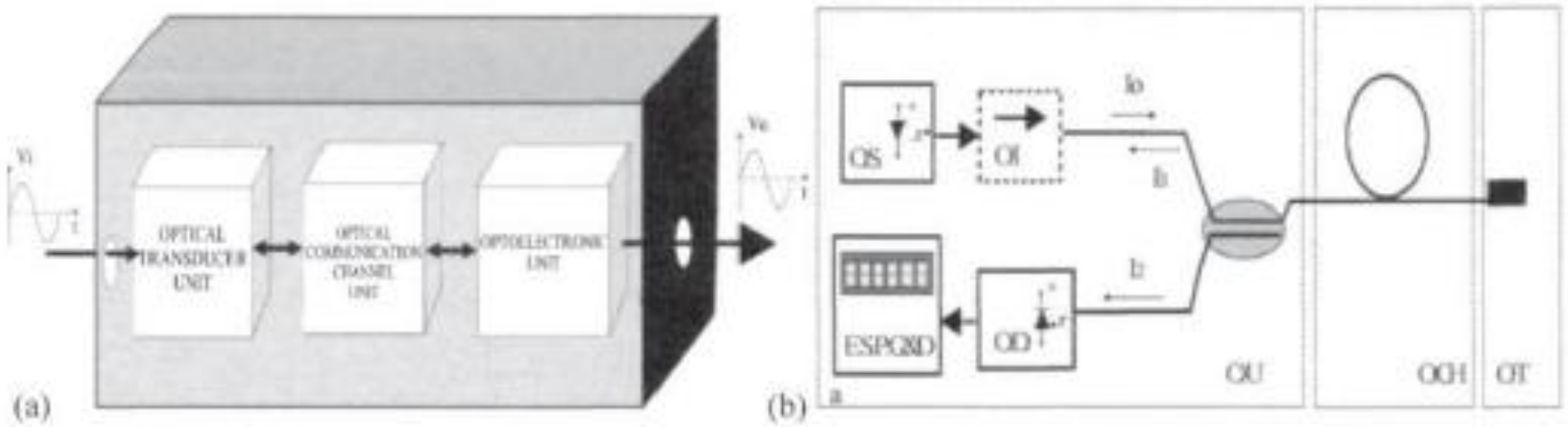
# Fibre Optic Sensing Technology

- a sensor system or sensor is usually made up of a transducer device, a communication channel and a subsystem for generating and/or detecting, treating, processing, and conditioning the signal, all of these being either integrated or not integrated. If luminous radiation is used in any of the subsystems, the photonic system is understood to be a Photonic Sensor (PS) or, as is also commonly used, Optical Sensor (OS), in which the transducer is the subsystem that usually determines and characterizes the sensor




a) Conceptual block diagram of a general Optical Sensor System; b) example of an OFS architecture. (OU: optoelectronic unit; OCH: optical channel; OT: optical transducer, OI : optical insulator, OS : optical source, OD : optical detector, ESP.C&D : Electronic signal processing conditioning and display unit).

# Optical Sensor Types

- *Domain and type of the magnitude*

a) Mechanical: Vibration including the measurement of displacement, velocity, acceleration, force and pressure. Optical effects produced by seismic masses, beams, membranes and laser velocimetry are representative examples of techniques used for the measurement of these magnitudes with or without contact. New concepts, like diffraction gratings in fibres, stimulated scattering etc. are currently being used,

b) Thermal: Temperature can be measured due to the dependence of the emission spectrum, on the absorption, the refractive index, the transmission parameters, the period of diffraction gratings, non-linear effects like scattering, etc. produced in materials and devices,

c) Electromagnetic: Electric fields, electric current and magnetic fields, can be transduced and measured, using electro-optic, magneto-optic and Faraday effects, etc. in photonic materials and devices,

d) Radiation: X-rays and nuclear radiation which produce luminescence or fluorescence effects, among others, in photonic materials and devices are used for their transduction and measurement,

e) Chemical Composition: Modifications in luminous radiation (absorption, fluorescence, decay times, etc) due to the chemical concentration are used for their transduction and measurement,

f) Flow and Turbulence of Fluids: Among other techniques, the dispersion and absorption provoked by particles in suspension, are used for their measurement,

g) Typical Magnitudes in the Biomedical field, such as acidity, oxygen, carbon dioxide, blood flow, gastric fluid flow, intracranial pressure, temperature monitoring, DNA identification, etc., are measured, among others, through techniques like absorption and fluorescence which, in addition, can be used in conjunction with neural nets.

# Optical Sensor Types

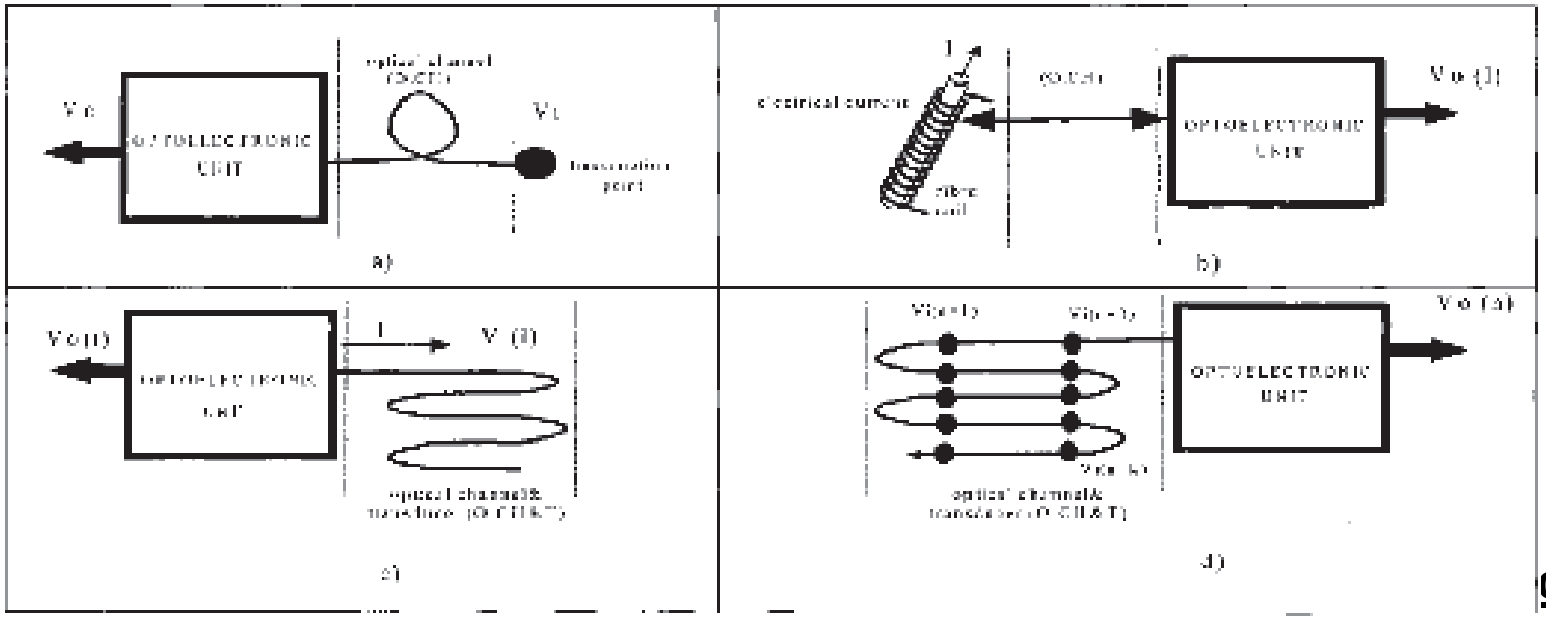
- *Spatial distribution of the measurand*

*i) Point, when the measurement is carried out at discrete points accessed by different channels.* In other words each sensor detects at only one point.

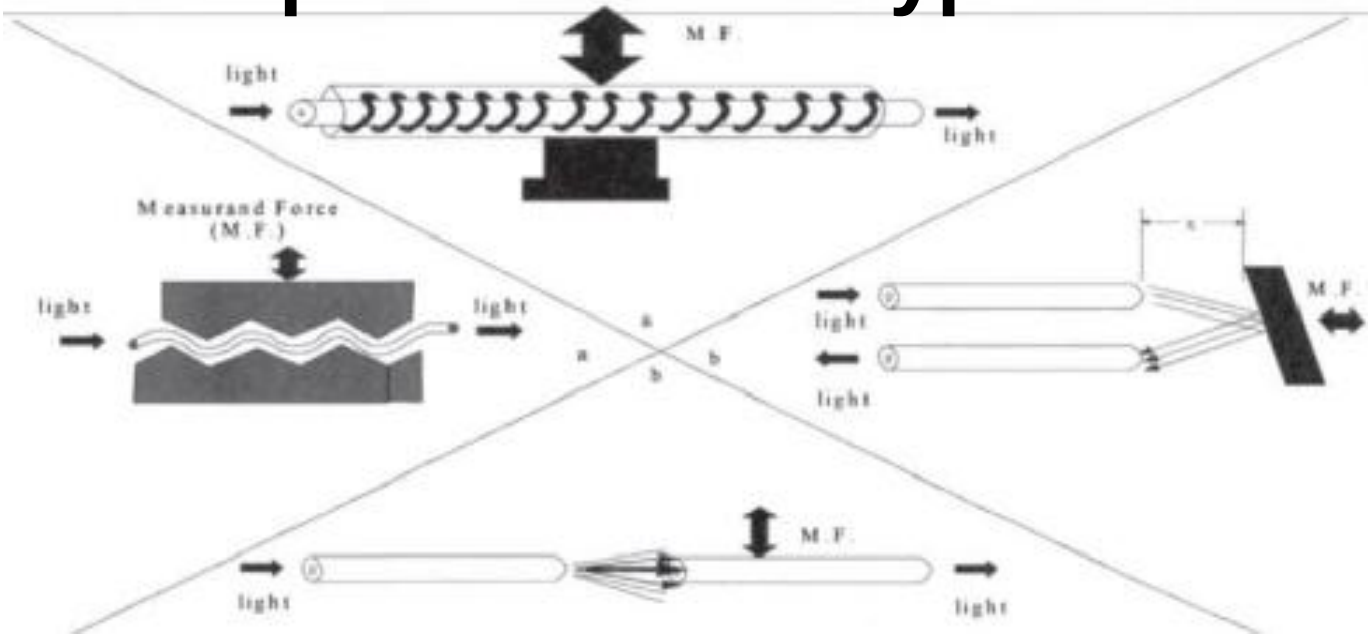
ii) *Integrated, when the measurement is integrated from all the values of the object variable* contributing to only one resultant value.

iii) *Distributed, when they can measure the value of the state of the measurand along a line of* space to be measured with a given spatial resolution.

iv) *Quasi-distributed, have the capacity to measure the value of the state of the variable at* discrete points of space situated in a single optical channel,



# Optical Sensor Types




a) Intrinsic: optical sensors/transductors in which the optical waveguide is used to carry out the transduction by modifying, in some way, its intrinsic characteristics of transmission or reflection depending on the magnitude to be measured. In this type of sensor, the luminous radiation remains totally or partially within the waveguide.

b) *Extrinsic: sensors/transductors in which the interaction between light and the measurand is* produced in an external optical device (which is affected by the magnitude to be measured) and the modulated light returned to the incoming or other waveguides. In this type of transducer the light modulation processes are carried out outside the optical waveguide.

# Optical Sensor Types

- *Manufacturing technology of the transducer*

According to the manufacturing technology of the transducer, the fibre optic sensors can be of:

a) *Optical Fibre Technology. Those where the optical devices (eg. waveguides such as those* in the previous section) are optical fibres. Sensors made with optical fibre technology,

b) *Integrated Optic Technology. Those where the optical devices (eg. the mentioned* waveguides) are made in integrated optic technology ,

c) *Integrated Opto-electronic. Those which 'harmoniously' combine photonic devices (optical,* opto-electronic), and even electronic devices for the generation and/or detection, for the treatment and/or processing and/or conditioning of the signal, all integrated on a single substrate. They can even integrate the whole set making up the sensor system and,

d) *Hybrid Optic, Technology. Those which harmoniously combine photonic and/or electronic* parts or devices in different technologies (fibre and/or integrated) on the same substrate. They can also completely integrate the whole set constituting the sensor system. This group can also include those types of sensors that, whilst being conventional, use the optical fibre as a communication channel to supply the energy and provide the communication channel. These are called optically tele-fed and tele-interrogated sensors.

# Where should Optical Fibre Sensing Technology be Used?

a) Measurement and monitoring in environments with strong electromagnetic radiation: transformers, alternators, electric motors, induction and microwave ovens, power supply lines etc.

b) Areas or spaces and components in which the nuclear radiation can be dangerous for health, like in nuclear power plants.

c) In aerospace and transportation in the new generation of aircraft for both the communication networks and sensor devices due to the high bandwidth low weight and the immunity to electromagnetic fields (EMI) and the avoidance of undesired electromagnetic contamination (EMS).

d) In medicine and biotechnology for the performance of 'in-vivo' medical measurements such as pressure, analysis and pH of blood, temperature, vision, surgical etc. There is a special use in radiotherapy thanks to EMI and its capacity not to produce EMS.

e) Point and multipoint measurement and monitoring of chemical compounds (methane CH4, carbon monoxide CO, carbon dioxide CO2, ozone O3, oxides of nitrogen, chlorines or fluorine compounds etc). Thanks to the use of photonic nets which can be situated in representative points or points of interest in the chemical, petrochemical, mining or environmental protection Sectors.

f) Applications which require great sensitivity to the object variable such as accelerometers, hydrophones, gyroscopes and electric and magnetic field measurement devices.

g) Distributed and quasi-distributed sensing of variables (temperature, cables, fibre-optic communications cable networks, CATV etc.). Other uses include civil and geotechnical engineering structures, construction, alternator windings in large electrical generation machines, aeronautical structures, in buildings and others

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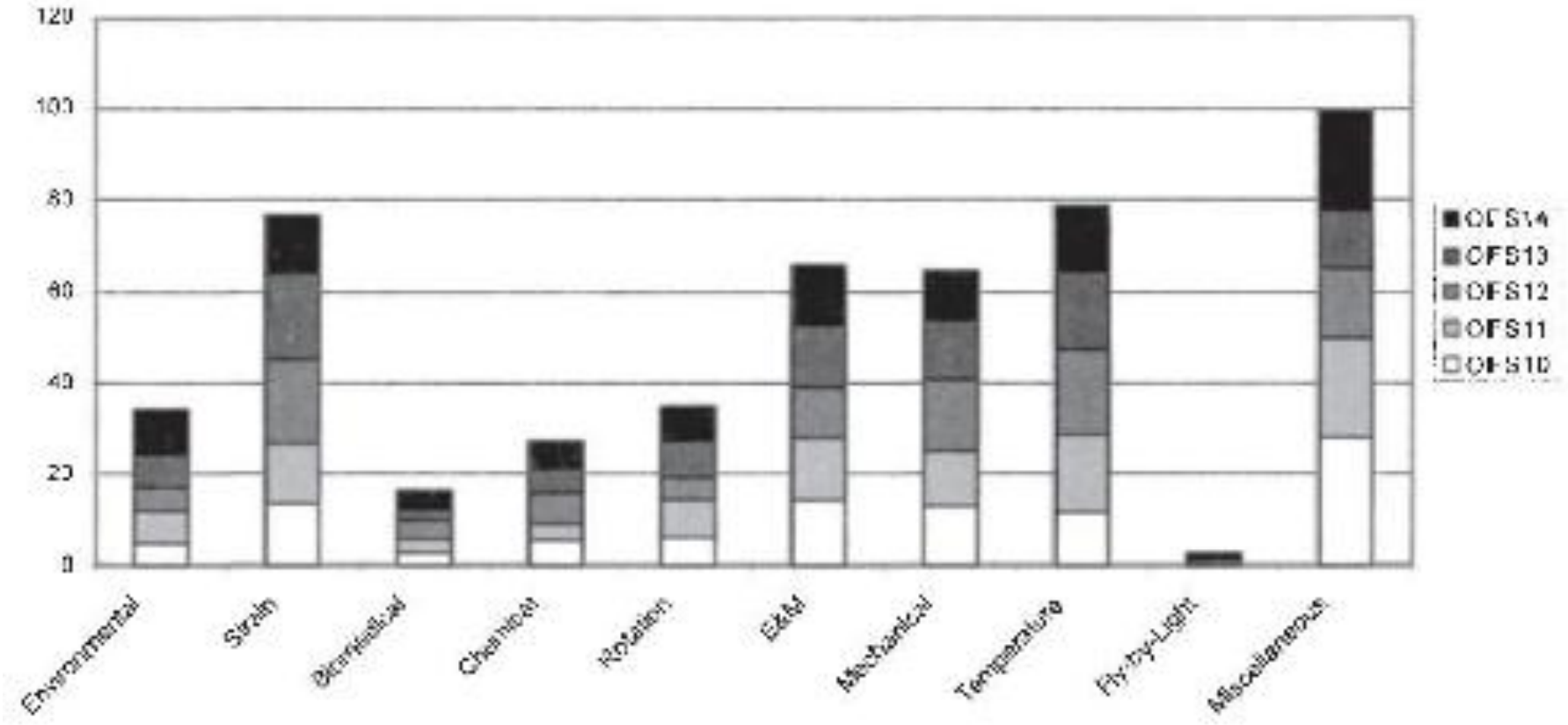


- R&D results: academic publications, per topic area, presented in the last five OFS international conferences.

# Optical Fibre Sensing Technology at University of Oradea

- Two type of systems were acquired in last year:
- **Jaz platform from OCEAN OPTICS**
- **NI PXIe-4844 Optical Sensor Interrogator for Fiber Bragg Gratings**


# Jaz Modular Spectroscopy

- Jaz® – an Ocean Optics product – is a modular measuring suite.
- Jaz is a community of stackable, modular and autonomous instruments that combine to create the ultimate in smart sensing for lab, field and anywhere your work takes you.
- Jaz is unfettered by the limits of traditional optical sensing instrumentation. Its unique features and expandable platform make it uniquely suited for field applications, remote sensing, process flow, quality assurance and more.
- Jaz brings an unparalleled level of flexibility, with its replaceable slits.

Ocean Optics

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### Jaz Spectrometer Module
- Benchmarked to the performance standards of the USB2000+ Spectrometer
- 200-1100 nm Sony ILX511B CCD array detector
- Multiple gratings available

### Jaz DPU Module
- 128 x 64 OLED display
- Choice of two screen/button orientations
- Embedded microprocessor for data processing
- Custom programmable scripting

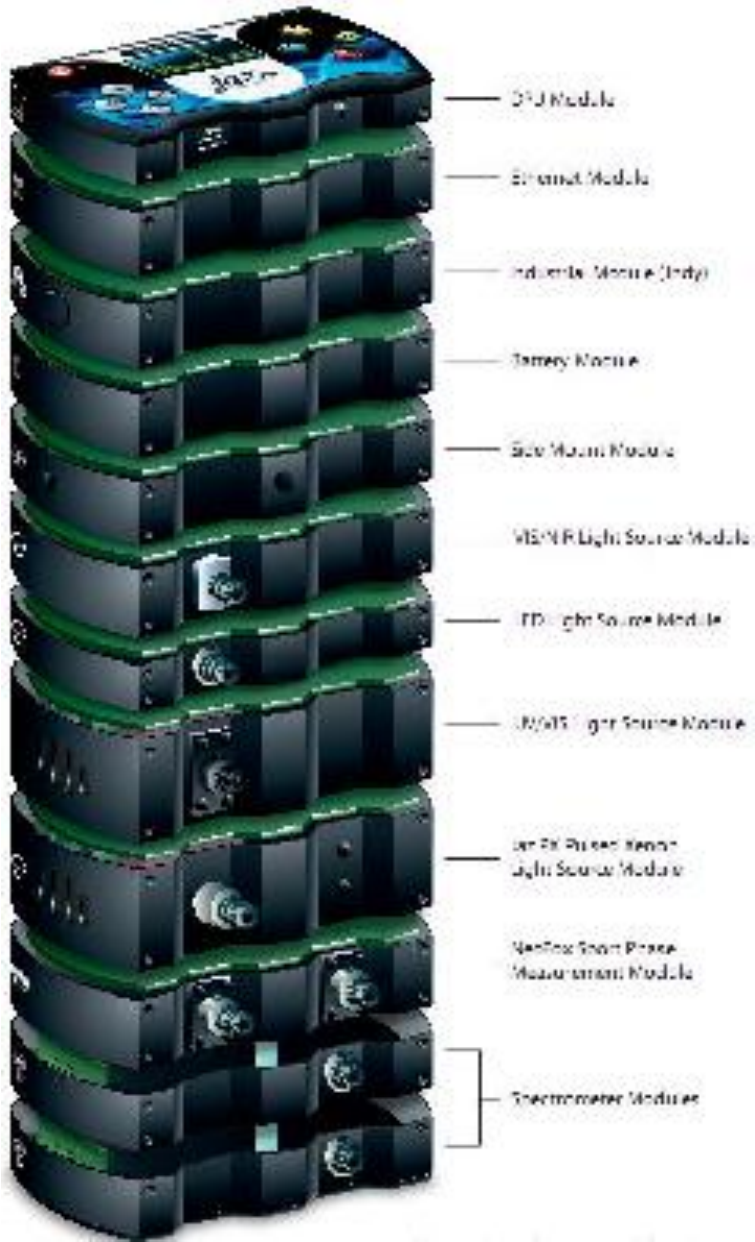
### Jaz Ethernet Module
- 100 Mbps, IEEE 802.3 compliant connectivity
- SD card slot
- Enables remote access via any computer on the same network
- Single-cable solution that provides PoE

### Jaz Industrial Module
- Communicates with RS-232 and RS-485
- Provides analog and digital inputs/outputs

### Jaz Battery Module
- Lithium-Ion battery for up to 8 hours of use
- Rechargeable from Ethernet, USB or external power supply
- 2 SD card slots for memory and other functions

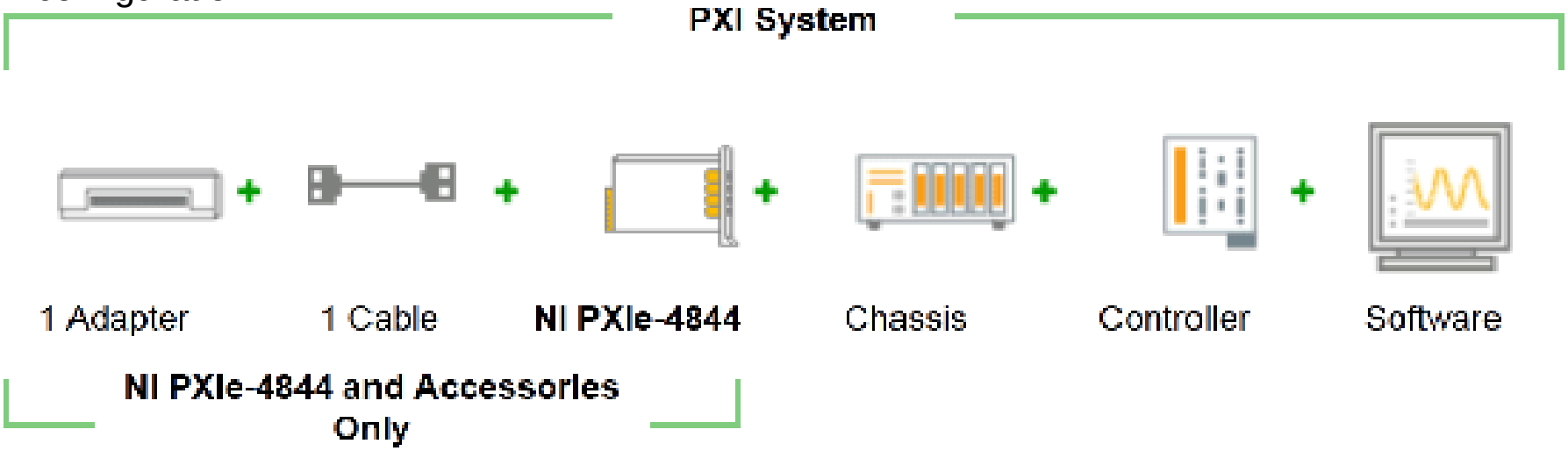
### Jaz Light Source Modules
- VIS-NIR Tungsten Halogen
- UV-VIS Deuterium-Tungsten Halogen
- Pulsed Xenon
- LED options

### Jaz Side Mount Module
- Lets you affix Jaz to tripod, breadboard and more
- Has ¼"-20 threaded mounts on three sides of the module



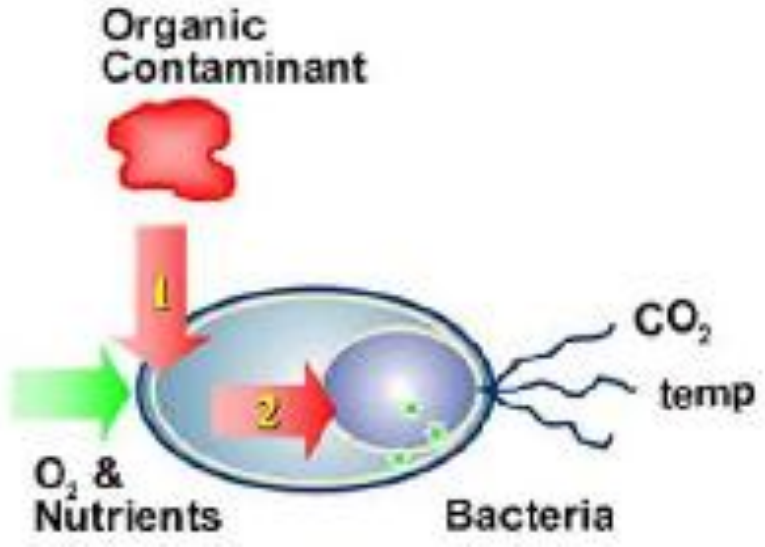
# NI PXIe-4844 Optical Sensor Interrogator for Fiber Bragg Gratings

- 4 optical channels, 10 Hz full spectrum scan frequency, 1510 to 1590 nm wavelength range
- FBG sensors are nonconductive, electrically passive, and immune to EMI
- Daisy chain dozens of sensors on a single fiber over long distances (over 10 km)
- Compatible with extrinsic Fabry-Perot, long-period grating (LPG), and other optical sensors
- No external calibration required with onboard NIST-traceable reference
- NI-OSI LabVIEW driver and NI-OSI Explorer for automatic sensor detection and configuration



# Developed applications at University of Oradea

- Integrated system of monitoring the temperature and $CO_2$ level to monitoring the bacteria growth in the project “Realization of Hungarian-Romanian R&D Laboratory for the Development of Major Projects in Polluted Terrain Cleaning” HURO/0802/100

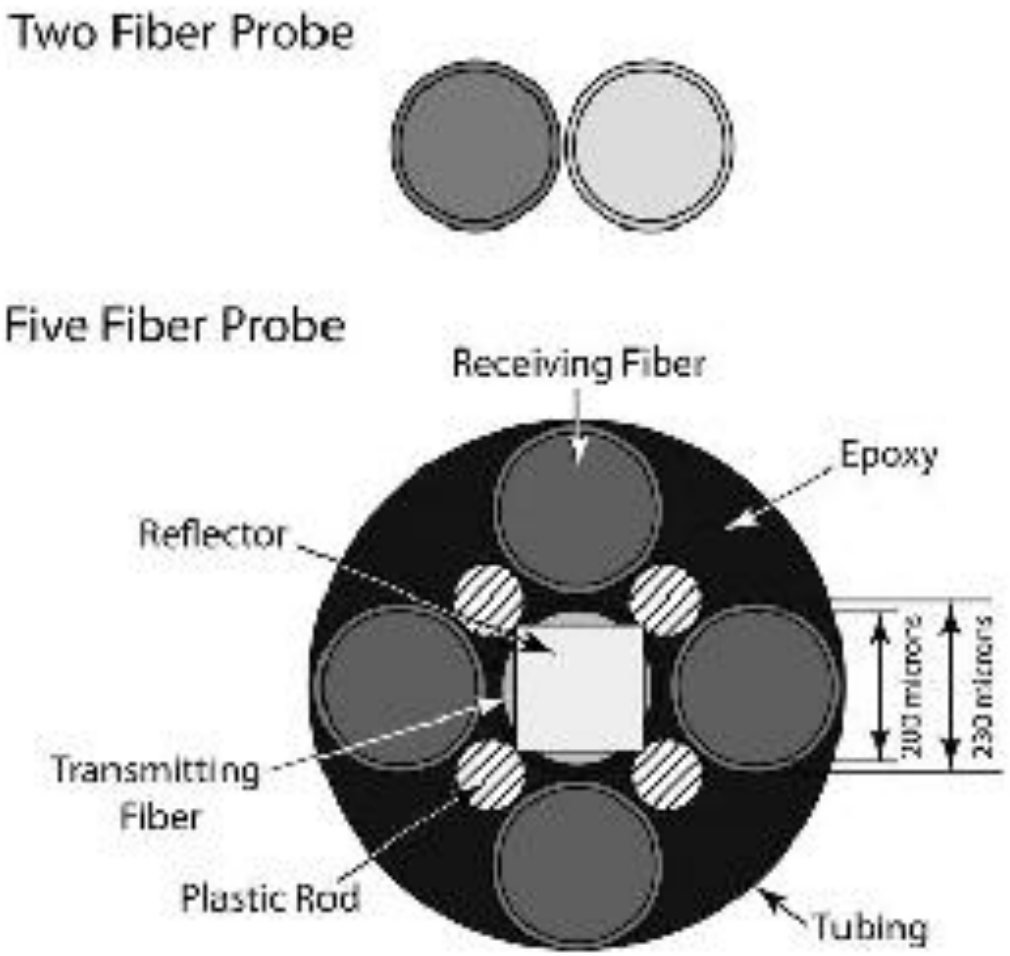


# Developed applications at University of Oradea

- new optical fiber sensors and the software of data acquisition to monitories fermentation and hydrolysis reaction in pure and real systems - in the project “New method and system for real time fermentation process monitoring” HURO 1001/121/2.2.2- BIOETHANOL;

- We are working to determine in real time the kinetic of starch hydrolysis reaction, the rate of ethanol fermentation and the concentration of final products was used (UV-VIS) **Ultraviolet–visible spectroscopy** .
- Was developed a system for ethanol concentration monitoring system using artificial neural networks.

# Developing a cantilever-probe directional sensor



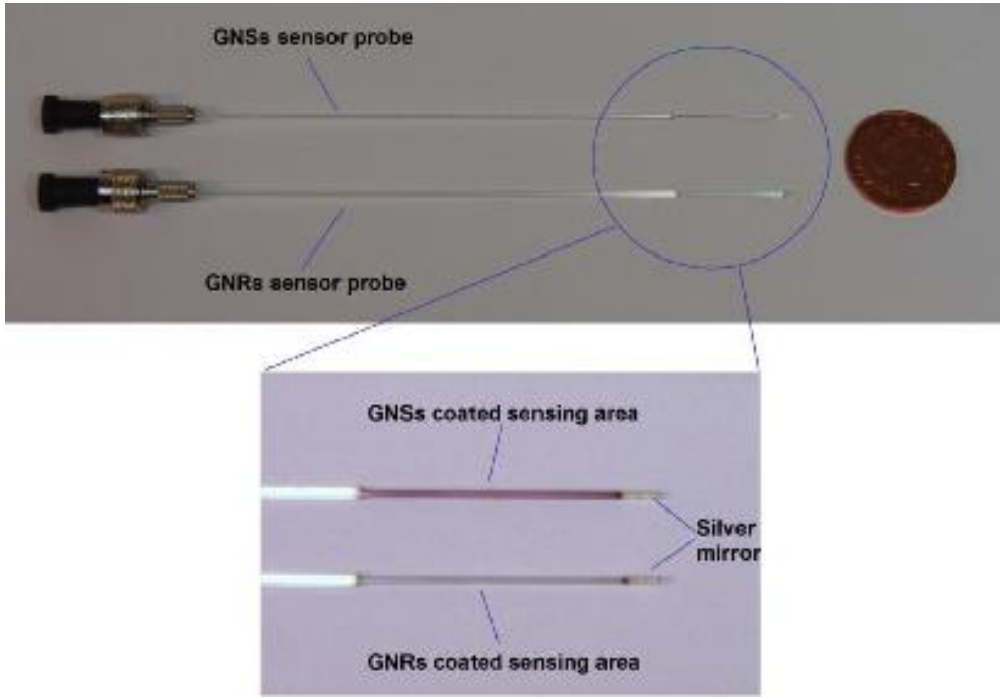

Cross section of the two-fiber probe (upper) and the design for a proposed five-fiber probe for displacement measurement in two orthogonal directions (lower). The reflector is shown only for the five-fiber probe. (The five fiber design effectively has two two-fiber probes along each axis.)

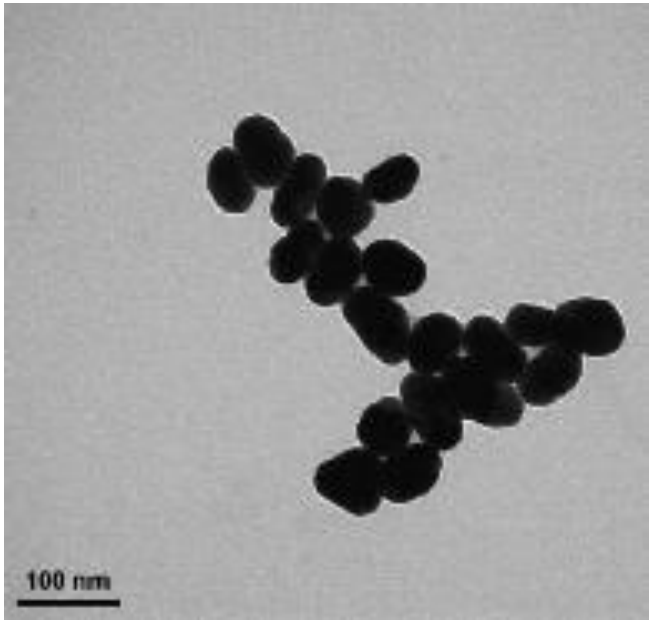
## Wavelength-based localized surface plasmon resonance optical fiber biosensor

- Jie Cao, Minh Hieu Tu, Tong Sun, Kenneth T.V. Grattan

*Sensors and Actuators B 181 (2013) 611– 619*

Two types of localized surface plasmon resonance (LSPR)-based optical fiber biosensors using gold nanospheres (GNSs) and gold nanorods (GNRs) have been developed and their performance characteristics evaluated and cross-compared successfully in this paper.



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# SuperDog

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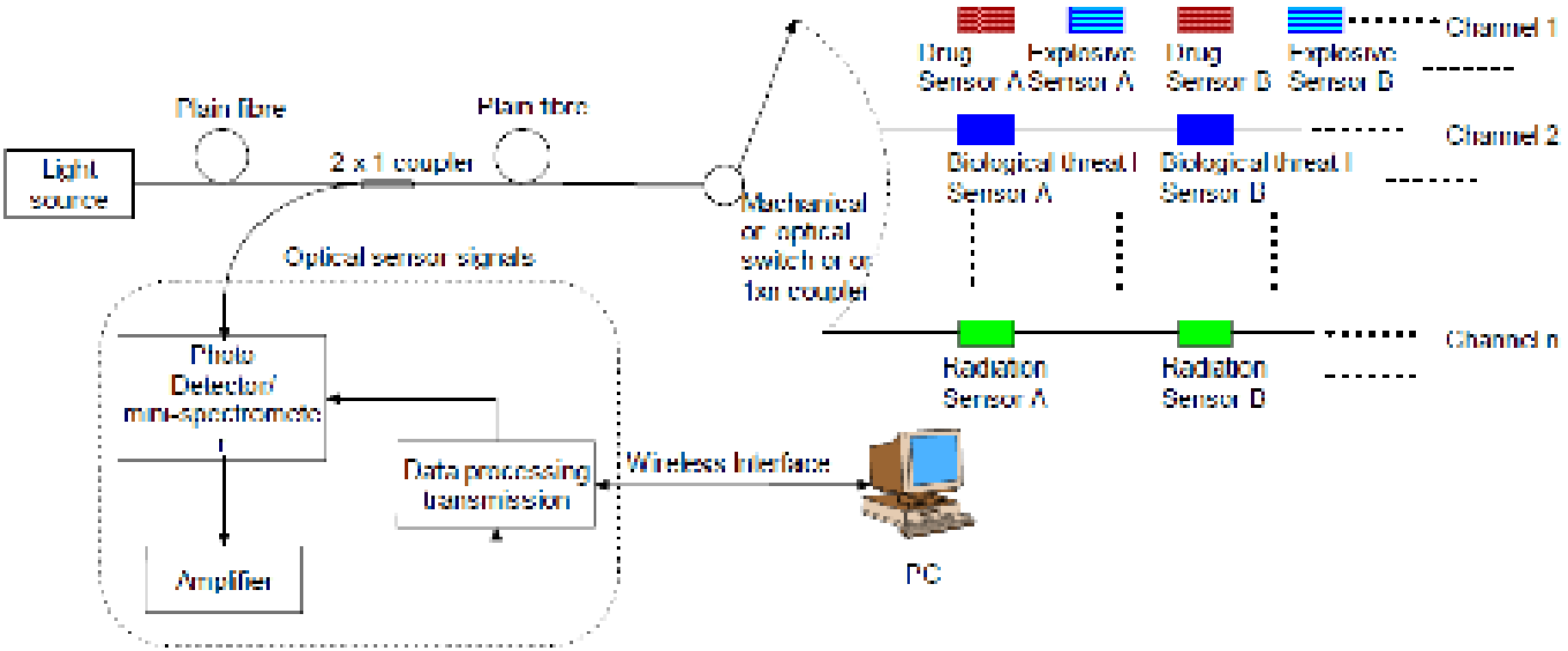
SUPERDOG concept is its inherent integration of creating critical sensor performance with robotic control/navigation both to allow an optimized (and flexible) 'sniffing' strategy to be realized and to enable the most effective communications with the security personnel.

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# *Workshop*

# SMARTMAT project

## *Hungarian-Romanian Research Platform for Smart Materials Research Projects Support*

*HURO/1101/191/2.2.1*

***Baile Felix 29. 05. 2013***

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# The part of project aims

• To develop a joint Hungarian – Romanian research platform in the field of smart and other new materials which will support the development of fundamental research projects.

• We would like to purchase a plastic deformation Final Element Method (FEM) softwares. Which can be used to modelling the large deformations and thermal testing.

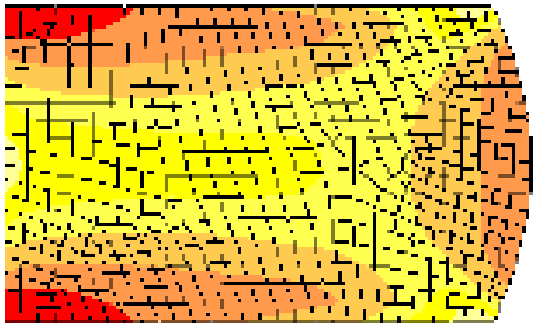
# Softwares

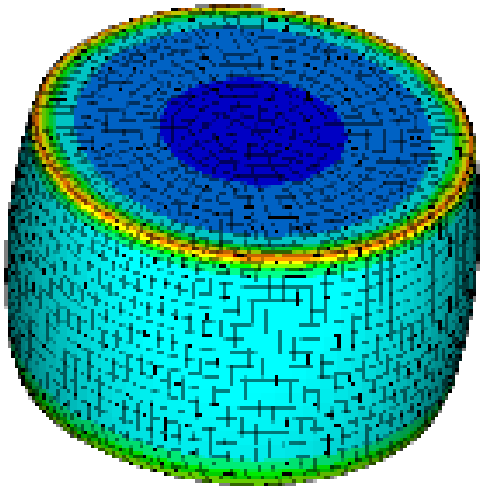
Our goal is to obtain the research (PhD), education (thesis, diploma work) is a well-fitting software, which demands the utmost covers.

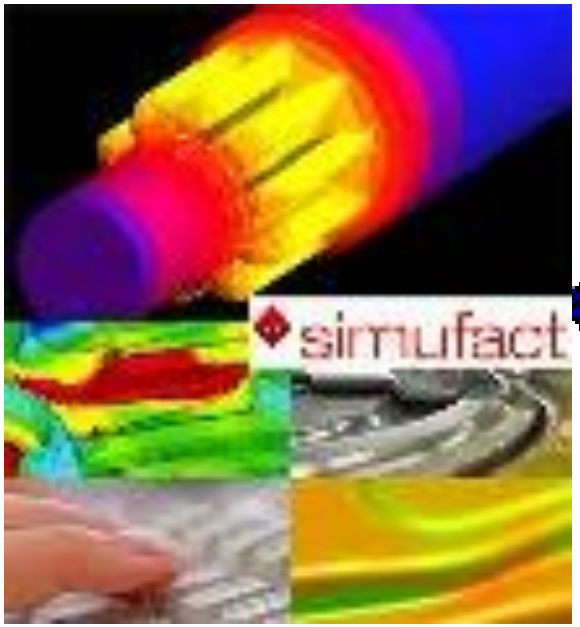
MSC Software

simufact

AUTODESK.



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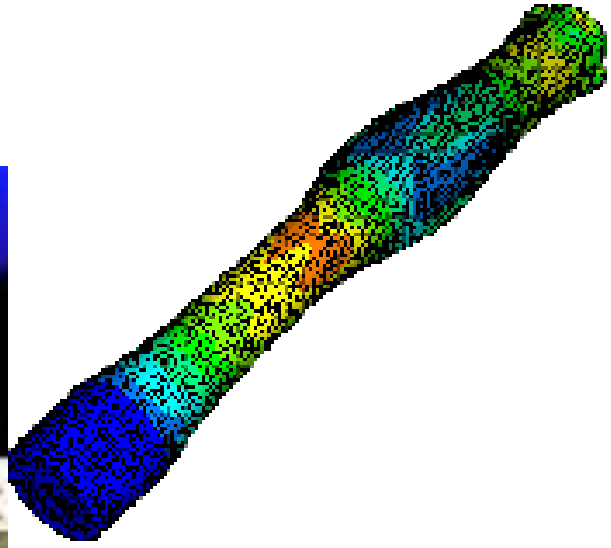

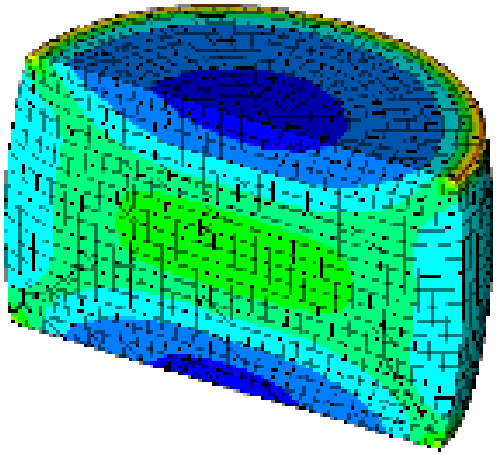

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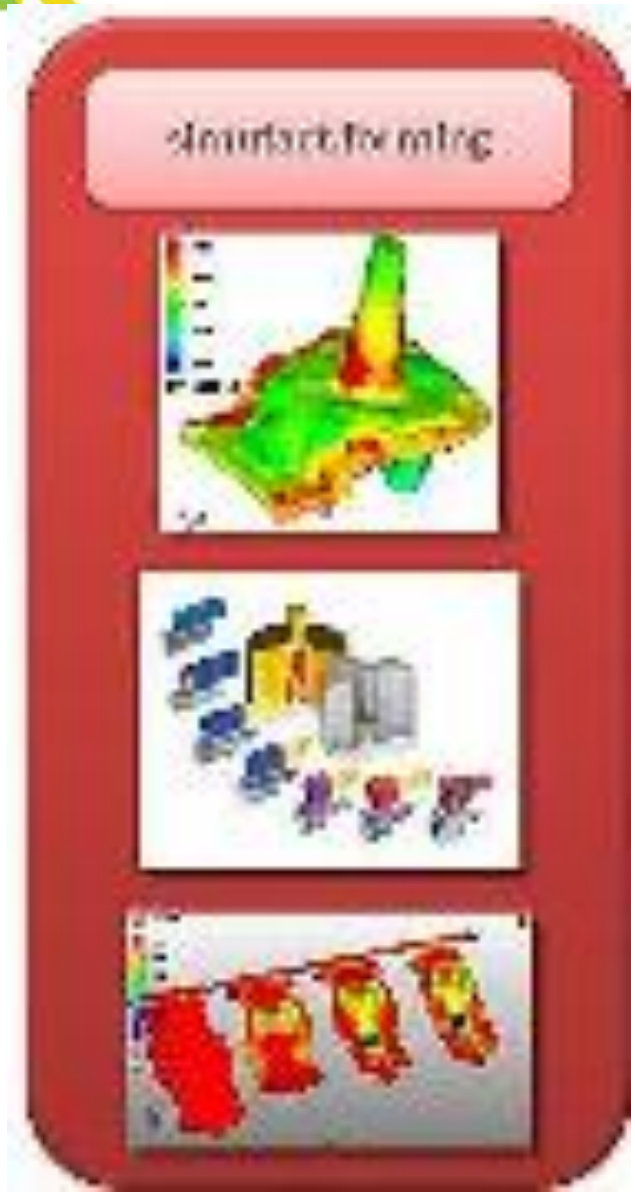

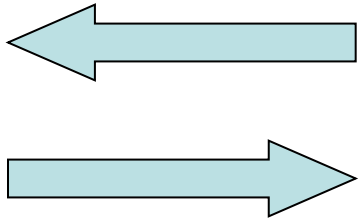



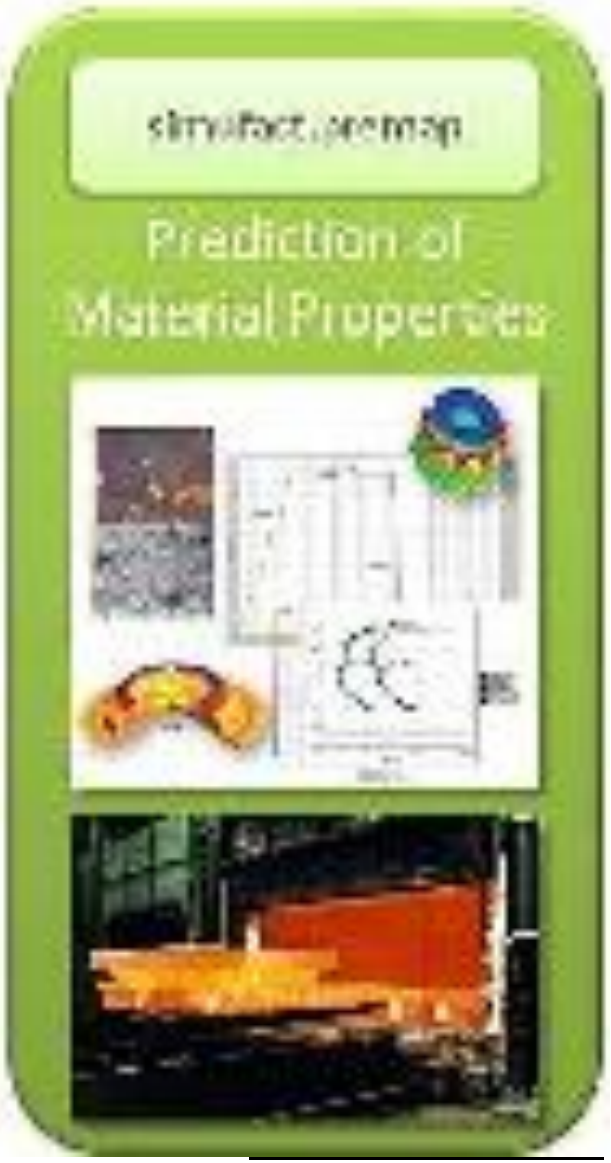

# Thank You for your attention!

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# Presentation of SMARTMAT Laboratory in University of Oradea

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# Activities of Project Partner Team

List of activities:

- Preparation activities
- Project management
- Communication activities
- Upgrade of laboratory infrastructure
- Acquisition of laboratory equipment
- Integration of laboratories in the research platform

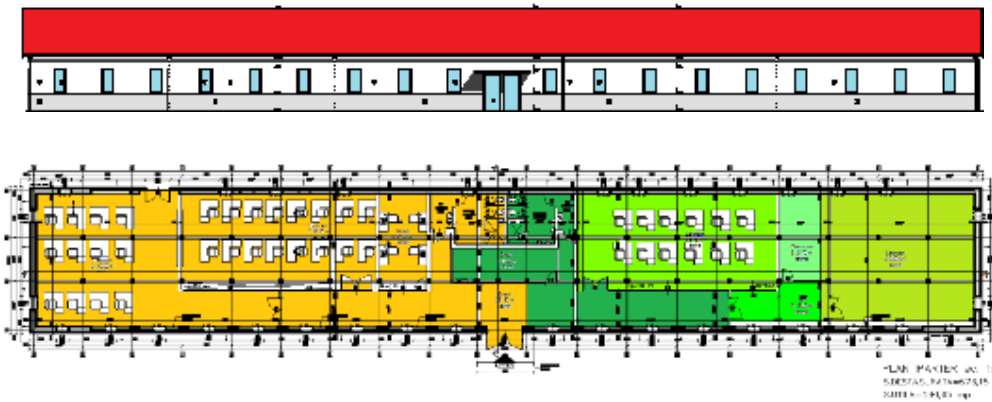
# Activities of Project Partner Team: Communication activities

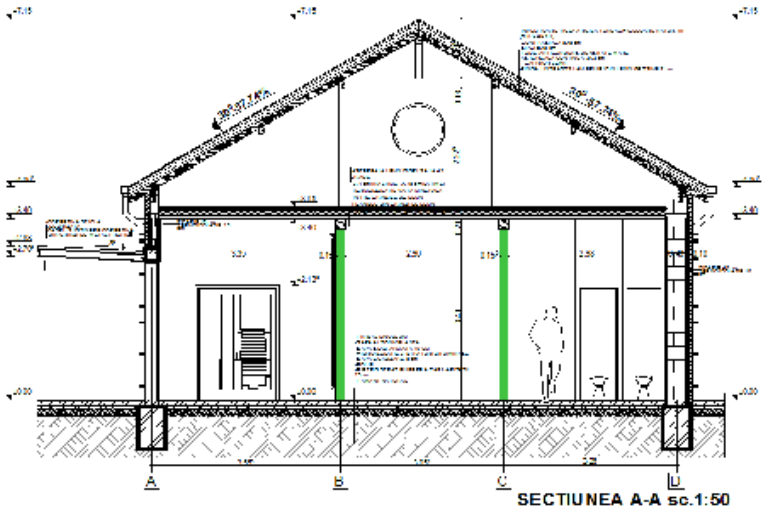
1. Project webpage design
2. Participation at Opening Day in Debrecen
3. Placement of visibility billboard



# Activities of Project Partner Team: Upgrade of laboratory

1. Tender for building design
2. Design of building upgrade
3. Tender for building upgrade
4. Building upgrade works





# Activities of Project Partner Team: Acquisition of laboratory equipment

1.Meetings and workshops
With equipment suppliers:
**MDT, Agilent, Park, Keyence.**
2. Received information from: **Asylum, JPK Instruments, Schaefer (AIST Nano)**




# Activities of Project Partner Team: Acquisition of laboratory equipment

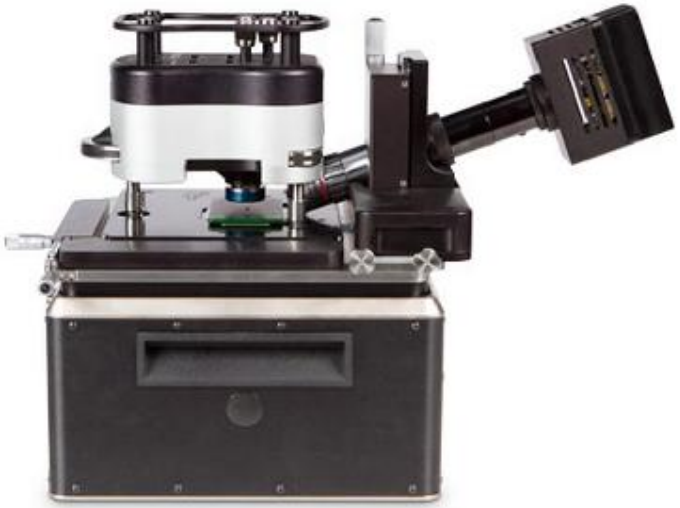
- Park Systems: NX10


# Activities of Project Partner Team: Acquisition of laboratory equipment

- Agilent: 5500 SPM & G200 Nanoindenter

# Activities of Project Partner Team: Acquisition of laboratory equipment

- Asylum Research: MFP 3D



# Activities of Project Partner Team: Integration of laboratories in the research platform

To establish:

- Cooperation protocols
- Platform management structure
- Communication between laboratories

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# *Workshop ONE*

# SMARTMAT project

## University of Debrecen & Univerity of Oredea

***Hungarian-Romanian Research Platform for Smart Materials Research Projects Support***

*HURO/1101/191/2.2.1*

***Oradea 29-30. 05. 2013***

www.hungary-romania-cbc.eu
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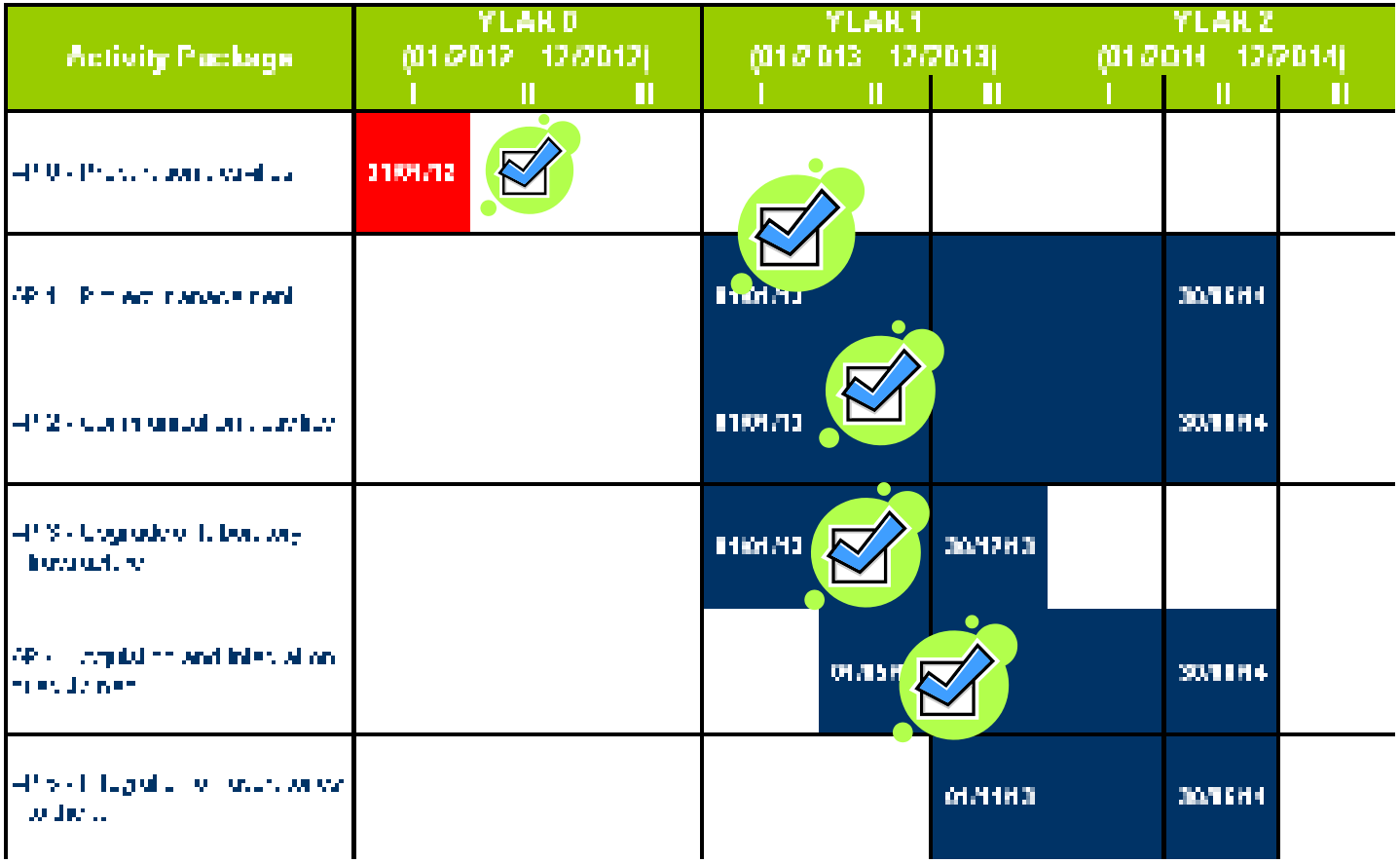
# The project aims

• To develop a joint Hungarian – Romanian research platform in the field of smart and other new materials which will support the development of fundamental research projects.

• The platform will integrate 3 laboratories, 2 at the University of Debrecen with the purpose of investigating smart and composite materials at **macro**-, and **micro**-scales and 1 laboratory at the University of Oradea where **nano**-scale properties can be analyzed.

# Timeframe of project



# AP0. Preparation activities

Results and outputs :

- Meeting record (minute) about staff meeting
- Study regarding the state of the art of composite and nano materials and the relating equipments
- Fesability study
- Signed Documents (Application Form + Annexes)

# AP1. Project management activities

Results and outputs :

- ERDF Subsidy Contract
- Financial reports
- Supervising documents of the activities
- Progress reports
- Verification of the fulfillment of the assumed duties

# AP2. Communication activities

I. MARKETING

1. Webpage: It is important to have an own webpage of the project, where all the important information can be found. We are planning to refresh the webpage continuously with news and research results regarding to the project.

2. Brochure. It is necessary to publish brochures, because these tools have an important role in communication.

II. PR TOOLS

1. Organizing opening and closing events. The aim is to increase the background knowledge of the researcher, students and media, and to generate public appearance.

2. Organizing workshop – appearance on professional events. An important factor of the efficient communication is that the professional interested parties meet with the dissemination of project activities and research results. 3. Communication materials-tables, plaques.

# AP3. Upgrade of laboratory infrastructure

Results and outputs

- Preparing technical specification of laboratories

- Preparation of public procurement documents and procedures IN PROGRESS ➡ 07th JUNE

- Realization of laboratory works and verification of work conformity with technical plans.

# AP4. Acquisition and integration of equipments

Results and outputs

- Monitoring the availability of required equipments.
- Studies regarding equipment availability on the market

- Public acquisition, preparing of documents and procedures. Generating equipment specification and compliance with procedures

- Commissioning, testing, verification, validation of equipments

# AP5. Integration of laboratories into the SMARTMAT research platform

**SMARTMAT scientific website and database** –
electronic communication interface between laboratories, platform management, external partners and wide public;

**SMARTMAT project website** –
will be still functional after project termination, disseminating information about research project results, open to the wide public;

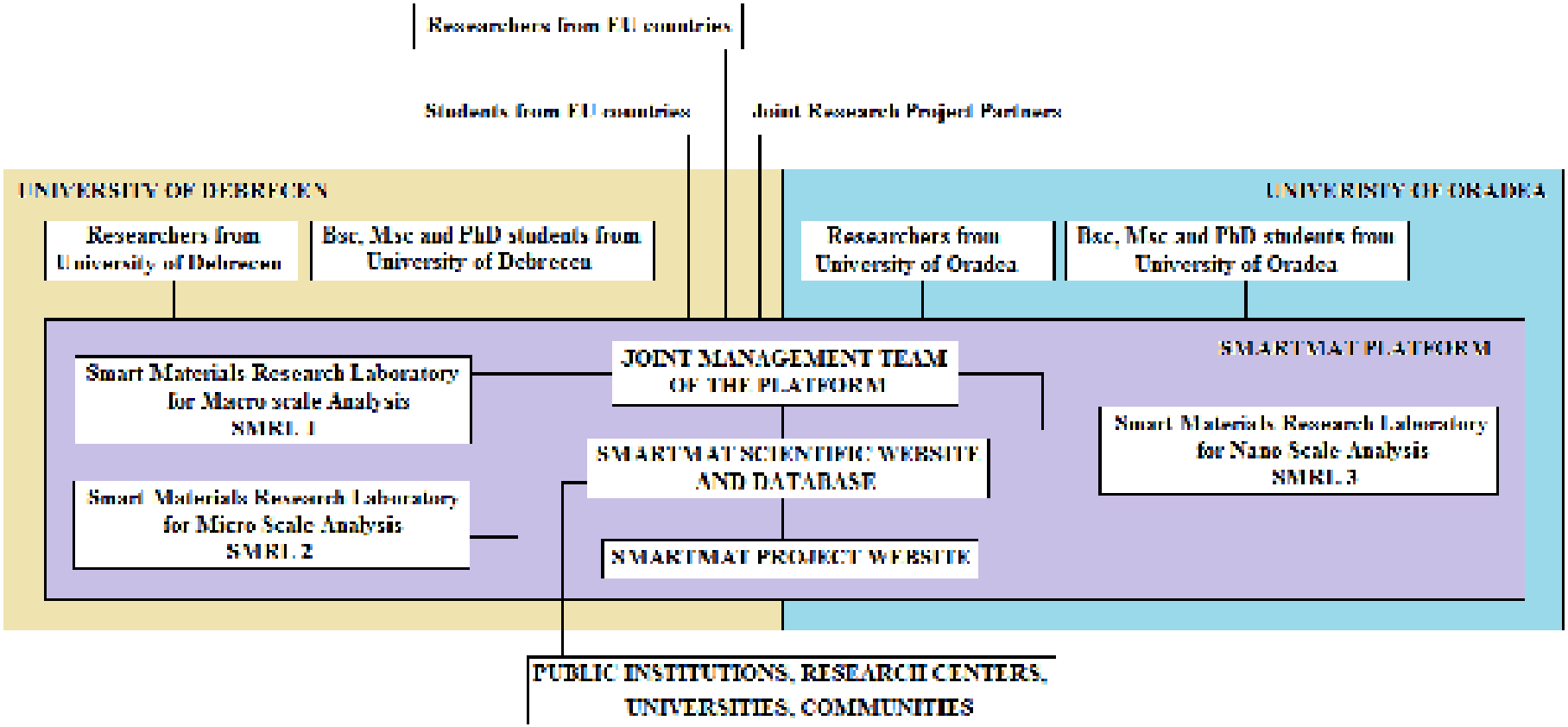
**Smart Materials Research Laboratories –** operation, equipments, staff;

**Internal research and education partners –** how they can access platform resources;

**External research and education partners –** how they can access platform resources;

**Joint research project partners –** public institutions, research centers, universities

# SMARTMAT research platform



# SMARTMAT LABS

## Smart Materials Research Lab for Macro scale SMRL1-HUN

**500 kN ELECTROMECHANICAL TESTER**
**30 TN HYDRAULIC FLEXURAL TESTING FRAME**
**RapidAir 457 system for image analysis**
**ZK 75 HE Zyklos Concrete mixer**

## Smart Materials Research Lab for Micro scale SMRL2-HUN

**TableTOP Electron microscope + EDX detector**
**Samlpe preparation for SEM**
High temperature rheology measurement system for viscosity measuring of liquid metals and vacuum system
**Software for deformation modeling and simulation**

## Smart Materials Research Lab for Nano scale SMRL3-ROM (in a new clean room)

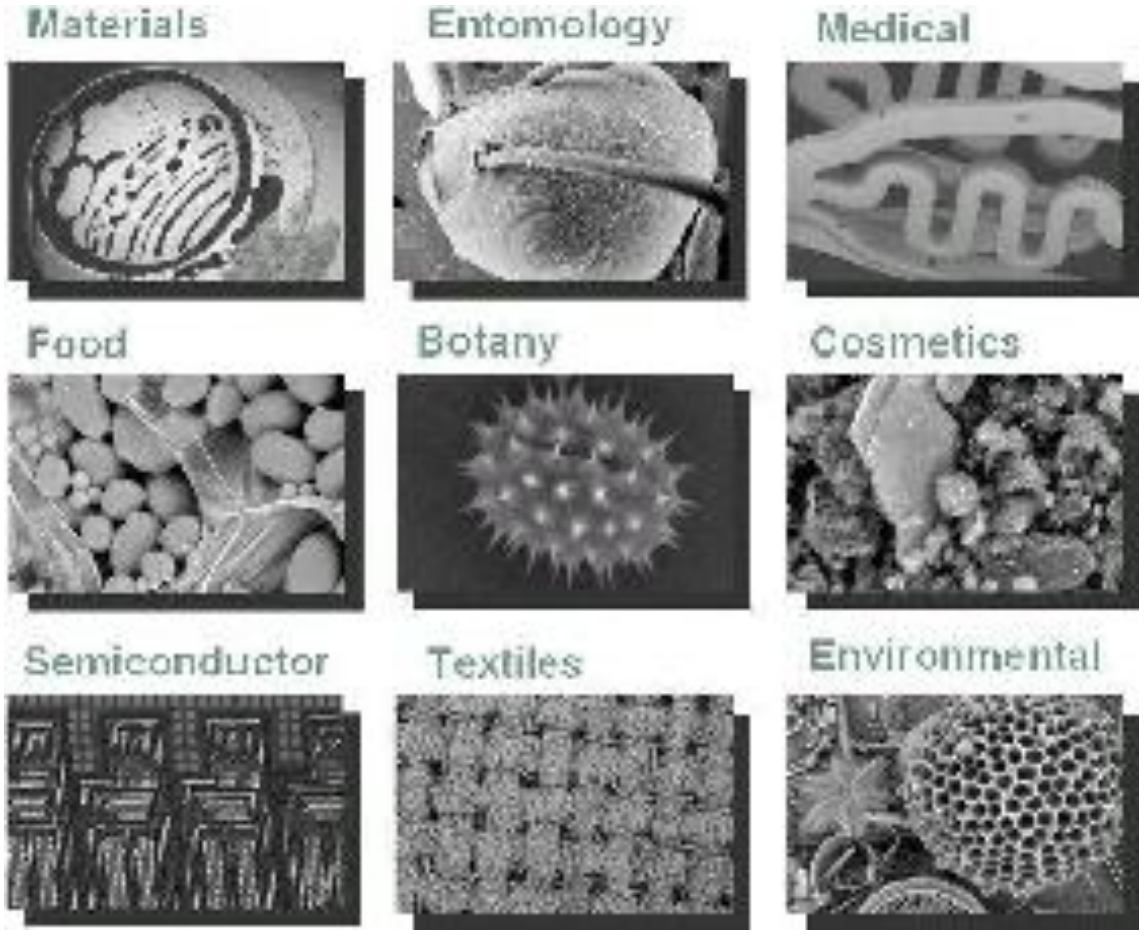
Atomic Force Microscope and accessories
Atomic Absorption Spectroscope

# SmartMat Research Lab for Micro scale - SMRL2

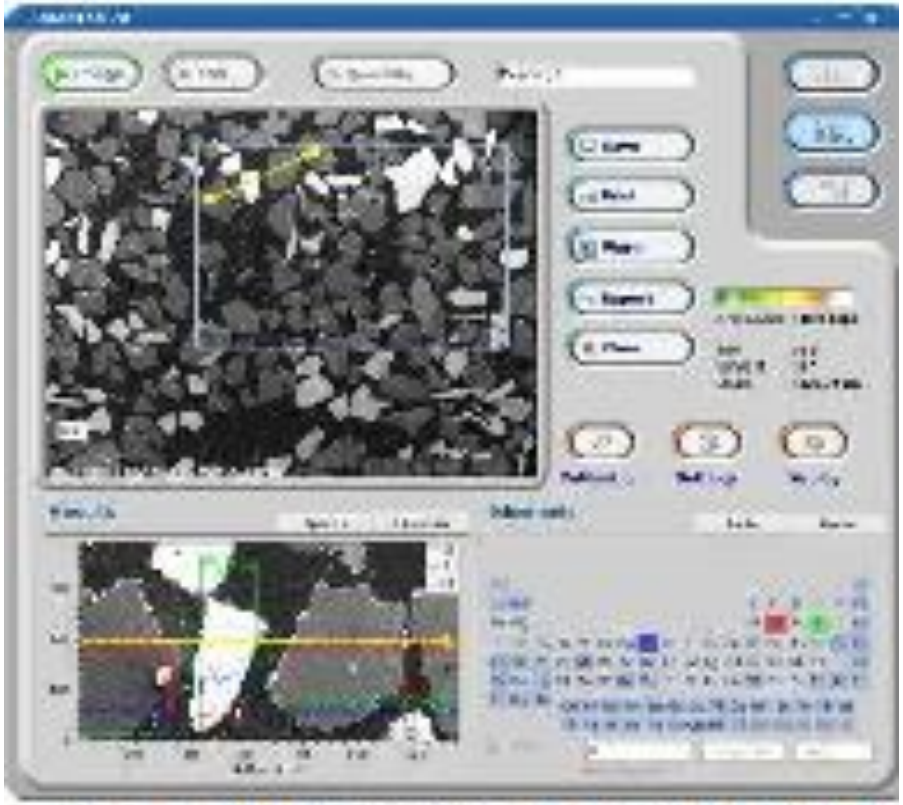

## TableTOP SEM + EDX

**It's simplicity enables all users, even those Scanning Electron Microscopy (SEM), to image their samples and to obtain high quality, high resolution images within minutes. No special sample preparation such as coating with metal films is required for hydrated**

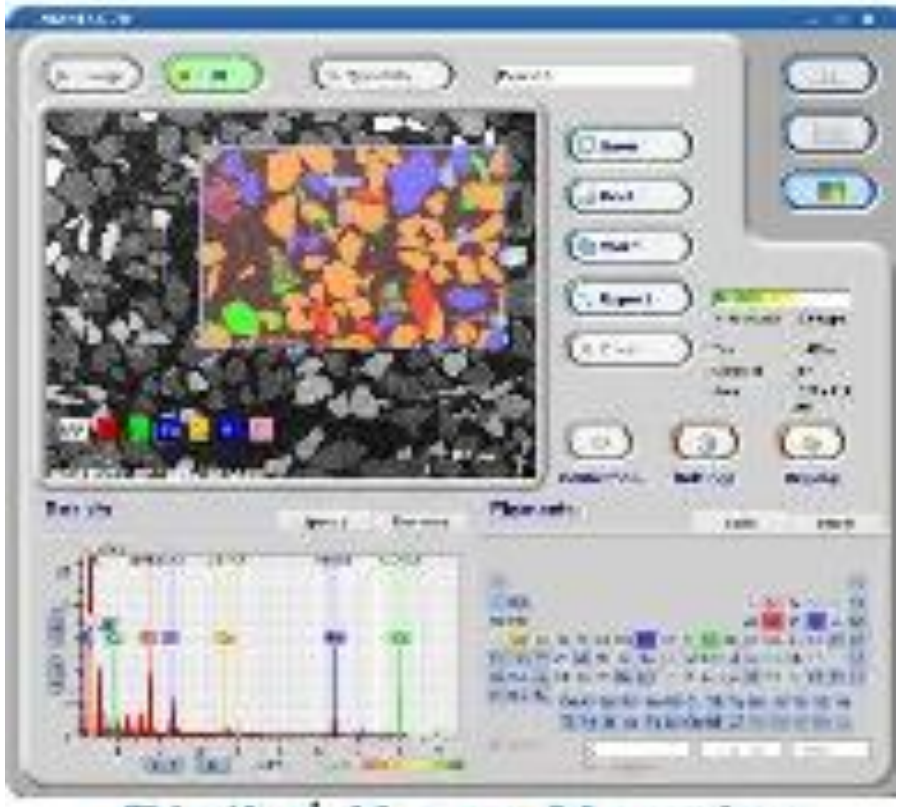
# SmartMat Research Lab for Micro scale - SMRL2



# SmartMat Research Lab for Micro scale - SMRL2



Line Profile Analysis (LPA)



Digital X-ray Mapping

# SmartMat Research Lab for Micro scale - SMRL2

**Automatic and manual cut-off machine**

**- Very high cutting capacity**

**- Constant feed speed principle ensures minimum deformation**

**- Automatic feed speed reduction for fast cutting of hard materials**

**- Quick positioning and freely selectable stop position functions**

**- Effective cooling system reduces the risk of burns**

# SmartMat Research Lab for Micro scale - SMRL2

**The LaboForce specimen movers**

**- Semi-automatic fine grinding and polishing of single specimens**
**- Quick and easy operation**
**- High quality finished specimens**
**- Automatic dosing unit**
**- Adjustable drip lubricator**

# SmartMat Research Lab for Macro scale

# SmartMat Research Lab for Macro scale

# SmartMat Research Lab for Micro scale

# SmartMat Research Lab for Micro scale

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# *Hungarian-Romanian Research Platform for Smart Materials Research Projects Support SMARTMAT*

## *HURO/1101/191/2.2.1*



***Workshop, Baile Felix, 2013.05.29-30***

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# Main data

- The project will be fulfilled in **18 month** (2013 January-2014 June), with the collaboration of the University of Debrecen (Lead Partner) and University of Oradea (Project Partner 1)

- 19 experts in Romanian side
- 7 experts in Hungarian side

# Project budget

- Project budget had been constructed according to HURO/1101 guidelines national co-financing and own contributions in the proper rate.
- Includes all the costs on Romanian and Hungarian side.
- Is balanced between Hungarian and Romanian partners.
- Financial management is assured by University of Debrecen and University of Oradea

Total budget is 1.195.121 Euro.

598.271 Euro – HU; 596.850 Euro - RO

# Management

***Hungarian side***

Dr. Budai István - project manager
Pintér Arnold - financial manager
Csáky Imre - project assistant

***Romanian side***

Dr. Vesselényi Tibor - project manager
Dr. Radu Tarca - financial manager
Dr. Prada Marcela -project assistant

# Objectives

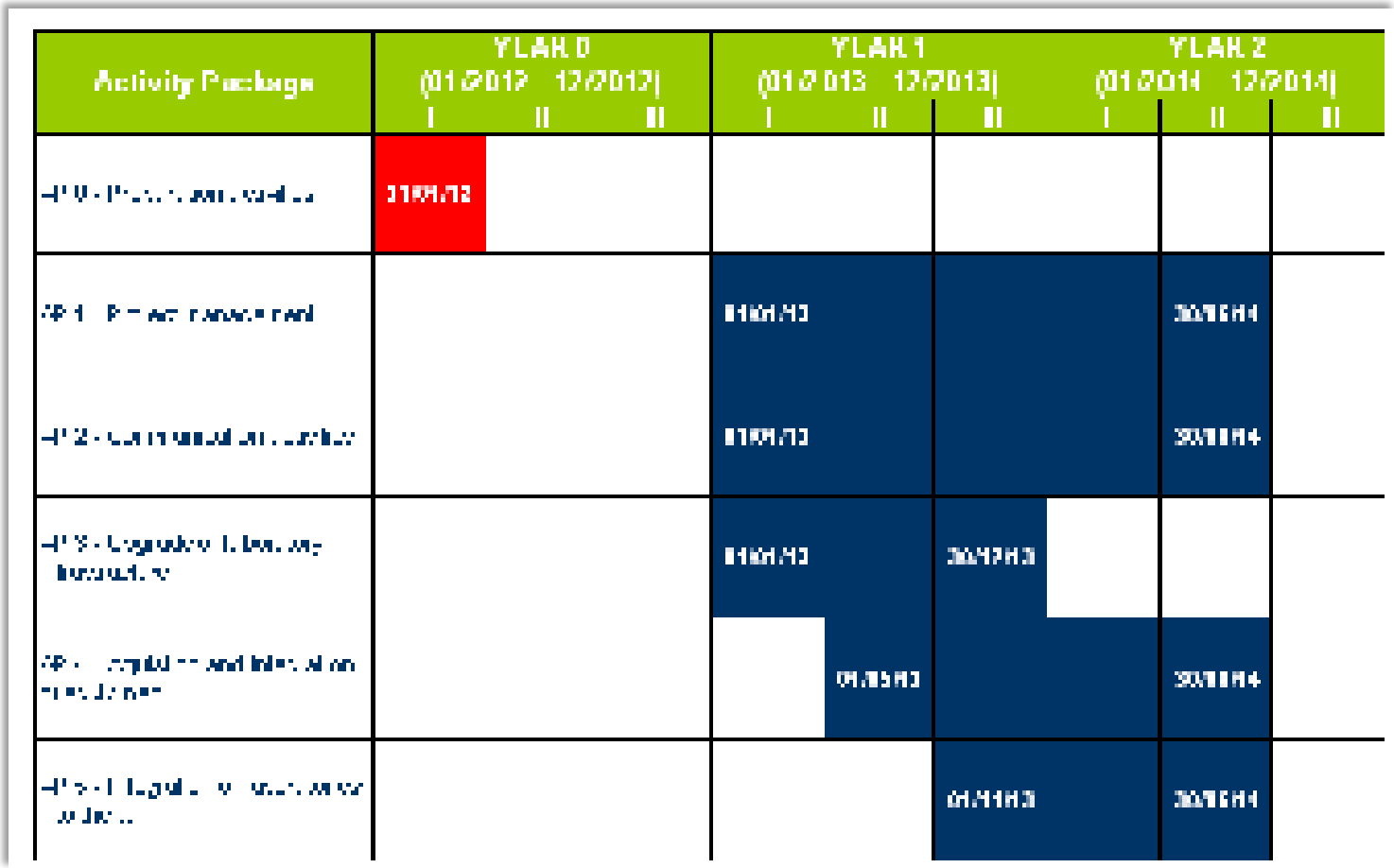
***General objective:***

➢ To develop a Hungarian-Romanian bilateral platform in the field of new materials, for new joint research projects support

***Specific objectives:***

➢Development of research laboratories, platform structure
➢Expert database and platform validation

# Timeframe


Activity Package
YEAR 0
(01/2012 - 12/2012)
I
II
III
YEAR 1
(01/2013 - 12/2013)
I
II
III
YEAR 2
(01/2014 - 12/2014)
I
II
III

# Preparation activities

- *Study regarding the state of the art of composite and nano materials and the relating equipments*
- *Feasibility study*
- *Project documentation*

# Project management

The project management team coordinate, supervises and verify the fulfillment of the activities. They are in permanent contact with the managing authorities too.

- *ERDF Subsidy Contract*
- *First period partner progress report (January-April 2013)*

# Communication activities

- Webpage
- Brochure, flyers, promotional materials
- Press release
- Opening and closing events.
- TV, Radio interviews
- Workshops 2 in Hungarian side, 2 in Romanian side

# Opening Event in Debrecen

# Upgrade of laboratory infrastructure

- Preparing technical specification of laboratories
- Preparation of public procurement documents and procedures
- Realization of laboratory works and clean room and verification of work conformity with technical plans.

# Acquisition and integration of equipments

- Monitoring the availability of required equipments.

- Public acquisition, preparing of documents and procedures.
- Commissioning, testing, verification, validation of equipments

# Integration of laboratories into the SMARTMAT research platform

- Research platform institutional and operative structure realization
- education partners
- Setup of communication platform between laboratories
- Development of laboratory handbooks and research application guidelines
- Development of scientific website and data base

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# SMARTMAT project

## University of Debrecen & Univerity of Oredea

# MakroLab

University of Debrecen, Faculty of Engineering Department of Civil Engineering

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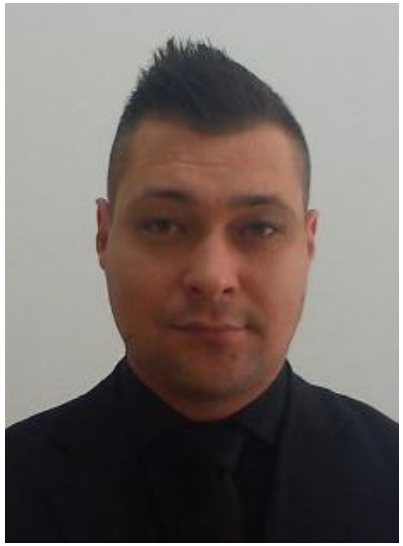
**www.huro-cbc.eu**

# Staff

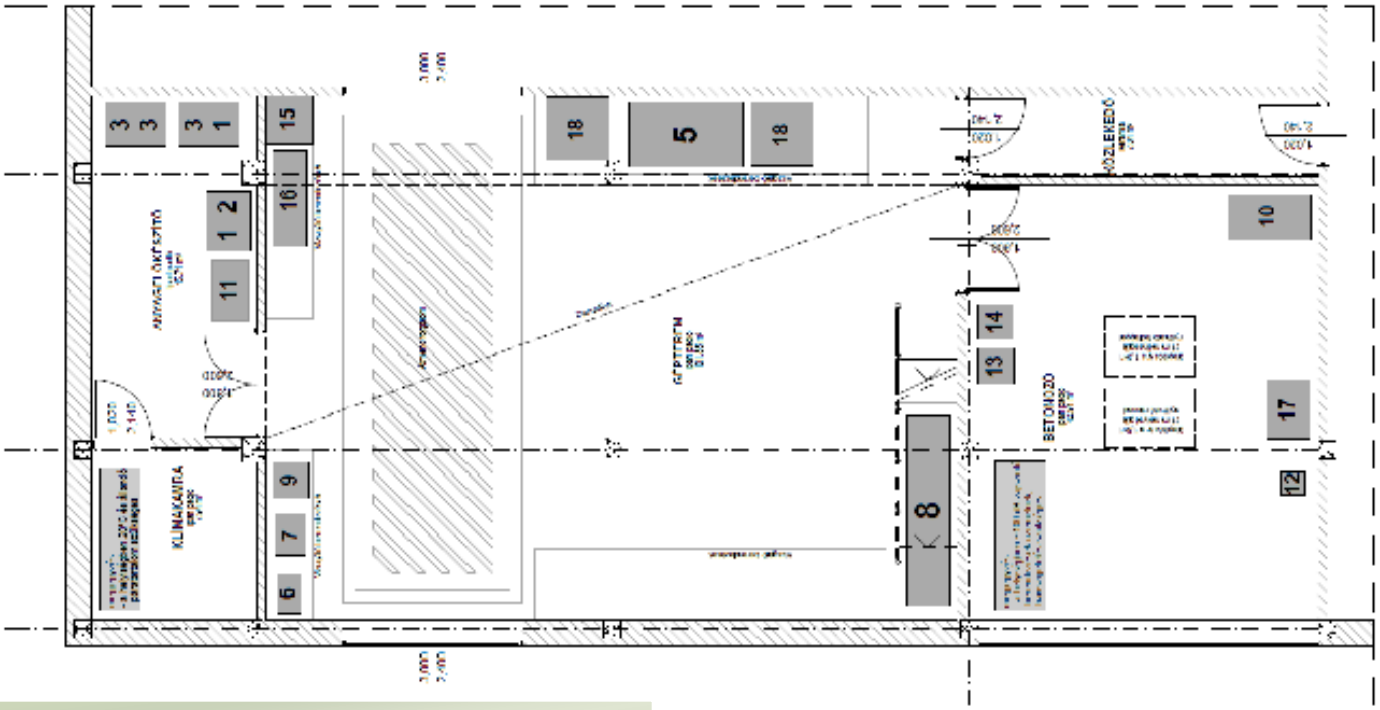
Dr. KOVÁCS Imre Ph.D.
Civil Engineer
Head of Department
***Structural experts***

Dr. NEHME Kinga Ph.D.
(born: PANKHARDT Kinga)
Civil Engineer
***Construction material expert***



KOVÁCS József
Architect
Civil Engineer
***Laboratory equipment expert***

# University of Debrecen, Faculty of Engineering
# Department of Civil Engineering
# Materials and Structures Laboratory



# 500 kN ELECTROMECHANICAL TESTER GENERAL DESCRIPTION

*CHARACTERISTICS:*

- CAPACITY: 500 KN.
- MAX. DISTANCE BETWEEN TWO PLATEN (mm): 2.000
- RELATIVE ERROR OF READEING + 1 OF APPLIED LOAD AND BETWEEN 2 AND 100% OF NOMINAL.
- FACE MEASURING POSITION: 0,1% CLASS 1.
- SPEED ACCURACY: + 1%
- CROSSHEAD DISPLACEMENT: AUTOMATIC PC CONTROLED
- MAX. WORKING SPEED: 0 – 100 mm/min.
- HORIZONTAL SPAM: 610 mm.

# 500 kN ELECTROMECHANICAL TESTER GENERAL DESCRIPTION

*RELATED RESEARCH TOPICS:*

- Steel and non metalic reinforcement: tensile strength, modulus of elasticity, stress - strain diagram
- Glass/steel/carbon fibre reinforced composite material: modulus of elasticity, stress - strain diagram
- Flexural and compressive strength of fibre reinforced mortar
- Different tests on soil specimens
- Differemt tests on rock specimens

# 500 kN ELECTROMECHANICAL TESTER GENERAL DESCRIPTION

*ROLE IN EDUCATION:*

- Construction Materials I.-II.; RC Structures I.-III., Steel Structures I.-III., Composite Structures, Timber & Masonry Sructures, Laboratory Practice
- Properties of Concrete, Mortar, Ceramic, Wood and other materials
- Behaviour of Column, Beam, Wall and Slab models

# 30 TN HYDRAULIC FLEXURAL TESTING FRAME GENERAL DESCRIPTION

*CHARACTERISTICS:*

- CAPACITY: 30 TN
- HYDRAULIC CILINDER: DOUBLE EFFECT
- PISTON TRAVELLING DISTANCE: 350MM
- FACE MEASURING POSITION: 0,1% CLASS 1.
- SPEED ACCURACY: + 1%
- PISTON DISPLACEMENT: AUTOMATIC PC CONTROLED
- HORIZONTAL SPAM: 6 MTS
- TESTING WIDTH: 70 CENTIMETERS

# 30 TN HYDRAULIC FLEXURAL TESTING FRAME GENERAL DESCRIPTION

*RELATED RESEARCH TOPICS:*

- Testing of Beams, Slabs or other specimens in bending
- Plate Shear test
- Analisys of full-scale structural models in different loading

# 30 TN HYDRAULIC FLEXURAL TESTING FRAME GENERAL DESCRIPTION

*ROLE IN EDUCATION:*

- Materials and Structural Testing Lab
- Reinforced Concrete, Prestressed Concrete, Composite structural element

# RAPIDAIR 457 SYSTEM

RapidAir 457 is an automated image analysis system performing analysis of the air void distribution of hardened concrete according to the ASTM C 457: „Standard method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete”

# RAPIDAIR 457 SYSTEM

*RESEARCH ACTIVITY:*

- Surface analysis of
  - full self compacting or conventional concrete after/during frost resistance test
  - Microstructural analysis of construction materials
  - Analysis of bond surface in different cementitious materials
  - Analysis of corrosion effect

# RAPIDAIR 457 SYSTEM

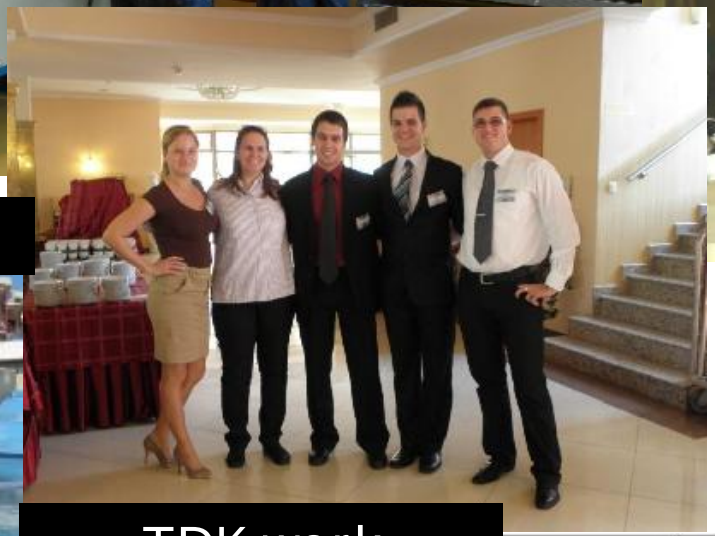
*ROLE IN EDUCATION:*

- Construction Materials I.-II.; RC Structures I.-III., Steel Structures I.-III., Composite Structures, Timber & Masonry Sructures, Laboratory Practice
- Properties of Concrete, Mortar, Ceramic, Wood and other materials
- Behaviour of Column, Beam, Wall and Slab models

laboratory work

factory visit

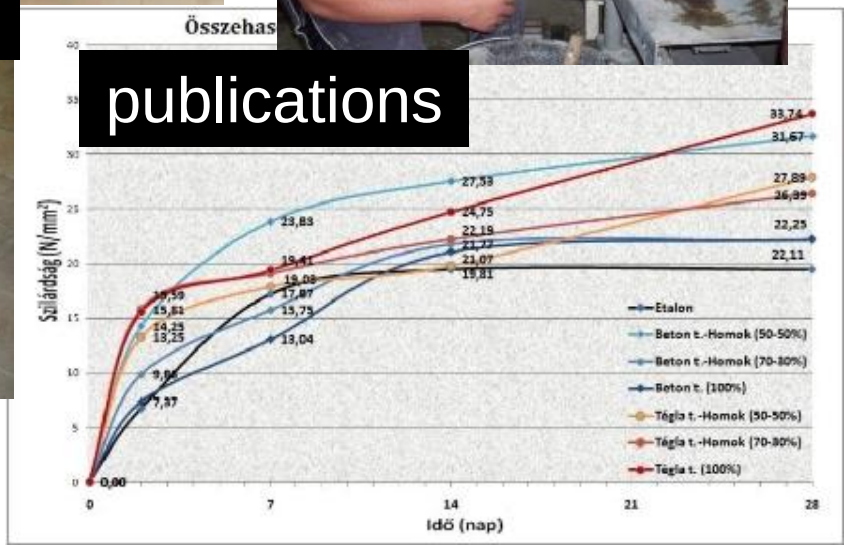
lectures



conference attendance

TDK work

publications




OTDK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# STATE OF ART IN NANOINDENTATION

**Eng. BLAGA FLORIN PhD.**

**University of ORADEA**

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**- What can be measured:**

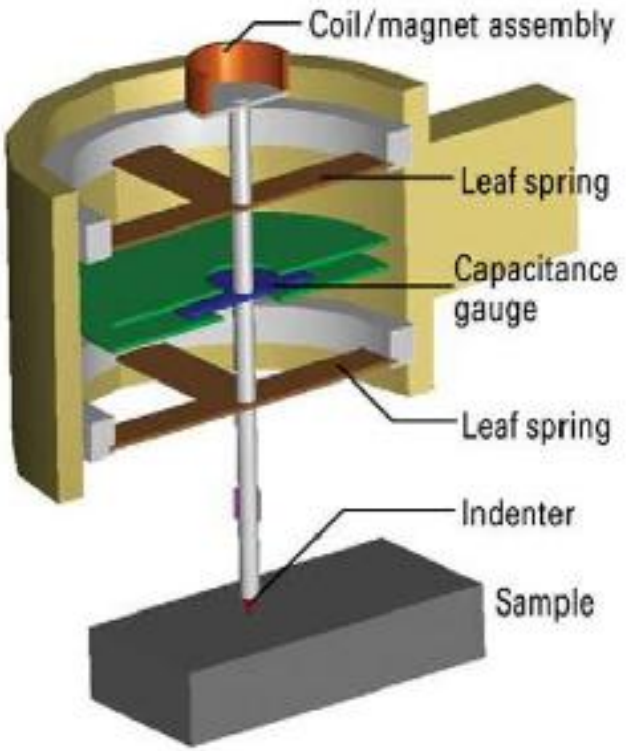
- Hardness
- Elastic modulus
- Peak force
- Peak displacement
- Work of indentation for each test
- Wear testing
- Ramped scratch testing for critical loads

**- Standard head for a wide variety of application:**

- Displacement resolution <0.01 nm
- Total indenter travel 1.5 mm
- Maximum indentation depth >500 µm

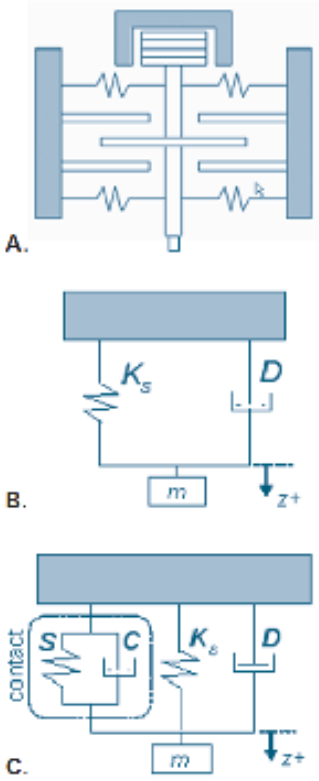
**- Loading capability**:

- Maximum load (standard) 500 mN
- Load resolution 40 nN
- Contact force <1.0 µN
- Load frame stiffness ~5 x $10^6$ N/m



# Continuous Stiffness Measurement

## Quasi-Static and Dynamic Methods

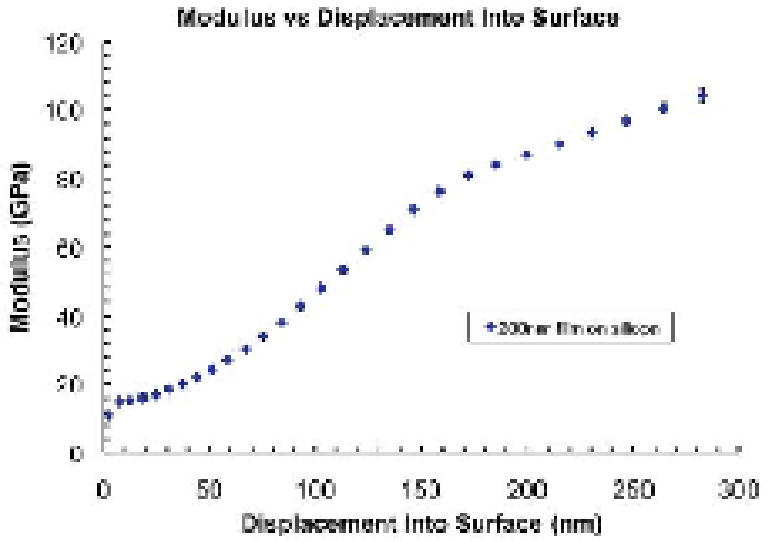



**A.** Schematic of free-hanging indenter.
**B.** Dynamic model of instrument alone (no contact).
**C.** Dynamic system model during testing.

- In conventional quasi-static indentation testing, the stiffness of contact is determined by analyzing the force vs. displacement curve during unloading. This depth-sensing method provides a single measurement for the given indentation depth
- The Continuous Stiffness Measurement (CSM) option, satisfies application requirements that must take into account dynamic effects, such as strain rate and frequency
- With the CSM option, the Nano Indenter applies a load to the indenter tip to force the tip into the surface while simultaneously superimposing an oscillating force with a force amplitude generally several orders of magnitude smaller than the nominal load

- The CSM option offers a means of separating the in-phase and out-of-phase components of the load-displacement history.

- This separation provides an accurate measurement of the location of initial surface contact and continuous measurement of contact stiffness as a function of depth or frequency, thus eliminating the need for loading cycles.

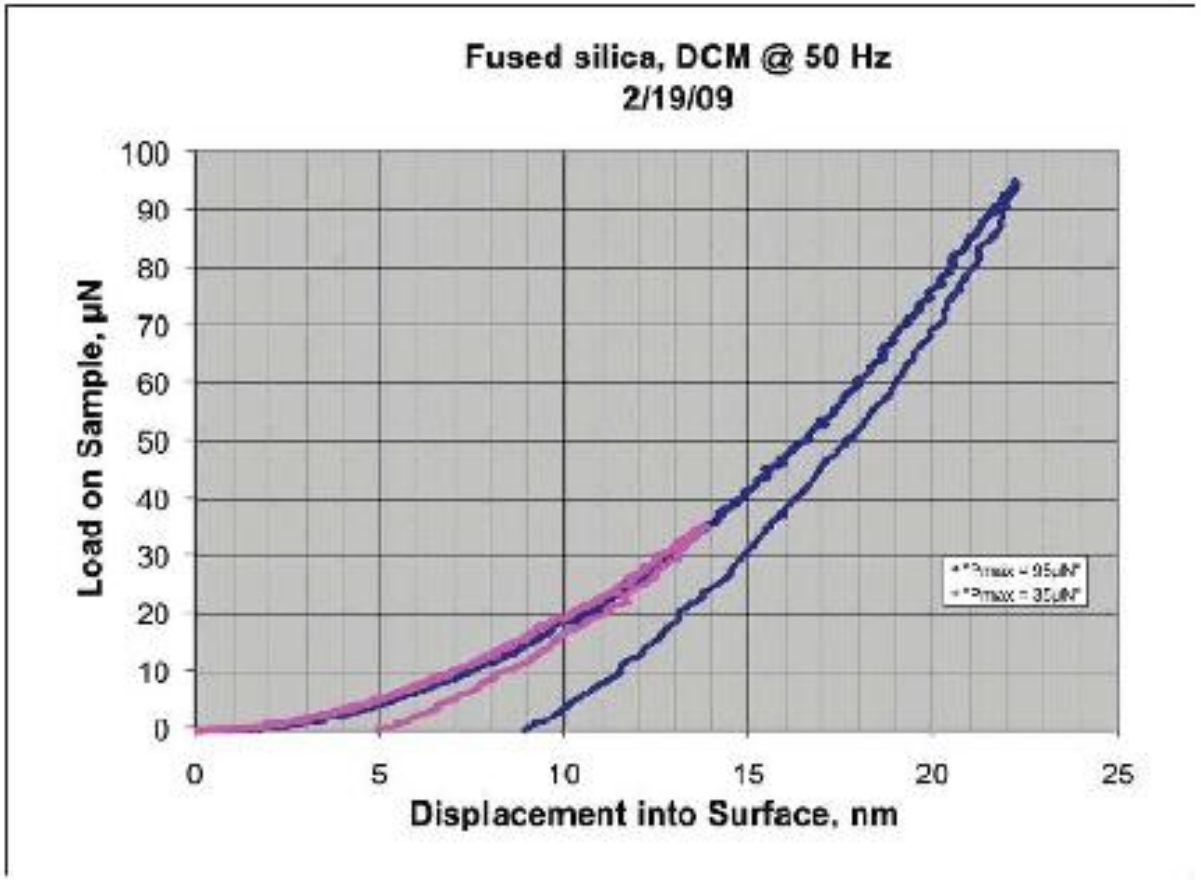



**Characterization of function of depth on thin film that was applied to sample using CSM**

# Dynamic Contact Module

- The Dynamic Contact Module (DCM) option extends the range of load-displacement experimentation down to the surface contact level.

- With this option, Nano Indenter users can study not only the first few nanometers of an indentation into the surface of a material, but even the pre-contact mechanics.




**Two force-displacement curves for two different tests on fused silica.**

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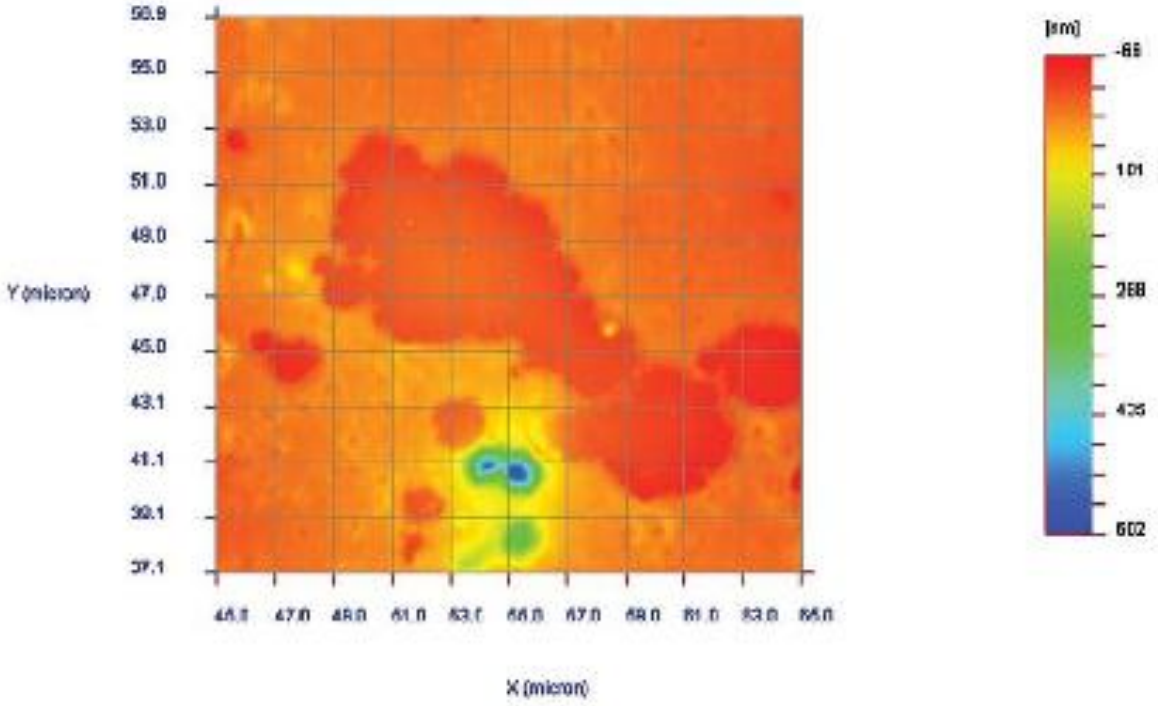


**A polished nickel particle (orange-red), embedded in epoxy (orange), tested on the NanoIndenter G200. The small yellow dots in a line traversing the length of the particle are residual indentation impressions of less than 75 nm deep. (Note: the sixth dot is larger than the others, because it landed in the epoxy, which is softer than the nickel.) Image size: 256 x 256 µm**

## DCM Indentation Head Option Specifications

| | |
|---|---|
| Displacement resolution | 0.0002 nm |
| Maximum indentation depth | >15 µm |
| Loading column mass | <100 mg |
| Load application | Coil / magnet assembly |
| Displacement measurement | Capacitance gauge |
| Typical leaf spring stiffness | ~100 N/m |
| Typical damping coefficient | 0.02 Ns/m |
| Typical resonant frequency | 180 Hz |
| Loading capability | |
| Maximum load | 10 mN (1 gm) |
| Load resolution | 1 nN (0.1 µgm) |

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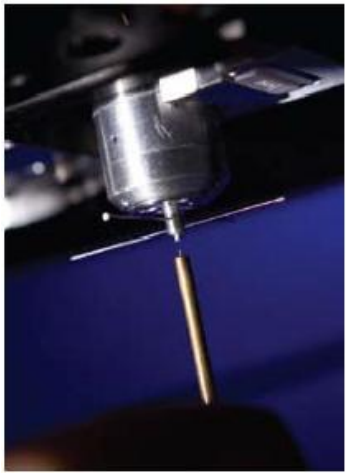
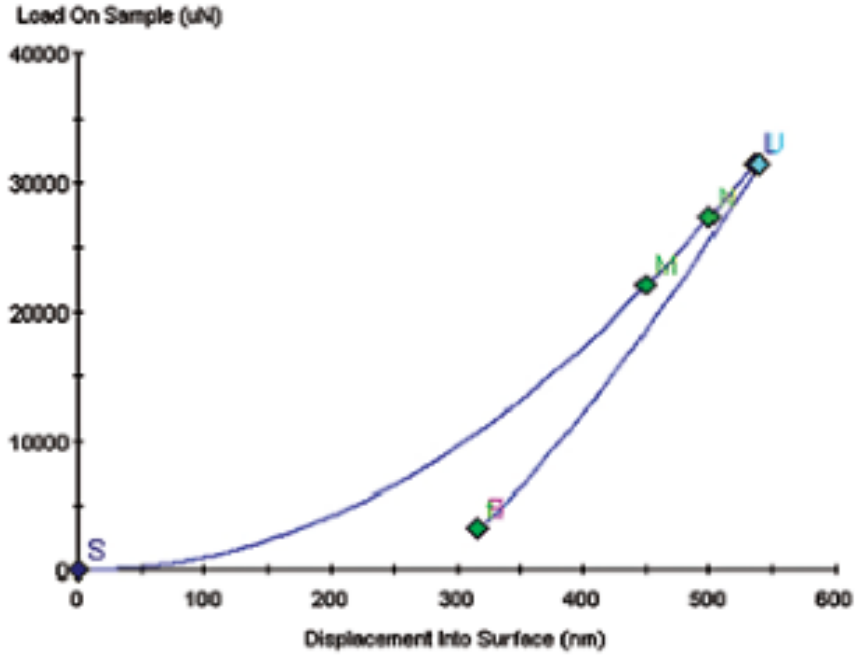
# New Enhanced Dynamic Contact Module II (DCM II) Option

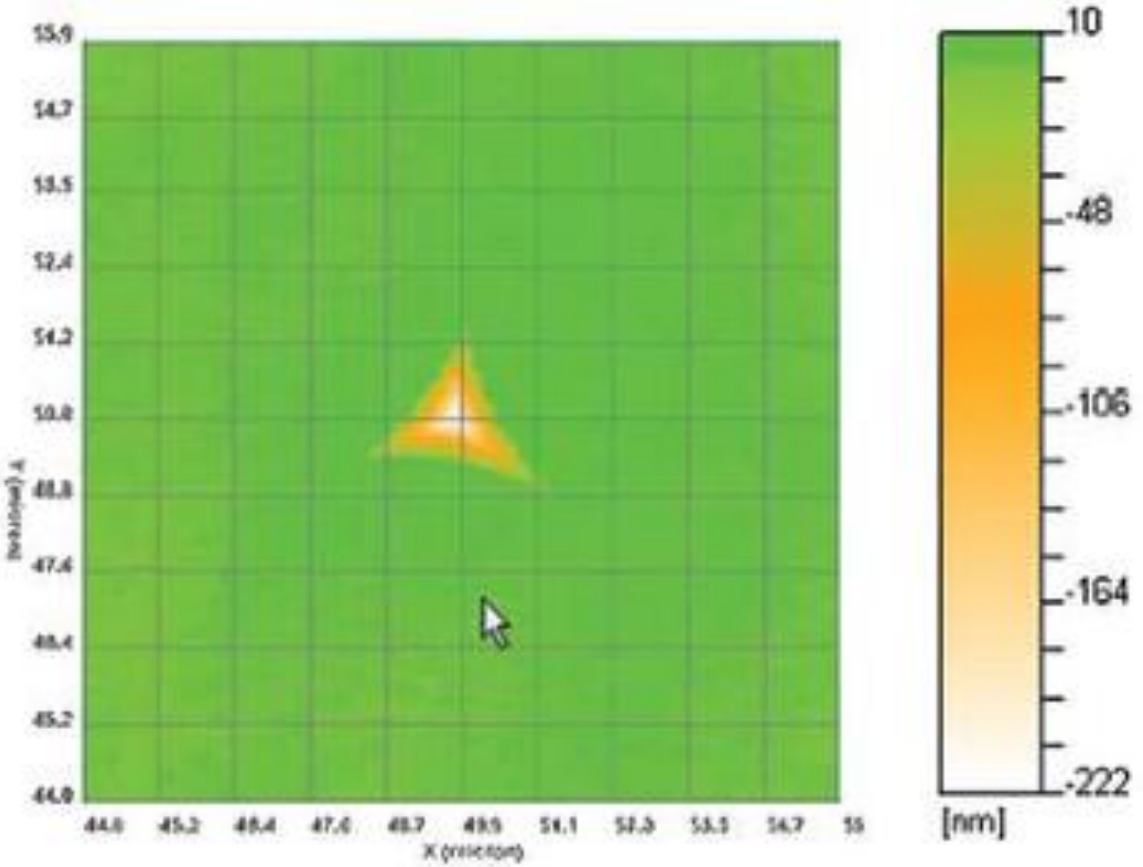
Dynamic Contact Module II



Easy Tip exchange




Displacement curve on fused silica sample




**Indenting on fused silica image captured in NanoVision software**

# Software Unprecedented Functionality for Nanoindentation and Tensile Testing

- Newly integrated tool simplifies determination of indenter area function and load-frame stiffness;
- Exclusive new test methods for polymers and substrate- independent thin film materials;
- Standard batch of tests comprising 25 or more samples can be set up in 5 minutes or less;
- Ability to plot 2D graphs and export directly to Microsoft Excel as viewed on monitor;
- New data organization structure organizes sample files into projects and subprojects;
- Convenient PDF printer to replace need for hardware printers.

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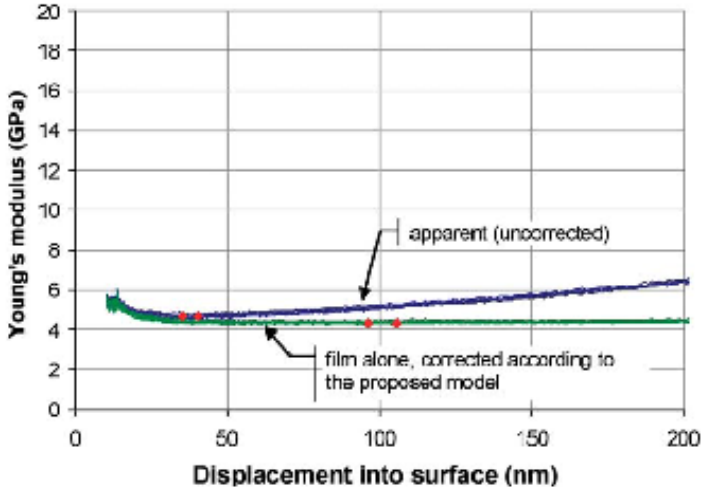
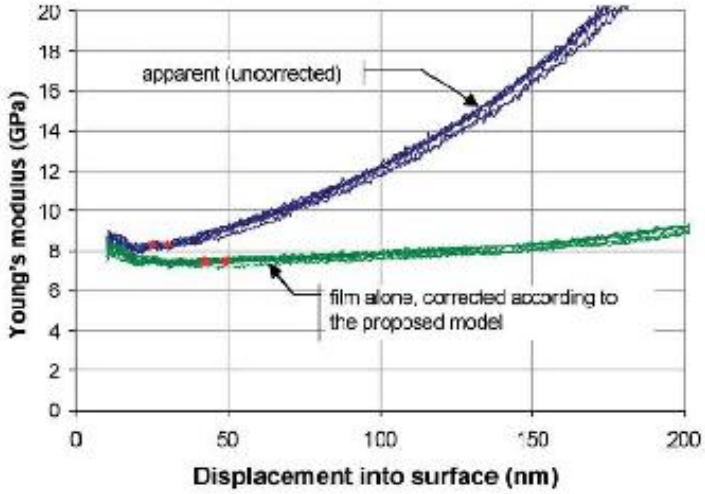



Low-k 1 on silicon substrate, tf = 1007nm




Low-k 2 on silicon substrate, tf = 445nm

## Table1. Results

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|
| | | | Results, standard | | | Results using new method | | |
| Sample | N | Thickness nm | Range[4] nm | $E_u$ GPa | $\sigma(E_u)$ GPa | Range[5,6] nm | $E_f$ GPa | $\sigma(E_f)$ GPa |
| low-κ 1 | 8 | 1007 | 35-40 | 4.69 | 0.07 | 95.9-105.4 | 4.34 | 0.06 |
| low-κ 2 | 8 | 445 | 25-30 | 8.23 | 0.13 | 42.2-46.8 | 7.46 | 0.12 |

**•1 and 2 show the Young's modulus as a function of penetration depth for each sample.**
**•The blue traces are the values without any correction for substrate influence.**
**•The red diamonds show the range used to calculate the (uncorrected) Young's moduli in the fifth column of Table 1.**
**• The green traces of Figures 1 and 2 are the values calculated according to the new method.**
**•The red diamonds, placed automatically at 10% of the film thickness show the range used to calculate the Young's moduli in Table 1, Column 8 and are significantly lower than what was previously reported for these samples (Table 1,Column 5).**

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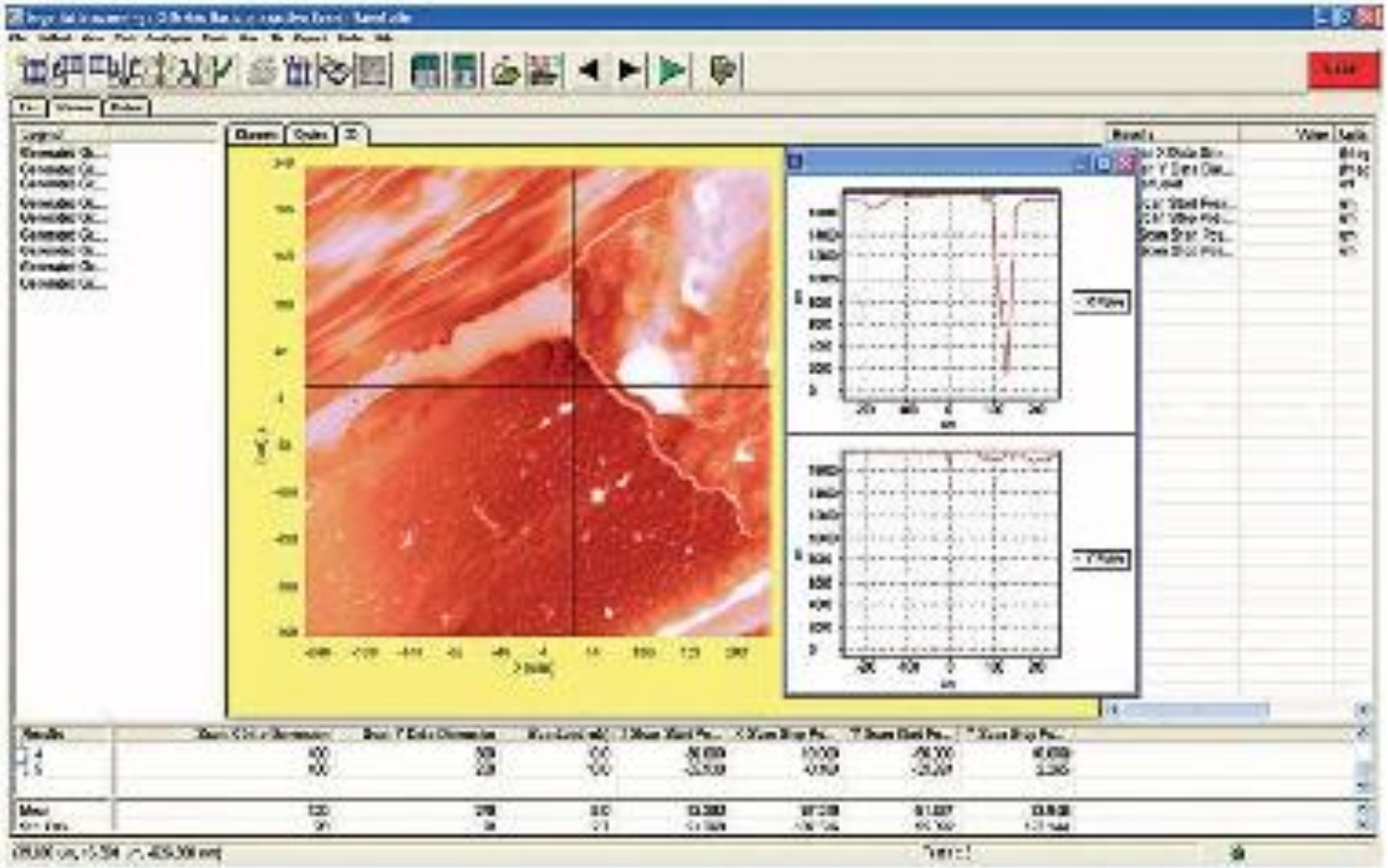
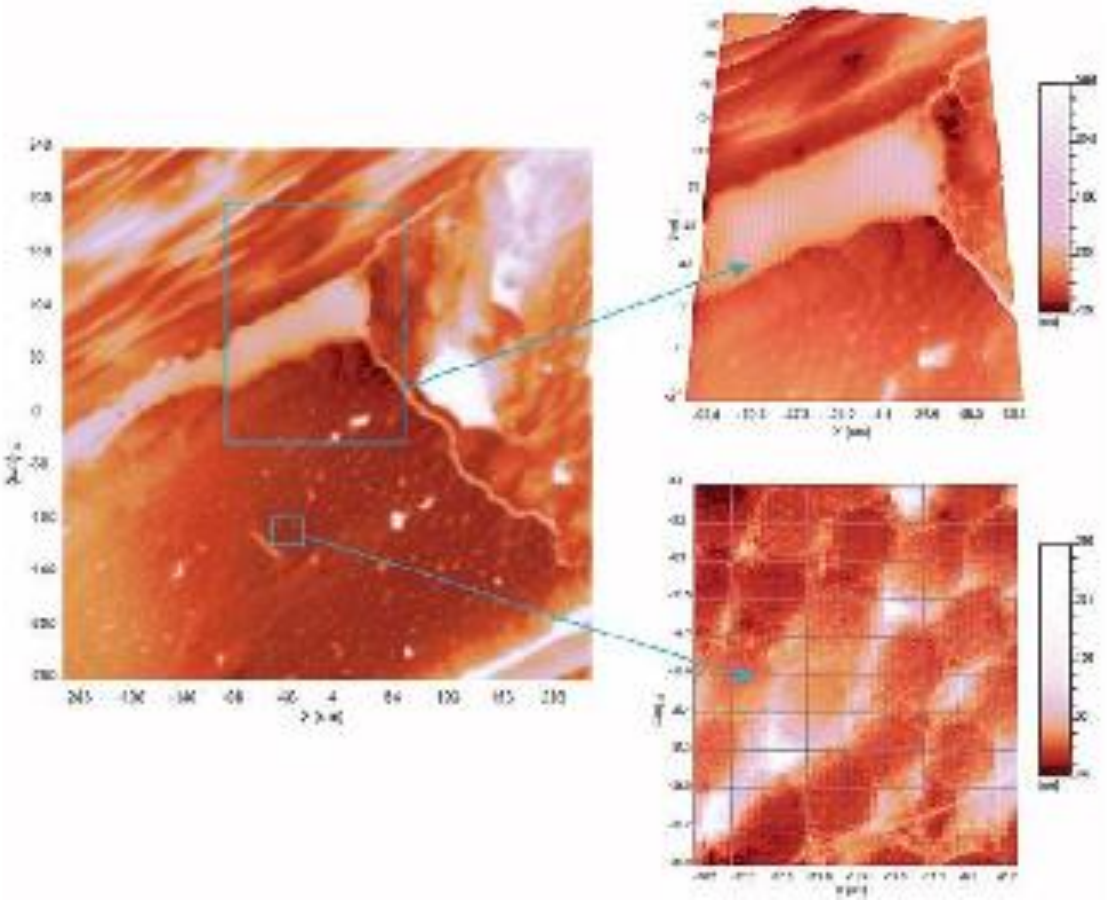


**Original 500 x 500µm survey scan. Carbon fibers are standing on end in the lower part of the image and in the upper left they are laying flat**



- **Left: 500 x 500µm scan of carbon fibers over 18µm of height deviation.**
- **Right top: 40µm scan of feature shown in 3D.**
- **Bottom right: 40µm scan of carbon fiber ends after polynomial leveling.**

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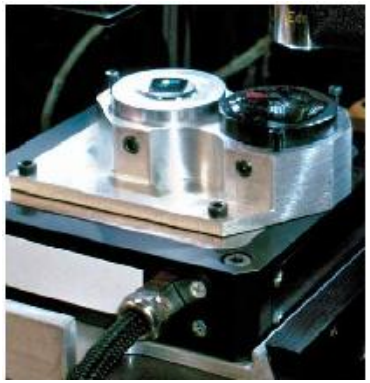
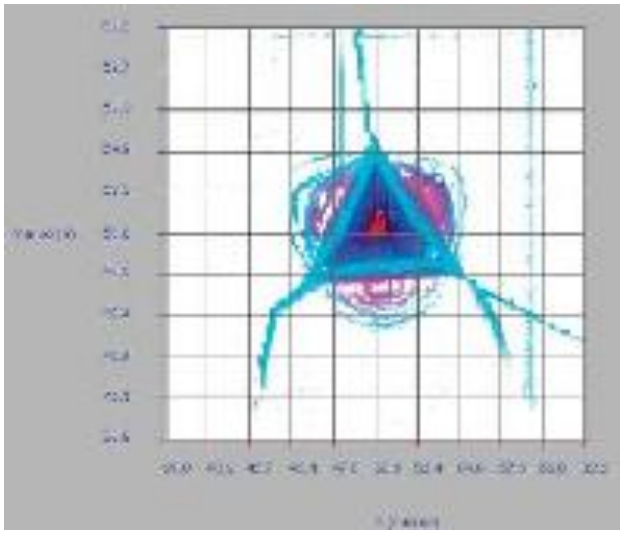
# Nano Vision Microscopy Module



Nanopositioning scanning stage



NanoVision enables rapid setup of automated indent and scan routines. Resultant scans make fracture toughness measurements easy

- Create quantitative high-resolution images;
- Target indentation test sites with nanometer-scale precision;
- Examine residual impressions in order to quantify material response phenomena such as pile-up, deformed volume, and fracture toughness;
- Full integration with NanoSuite software, including:
  - Parallel control of nano positioning stage and all other system hardware in NanoSuite;
  - NanoSuite 3D software;
  - NanoSuite methods that integrate indentation and imaging.
- User control over scan area, resolution and speed;
- Includes 10 NanoVision sample pucks

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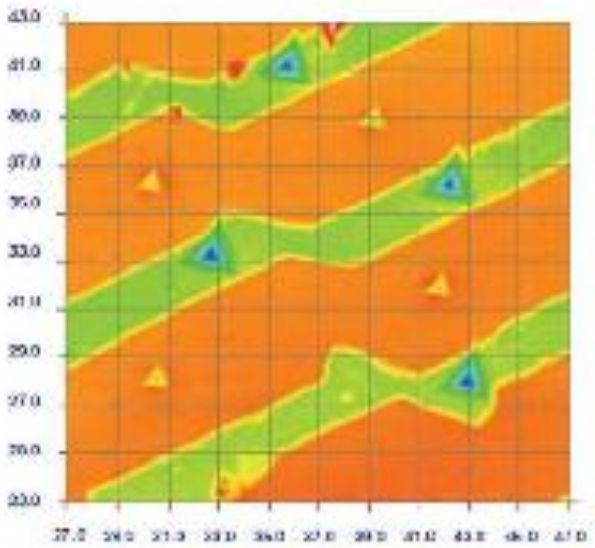
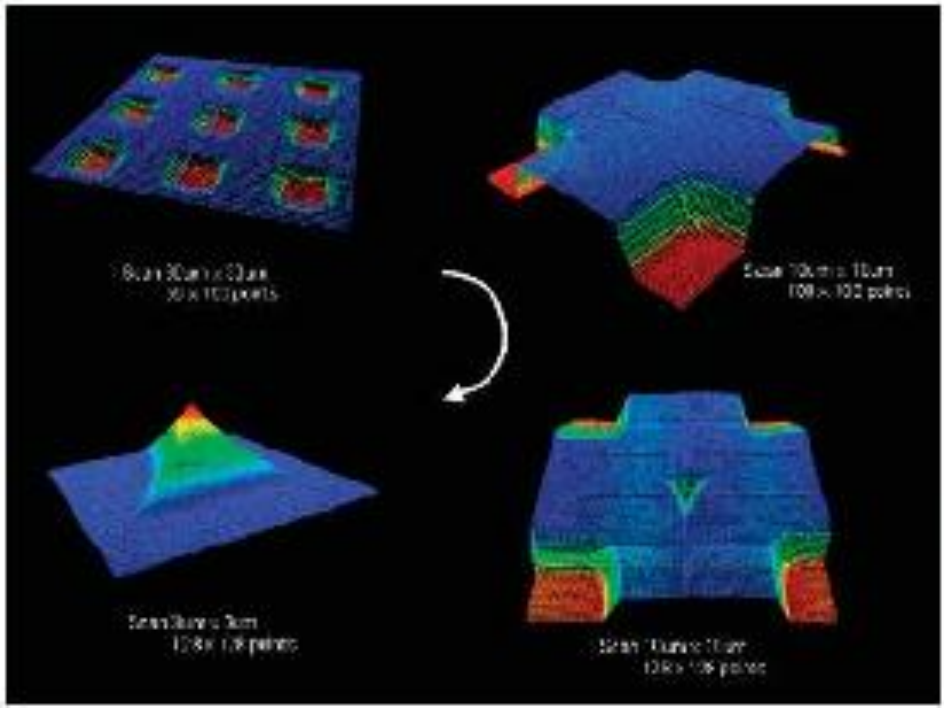


**NanoVision probes the surface of the sample to create a topographical image, which can then be used to select test sites. Nano Vision was used to test individual phases of chrome and chrome-silicide.**



**NanoVision option was used to scan a grid with known step heights in order to demonstrate topological accuracy. One advantage of using an indentation system for image generation is that you can rely on the measured displacements. Surface topology is measured with the same system that is used to measure material properties, so displacement measurements must be accurate and repeatable**

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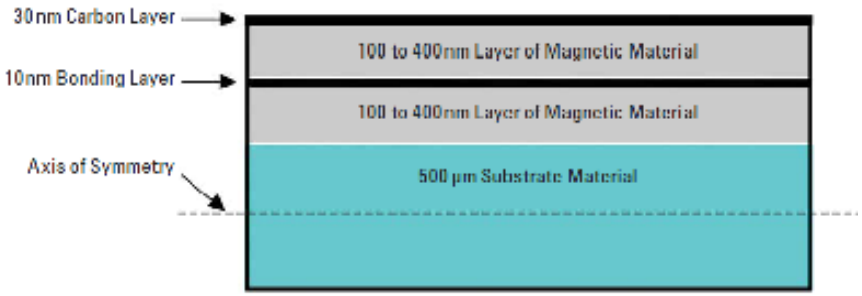
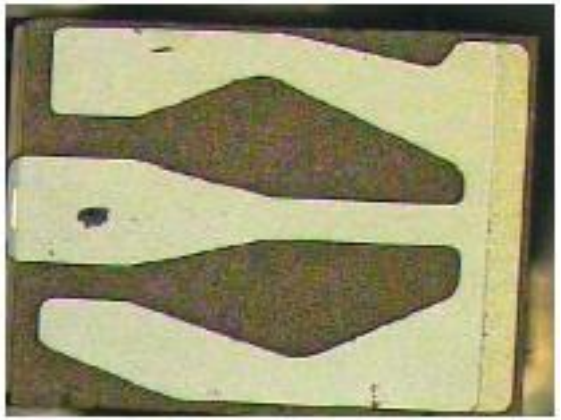
# Scratch Testing of Hard Drive Components




**Cross-sectional diagram of the hard disk samples showing the 30 nm coating of DLC**



**The slider removed from the gimbal device of a hard drive. Sliders typically have a 2 nm to 4 nm coating**

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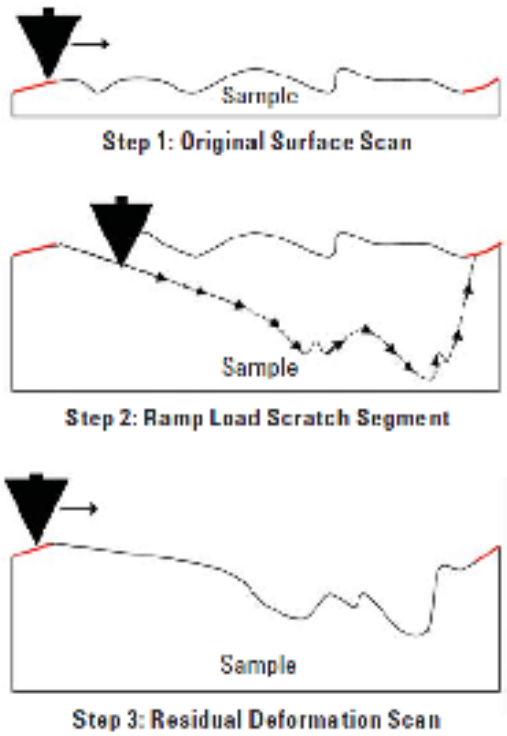



**Diagram of the three-step ramp load scratch test. Red lines show the areas of pre and post profile scans used to perform leveling of the 3 steps.**

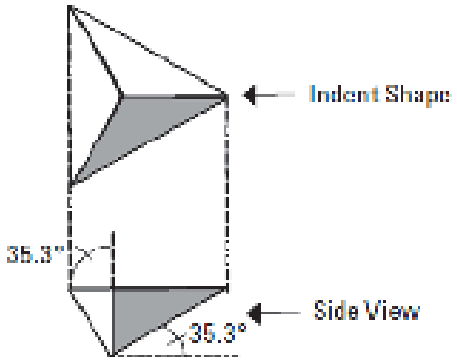



**Diagram of a cube corner tip.**

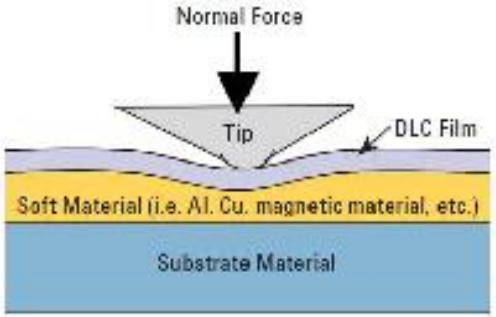
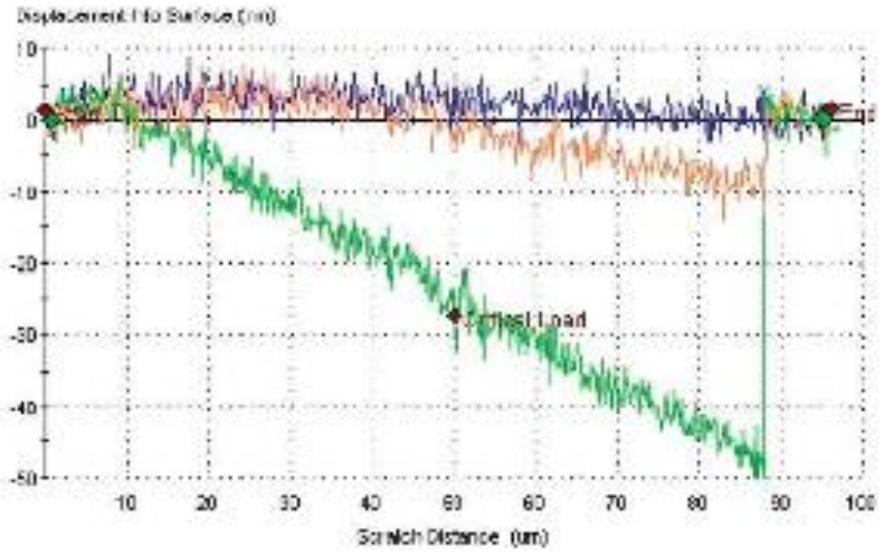
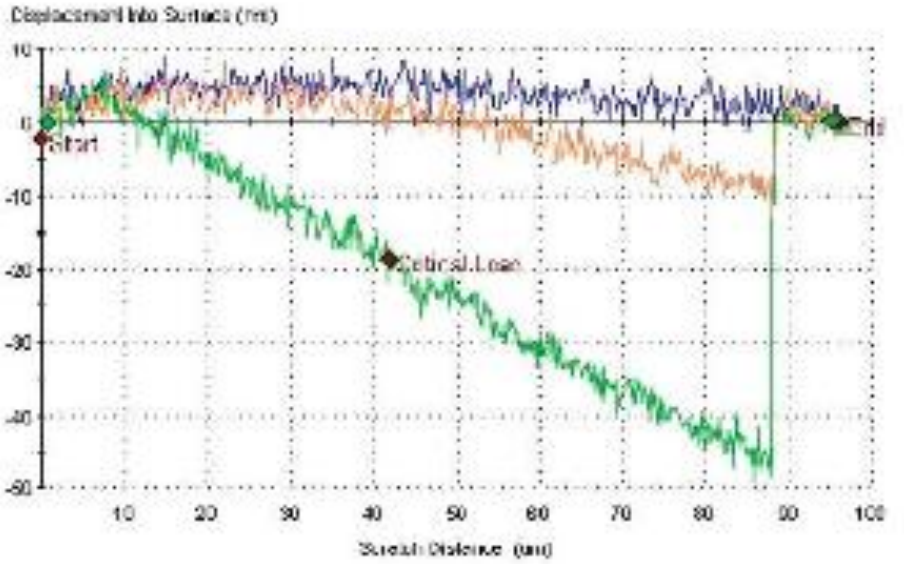


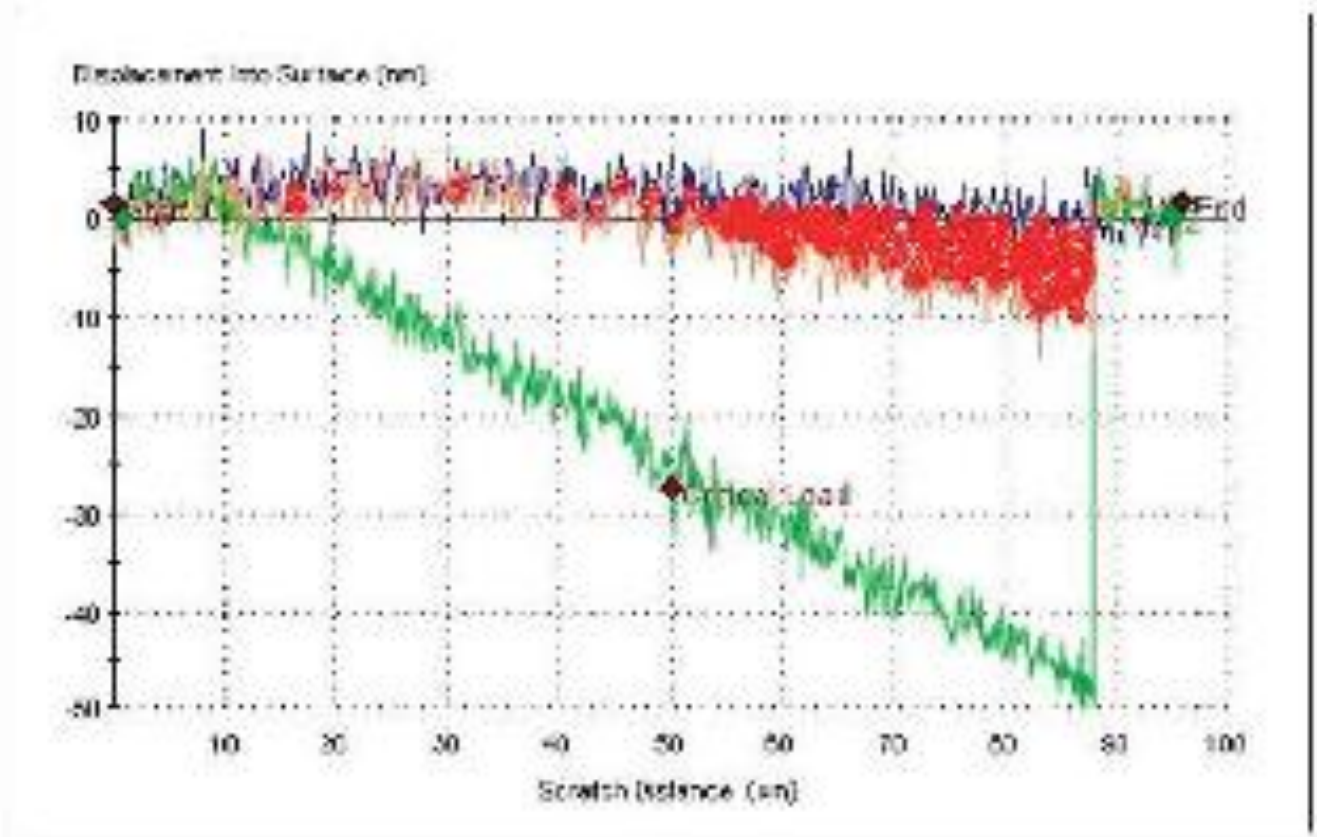

**Diagram of the deformation that occurs during testing of a DLC film on soft material. Notice that the film material elastically deforms while the underlying soft material accommodates the bulk of the deformation**




Typical displacement curves for the scratch test performed on hard disk Sample 1




Typical displacement curves for the scratch test performed on hard disk Sample 2




**The Residual Deformation was calculated by determining the area between the original surface scan and the residual deformation scan. The area of deformation is shown in red.**

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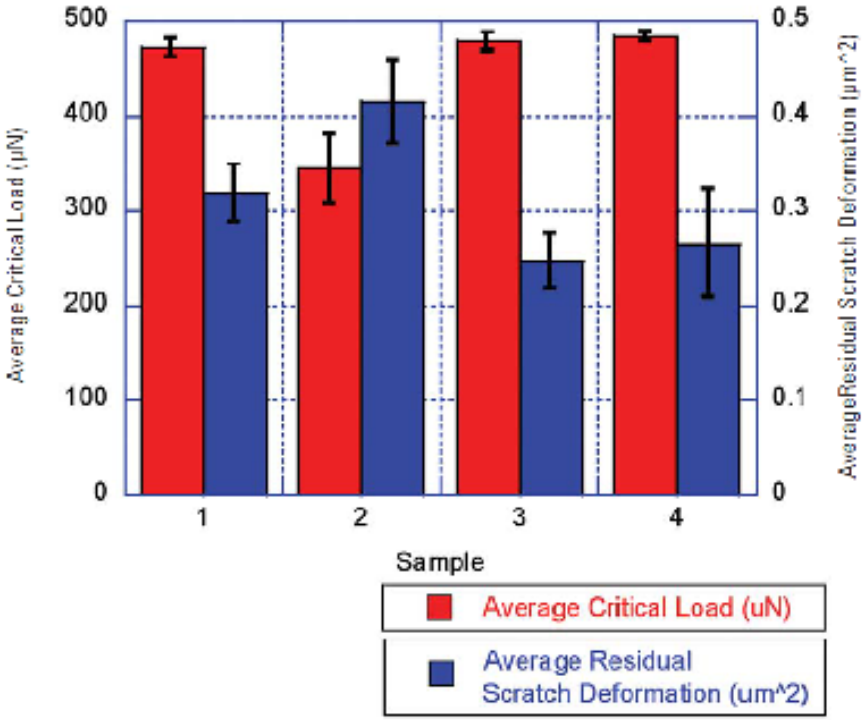
| Sample Number | Critical Load<br>μN | Penetration at Critical Load<br>nm | Residual Scratch Deformation<br>μm² |
|---|---|---|---|
| 1 | 471.3 ± 9.5 (1σ) | 26.3 ± 0.6 (1σ) | 0.318 ± 0.030 (1σ) |
| 2 | 343.7 ± 36.6 | 18.7 ± 2.5 | 0.414 ± 0.044 |
| 3 | 478.0 ± 9.5 | 28.0 ± 1.0 | 0.246 ± 0.029 |
| 4 | 483.0 ± 5.2 | 28.0 ± 2.6 | 0.265 ± 0.057 |

**Scratch test results for the hard disk samples**




**Scratch test results for the hard disk samples. The error bars represent one standard deviation.**

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# Thank You !

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