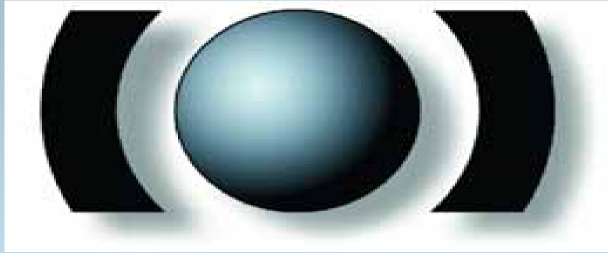


International
Laser Center

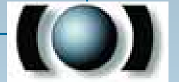
“Nano” activities
at

ILC
Bratislava

F. Uherek

Outlook

1. Briefly about ILC
2. Current “nano” activities
3. Technical base of “nano” activities



1. Briefly about ILC

Organization Structure of ILC Bratislava

Ministry of Education

ILC

Prof. D. Chorvát
director

Administration

Department of Laser Technology

Education

Department of Biophotonics

Prof. F. Uherek

vice-director, head of dept.

Cooperation with universities

Dr. D. Chorvát

head of dept.

Department of Laser Technology

Laboratory of Laser Microtechnology — Dr. J. Bruncko

PLD, laser microtechnology

Laboratory of Material and Surface Analysis — Prof. J. Kováč

SEM, SNOM, AFM, STM, Conf. Micr.

Laboratory of Femtosesond Spectroscopy — Dr. I. Bugár

Ti:sapphire, Cr:forsterite

Laboratory of TOF SIMS — Dr. D. Velič

TOF SIMS - ION TOF IV.

Laboratory of Applied Optics — Dr. M. Držík

Laser vibrometry, interferometry,
OA testing, laser deflectometry

Laboratory of Information Technology — Dr. J. Chovan

Fiber Communication Systems,
OE device characterisation

Department of Biophotonics

Laboratory of Biomedical Imaging and Visualisation

Optical tomography, data vizualisation, modelling of biosystems

Laboratory of Rapid Prototyping and Reverse Engineering

Noncontact optical measurement, reconstruction of surface and volumetric structures, stereolitography

Laboratory of Laser and Scanning Microscopy

3D (multispectral) imaging and analysis of processes in isolated cells and tissues

Laboratory of Laser Spectroscopy

Laser induced luminiscence, femtosecond time-resolved and non-linear spectroscopy

Laboratory of Applied Biophysics

Raman spectroscopy and imaging, PDT

Laboratory of Clinical and Applied Pharmacology

Molecular biology, applied photonics

2. Current “nano” activities at ILC

Current “nano” activities at ILC

1. Participation in “NANOEXCEL” agenda

   Preparation the agenda for “National center of excellence”

2. Realization of projects in field of nanotechnology

   Slovak, bilateral and EU projects

   N2T2, COST P11, SK-CZ, SK-D, VEGA, APVV and applied research

3. Finalization of technical base for nanotechnology at ILC

   Finishing the contract of ILC (between ILC and ILC MSU)

3. Technical base of “nano” activities at ILC

Laboratory of Laser Microtechnology

Laboratory of
Laser Microtechnology

Areas of activity

- Pulsed Laser Deposition (PLD)
- Laser microprocessing - cutting, drilling, graphical processing, attention to microprocessing of the nonstandart materials with extremly hard coating (diamond layers, ceramics, etc.)
- Laser welding and cutting

Equipment

- Pulsed Nd:YAG laser Quanta Ray Pro 250
- Two vacuum systems for pulsed laser deposition of thin films
- CW Argon laser Spectra Physics 2017, CO_2 laser 100 W
- Nd:YAG laser 800W, DPSS Nd:YAG laser 20 W and 50W

Laboratory of Laser Microtechnology

Pulsed Nd:YAG laser Quanta Ray Pro 250

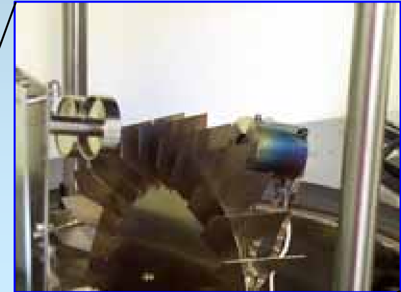
Repetion Rate: 10 Hz
Pulse Width: 8 – 12 ns

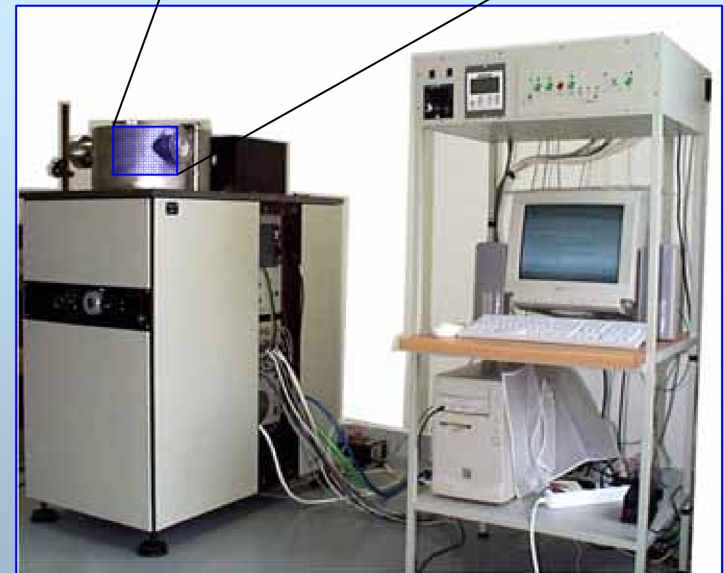
Wavelength (nm)	Energy (mJ/pulse)
1064	1500
532	800
355	425

Laboratory of Laser Microtechnology

Pulsed Laser Deposition of thin film – Vacuum system I.

- Vacuum up to 1.10^{-4} Pa
- Heated substrate holder up to 700°C
- Holder for two targets
- Mechanical droplets separator
- Multilayers film deposition
- Actual available targets: Pt, Au, Cu, Ni, Ti, Zn, W/Ti, Mo, Al





Laboratory of Laser Microtechnology

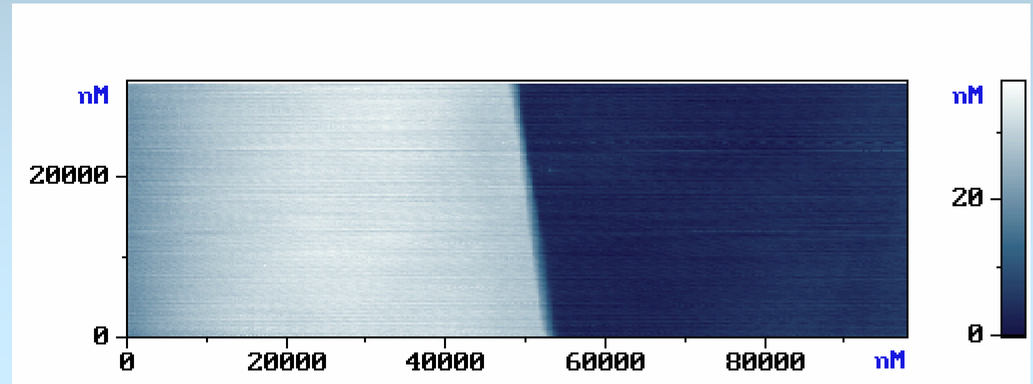
Pulsed Laser Deposition of thin film – vacuum system II.

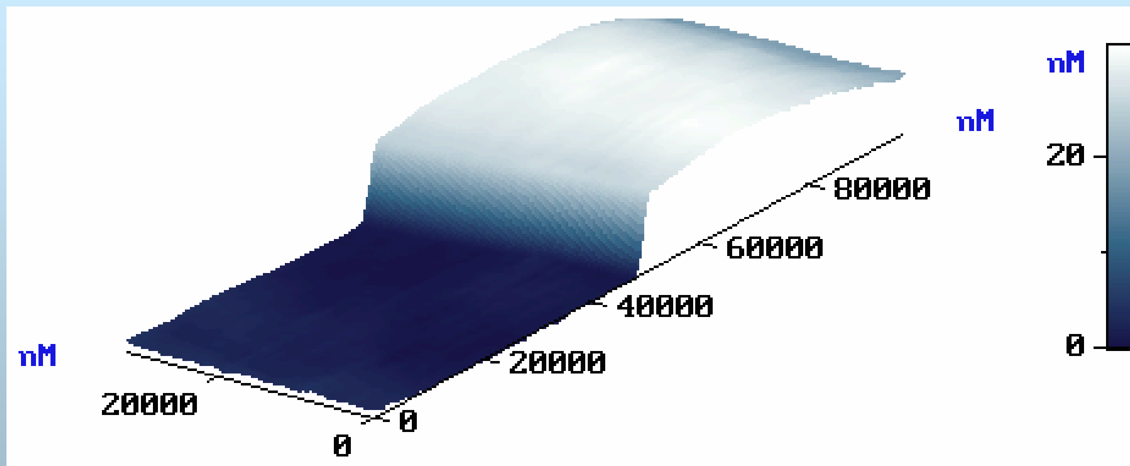
- Vacuum up to 1.10^{-4} Pa
- Heated substrate holder up to 870°C
- Cooled substrate holder up to liquid nitrogen
- Holder for two targets
- Reactive deposition
- Multilayers film deposition
- Actual available targets: ZnO, YBCO
  MgO, LSMO, ...

Results

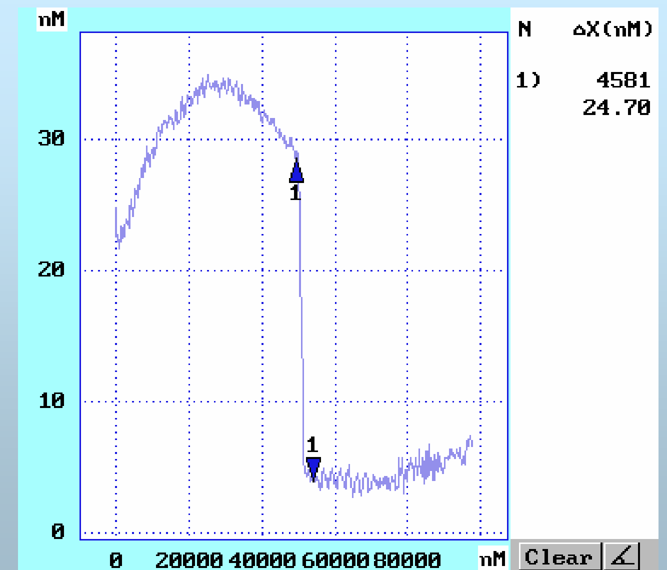
Molybdenum thin films (PLD)

Substrate	Si (111)
No. of pulses	3000
Dep. temp.	22°C
Thickness	25 nm

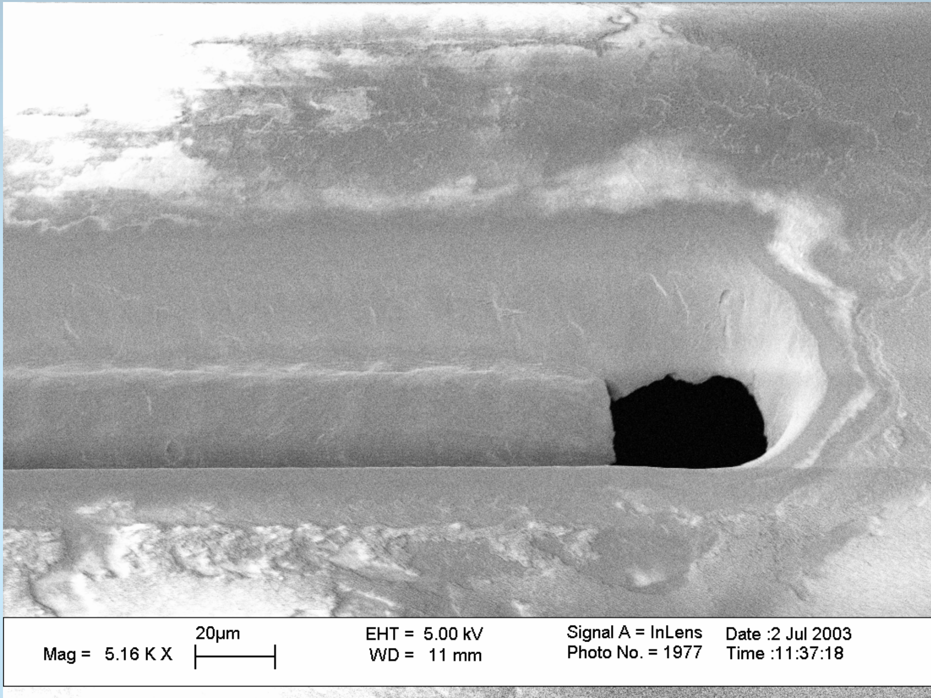


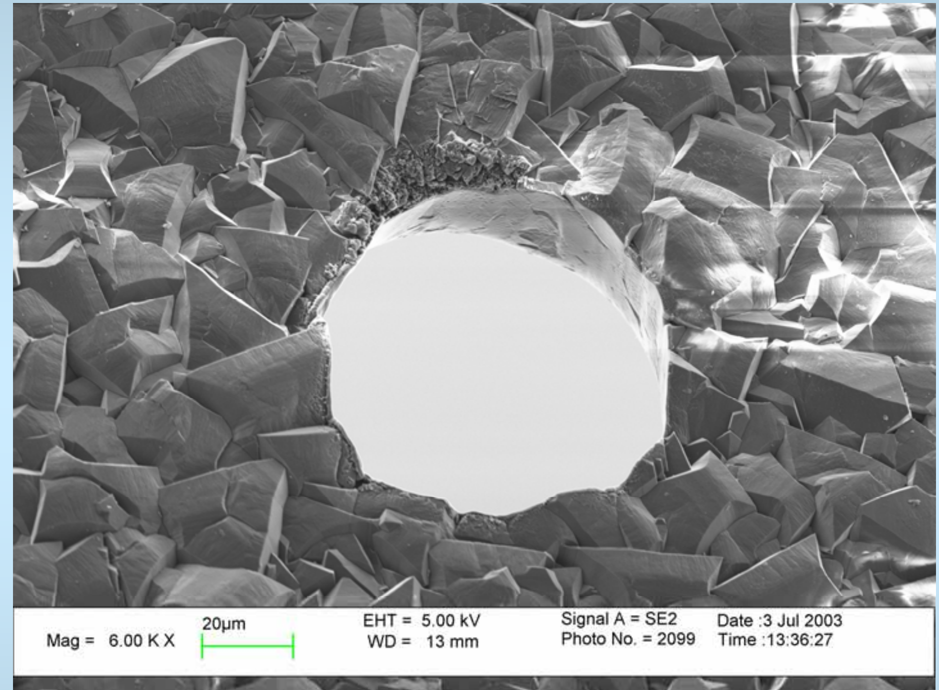


AFM picture of Mo layer in contact mode

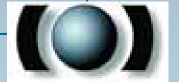


Results





Sapphire and diamond scribing and drilling



Laboratory of Material and Surface Analysis

Laboratory Equipments - LS AFM

Large sample AFM (NT-MDT Solver P7LS)

The general view of the unit

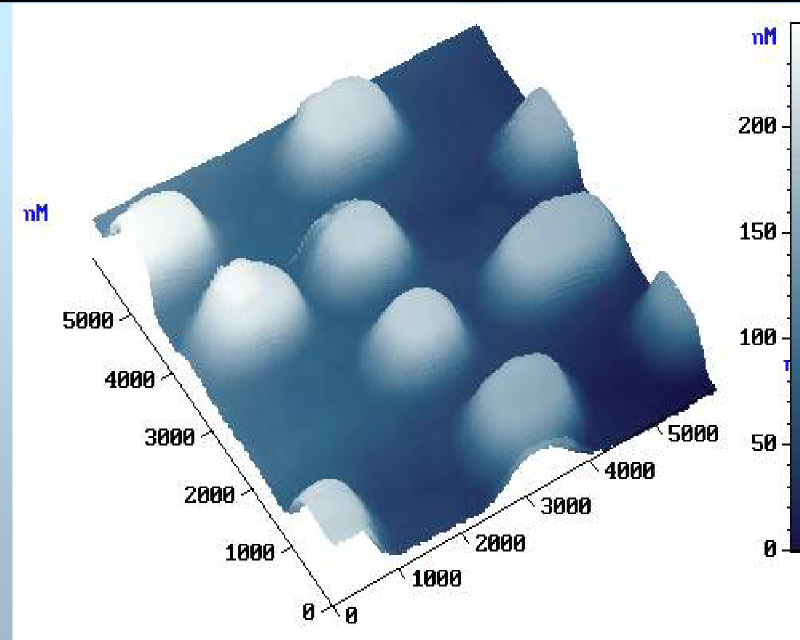
1 - measurement module, 2 - monitor, 3 - electronic control module (controller), 4 - computer with the required interface card and the software

Laboratory of Material and Surface Analysis

Atomic Force Microscopy

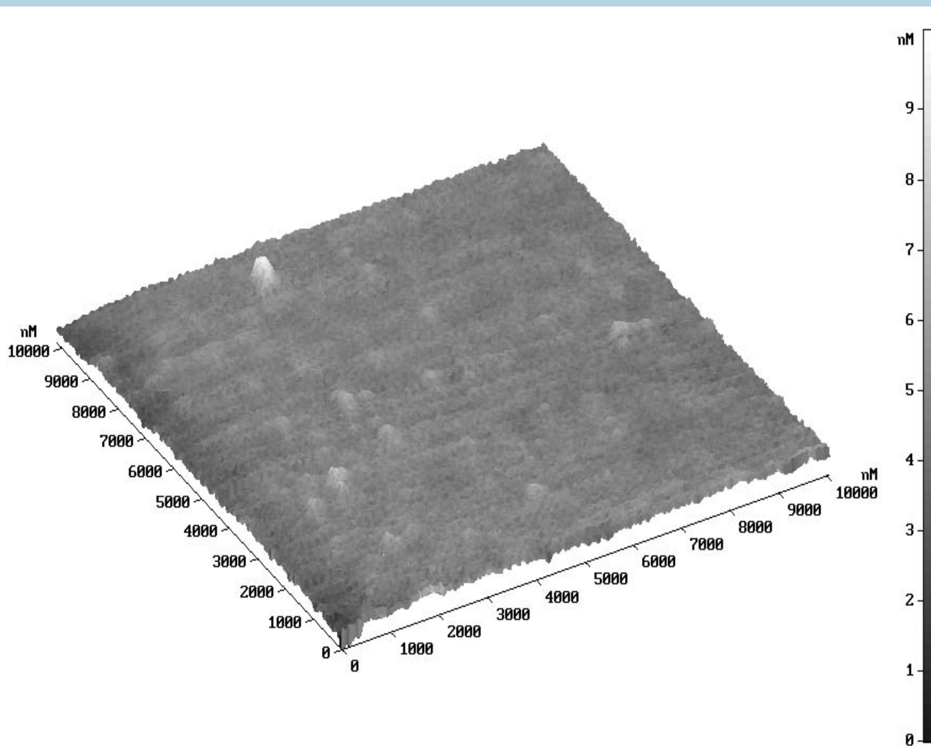
Solver P7LS NT-MDT

Scannig range	90x90x5 μm
Z resolution under appropriate conditions	0.1 nm
Sample size	Diameter up to 250 mm

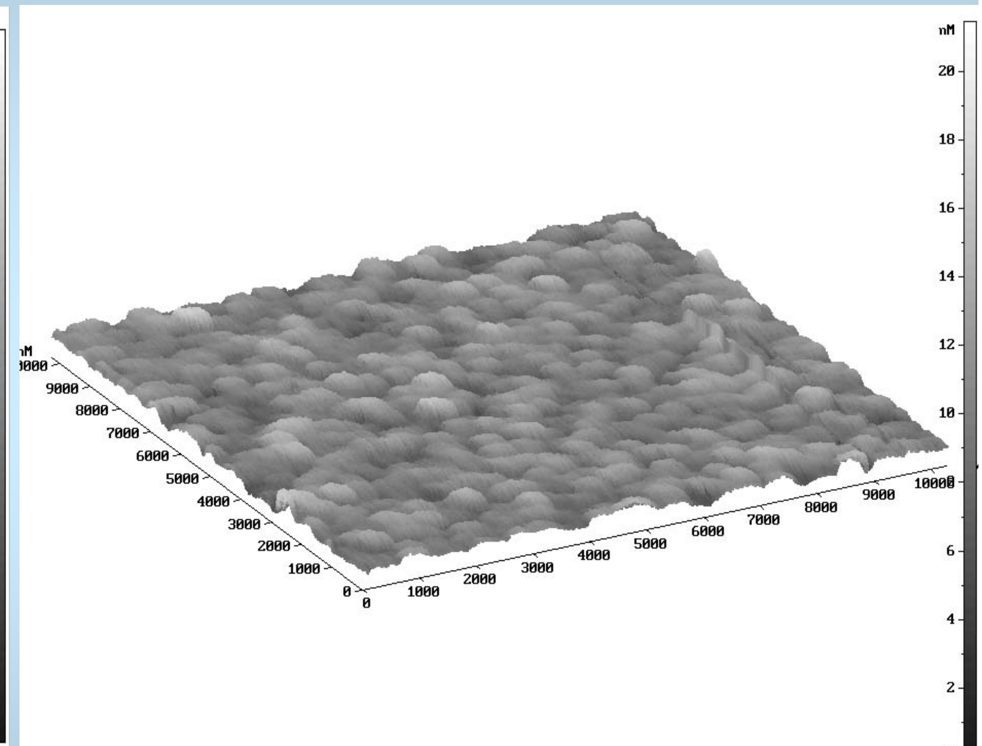


Laboratory Equipments - LS AFM

Measurement results



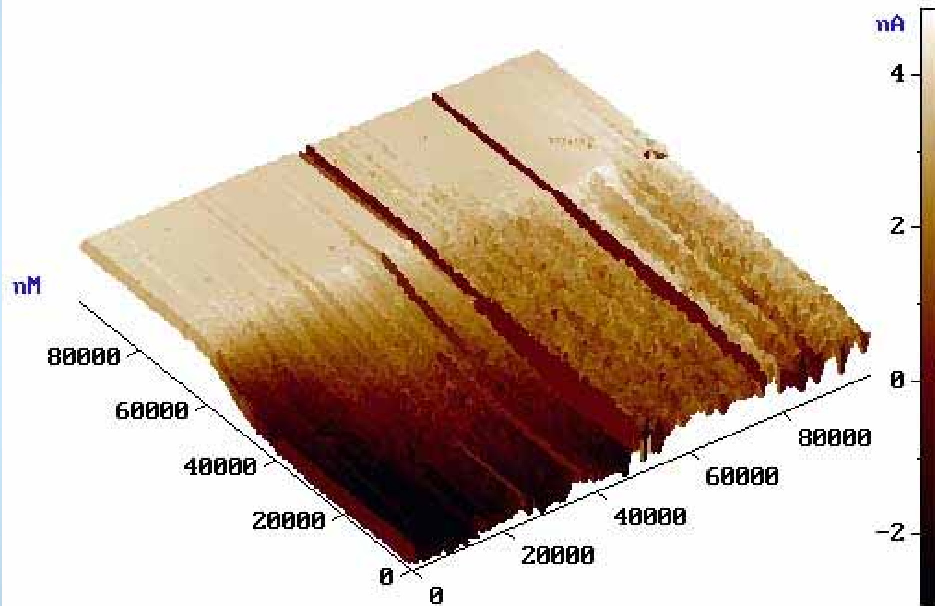
Contact mode – MgO topography



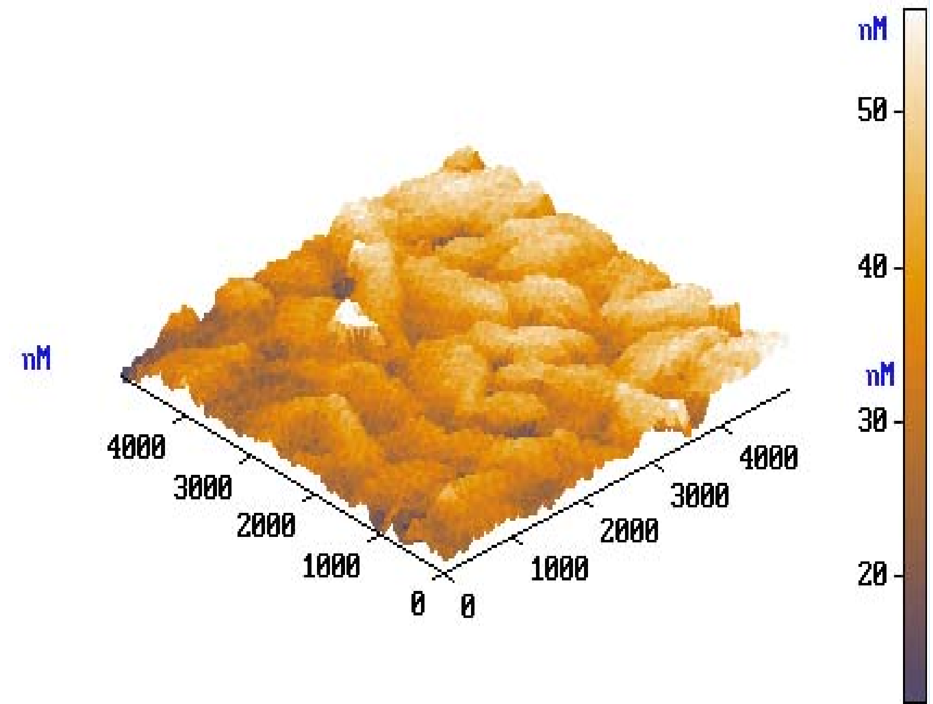
Contact mode – ZnO topography

Laboratory Equipments - LS AFM

Measurement results



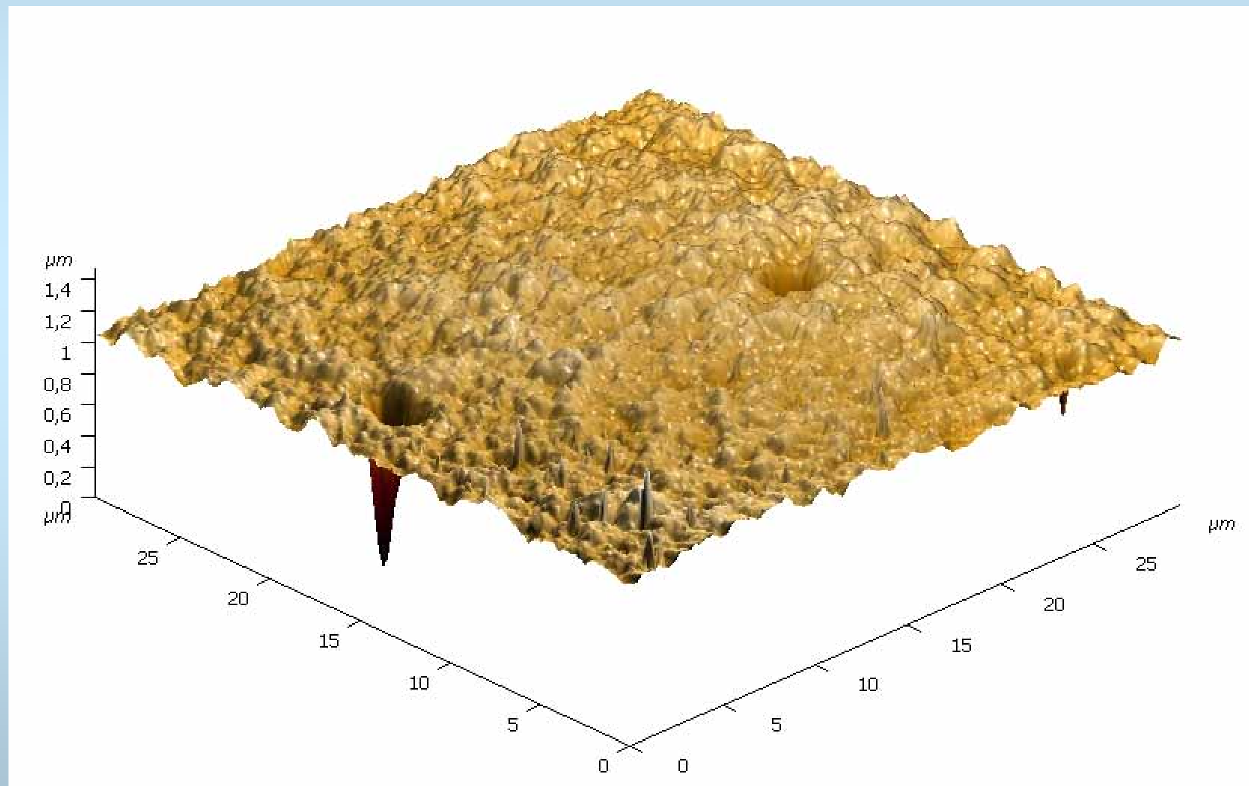
Semicontact mode – LiNiO EFM



Semincontact mode – Organic material

Laboratory Equipments - LS AFM

Measurement results



GaN – surface analysis, contact mode

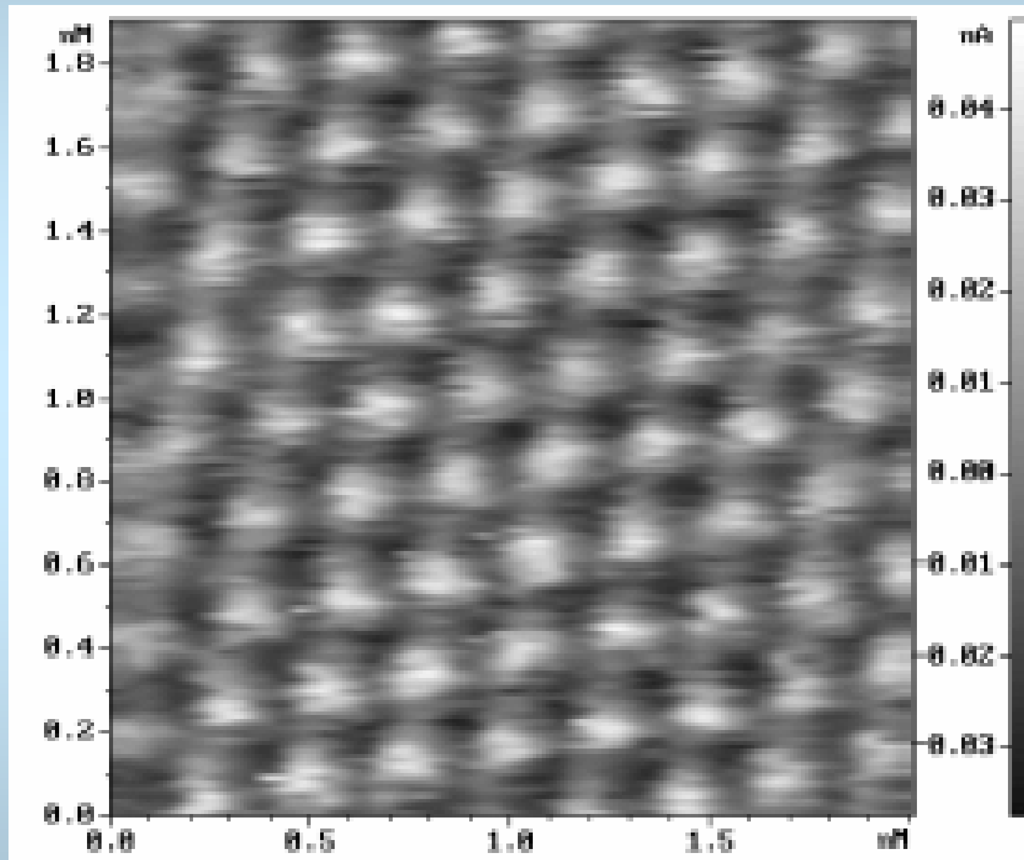
Laboratory Equipments - FA STM

Free-air STM (NT-MDT Smena)

Measuring head STM SFS012 with attachment designed for making measurements with atomic resolution AA010, without motorized approach but provided with back foot

Laboratory Equipments - FA STM

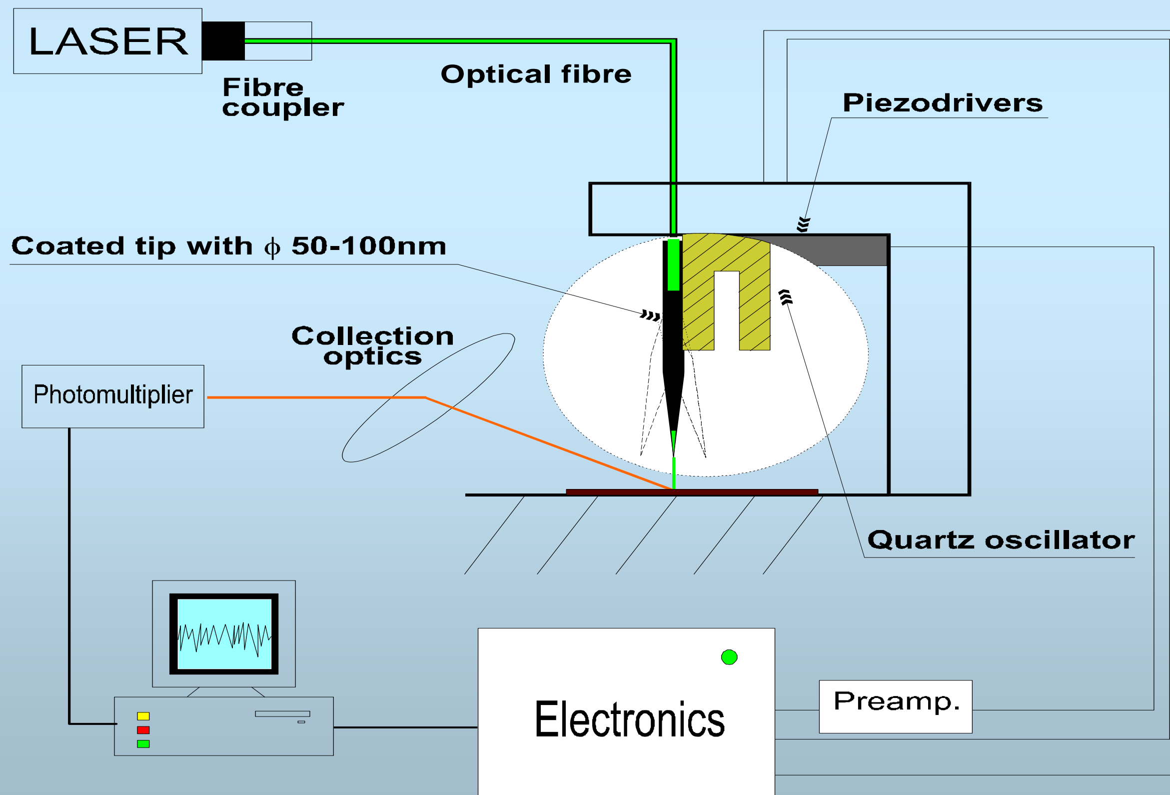
Measurement results

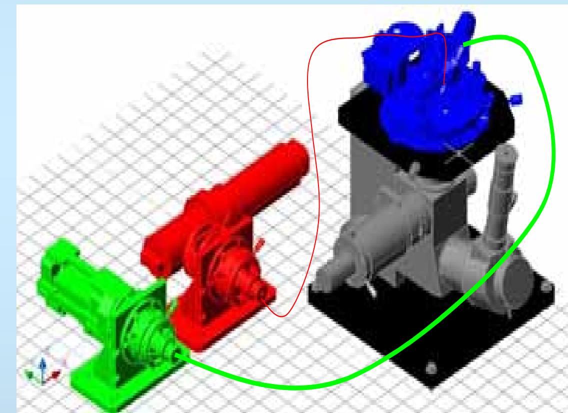


Pyrolytic graphite atomic resolution achieved by the method of “constant height” (Z=const)

Laboratory of
Material and Surface Analysis

SCANNING NEAR-FIELD OPTICAL MICROSCOPY

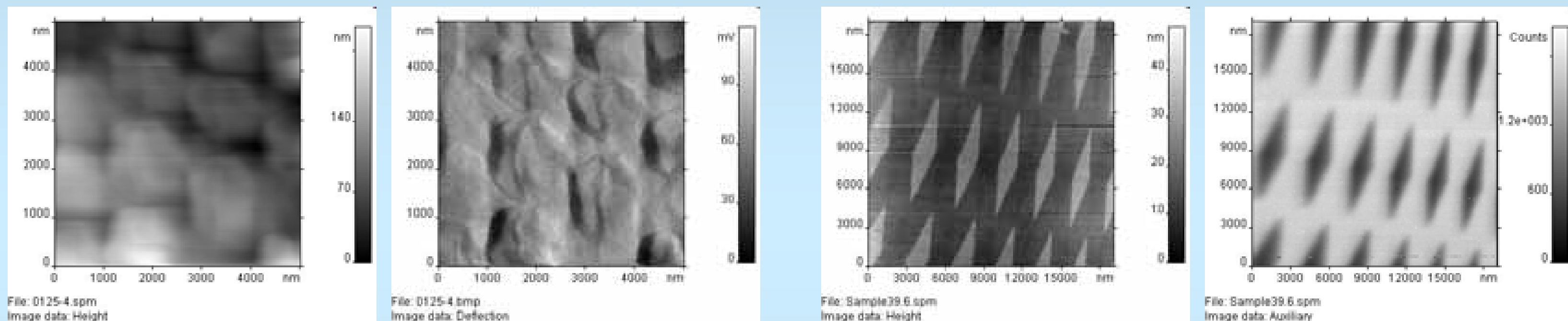




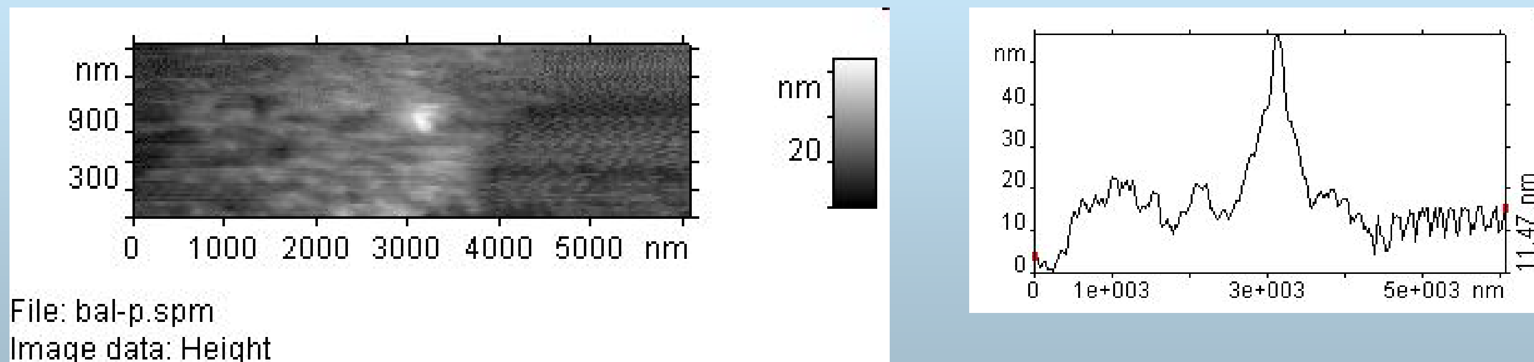
Lateral resolution ~ 100 nm

SCANNING NEAR-FIELD OPTICAL MICROSCOPY

Height, Deflection, and Optical Images of Test Pits and Lattice

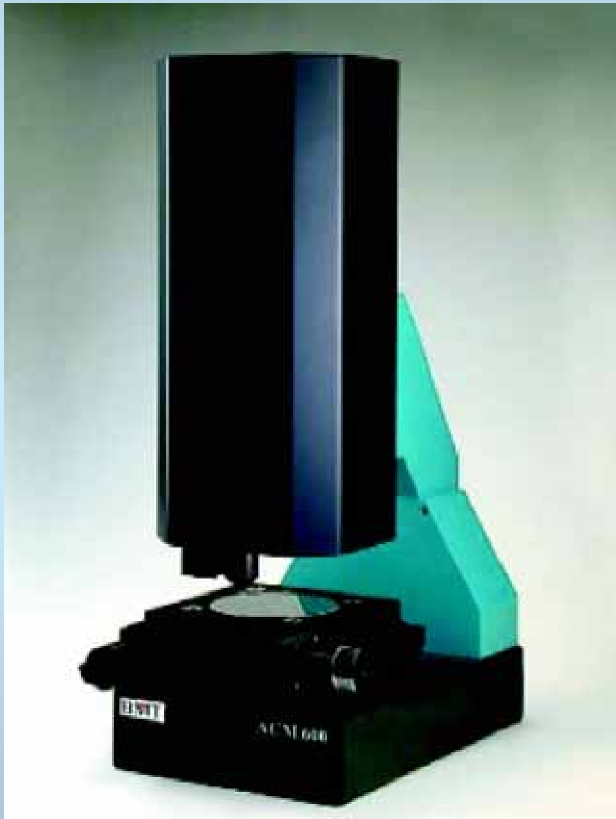


Height Image and Profile of Metal Clusters (courtesy of Dr. Majkova)



Laboratory Equipments - CMP

Confocal Microscope Profiler ACM 600 (BMT)



Fast non-contact quantification and qualification of 3D structures with high fidelity of profile reproduction

Measurement area from approx. 0.1 mm^2 to approx. 0.7 mm^2

Lateral resolution up to 1 µm, vertical max. 5 nm

Fast optics due to micro-lens disk

Measurement results are independent of material characteristics

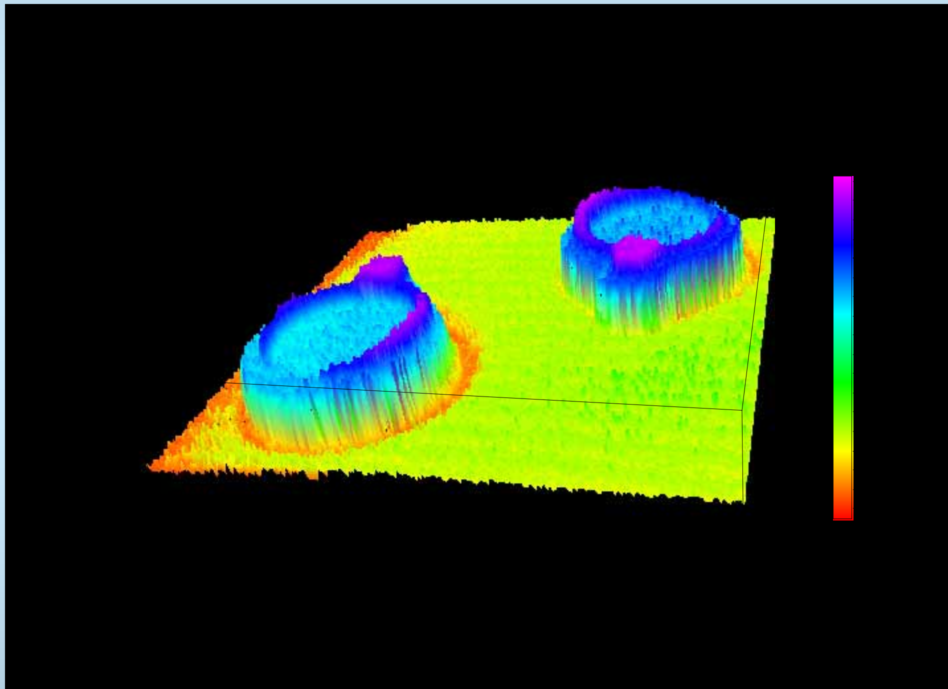
Fully automated measurement procedure

Use as ordinary microscope after motorised retraction of micro-lens disk

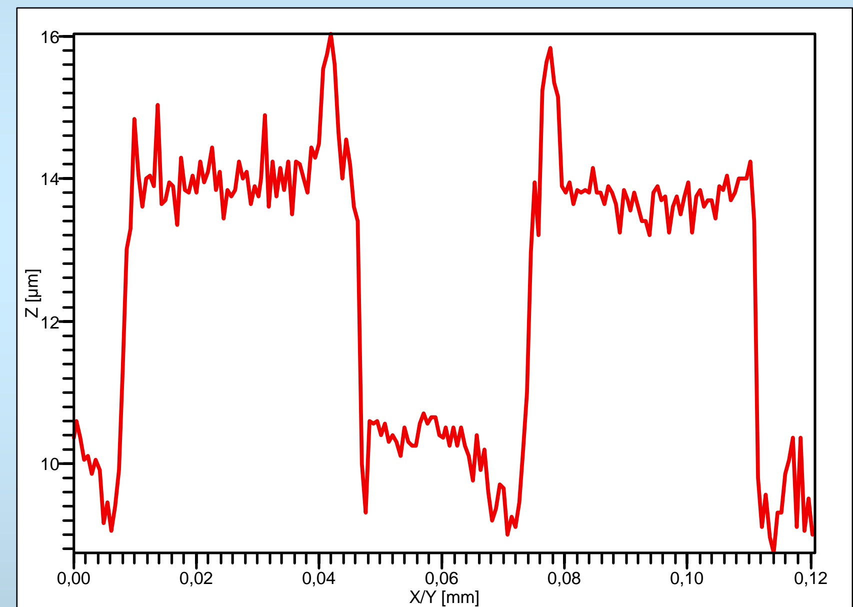
Vertical scanning with piezo drive or stepping motor

Laboratory Equipments - CMP

Measurement results



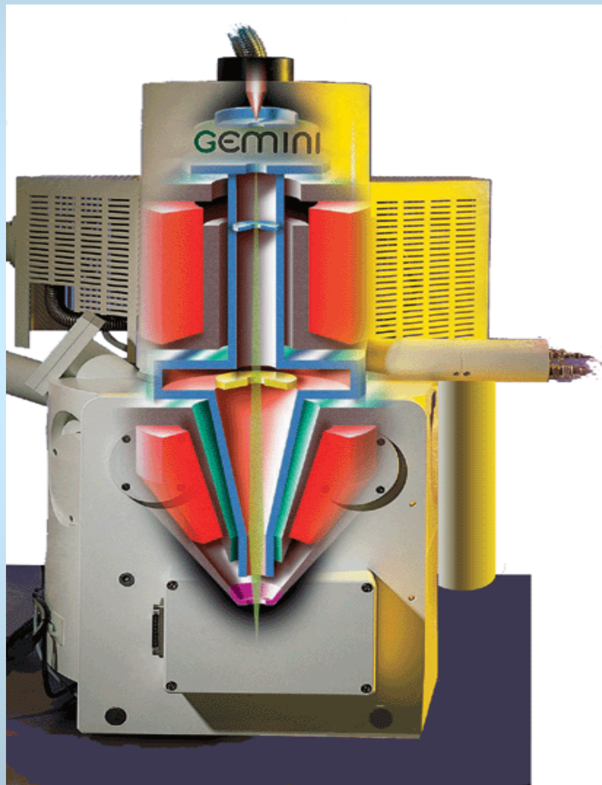
3D profile



2D profile

Laboratory of Material and Surface Analysis

Field Emission Scanning Electron Microscope - LEO 1550



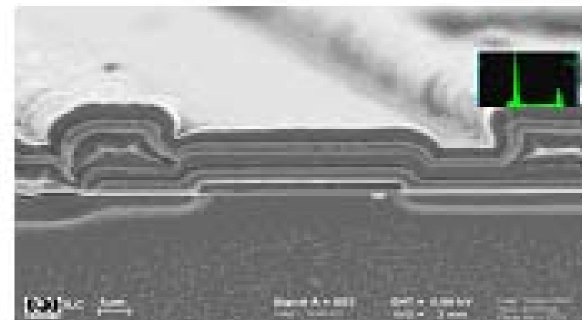
Essential properties

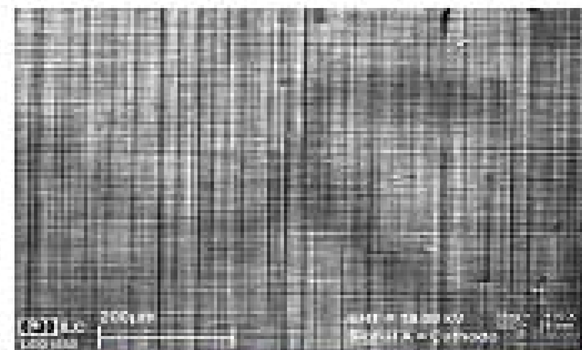
• *Resolution at WD=2mm*	**1.0nm** @ 20kV 2.3nm @ 1kV
• *Acceleration voltage*	100V to 30kV
• *Probe current*	4pA to 10nA
• *Current stability*	0.5% per hour
• *Beam blanking system*	$t_{ON,OF}$ < 20ns
• *Specimen chamber*	330mm inner diameter 270mm height

- *Motorized PC-controlled 5-axis stage*
- *Integrated data acquisition, processing and archiving*
- *Dry vacuum*

Field Emission Scanning Electron Microscope - LEO 1550

Field Emission Scanning Electron Microscope - LEO 1550



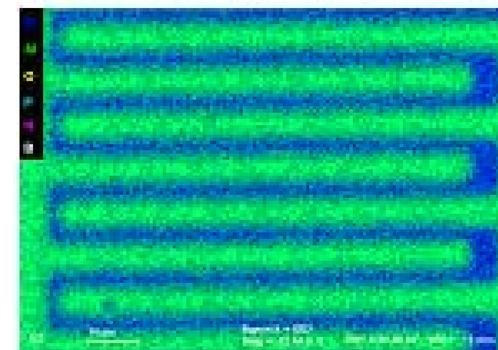


Detector system

Secondary electrons (SE)
in-lens and in-chamber configuration
▪ *Back-Scattered electrons (BSE)*
0.1 Z atomic resolution at Z=30, down to 1.5kV
▪ *Energy Dispersive X-ray Spectroscopy (EDS)*
energy resolution < 129eV, MnKα at 1.000cps
▪ *Electron Beam Induced Current (EBIC)*
voltage offset ±15V, DC coupled
▪ *Integral Cathodoluminescence (CL)*
300nm to 2000nm wavelength range
▪ *Specimen current (SC)*

Applications

- surface topography and morphology: SEI, SEII, BSE
- chemical composition analysis: EDS
- material composition mapping: EDS, CL, BSE
- semiconductor materials and structures composition mapping, defects identification: EDS, CL
- semiconductor device functional analysis: voltage contrast, EBIC, SC
- large scale detailed mapping of samples



Laboratory of SIMS

Laboratory of TOF SIMS

TOF-SIMS IV-100
(ION TOF)

Characteristics

•High Mass Resolution

> 10 000

•High Mass Acuracy

1-10 ppm

•High Mass Range

up to 10 000 u

•High Lateral Resolution

< 100 nm

•Depth Resolution

< 1 nm

Options

Surface Spectroscopy

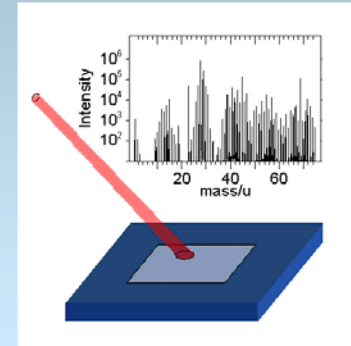
- elemental and molecular information
- ppm sensitivity
- mass resolution >10 000

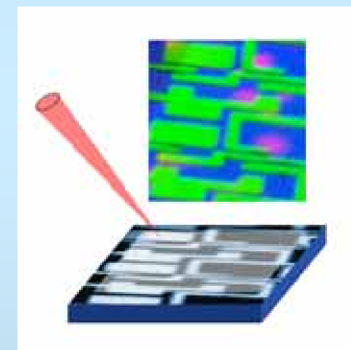
Surface Imaging

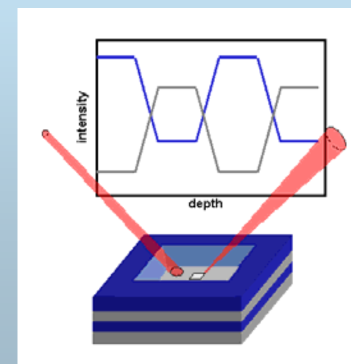
- lateral resolution < 100 nm
- parallel mass detection

Depth Profiling

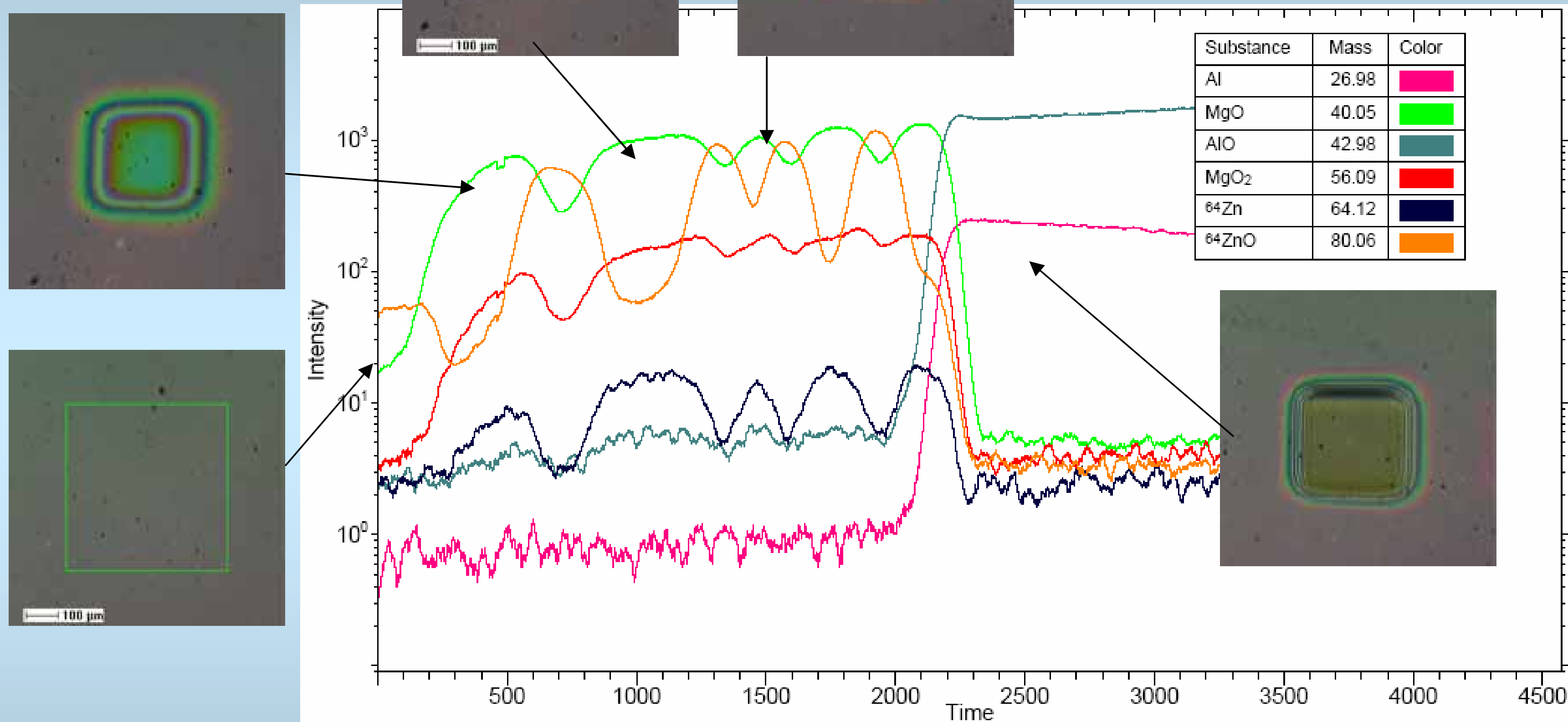
- depth resolution < 1 nm
- thin layers from 1 nm to > 10 µm
- ideal for insulators







MgZnO multivrstvy



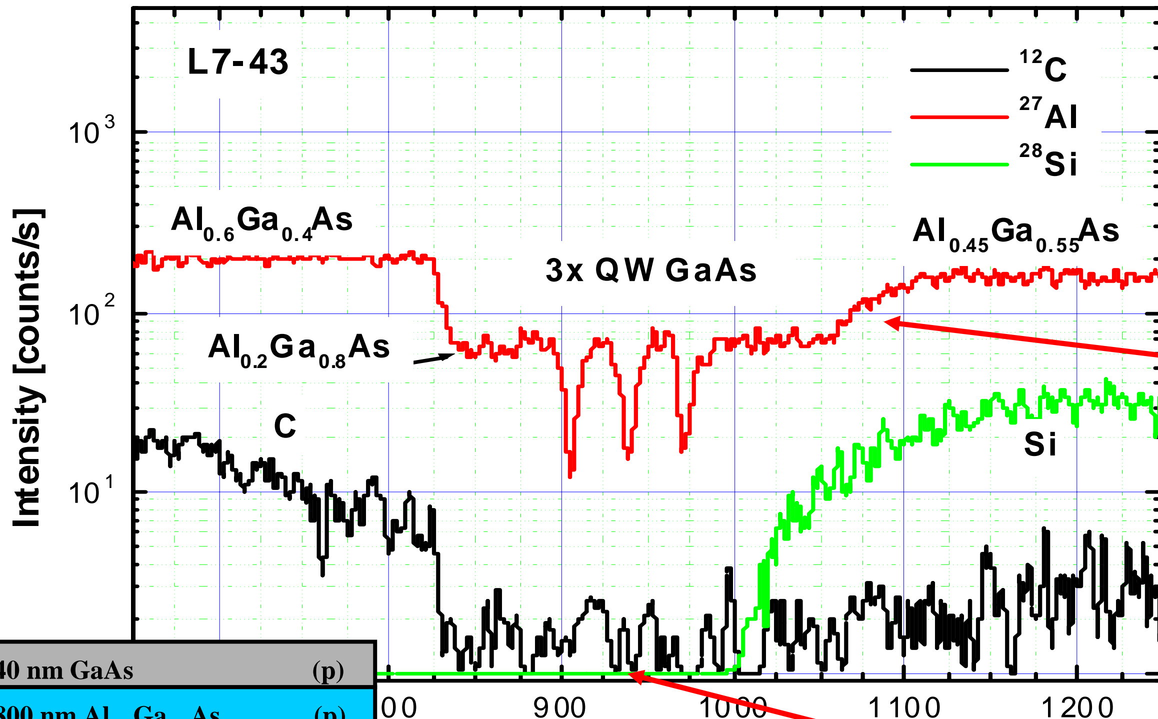
PI: Au^{+} 25 keV 50x50µm², Sputtering with Cs^{+} 2 keV 300x300µm², negative ions

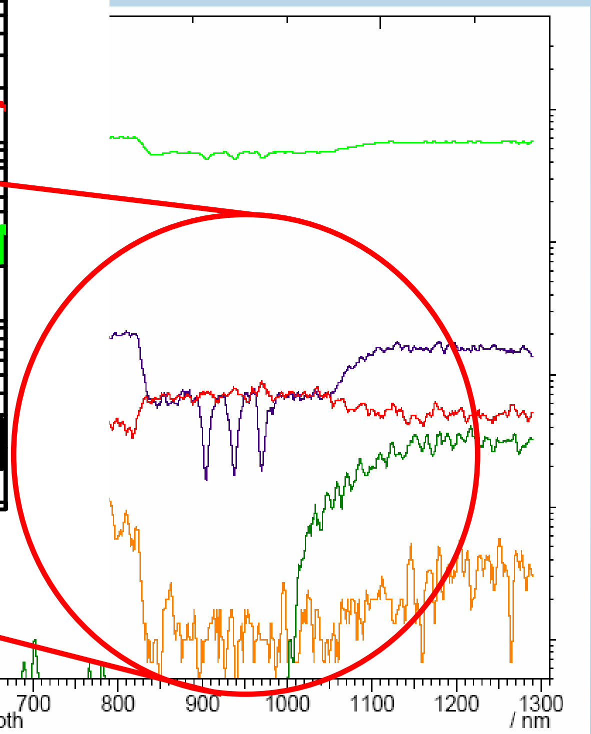
E 376, $(Mg_xZn_{1-x}O)(Mg_{1-y}Zn_yO)$ vrstvy na zafíre
Uni Leipzig, pripravené PLD

Laserová štruktúra L7-43

International Laser Center

L7-43, Uni Leipzig



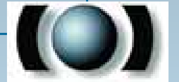


Layer	Doping
40 nm GaAs	(p)
800 nm $Al_{0.6}Ga_{0.4}As$	(p)
Active region 171 nm	(UD)
1300 nm $Al_{0.45}Ga_{0.55}As$	(n)
GaAs buffer	(n)
GaAs substrate	(n^+)

Active region layer	Doping
48 nm $Al_{0.2}Ga_{0.8}As$	(UD)
9 nm GaAs	(UD)
24 nm $Al_{0.2}Ga_{0.8}As$	(UD)
9 nm GaAs	(UD)
24 nm $Al_{0.2}Ga_{0.8}As$	(UD)
9 nm GaAs	(UD)
48 nm $Al_{0.2}Ga_{0.8}As$	(UD)

...µm², Sputtering with Cs^+ 0.5 keV 250x250µm², positive ions

J. Ková... h, SIMS investigation of semiconductor laser ... MOVPE, SIMS Europe 2004, September 2004, Münster, Germany



Laboratory of Applied Optics

Laboratory of Applied Optics

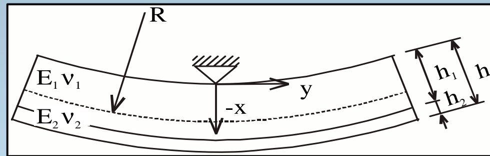
Equipment:

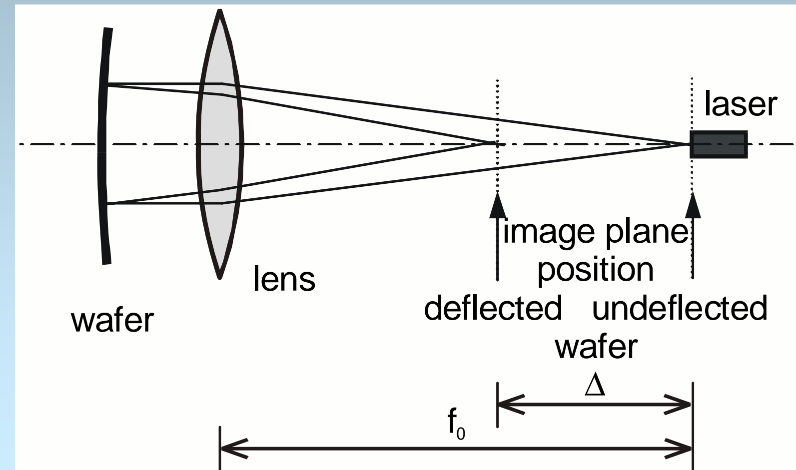
- laser Doppler vibrometer - Polytech
- single frequency DPSS laser 532 nm, 50 mW
- optic sets for holography and interferometry
- laser deflectometry
- opto-acoustic defectoscopy

Applications:

- measurements of the vibration frequencies, velocity and displacement amplitudes of vibrating surface
- measurements of deformations of the objects,
- analysis of the vibrations of the automobile engine, chasis, wheels, etc.

Multilayer structures – residual stresses analysis





Factors for residual stresses developing

- mismatch of thermal expansion coefficients of layers (**thermal stress**)
- microstructural changes, mutual diffusion of materials at their interface, adhesion (**intrinsic stress**)
- thermal/humidity gradients

Autocollimation optical setup – plate deformation measurement

Accuracy: ±**1 MPa** for residual stress

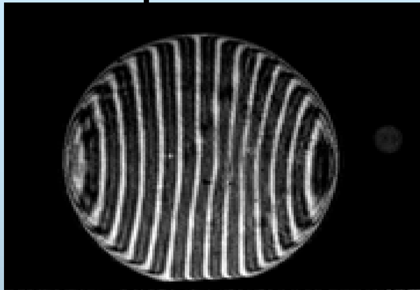
Range of measurement: up to **10 GPa**

Anisotropic thin layer stress state evaluating

Plotting of the measured stress-temperature curve and **superposition** of theoretically calculated thermal stress

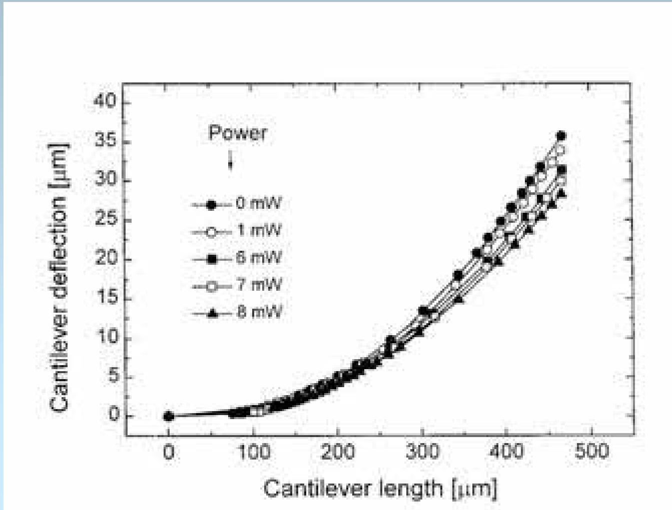
Separation of thermal/intrinsic components

Slope contours



Method based on binary grid optical filtration stress analysis of thin plates elements

Microinterferometer

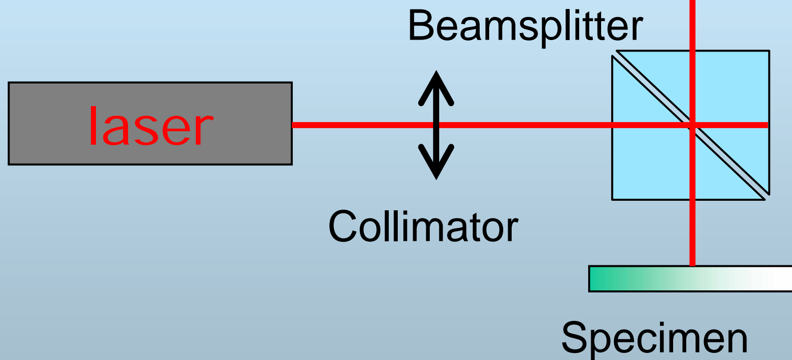


λ = 532 nm, 633 nm, 650 nm

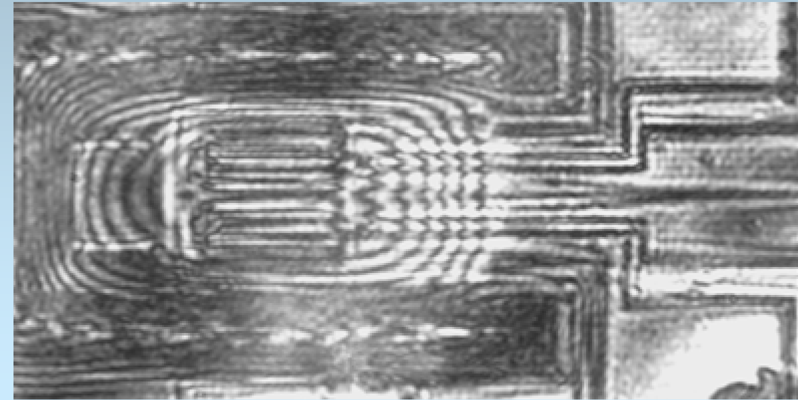
$$w = m\frac{\lambda}{2n}, \quad m = 0, \pm 1, \pm 2, \ldots$$

n – index of refraction

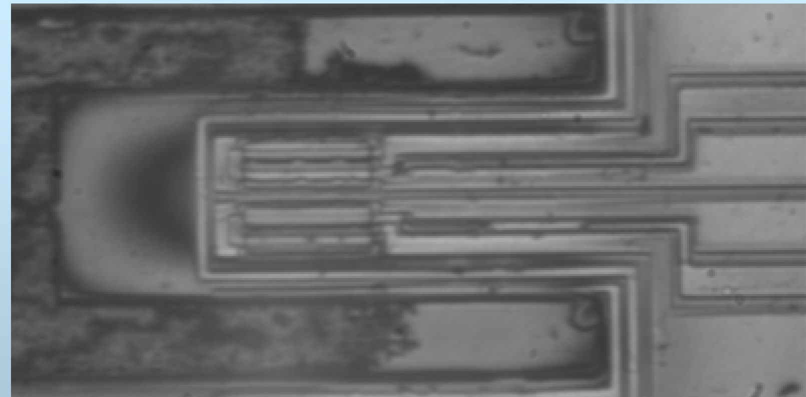
CCD



Tolansky interferometer

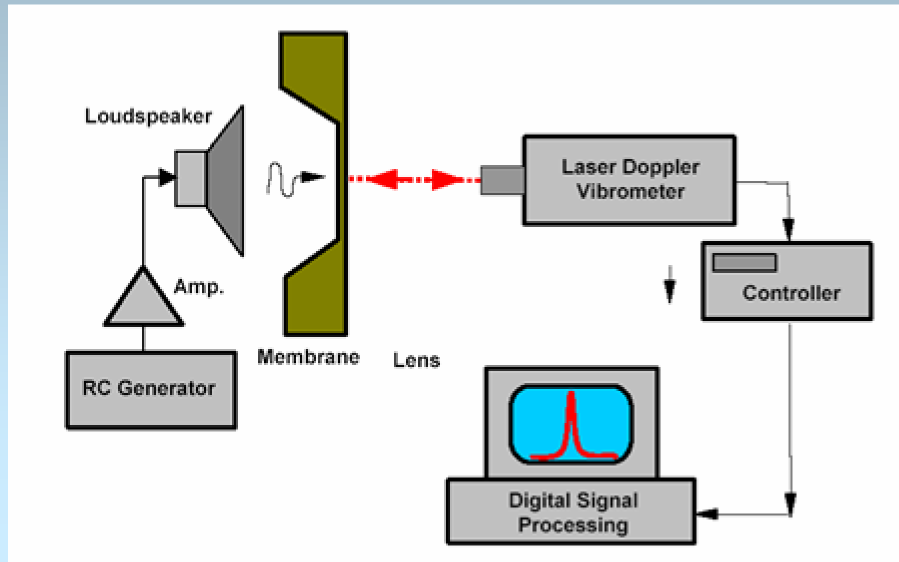


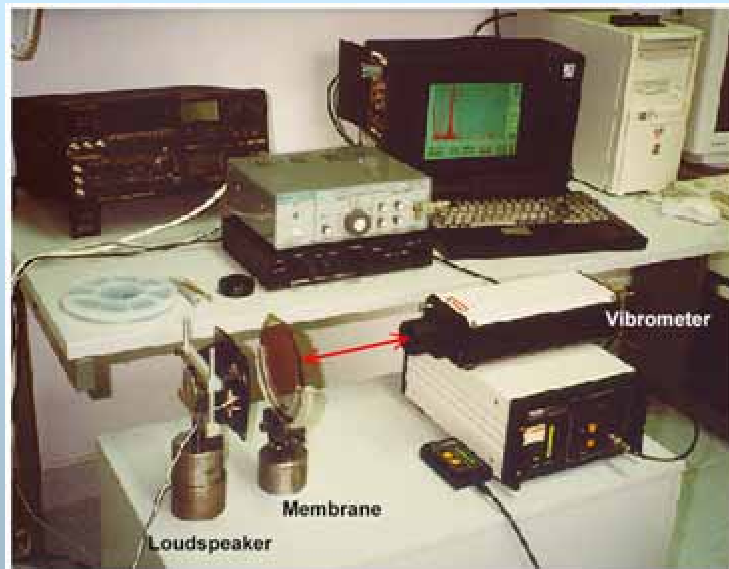
- Interference pattern



- Polyimide thin film interference

Si MEMS stress measurement





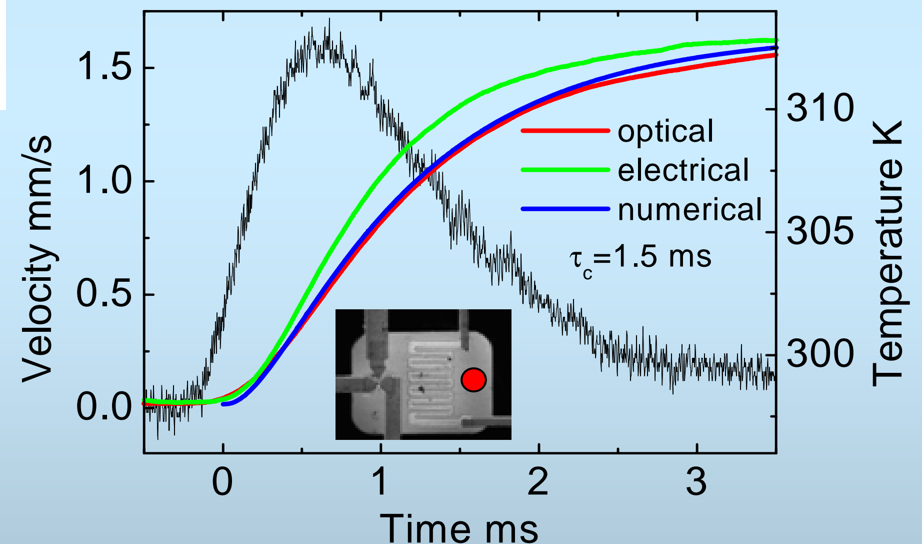
Evaluation of stress state of MEMS

In-situ stress measurement

at the various deposition processes inside vacuum chamber

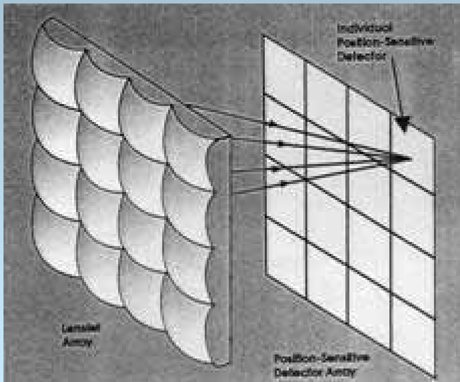
Laser Doppler Vibrometer

- Polytec OFV-303
- *Vibration amplitudes:* **1÷10 nm** and more
- *Frequency range:* **20 kHz**



Laser Doppler Vibrometer -besides the vibrational measurement also **one-shot events detection.**

Silicon wafers flatness diagnostic measurement – Shack-Hartmann



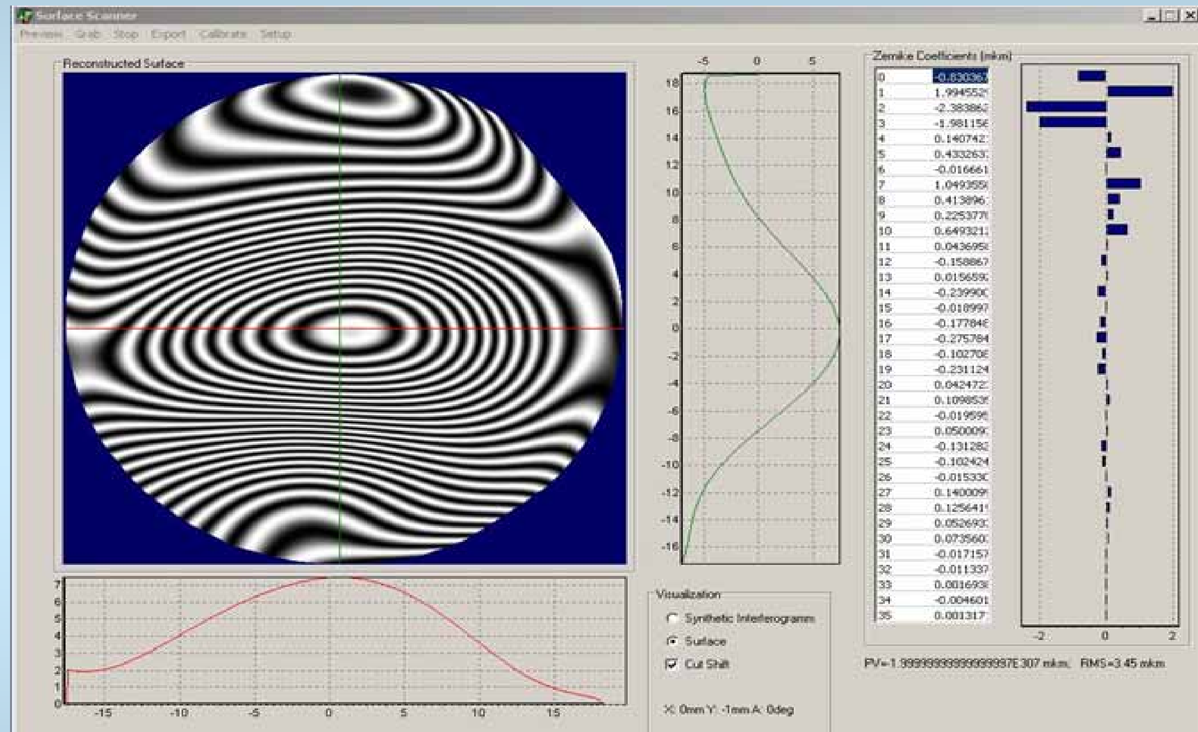
Light source:
LD 670nm/1 mW

CCD camera:
VCCI-A LCL 902C
Watec Am. Co.

Resolving power:
better then **3 nm**

Maximum substrate diameter:
100 mm (4 inches)

Absolute and relative way of measurement:
flat etalon or arbitrary reference plane



Complete Silicon (GaAs) wafers surface characterisation

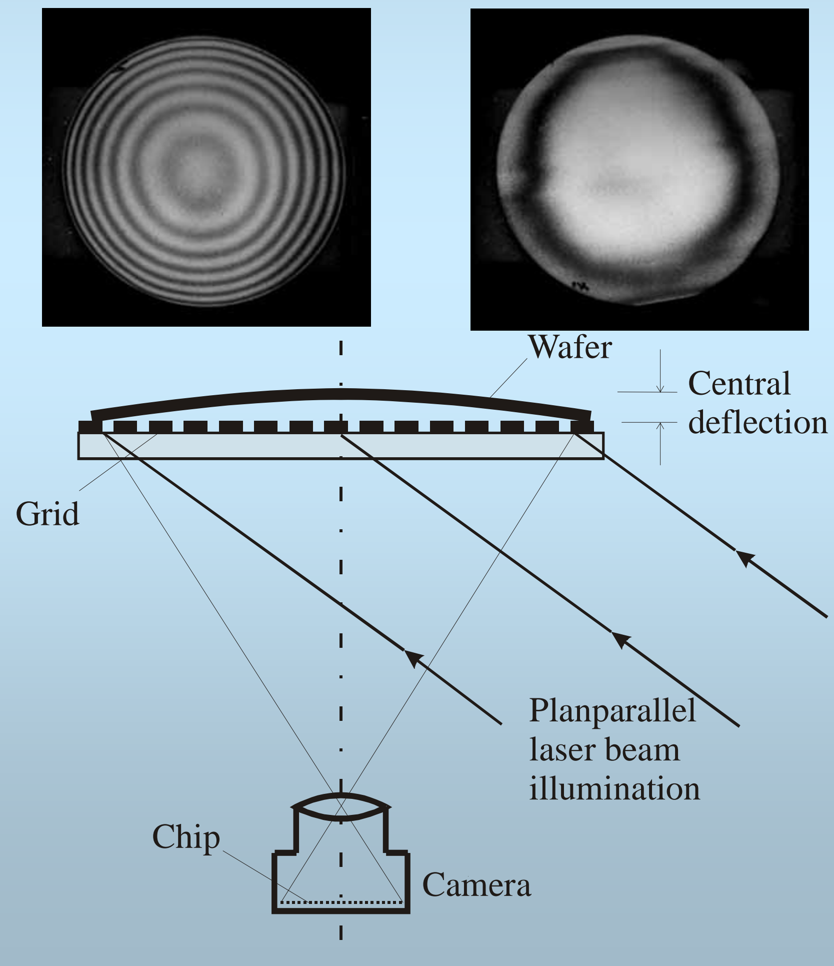
Global flatness expressed in
- TIR – total indicator reading
- or FPD – focal plane deviation
- or BOW – central deflection

Global thickness
- TTV – total thickness variation

Methods used
- Autocollimation optical setup
- Confocal measurement system
- Holographic/speckle interferometry

- Surface texture analysis
  - roughness and waviness
  - R_a – arithmetic average
  - R_q – RMS of roughness
- Surface roughness anisotropy

Global flatness of diffuse-like surfaces
- moire microprofilometry

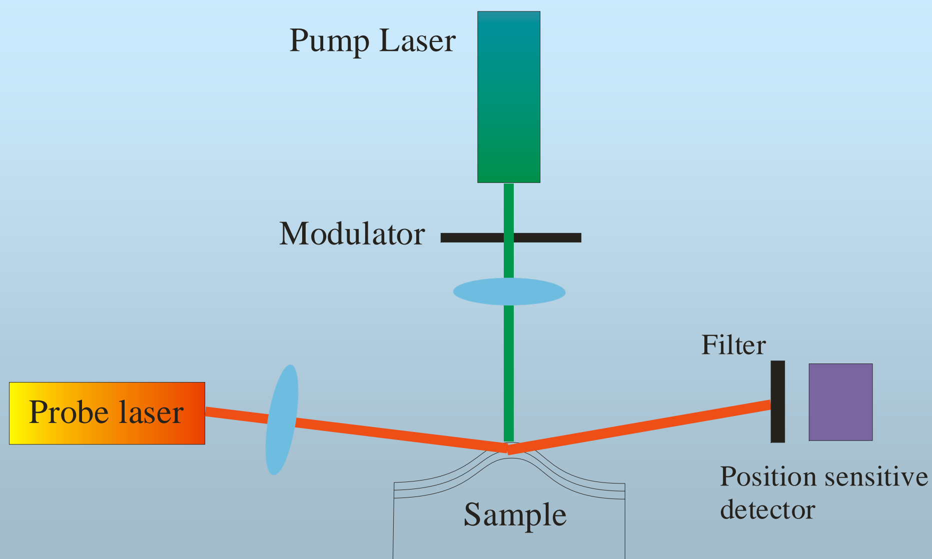


Thermal properties analysis

- **Specific heat**
- **Thermal diffusivity**
- **Thermal conductivity**

Laser Excitation and Deflectometry

Pulsed photothermal deflection effect



Deflectometer

Laser excitation: **Nd:YAG** 1.06 μm/**200mW**

Laser probe: **He-Ne** LGN-303, **2mW**

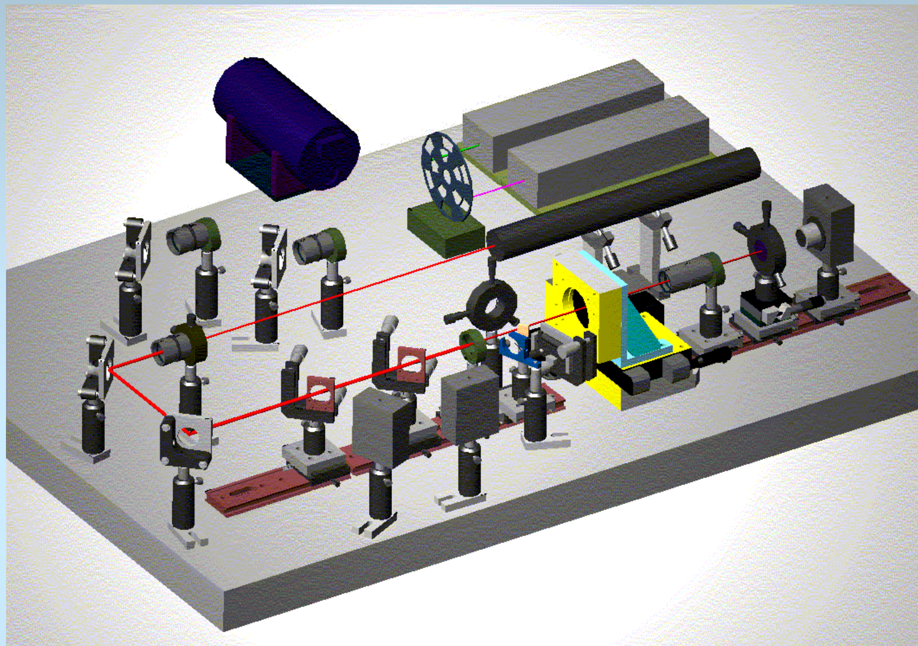
Detector: photodiode **FD-19**

Lock-in amplifier: Stanford Research System

Chopper: **10÷2000Hz**

Oscilloscope, CCD camera, adjustment equipment

Laser based thermoelastic evaluation of thin layers material properties



Laser based ultrasonics

⇒ *E* – Young's modulus
n – Poisson's ratio
ρ – density
of the thin layer material

Thin layers < **1 μm**

⇒ it needs very short laser pulses **1 ÷ 10 ns**,

⇒ at least **1 mJ** energy Nd:YAG laser 1064 nm

⇒ frequency of surface acoustic wave (SAW) up to **1 GHz**

Laser based photothermal deflection spectroscopy (PSD)

contactless laser pulse excitation
laser probe sensing

evaluation of thermal parameters

- *thermal conductivity*
- *thermal diffusivity*
- *specific heat*

Precise time of flight of SAW wavefront determination

- autocorrelation wave signals processing

Laboratory of Femtosecond Spectroscopy

Laboratory of Femtosecond Spectroscopy

Areas of activity:

- Fluorescence study for OLED and semiconductor heterostructures development
- Fluorescence study of host-guest molecular complexes
- Supercontinuum generation in photonic crystal fibers
- Pump-probe spectroscopy of metal nanoparticles

Equipment:

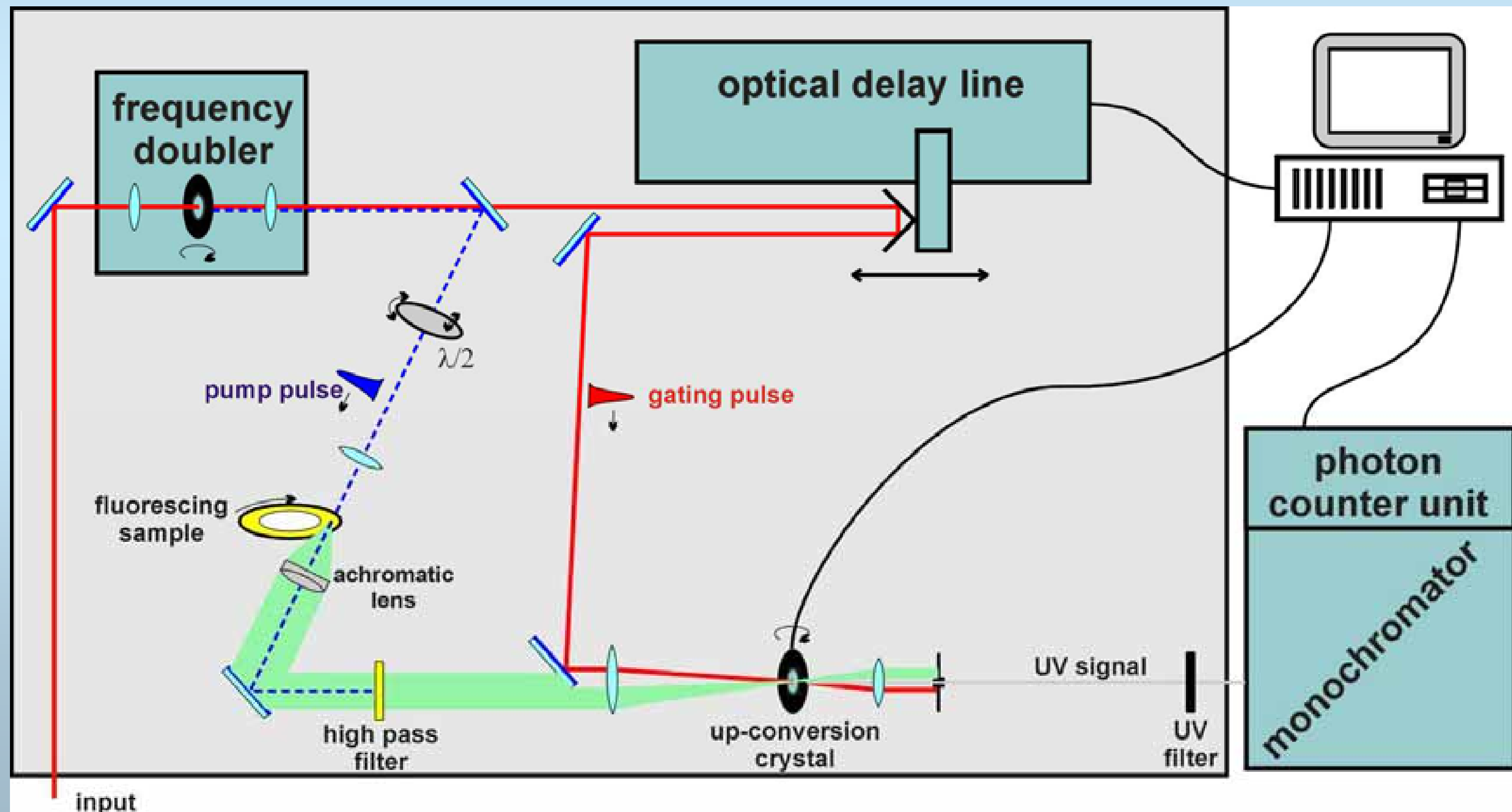
- Sources of femtosecond laser pulses
  - TiF50 Ti:sapphire fs oscillator (CDP)
  - MPA50 Ti:sapphire fs amplifier (CDP)
  - CrF65 Cr:forsterite fs oscillator (PCC RAS)
  - Frequency conversion system of fs pulses (CDP)
- Equipment for femtosecond spectroscopy
  - Fluorescence up-conversion system (CDP)
  - Pump-probe measurement system (CDP)

Femtosecond time-resolved fluorescence study

APPARATUS:

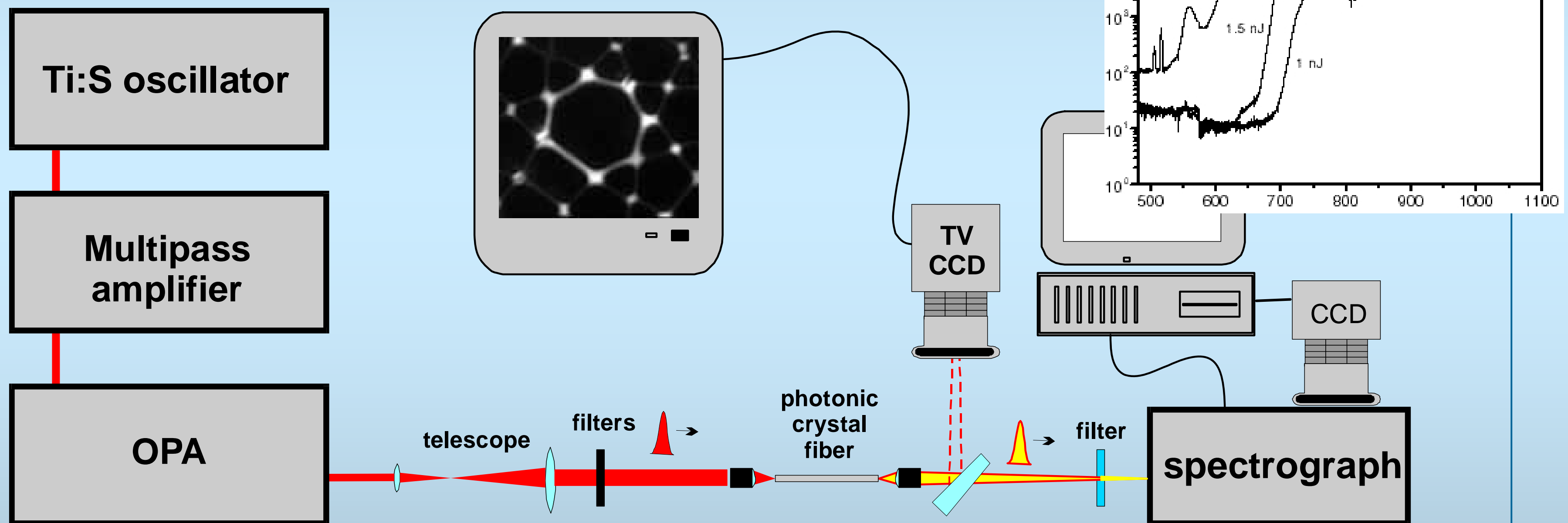
- **Ti:Sapphire oscillator CDP TiF50,** 50 - 100 fs, 760 - 830 nm (100 fs), 80 MHz, 400 mW
- **Cr:Forsterite oscillator PCC RAS CrF65,** 50 - 65 fs, 1250 - 1275 nm, 120 MHz, 450 mW
- **Fluorescence up conversion system CDP FOG100**

  6.25 fs – 600 ps, 370 – 1500 nm, 4 kHz – 100 MHz



Supercontinuum Generation in Photonic Crystal Fibers

Cooperation with the group of prof. Zheltikov in the ILC of Moscow State University and with TE Glass Structures Institute, Saratov, Russia



Experimental apparatus in the Laboratory of Femtosecond Spectroscopy in ILCB

Our Location and Contact Information

International Laser Center
Ilkovičova 3
Bratislava
812 19
Slovak Republic

tel.: +421 / 2 / 654 21 575
fax.: +421 / 2 / 654 23 244

uherek@ilc.sk
http://www.ilc.sk