CEITEC NANO

Research Infrastructure
BRNO | CZECH REPUBLIC

BRNO UNIVERSITY OF TECHNOLOGY

UNIVERSITAS MASARYKIANA BRUNENSIS

About CEITEC Nano Research Infrastructure

CEITEC Nano Research Infrastructure provides complex equipment, expertise and methods for nanotechnology and advanced materials R&D. The CEITEC Nano facilities for nanofabrication, nanocharacterization, structural analysis and X-ray tomography enable the user to carry out complete fabrication of nanostructures and nanodevices and their characterization down to the sub-nanometre level in an entirely clean environment. The nanofabrication laboratory is located in a class 100 cleanroom with an area of 356 m^2 and the nanocharacterization laboratory is located in a class 100,000 cleanroom with an area of 1,337 m^2. The structural analysis laboratory occupies a further 300 m^2 of class 100,000 cleanroom. Originating from the CEITEC project, the research infrastructure was financed by EU structural funds between 2011–2015 and began full operation in September 2016. The initial investment was 240 million CZK for the cleanroom technology and 660 million CZK for the equipment. Currently, CEITEC Nano is acknowledged in the national Roadmap of Large Research Infrastructures for 2016–2022 and its running costs of approx. 40 million CZK per year are covered mainly by the Czech Ministry of Education, Youth and Sports and partially by Brno University of Technology and by Masaryk University.

CEITEC Nano labs

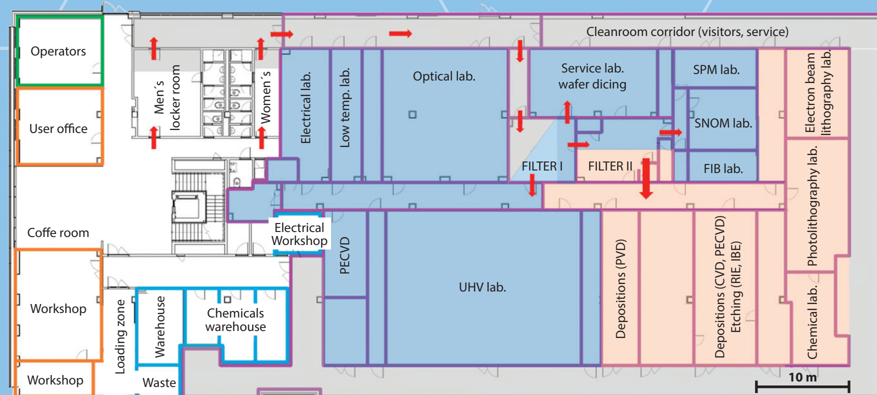
Nanofabrication	Nanocharacterization	Structural analysis
Lithography - EBL, FIB, UV, laser scanning, nanoimprint **Chemical lab** - Wet benches, multiple processes **(Plasma)chemical and thermal processes** - LPCVD, ALD, MOCVD - PECVD, RIE, DRIE - IBE, RIBE, CAIBE **Depositions (PVD)** - Magnetron sputtering - E-beam evaporator - IBAD **Packaging** - Wire bonder - Laser dicer	**Microscopy** - SEM/FIB, SPM, SNOM, Kerr microscopy **UHV techniques** - XPS - Scanning Auger microscopy - SIMS, LEIS - UHV complex system **Elmag measurements** - 9T, 1.4K ppms - LT probe station - Automated probe station - High-temperature probe station **Optical measurements** - VUV-VIS-MIR spectroscopic ellipsometers and spectrometers, FTIR microscope	**Microscopy** - HRTEM - HRSEM with EDS, WDS - FIB/SEM - Sample preparation (TEM lamella, sample polishing) **X-ray diffraction** - High-resolution X-ray diffractometer - Powder diffractometer **X-ray CT** **Computed tomography** - Micro CT - Nano CT

Users

Academic users: most users of the research infrastructure come from academic institutions in the region and worldwide. Academic users typically use the infrastructure via a self-service system. They get training and support from CEITEC Nano staff and then do the nanofabrication and/or measurement on their own. The instrument hours are free of charge; the academic users pay only a service fee of 30 000 CZK per year per user.

Commercial users: the research infrastructure also attracts high-tech companies giving them access to state-of-the-art equipment for nanotechnology and materials science. They typically order measurement or nanofabrication services and their samples are processed by CEITEC Nano staff. Commercial users pay an hourly charge which includes the full instrument running cost and instrument depreciation. The hourly charge varies between 1600–7000 CZK per hour, depending on the equipment.

Nanofabrication and Nanocharacterization cleanroom



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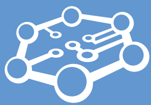
MŠMT MINISTRY OF EDUCATION, YOUTH AND SPORTS

European Union European Regional Development Fund

nano.ceitec.cz



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SERVICES FOR INDUSTRIAL USERS

Mechanical & automotive engineering

ABOUT

CEITEC Nano facilities are equipped with state-of-the-art equipment for multiple analytical tasks which can be used by the mechanical engineering and automotive industry in process development, quality control and failure analysis.

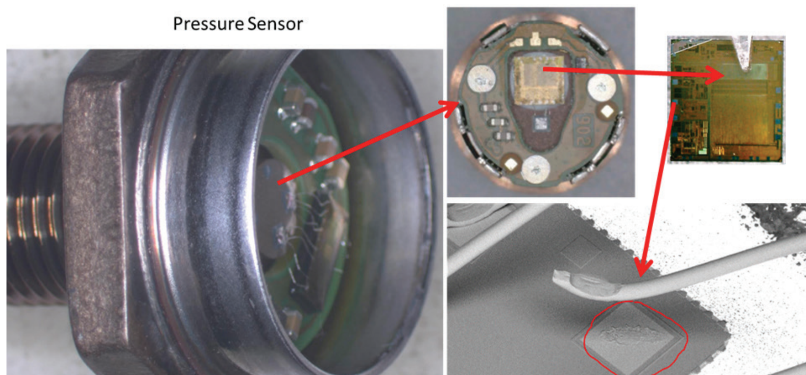
EQUIPMENT

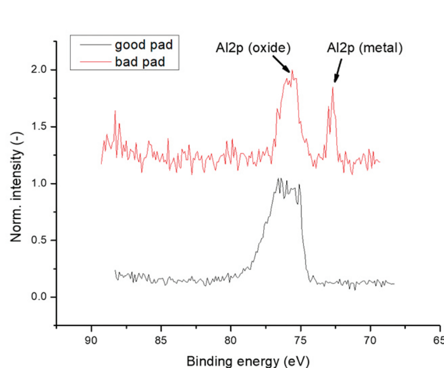
Optical, electron and scanning probe microscopies. TEM lamela preparation (FIB and classical). Metallographic sample preparation. Optical, X-ray, electron and ion spectroscopies. X-ray computed tomography. Electric and magnetic measurements. Fully equipped nanofabrication facility.

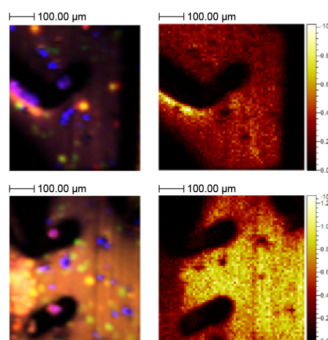
APPLICATION EXAMPLES

1. Failure analysis

Multiple analytical techniques were used to determine the source of bad adhesion of wire bonding pads in pressure sensors.







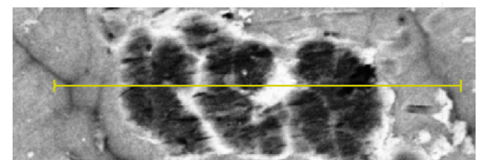
Techniques used:
SIMS, XPS, SEM, FTIR microscopy

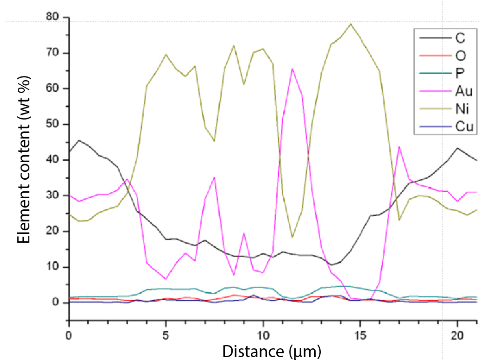
MORE INFO

Web: http://nano.ceitec.cz

2. Analysis of wear patterns

Wear patterns were analyzed for suspected oxidation. No oxidation was proved; the patterns were separated particles from the matching counterpart.

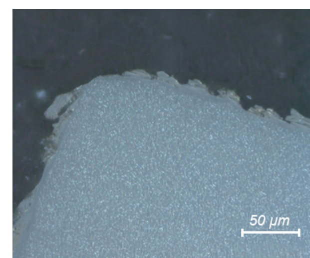


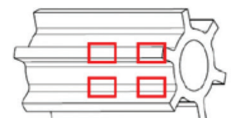


Techniques used: SEM, EDX

3. Metallographic evaluation

Evaluation of electro-discharge machining for the aerospace industry. Assessment of potential presence of α modification of TiO_2 in the corrosion layer. The corrosion layer is not present; nonetheless, numerous surface cracks found. Modification to the machining parameters recommended.





Techniques used:
Sample preparation, Optical Microscopy

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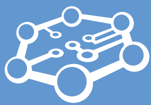

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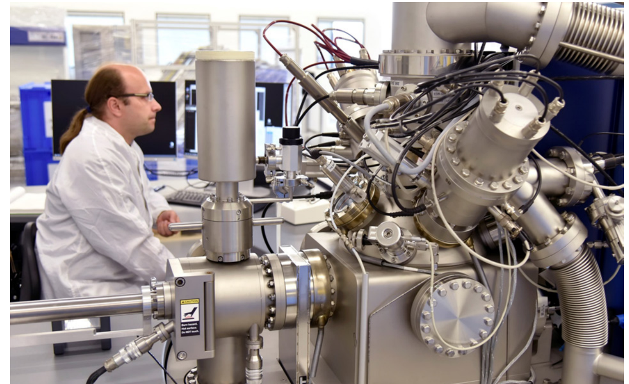
SERVICES FOR INDUSTRIAL USERS

Semiconductors & Materials Engineering

ABOUT

CEITEC Nano facilities are equipped with state-of-the-art equipment for multiple analytical tasks which can be used in material design and science, material process control, semi-conductor industry and failure analysis.



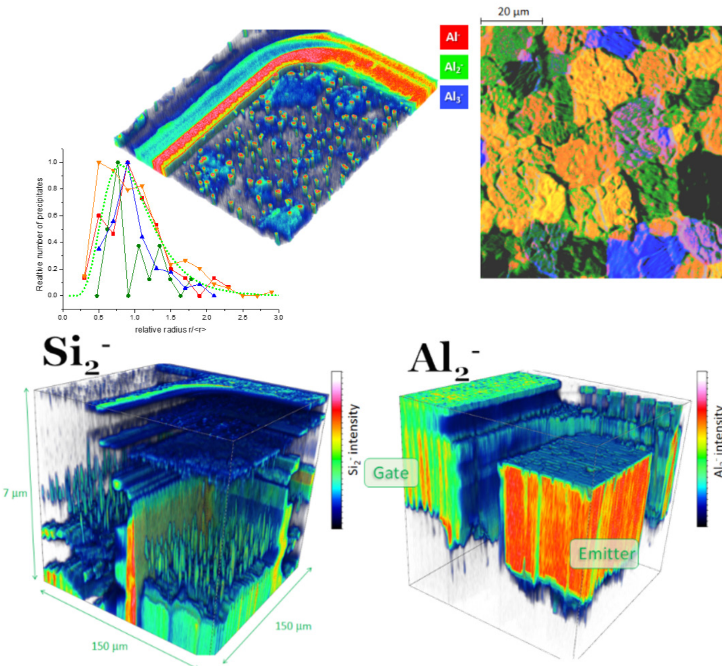
EQUIPMENT

Optical, electron and scanning probe microscopes (OM, SEM, TEM, SPM). TEM lamella preparation using focus ion beam (FIB). Material analysis using secondary ion mass spectrometry (SIMS), energy dispersive X-ray spectrometry (EDX).

APPLICATION EXAMPLES

1. Al contacts – Si precipitates

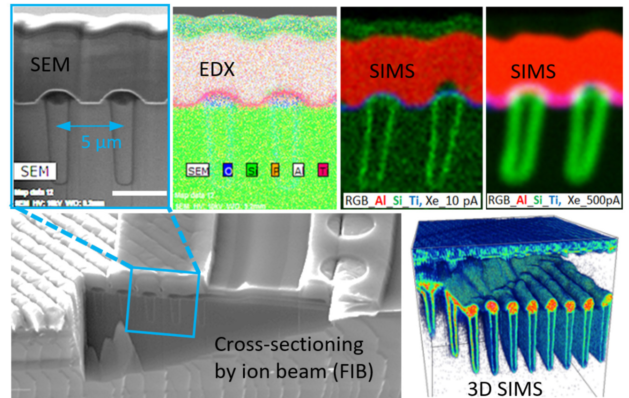
3D material structural analysis with high mass, spatial and depth resolution.
Resolving of Al grains, Si precipitates in grains, grain boundaries and Si/Al interface. 3D precipitates size evaluation.



Techniques used: SIMS, FIB+SEM

2. TIGBT structural analysis

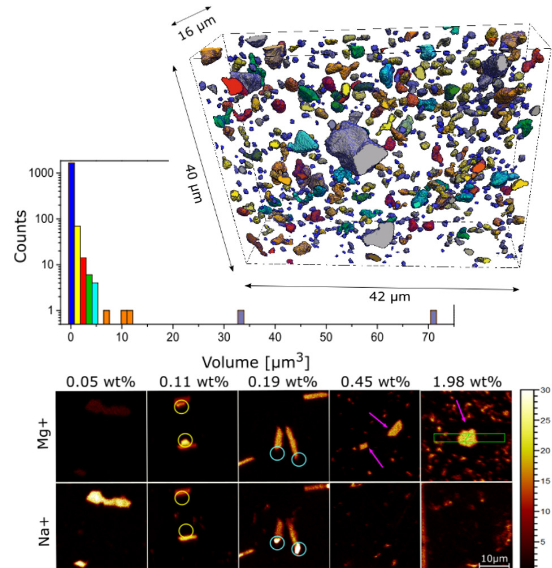
Structure analysis of side part of TIGBT transistor.



Techniques used: FIB+SEM, EDX, SIMS

3. $MgAl_2O_4$ spinel in Al_2O_3

3D analysis of size distribution of spinel particles in ceramics.



Techniques used: SIMS, SEM

MORE INFO

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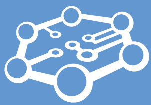

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SERVICES FOR INDUSTRIAL USERS

Semiconductor, Chemistry, Mechanical and Materials Engineering

ABOUT

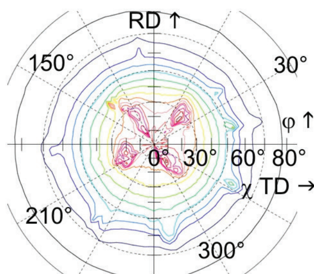
The CEITEC Nano X-ray laboratory solves the material analysis tasks by comprehensive analytical methods, utilizing the phenomenon of X-ray diffraction and scattering. The need for precise determination of phase composition, crystal quality and other quantities accompanies a material since its development throughout its mass production.

EQUIPMENT

X-ray diffractometers are adapted for phase composition measurement (e. g. amount of austenite in heat treated steels, amorphous phase in polymers, purity of ceramics, products quality control), crystal quality and orientation of single-crystals (needed in semiconductor or optics applications), stress and texture measurement (e. g. in geared wheels, metal sheets, turbine blades).

APPLICATION EXAMPLES

Pole figure (1 1 3) diffraction CrMnCoFeNi HEA single-crystal.
(Texture measurement/orientation of single-crystal.)



Civil Engineering – Phase analysis

Ceramics – Phase analysis

Mechanical Engineering – Phase analysis, stress and texture measurement

Geology – Phase analysis and texture measurement

Pharmacy – Phase analysis

Archeology – Phase analysis

Chemical indrustry – Phase analysis

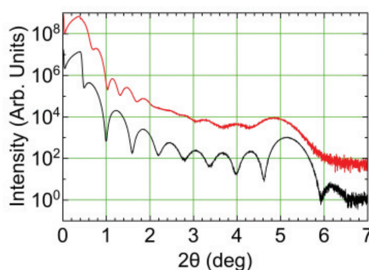
Semiconductors – Crystal quality, thin films analysis

Art – Phase analysis

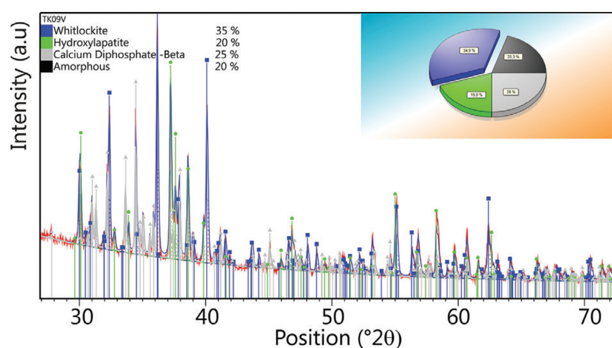
Polymers – Phase analysis, amorphous phase

Single-crystals – Quality and orientation of single crystals

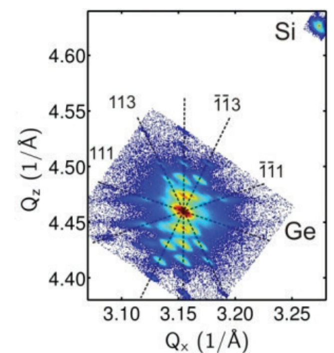
X-ray specular reflectivity on a thin film of an organic semiconductor and a similar layer with a metal electrode above.
(XRR, reflectivity measurement)



Qualitative and quantitative phase analysis of bio-ceramic.
(Phase analysis)



HRXRD Reciprocal space map on facetted Si/SiGe heterostructures.
(single-crystal quality; reciprocal space mapping)



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High-Resolution Transmission Electron Microscope FEI Titan Themis3 60–300

DESCRIPTION

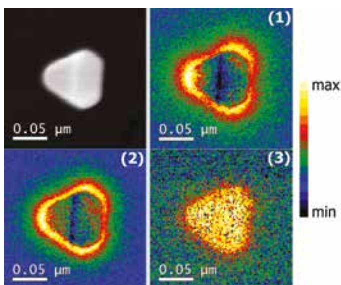
The TEM FEI Titan™ Themis provides easy access to atomic information about electron transparent samples. The microscope combines high-brightness X-FEG module with monochromator, 3-lens condenser, SUPER-X EDX detector, image (Cs)-corrector and high-end GATAN GIF Quantum ERS/966 energy filter for EELS and EFTEM to achieve comprehensive results. Thanks to new enhanced piezo stage, FEI Ceta 4k×4k 16-bit CMOS camera, multiple STEM detectors (BF, ADF and HAADF), variation of analytical holders (ST, DT, Tomographic and Cryo), implemented Lorentz lens and sophisticated SWs for data acquisition and post-processing the microscope can be used as a complex and automated tool for materials science.

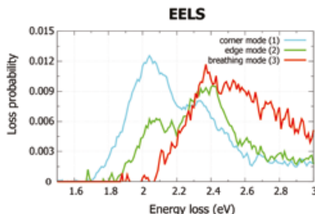
RESOLUTION and DETECTION LIMITS:			
		STEM RESOLUTION (300 kV):	≤ 136 pm
TEM INFORMATION LIMIT (300 kV):	≤ 70 pm	PIEZO STAGE STEP:	≥ 20 pm
TEM INFORMATION LIMIT (120 kV):	≤ 90 pm	EDX ENERGY RESOLUTION:	≤ 136 eV
TEM INFORMATION LIMIT (60 kV):	≤ 100 pm	EDX DETECTION LIMIT:	Z ≥ 5 (Boron)
LORENTZ TEM RESOLUTION (300 kV):	≤ 2 nm	EEELS ENERGY RESOLUTION:	≤ 0.2 eV

ACCELERATOR – offers minimized knock-on damage of beam sensitive samples, high contrast for light compounds or penetration power for dense materials by alignment of high tensions to 60 kV, 120 kV or 300 kV, respectively.
MONOCHROMATOR – narrows the energy spread of electron source to achieve 70 pm lateral resolution in HR-TEM or high energy resolution in EEL spectroscopy as low as 100 meV.
ChemiSTEM – is a FEI ultimate EDX technology for fast chemical analysis. It includes high brightness X-FEG Shotky electron gun, SUPER-X spectrometer with 4x30mm2 SSD windowless detectors offering 0.8 srad solid angle, and 100,000 spectra/sec fast data acquisition.
IMAGE CORRECTOR – is spherical aberration (Cs)-corrector, CEOS GmbH, which boosts the resolution of the HR-TEM mode to the sub-Angström level. It minimizes the effect of delocalisation in HR-TEM imaging which enables determination of artefact free atomic coordinates.
LORENTZ LENS – is within the lower pole piece of the objective lens and enables imaging of magnetic structures in field-free conditions (the objective lens is turned off). Focusing the Lorentz lens allows imaging of magnetic domains with different properties.
GIF QUANTUM ERS/966 – is a post-column energy filter, Gatan, Inc., alowing to form energy-filtered images, energy-filtered diffraction patterns and electron energy-loss spectra. The Quantum™ ERS is perfectly matched to monochromated electron beam, enabling high-resolution EELS of both core-level and valance state transitions. The system includes high speed spectrum imaging and DualEELS™ with hardware synchronized for ultrafast S/TEM EELS spectrum imaging at two energies.

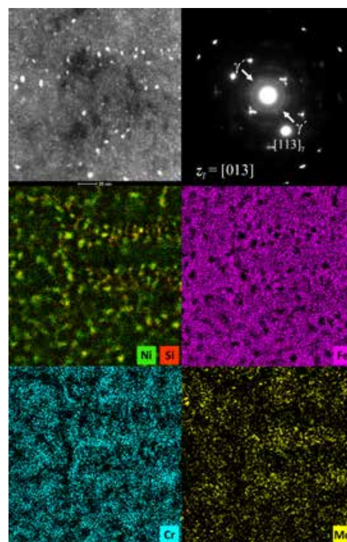
APPLICATIONS RESULTS

Monochromated STEM-EELS:
Plasmon resonances in a gold nanotriangle [1]

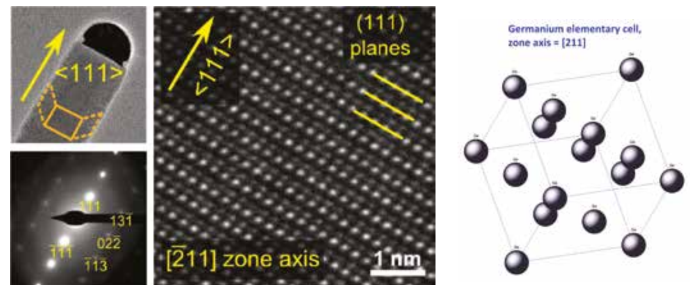




STEM-EDX:
Radiation-induced nanoprecipitates in a 316 stainless steel irradiated by self-ions [2]



HR-TEM (300 kV):
Atomic structure of germanium nanowires grown in hydrogen atmosphere [3]



REFERENCES

1. M. Horák, V. Křápek, T. Šikola, *Fine Mechanics and Optics*, Vol. 62, No. 11-12, 2017.
2. J. Michalička, Z. Jiao, G.S. Was, Radiation-Induced Precipitates in a Self-Ion Irradiated Cold-Worked 316 Austenitic Stainless Steel Used for PWR Baffle-Bolts, *18th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors*, Portland, USA, 2017.
3. M. Kolíbal, T. Pejchal, T. Vystavěl, T. Šikola, *Nano Letters*, 16 (8) (2016) 4880–4886.

MORE INFO

Guarantor: **Jan Michalička** (jan.michalicka@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/high-resolution-scanning-transmission-electron-microscope-fei-titan-themis-60-300-cubed/

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Scanning Electron Microscope with Focused Ion Beam

FIB-SEM TESCAN LYRA3

DESCRIPTION

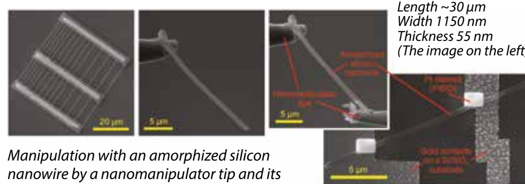
The SEM/FIB is a type of microscope where a focused electron/ion beam is scanned over the sample to generate an image of the surface or to modify it with nanometric resolution (usually better than 10 nm). The image is formed by detecting secondary and backscattered electrons emitted from the impact place of a particle beam. The Gas Injection System (GIS) provides a gas inlet for gaseous precursors, thus allowing deposition and enhanced or selective etching on the sample surface using advanced surface chemistry. The microscope is equipped with two closed loop nanomanipulators (optionally two more can be installed), which allows measurement of 2-probe or 4-probe current-voltage characteristics. The tool is equipped with Electron Dispersive X-Ray spectroscopy analyser (EDX) for elemental analysis. Applications include positive/negative lithography, sample imaging and modification, electrical measurements and basic chemical and elemental analysis.

SPECIFICATIONS

SEM	
Product name	LYRA 3 XMH
Emitter	Schottky cathode
Acceleration voltage	200 V–30 kV/50 V–30kV in Beam Deceleration Mode (BDM)
Probe current	2 pA–200 nA
Detectors/ Resolution	SE/ 1.2 nm @ 30kV / 2.5 nm @ 3kV
	SE (BDM) / 1.5 nm @ 3kV
	In Beam BSE / 2.0 nm @ 15kV
	BSE / 2.0 nm @ 30kV
	TE
	EBIC
Chamber vacuum	< 9e-3 Pa (<5e-4 Pa reachable)
Specimen stage movements	Compucentric, fully motorized
	X: 130 mm (–65 mm to +65 mm)
	Y: 130 mm (–65 mm to +65 mm)
	Z: 100 mm
	Rotation: 360° continuous
	Tilt: –30° to +90° mm
Decontaminator/Plasma Cleaner	

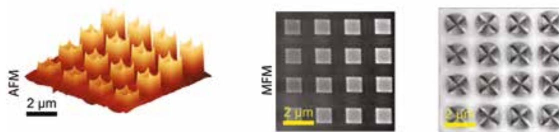
FIB	
Ion column	Canion
Emitter	Ga LMIS
Acceleration voltage	500 V–30 kV
Probe current	1 pA–40 nA
SEM - FIB coincidence at	WD 9 mm (SEM) WD 12 mm (FIB)
SEM - FIB angle at	55°
GIS	
Number of channels	5
Type of precursors	SiO_x, H_2O, F, PT, W, (Co)
Nanomanipulators	
Type	Smaract (closed loop)
Position	2 at chamber roof 2 optional at SEM stage
Analytical (EDX) detector	
Type	BRUKER XFlash 5010
Energy resolution	≤ 129 eV @ MnKα
	59 eV @ FKα
	52 eV @ CKα

RESULTS

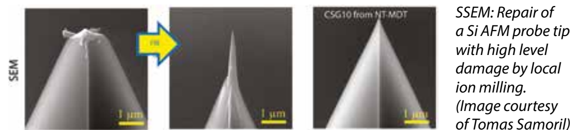


Resitivity 1.7×10^{-2} Ωm of the nanowire with the parameters: Length ~30 µm, Width 1150 nm, Thickness 55 nm (The image on the left)

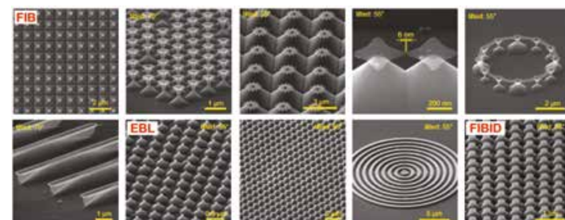
Manipulation with an amorphized silicon nanowire by a nanomanipulator tip and its resistance measurement by use of two nanomanipulators and gold contacts on Si/SiO_2 substrate. (Image courtesy of Tomas Samoril)



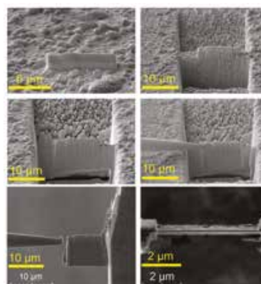
Focused Electron Beam Induced Deposition (FEBID) of Magnetic Nanostructures by using Dicobalt octacarbonyl $Co_2(CO)_8$ precursor characterized by AFM (Atomic Force Microscope) and MFM (Magnetic Force Microscope). (Image courtesy of Michal Urbanek)



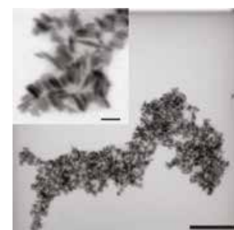
SSEM: Repair of a Si AFM probe tip with high level damage by local ion milling. (Image courtesy of Tomas Samoril)



Fabrication of micro- and nanostructures by selective wet etching (KOH) of Si (100) with FIB, EBL or FIBID mask. (Image courtesy of Tomas Samoril)



TEM lamella preparation. (Image courtesy of Eva Kolibalova)



Characterization of CuO/ZnO nanocomposite by transmitted electron (TE) detector in bright field. (Image courtesy of Jan Cechal)

MORE INFO

Guarantor: **Tomáš Šamořil** (tomas.samoril@ceitec.vutbr.cz)

Web: http://nano.ceitec.cz/focused-ion-beam-scanning-electron-microscope-tescan-lyra3/

Correlative Probe & Electron Microscopy

LiteScope™

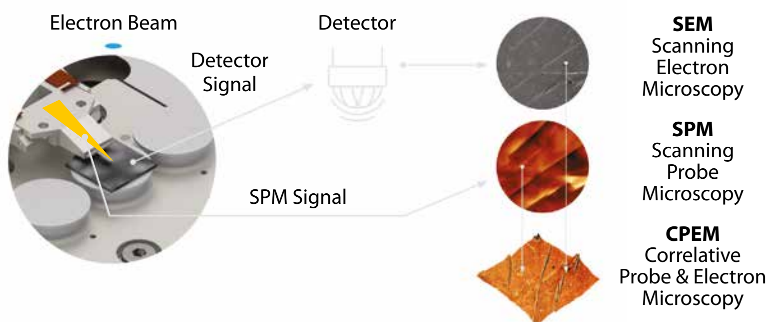
DESCRIPTION

The LiteScope™ is a unique Scanning Probe Microscope SPM integrated to TESCAN LYRA3 FIB-SEM. The combination of complementary SPM and SEM techniques enables use of the advantages of both commonly used microscopy techniques without breaking the vacuum. Complex sample analysis, including the characterization of surface topography, mechanical properties, electrical properties, chemical composition, magnetic properties and others, can be easily performed using LiteScope™ and its range of replaceable probes.
Furthermore, LiteScope™ opens up a completely new field of novel measurement techniques: so-called Correlative Probe and Electron Microscopy (CPEM). It enables both SPM and SEM measurements to be taken in the same place, at the same time, and using the same coordination system.
Moreover, in combination with Focused Ion Beam (FIB) or Gas Injection System (GIS) LiteScope™ offers quick and easy 3D inspection of the fabricated structures.

LiteScopeTM mounted on FIB-SEM stage

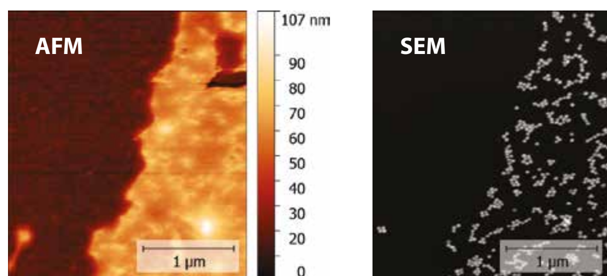
PRINCIPLE

A sample is attached to a piezoelectric scanner which provides the scanning motion under a stationary probe tip. The probe tip is guided to the area of measurement and the offset between probe tip and electron beam is set at a fixed value, approximately a few hundred nanometers. During the scanning procedure, the electron beam stays still.

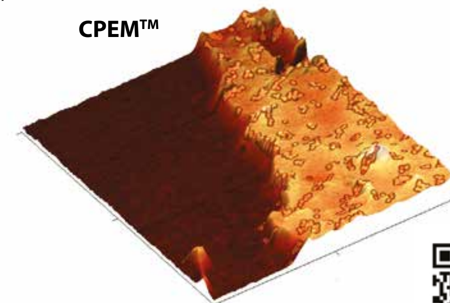


DIVERSE APPLICATIONS

Graphene-veiled gold nanoparticles hybrid structures



AFM topography demonstrates the structure of graphene, how it overflows the golden nanoparticles. However the SEM image does not provide any information about graphene, but shows the distribution of nanoparticles. CPEMTM combines the best of each measurement method and shows the topography in 3D with highlighted nanoparticles.



SPECIFICATIONS

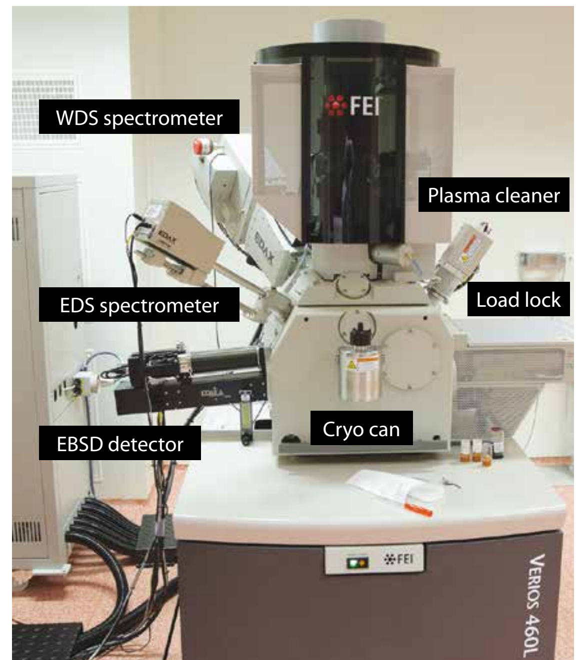
Vacuum working range	10^5 Pa to 10^{-5} Pa
Scanning range X, Y, Z	100 µm × 100 µm × 100 µm
Maximum sample size	10 mm × 10 mm
Maximum sample height	8 mm
Resolution	up to 0.4 nm

MORE INFO

Guarantor: **Tomáš Šamořil** (tomas.samoril@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/focused-ion-beam-scanning-electron-microscope-tescan-lyra3/

Scanning Electron Microscopy

VERIOS 460L



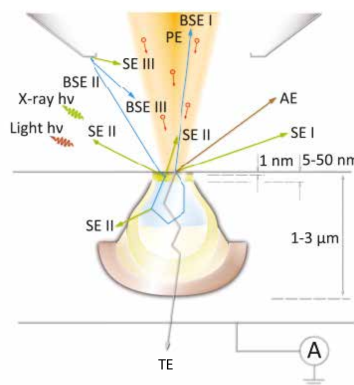
DESCRIPTION

The Thermo Fisher Verios 460L field-emission scanning electron microscope (FESEM) offers sub-nanometer resolution over a wide energy range (0.7 nm @ 1 keV, 0.6 nm @ 2-30 keV) with excellent materials contrast. Its extraordinary low-voltage performance provides extremely precise, surface-specific information even on insulating samples with no conductive coating. The microscope is equipped with a wide array of imaging and analytical detectors for structural and compositional analysis.

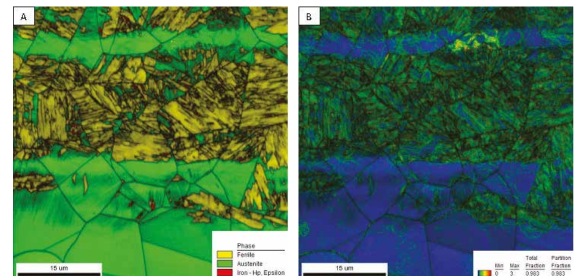
PRINCIPLE

In the SEM a finely focused electron beam is produced and scanned over the sample under vacuum to obtain the image. The incident electron beam interacts with the sample and generates a number of signals each carrying a specific type of information. The intensities of these signals can be measured by a variety of detectors. The most commonly imaged signals are secondary electrons (SEs) and backscattered electrons (BSEs).

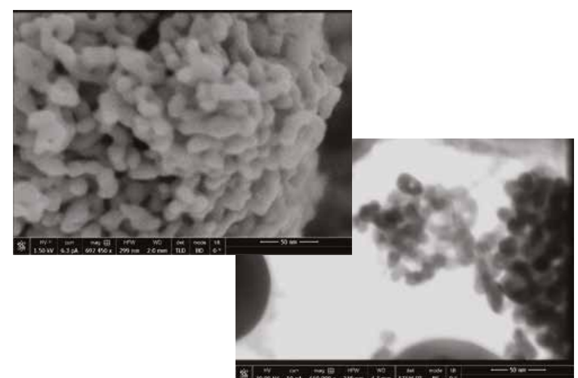
PE	primary electrons
AE	auger electrons
SE	secondary electrons
BSE	backscattered electrons
SE I	generated by PE in sample
SE II	generated by BSE in sample
SE III	generated by BSE on column/chamber
SE IV	generated by PE in column
BSE I	PE scattered in sample (elastically)
BSE II	PE scattered in sample (inelastically)
BSE III	BSE I scattered on column/chamber
TE	transmitted electrons



APPLICATION EXAMPLES



EBSD analysis of a 301LN steel sample: A) Phase map indicating regions of Austenite, Ferrite and ε-martensite; B) Map of Kernel Average Misorientation parameter, indicating local lattice distortion as a measure of strain.



Titanium dioxide particles (uncoated) observed in secondary electron signal in beam deceleration mode (left) and in STEM mode (right).

SPECIFICATIONS

Electron optics		Imaging detectors (detected signal)	Analytical detectors (detected signal)
Column type	Elstar UC	TLD-SE (SE I, SE II, SE IV)	EDS EDAX SDD Octane Super (X ray)
Source type	Schottky FEG with monochromator	TLD-BSE (BSE I, BSE II)	WDS EDAX TEXS HP XM 4 (X ray)
Imaging modes	field free	ICD (BSE I)	EBSD EDAX DigiView IV (BSE, TE)
	XHR immersion	MD (BSE I, BSE II)	**Accessories**
	EDS optimized	ETD (SE I,SEII, SE III, BSE II, BSE III)	Beam current measurement (integrated + external)
	Beam deceleration	FSD (BSE)	IR camera for viewing sample/column
Probe current	0.8 pA to 100 nA	CBS (BSE II, BSE III)	Chamber mounted Nav-Cam+
Landing Energy	20 eV to 30 keV	STEM (TE)	Plasma cleaner
			Cryo cleaner

Sample stage movements		Maximum sample sizes	
X, Y	100 mm	**Maximum size**	100 mm diameter
Z	20 mm	**Maximum sample thickness (via loadlock)**	15 mm including sample holder
Tilt	– 10 ° to + 60 ° eucentric	**Maximum sample thickness (via chamber door)**	27.8 mm including sample holder
Rotation	720 ° stroke	**Fully motorized loadlock**	

MORE INFO

Guarantor: **Ondřej Man** (ondrej.man@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/high-resolution-scanning-electron-microscope-fei-verios-460l/

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Scanning Near-Field Optical Microscope

Nanonics MultiView 4000

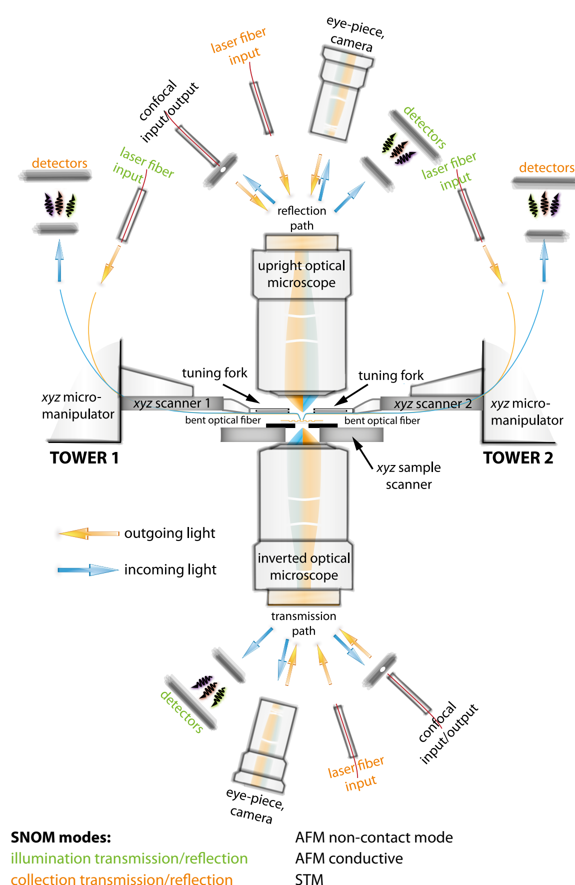
DESCRIPTION

A SNOM is a microscopic tool, which breaks the far-field light resolution limit by mapping the near-field light (evanescent waves) distribution of nanostructures. In order to achieve this, there is a very sharp optical probe (detectors/illuminators), which has an aperture of tens of nanometers. This scans the sample surface at a distance much smaller than the wavelengths of the light interacting with it. The optical resolution and structure size resolution is limited by the probe size, not by the wavelength of the incidental light (resolution ~100 nm). This technique provides for the capabilities of the basic experimental setup – illumination by a SNOM probe set to collect interacting light in reflection or transmission; to illuminate a sample in transmission or reflection and collection the interacting light via a SNOM probe.

FEATURES

- SPM techniques – SNOM, AFM
- two independent probe scanning systems, sample scanning
- combined SNOM collection, illumination with reflection and transmission modes
- probes – bent optical fibres on tuning fork according to applications
- optical and acoustic hoods
- confocal spectroscopy and photoluminescence (2µm spot, 400–900 nm)

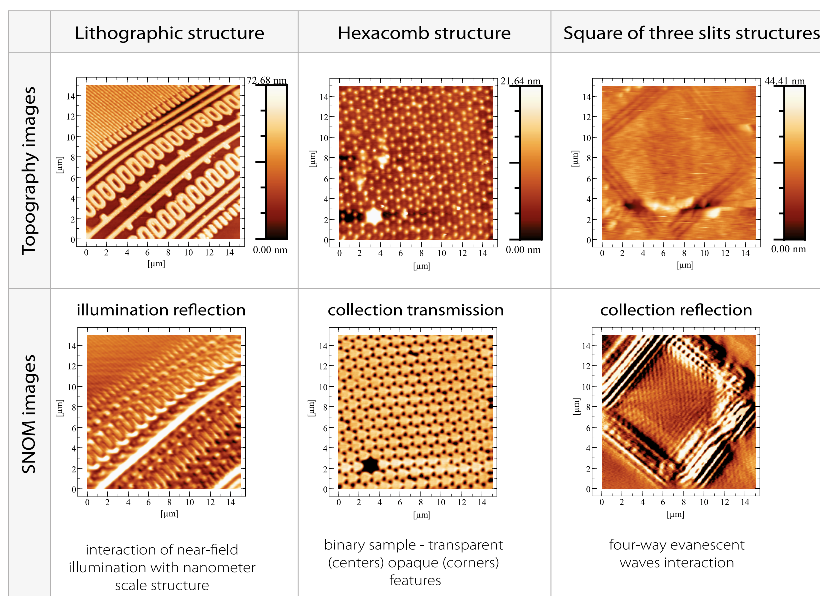
TECHNICAL SPECIFICATION



SNOM modes:
illumination transmission/reflection
collection transmission/reflection

AFM non-contact mode
AFM conductive
STM

APPLICATIONS



Lasers:
Nd:YAG λ = 532 nm
power 20 mW
HeNe λ = 632.8 nm
power 10 mW

Detectors:
Avalanche PhotoDiode (APD)
- λϵ<400, 1100> nm
- photoncounting mode
PhotoMultiplier Tube (PMT)
- λϵ<165, 650> nm

Optical microscopes:
upright – Olympus BXFM
inverted – Olympus BXFM
objectives – 50x NA, 10×

Manipulators:
xyz scanner 1: 40 µm × 40 µm × 30 µm
xyz scanner 2: 40 µm × 40 µm × 30 µm
xyz sample scanner: 80 µm × 80 µm × 30 µm
xyz micromanipulators: 5 mm × 5 mm × 10 mm

fiber probes optimized for selected measurement

MORE INFO

Guarantor: **Petr Dvořák** (petr.dvorak@ceitec.vutbr.cz), **Filip Ligmajer** (filip.ligmajer@ceitec.vutbr.cz)

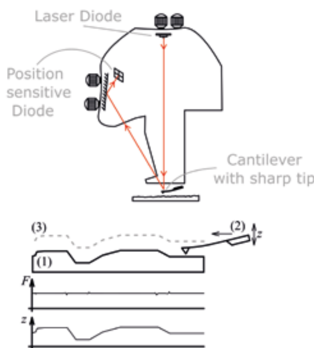
Web: http://nano.ceitec.cz/scanning-near-field-optical-microscopy-nanonics-imaging-mv-4000/

Scanning Probe Microscope

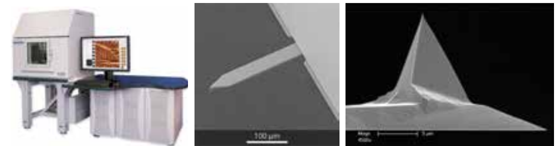
Bruker Dimension Icon

PRINCIPLE

The first scanning probe microscope was invented in 1981. Its inventors (G. Binnig and H. Röhrer) were awarded the Nobel price in 1986. This microscope uses interaction between the sharp tip and sample surface to measure topography. If the tip is sharp enough – ideally one atom at the very end – it is able to distinguish each atom on a measured surface.



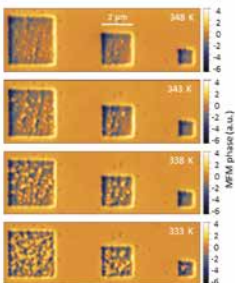
- similar principle to a turntable
- able to reach atomic resolution
- tip measures at all desired points separetly – scanning
- feedback keeps cantilever deflection (force) constant
- measured force combines attractive van der Waals forces and repulsive quantum-mechanical interactions



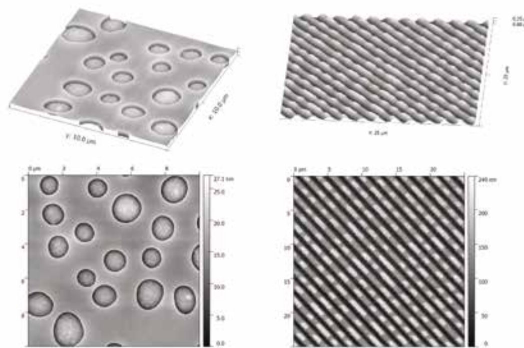
SPECIFICATIONS

Scanner range	90 µm (lat.) x 10 µm (vert.)
Scanner noise	< 0.15 µm (lateral) < 35 pm (vertical)
Sample size	Ø 210 mm × 15 mm (vert.)
Techniques	ScanAsyst Mode
	Non-contact Mode
	Contact Mode
	Tapping Mode
	Phase Imaging
	Force Spectroscopy
	Force Modulation
	PeakForce TUNA, QNM
	Lateral Force Microscopy
	Electric Field Microscopy
	Scanning Tunneling Microscopy
	Kelvin Probe Force Microscopy
	Magnetic Force Microscopy
	...

RESULTS



MFM of FeRh squares [1]



PS Balls on LDPE

DVD record

PUBLICATIONS

[1] Schánilec, V.; Horký, M. and col. Magnetic phase transition asymetry dependent on the spatial confinement of FeRh patterns

MORE INFO

Guarantor: **Dalibor Šulc** (dalibor.sulc@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/scanning-probe-microscope-bruker-dimension-icon/

Nanoindentor

Hysitron TI 950

DESCRIPTION

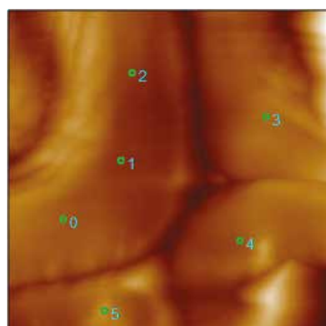
To measure nanomechanical properties of surface layers of bulk materials and thin films, nanoindentation measurement techniques are commonly used. An indenter is pushed into the sample until bulk plastic deformation occurs, and then unloaded. During the indentation process, the device continuously monitors the load and the position of the indenter relative to the surface of the specimen. The area of the indent is then calculated from knowledge of the area function of the tip of the diamond indenter.

FEATURES

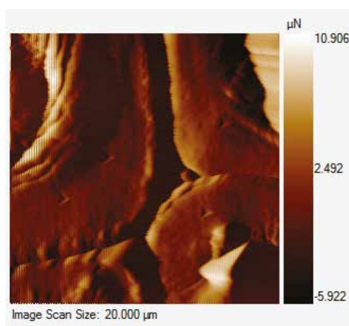
- **Quasistatic nanoindentation** – measurements of material mechanical properties such as hardness and elastic modulus
- **SPM imaging** – in-situ contact imaging of 3D profile of tested surface
- **Modulus Mapping** – large area mapping to provide quantitative information of material surface nanomechanical properties
- **Scratch testing** – evaluation of friction, wear resistance and coating adhesive strength
- **nanoDMA** – dedicated testing mode for dynamic mechanical behavior investigation of polymers and biomaterials
- **nanoECR** – in-situ electrical and mechanical measurements for material deformation and stress induced transformation behavior analyses
- **xSOL** – high temperature stage, can be added to provide feedback-controlled temperature accuracy during high temperature testing up to 800 °C
- **3D Omniprobe**

RESULTS

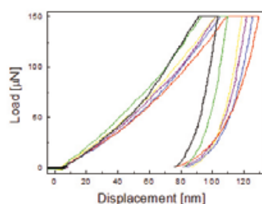
Nanoindentation tests of wood cell walls



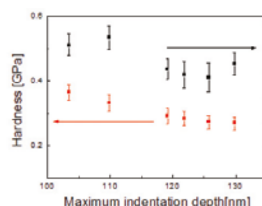
Automated positioning of indentation prints using SPM piezoscanner



Indentation prints in wood cell walls (spruce) imaged using the SPM piezoscanner



Load-displacement curves



Hardness and effective modulus results for different maximum indentation depths

SPECIFICATIONS

Standard transducer specifications
Normal load
Resolution: <1 nN
Noise floor: <30 nN
Imaging contact force: ≤70 nN
Normal Displacement
Resolution: <0.02 nm
Noise floor: <0.2 nm
Drift: <0.05 nm/sec
Maximum indentation depth: 5μm
High load transducer specifications
Maximum normal force: 2 N
Normal force noise floor: <0.02mN
Load resolution: <0.015mN
Displacement noise floor: <0.6nm
Displacement resolution: <0.03nm
Maximum normal displacement: 80 μm
Maximum lateral force: 5 N
Maximum scratch length: 150 mm
Stage specifications
X and Y stages
Travel: 250 mm × 150 mm
Encoder resolution: 500 nm
Z stage
Travel: 50 mm
Resolution: 3 nm

MORE INFO

Guarantor: Veronika Králová (veronika.kralova@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/hysitron-ti-950/

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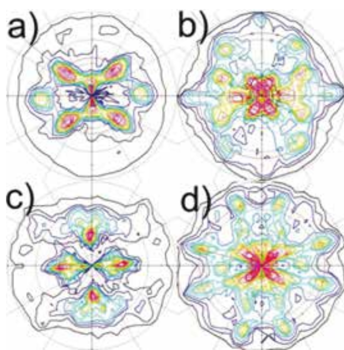
X-ray Powder Diffractometer

Rigaku Smartlab 3kW

DESCRIPTION

The Rigaku SmartLab 3 kW is an automatic X-Ray powder diffractometer with θ/θ goniometer. The Bragg-Brentano and Parallel Beam modes are complemented by use of additional accesories (Eulerian craddle, sample holders, etc.) to extend the range of measurement techniques.

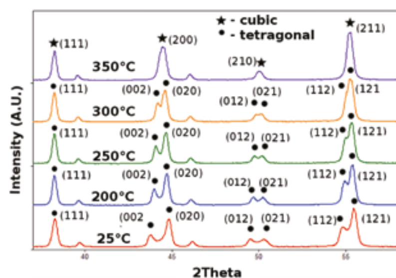
APPLICATION EXAMPLES



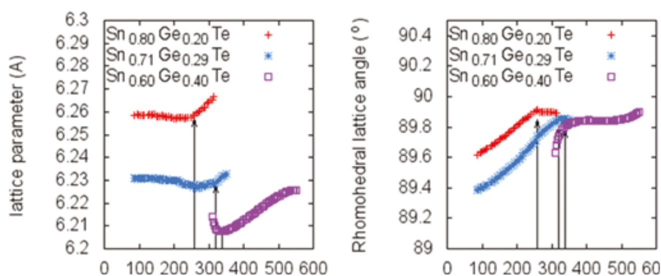
The pole figures represented texture/preferred orientation in the sample.

The Al rolled sheet is highly oriented along the planes a) (1 1 1) and c) (0 2 2).

The figures b) and d) belongs to (0 0 2) and (1 1 3) planes and shows the existence of big oriented crystallites.



Phase transformation of Perovskite powder during heating up to 350 °C in air, use of High Temperature Chamber HTK1600



Lattice parameter temperature dependence of the SnGeTe ternary alloy showing ferroelectric phase transition (denoted by black arrows). The experiment was performed using TTK450 chamber cooled with liquid nitrogen.

SPECIFICATIONS

Phase analysis	Determination of presence and amount of current phases in material
Texture	Distribution of preferential crystallographic orientation (texture)
Residual stress	Non-destructive measurement of residual stress
Reflectivity	Thickness of thin layers, its density, roughness of surface and interfaces
In-situ High temperature	In situ observation of material up to 1600 °C in vacuum and air
In-situ Low temperature	In situ observation of material at temperatures from -193 °C to +450 °C
In-situ Environmental	In situ observation of material up to 900 °C in reactive gasses or low vacuum

PUBLICATIONS

(1) Železný, V. et al. Temperature-dependent far-infrared reflectance of an epitaxial (BaTiO3)8/(SrTiO3)4 superlattice. Phys. Rev. B 95, 214110 (2017)
(2) Castkova, K. et al. Electrospinning and thermal treatment of yttria doped zirconia fibres. Ceram. Int. 43, 7581-7587 (2017)
(3) Tkachenko, S. et al. Isothermal oxidation behavior of experimental Ti–Al–Si alloys at 700 °C in air. J. Alloy. Comp., 694, 1098-1108 (2017)
(4) Abdel-Mohsen, A. M. et al. Novel chitin/chitosan-glucan wound dressing: Isolation, characterization, antibacterial activity and wound healing properties. Int. J. Pharm. 510, 86-99 (2016)
(5) Novak, M. et al. Two paramagnetic types of cookeite from the Dolni Bory-Hate pegmatites, Moldanubian zone, Czech Republic: Proximal and distal alteration producsts of Li-bearing sekaninaite. Can. Mineral. 53, 1035-1048 (2015)
(6) Castkova, K. et al. Chemical Synthesis, Sintering and Piezoelectric Properties of Ba0.85Ca0.15Zr0.1Ti0.9O3 Lead-Free Ceramics. J. Am. Ceram. Soc. 98, 2373-2380 (2015)
(7) Trunec, M et al. Effect of Phase Structure on Sintering Behavior of Zirconia Nanopowders. J. Am. Ceram. Soc. 96, 3720-3727 (2013)

MORE INFO

Guarantor: Pavla Roupcová (pavla.roupcova@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/x-ray-powder-diffractometer-rigaku-smartlab-3kw/

Rotating Anode X-ray Diffractometer for Thin Films

Rigaku Smartlab 9kW

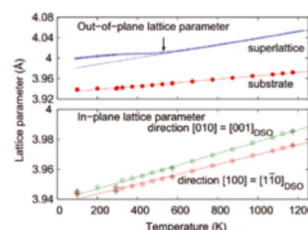
DESCRIPTION

The Rigaku SmartLab 9 kW is an automatic X-ray diffractometer dedicated the to characterization of thin films and nano-structures. Available non-destructive analytical techniques reveal information on a crystal structure, chemical composition, and physical properties of various materials such as semiconductors, metallic layers, and insulators. θ/θ goniometer with an in-plane arm allows a large variety of X-ray diffraction and scattering techniques. A series of optical elements can be used to adapt experimental conditions for particular experiment.

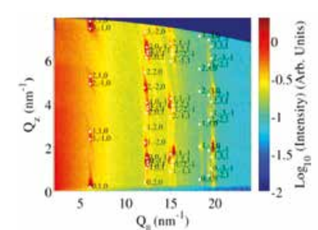
SPECIFICATIONS

Technique	Description
Coplanar X-ray diffraction (XRD)	Lattice parameters, lattice strain, chemical composition, inter-layer diffusion in thin films and multi-layers
Reflectivity and diffuse scattering (XRR)	Thickness of layers (~0.5–500 nm), surface and interface roughness, and roughness lateral correlations
Small angle X-ray scattering in 1D and 2D mode (SAXS)	Size distribution of particles and pores in solutions and light solid matrices, their mutual distance; size up to >≈ 100 nm
Grazing incidence small angle scattering (GISAXS)	Particles and pores size and their size distribution and mutual distance in thin films and nano-porous thin films
Grazing incidence X-ray diffraction (GIXRD)	In-plane lattice parameters and preferential crystallites orientation in thin films; depth resolved measurements
Texture (Pole figures) measurements	Distribution of preferential crystallographic orientation in thin films
Scanning micro-diffraction	Information as for techniques above with lateral resolution down to 0.2 mm on laterally inhomogeneous samples
Phase analysis and material characterisation	Crystal phases of materials, calculation of amount of present phases, crystallite size and crystallinity in thin films
In situ measurements at high temperatures	Annealing chamber DHS1100 (9kW) allows for in situ measurements at sample temperatures up to 1100 °C, in vacuum or in inert gas; hemispherical chamber dome allows to apply scattering techniques mentioned above
High speed 2D reciprocal space mapping	The pixel array detector HyPix-3000 with ultra high dynamic range and high sensitivity allows for fast data acquisition of 2D reciprocal space maps; higher time resolution is achieved in real-time in-situ experiments

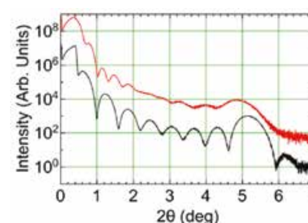
RESULTS



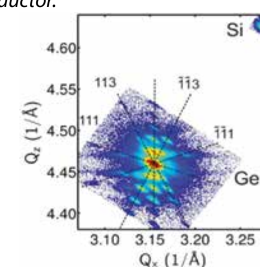
Temperature dependence of the lattice parameters in epitaxial $(BaTiO_3)_8/(SrTiO_3)_4$ Superlattice.



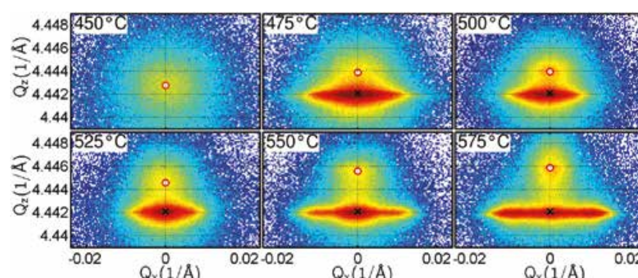
GIXRD reciprocal space map on a thin film of an organic semiconductor.



X-ray specular reflectivity on a thin film of an organic semiconductor and a similar layer with a metal electrode above.



High resolution (HR) XRD Reciprocal space map (RSM) on facetted Si/SiGe heterostructures.



HRXRD RSMs of epitaxial Ge microcrystals showing evolution of Ge peaks due to strain varying at different growth temperatures.

PUBLICATIONS

1) Železny, V. et al. Temperature-dependent far-infrared reflectance of an epitaxial $(BaTiO_3)_8/(SrTiO_3)_4$ superlattice. Phys. Rev. B **95**, 214110 (2017)
(2) Gablech. I. et al. Stress-free deposition of [001] preferentially oriented titanium thin film by Kaufman ion-beam source. Thin Solid Films **638**, 57 (2017)
(3) Wang Ch. et al. Mid-infrared ellipsometry, Raman and X-ray diffraction studies of $Al_xGa_{1-x}N$/AlN/Si structures. Appl. Surf. Sci. **421**, 859–865 (2017)
(4) T. Storzer, et al. Growth, Structure, and Anisotropic Optical Properties of Difluoro-anthradithiophene Thin Films. J. Phys. Chem. C **121**, 21011 (2017)
(5) Růžička J. et al. Structural and electronic properties of manganese-doped Bi_2Te_3 epitaxial layers. New J. Phys. **17**, 013028 (2015)
(6) Isa F. et al. Three-dimensional Ge/SiGe multiple quantum wells deposited on Si(001) and Si(111) patterned substrates. Semicond. Sci. Technol. **30**, 105001, (2015)
(7) Meduňa M. et al. Reconstruction of crystal shapes by X-ray nanodiffraction from three-dimensional superlattices. J. Appl. Cryst. **47**, 2030 (2014)

MORE INFO

Guarantor: **Ondřej Caha** (ondrej.caha@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/x-ray-powder-diffractometer-rigaku-smartlab-9kw/

X-ray Photoelectron Spectroscopy

KRATOS Axis Supra

Load lock | Main chamber | Surface science station

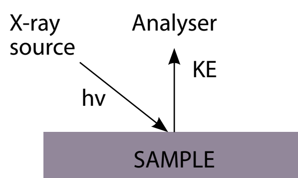
DESCRIPTION

XPS (X-ray Photoelectron Spectroscopy) is a method for characterisation of surfaces and ultra thin films. An X-ray beam irradiates the location on a sample from which electrons are emitted and some of them are collected in an analyser. In spectroscopy mode, the analyser continuously changes the energy of electrons which are counted by a channel plate detector. From the obtained XPS spectrum (number of counted electrons vs. binding energy), the elemental and also chemical composition of the sample surface can be determined. The parallel imaging mode is based on the parallel collection of electrons on one specified energy level and their 2D projection on the channel plate detector. The obtained image containes chemical information and could be used to set the precise location for the spectroscopy of a small area. The whole system has to be operated under Ultra High Vacuum conditions (pressure ~10^{-9} mbar or less) to prevent collisions of electrons with other particles before they reach the detector.

PRINCIPLE

hv ... energy of incident X-ray beam
KE ... kinetic energy of emited electron
BE ... binding energy which is necessary to emit electron from atom shell

BE=hv-KE



SPECIFICATIONS

XPS

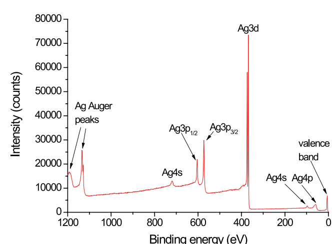
Spectroscopy	large area analysis 300×700 µm small area analysis 15 µm
Parallel imaging	lateral resolution 1 µm
Snapshot mode	quick spectrum measurement
Angle-resolved XPS	obtaines spectra for different emission angles, which changes information depth
Line and map scan	this modes use deflection electrodes to scan over area of interest
Detection limit	0.1 to 1 atomic %
Depth resolution	up to 8 nm

Other techniques

Ion beam etching	instrument is equipped with Ar cluster ion source which allows cleaning sample surface or depth profiling
UPS	Ultraviolet Photoemission Spectroscopy - UV lamp is used instead of X-ray source. This technique provides information on valence levels and work function of materials
Surface Science Station	extra chamber which allows preparation or modification of samples and consequently move them into the main chamber for analysis without exposure of samples to atmosphere

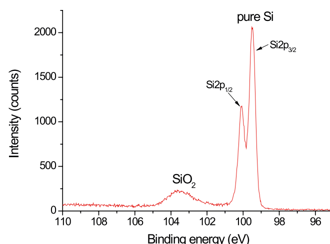
RESULTS

Survey spectrum of Ag clean sample



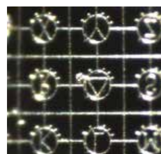
Sample surface was cleaned by ion beam etching Ar+ 5keV. Spectrum containes many Ag peaks but neither carbon nor oxygen peaks located at 285 eV, 530 eV, respectively.

Spectrum of Silicon substrate covered by native SiO_2

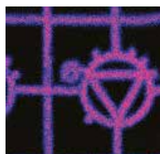


Spectrum shows detailed Si2p peak, which consists of two components: pure Si from substrate and SiO_2 from thin native oxide.

XPS parallel imaging of Au special grid



Optical microscope image of analysed location 800×800 µm



Parallel image obtained on Au4f peak using mode 400×400 µm

PUBLICATIONS

Šik, O. et al. Investigation of the effect of argon ion beam on CdZnTe single crystals surface structural properties. *Surf. Coatings Technol.* **306**, 75–81 (2016).

Vallejos, S. et al. ZnO Rods with Exposed {100} Facets Grown via a Self-Catalyzed Vapor-Solid Mechanism and Their Photocatalytic and Gas Sensing Properties. *ACS Appl. Mater. Interfaces* **8**, 33335-33342 (2016).

Manakhov, A., Čechal, J., Michlíček, M. and Shtansky, D. Determination of NH2 concentration on 3-aminopropyl tri-ethoxy silane layers and cyclopropylamine plasma polymers by liquid-phase derivatization with 5-iodo 2-furaldehyde. *Appl. Surf. Science* **414**, 390-397 (2017).

MORE INFO

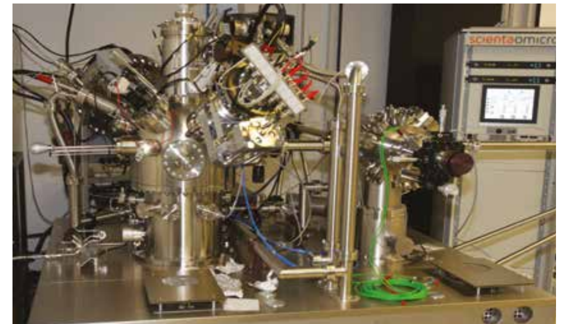
Guarantor: Josef Polčák (josef.polcak@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/x-ray-photoelectron-spectroscopy-kratos-analytical-axis-supra/

Surface analysis UHV system

NanoSAM Lab

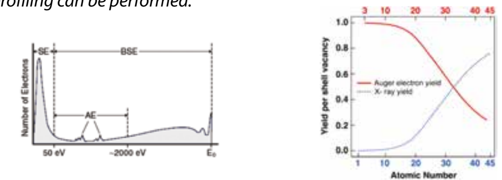
DESCRIPTION

The Nano SAM Lab is a dedicated surface analysis UHV system for high resolution structural and chemical analysis by Scanning Auger Microscopy (SAM), Scanning Electron Microscopy (SEM) and Secondary Electron Microscopy with Polarization Analysis (SEMPA) for the characterization of the magnetic domain structure. The instrument is designed for use together with the UHV Gemini high resolution electron column. It includes Matrix software and electronics for static Auger spectroscopy (AES) and SAM. In combination with UHV Gemini, Matrix provides an unsurpassed drift correction technology based on autocorrelation of subsequent SEM images. This opens up the possibility to perform long term AES measurements on very small features with low intensity, or elemental resolved SAM maps of nanostructures with a low concentration of elements of interest and/or low sensitivity factors. The NanoSAM Lab is equipped with high precision goniometer – mounted four axis UHV stage for the combination of high resolution SEM, SAM and SEMPA, which allows heating up to 750 K. Moreover, the NanoSAM Lab includes a preparation chamber which comprises a manipulator with the possibility of heating the sample to 1500 °C by resistive heating and 900 °C by radiative heating. The preparation chamber contains 8 flanges for user extensions.



AES

AES provides quantitative elemental and chemical state information from surfaces of solid materials usually in the area of material science. A focused electron beam scans across the sample surface which leads to the production of various signals including the emission of 'Auger' electrons. An electron energy analyzer measures the kinetic energies of the emitted Auger electrons, which are characteristic for elements present within the top 1-5 nm of the sample and intensity of an Auger peak. Hence, the identity and quality of a detected element can be determined. When used in combination with a sputtering ion source, compositional depth profiling can be performed.



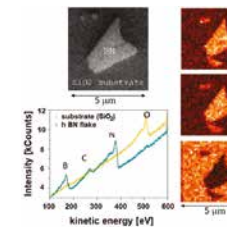
SPECIFICATIONS

SEM	
Electron column	UHV Gemini (FEG)
Emitter	Schottky cathode
Acceleration voltage	100 V – 20 kV
Probe current	min. 50 nA @ 15 keV
	min. 28 nA @ 3 keV
Detectors	Inlens SE detector
Resolution	< 3 nm @ 15 keV
	< 13 nm @ 1 keV
Specimen stage movements	XYZ: 10×10×10 mm
	Tilt: -60° to +60°
Stage heating	Up to 750 K
Maximum sample size	12×15×4 mm^3
Basic pressure	< 3 e-10 mbar
Image drift compensation	
4 electrical contacts up to 6 Ghz	
Preparation chamber	

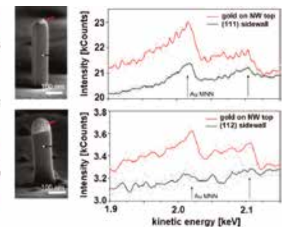
SAM	
SEM–SAM coincidence at	~ 22 mm
SEM–SAM angle at	+30°
Lateral resolution (@ 1nA)	< 6 nm @ 10 keV
	< 10 nm @ 5 keV
Analyzer resolution	> 420 KCPS no background
Image drift	< 10 nm/10 hrs
SEMPA	
SEM–SEMPA angle at	-60°
Resolution	< 50 nm
Image drift	< 10 nm/10 hrs
Detectors	Rotation Detector
Ion Source/ Charge Neutralization	
Neutralization	10 eV - 5 keV ions
Ion sputtering	@1 - 5 keV > 2 mA/cm^2
	min. 100 nA @ 15 eV

Chemical Composition Determination

b) AES of hexagonal Boron Nitride

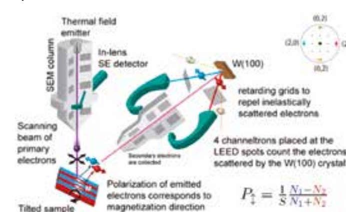


a) Au detection on germanium nanowires



SEMPA

SEMPA is a technique for direct characterization of the magnetic domain structure of a sample. Spin polarization of the secondary electrons (emitted by the primary beam) corresponds to the magentization direction in the material and can be measured in order to create a magnetization map of the sample. Spin detection is based on the SPLEED (Spin Polarized Low Energy Electron Diffraction) principle, i.e. diffraction from a single crystal surface of W(100) forming a few well defined diffraction spots - (LEED) spots.



The LEED spots exhibit intensity variations and asymmetries that depend on the energy and degree of spin polarization of the scattered electrons.

Magnetic domains of Iron Whisker

(a) The domain images show the magnetization distribution obtained in the two perpendicular polarization components. (b) Topography image obtained simultaneously by counting the electrons in all 4 main LEED spots. (With permission of Lukas Flajsman)

MORE INFO

Guarantor: Marek Otevřel (marek.otevrel@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/scanning-electron-microscopy-with-polarization-analysis-scienta-omicron-nanosam-lab/

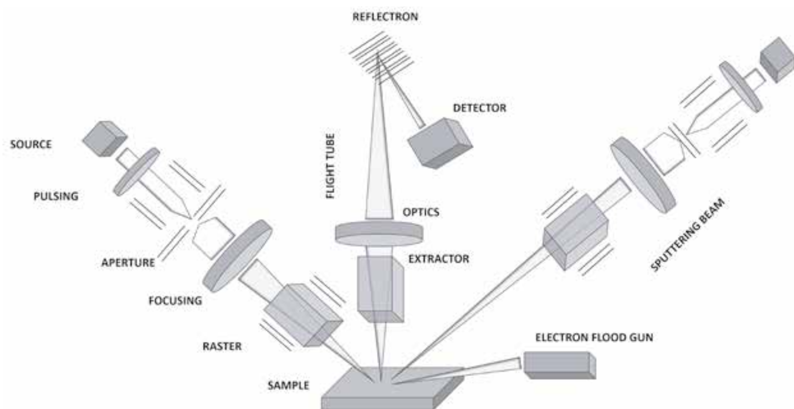
Secondary Ion Mass Spectrometry

Ion TOF.SIMS[5]

PRINCIPLE

The Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is a very sensitive surface analytical technique which provides detailed elemental information about the surface, thin layers, interfaces of the sample, and gives a full three-dimensional analysis. A finely focused ion beam sputters the sample surface and the exact mass of emitted ions and ion clusters is measured by the time-of-flight analyzer. From the exact mass and intensity of the SIMS peak, the identity of an element or molecular fragments can be determined.

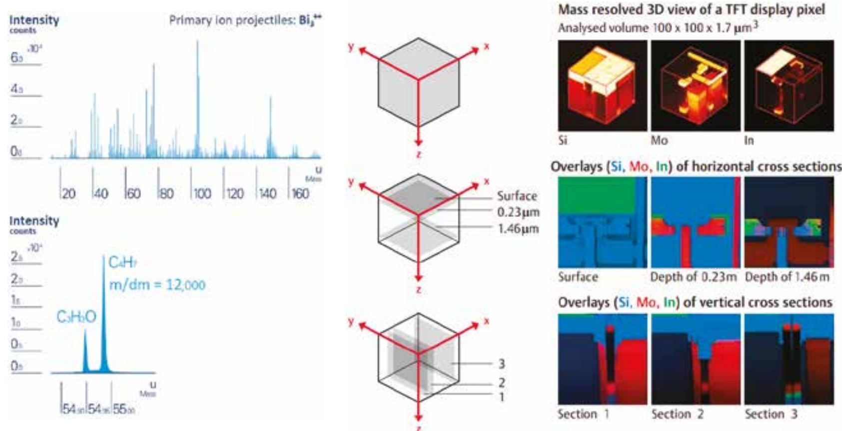
SCHEMA



SPECIFICATIONS

Primary Ions	$Bi1^+$, $Bi1^{++}$, $Bi2^+$ .. Bi_7^+, Mn^+ Energy: 30 keV
Sputtering Ions	Cs^+,O_2^+ Energy: 0.5 – 2.0 keV
Electron flood gun	Energy: <20 eV
Sample holders	Back side mounting stage: Sample size: 15 mm × 10 mm Top side mounting stage: Sample size up to 100 mm × 50 mm Heating/Cooling stage: Sample size: 10 mm × 10 mm Temp. range -130°C – 600°C Rotating stage: Sample size: ø10 mm
Surface spectrometer	High sensitivity (1–2 ML)
Surface imaging	High lateral resolution (<60 nm)
Depth profiling	Depth resolution better than 1 nm
	High mass resolution > 11 000 @ 29 u
	Sputter speed of up to 10 µm/h
3D Analysis	Parallel mass detection

RESULTS



left – High resolution mass spectrum of a PET sample.
right – Mass resolved 3D analysis of TFT display pixel.

MORE INFO

Guarantor: **Marek Otevřel** (marek.otevrel@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/secondary-ion-mass-spectroscopy-ion-tof-tof-sims5/

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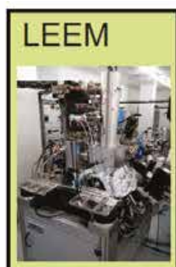
UHV Preparation and Analytical system

DESCRIPTION

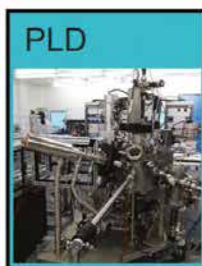
The complex UHV (Ultra-High Vacuum) system combines in-situ preparation and analytical methods for the study of surfaces and thin layers, both inorganic and organic. The configuration of the system allows the study of structures of sample surface layers, their chemical compositions and electronic structures in real time, on both a micrometer and nanometer scale. In order to maintain high bulk and surface purity during the preparation of the samples and their subsequent analysis the whole system is kept under ultra-high vacuum conditions with a pressure level in the order of 10^{-8} Pa. The complex UHV system is divided into eight major independent units, which are interconnected via an UHV linear transfer system. The individual units are designed to allow the users to work simultaneously. The said transfer system guarantees a seamless transfer of samples between the units under UHV conditions and it is able to store up to 15 samples.

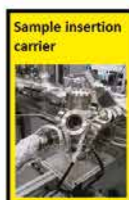
SPECIFICATIONS

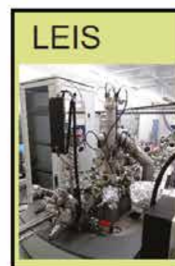
The pulsed laser deposition preparation chamber (PLD)	Preparation of heterostructural layers on an oxide basis.
The low-energy electron microscopy system (LEEM)	Photoelectron/Low Energy Electron Microscopy with a resolution of at least 10 and 5 nanometers, respectively.
The chamber for photo-emission analytical methods (PES)	X-ray Photoelectron Spectroscopy (XPS), angle resolved XPS, Ultraviolet Photoelectron Spectroscopy (UPS), angle dependent UPS. Analysis of the chemical composition (macroscopic level) including bonding of the tracked elements.
The multi-purpose chamber for the preparation of surfaces and thin layers (CD - custom deposition)	Preparation and deposition of metals, semiconductors based on nitrides and oxides equipped with effusion cells, a plasma source for atomic nitrogen and oxygen. Ability to build-in custom components – effusion cells, steaming tools etc.
The chamber for sample heating and sputtering (Prep.)	Cleaning and basic preparation of the substrates surfaces, deposition of organic materials, and other "unclean processes".
The chamber for STM and AFM scanning probe techniques (SPM)	Advanced Scanning Tunneling Microscopy (STM) and Atomic Force Microscopy (AFM) techniques achieving an atomic resolution on metal, semiconductor and oxide surfaces.
The molecular beam epitaxy preparation chamber (MBE)	Preparation of III-V semiconductors (Ga, In, Al, As, Sb, C, Si), heterostructures InAs, GaAs etc.
The low-energy ion scattering system (LEIS)	Analysis of back-scattered noble gas ions energies. Highly sensitive to the chemical composition of the surface of structures studied.



UHV linear transfer system







MORE INFO

Guarantor: **Marek Otevřel** (marek.otevrel@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/ultra-high-vacuum-preparation-and-analytical-system-uhv-cluster/

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Pulsed laser deposition

PRINCIPLE

Pulsed laser deposition (PLD) is a vacuum deposition using ablation of a target material with a pulsed ultraviolet laser. This technique is most often used for deposition of oxides, for example transition metal oxides; however metals can be deposited as well. The highlight of this technique is the in-situ monitoring of sample growth with reflection high energy electron diffraction (RHEED) that in principle enables the monitoring and control of the grown on monolayer scale. The PLD chamber is attached to the UHV cluster and thus the PLD grown samples can be analyzed within the cluster or additional materials can be deposited on the same substrate.

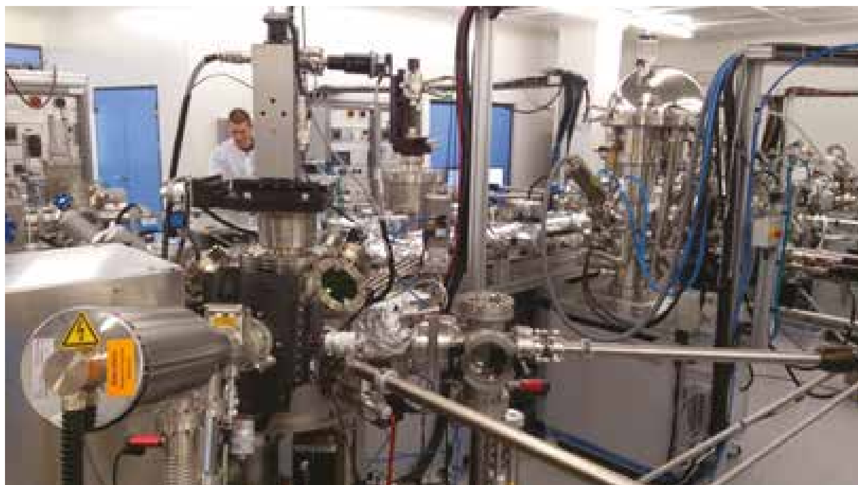
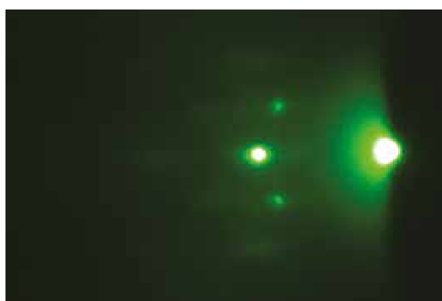
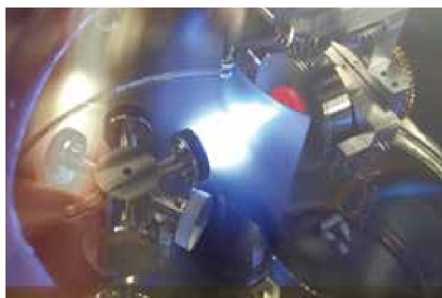


Photo of the PLD chamber with loadlock and RHEED gun (front) with laser focusing optics (left) attached to the UHV cluster (back and right)



RHEED pattern on LSAT substrate



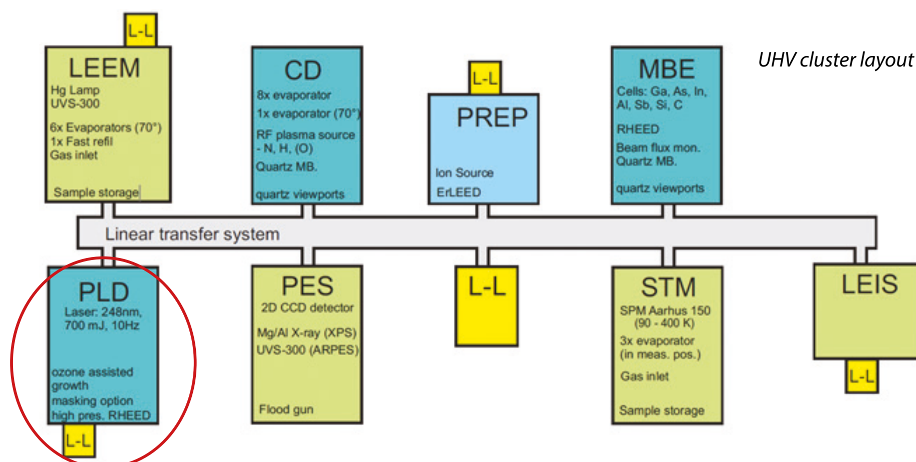
View into the PLD chamber during the preablation – cleaning of the target. The shining area is the plume due to the ablated target material. The sample is on the heater (the red spot) heated to 800 °C half hidden behind the shutter.

FEATURES

- In situ investigation with RHEED
- growth in oxygen, argon or in ozone atmosphere
- resistive heating up to 800 °C
- laser heating up to 1200 °C
- masking possibilities
- scanning with target or laser beam beam for improved homogeneity of the thin film
- up to 5 different materials can be combined during one growth
- sample size up to 10×10 mm

SPECIFICATIONS

UV laser enegy	700 mJ per pulse
UV laser wavelength	248 nm
base pressure	1×10^{-9} mbar



UHV cluster layout

MORE INFO

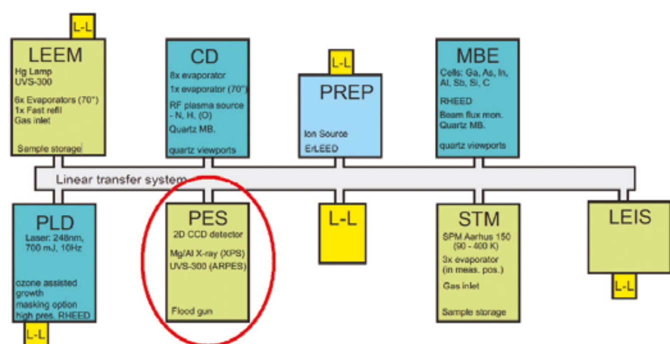
Guarantor: **Adam Dubroka** (dubroka@physics.muni.cz)
Web: http://nano.ceitec.cz/ultra-high-vacuum-preparation-and-analytical-system-uhv-cluster/

Photoelectron Spectroscopy Instrument

Complex UHV - PES

SPECIFICATIONS

The Photoelectron Spectroscopy instrument (PES) is a part of the complex UHV system which consists of eight deposition and analytical instruments allowing the transfer of samples between them at ultra-high vacuum conditions (pressure ~10^{-9} mbar or less). Hence, those samples manufactured/modified in a deposition system are not contaminated when transferred to any of the analytical instruments within the system.



The PES instrument is mounted in a vacuum chamber which is connected to the linear transfer system. The instrument can be isolated from the system by a gate vacuum valve and consists of several components:

- A non-monochromatic twin anode Mg/Al X-ray source XR50 which allows selection of the energy of radiation 1253.6 eV or 1486.6 eV, resp.
- A UV – source UVS-300 providing high flux and very stable ultra violet radiation of adjustable ratio of photon energies of 40.82 and 21.22 eV (He II / He I).
- A flood gun FG 15/40 for sample irradiating with a stream of low energy electrons to compensate sample charging caused by the photoemission from insulating samples.
- A motorized 5-axis stage equipped with an e-beam heater for sample annealing.
- A hemispherical energy spectrometer Phoibos 150 equipped with a 2D CCD detector which allows viewing of data in both dispersive and nondispersive directions to be able to obtain for example angle-resolved or spatially resolved information.

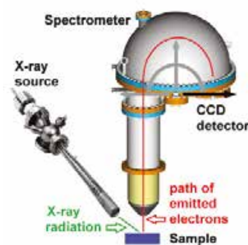
MORE INFO

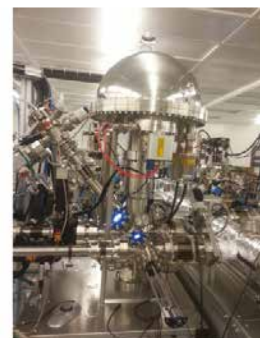
Guarantor: **Marek Otevřel** (marek.otevrel@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/ultra-high-vacuum-preparation-and-analytical-system-uhv-cluster/

METHODS

XPS – X-ray Photoelectron Spectroscopy is a method for characterization of surfaces and ultra-thin films based on emission of electrons from a sample irradiated by X-rays. Obtained XPS spectrum reveals the elemental and chemical composition of the sample surface.
UPS – Ultraviolet Photoelectron Spectroscopy – provides information on the valence band levels and the work functions of materials.
ARPES – Angle-Resolved Photoemission Spectroscopy is one of the most direct methods for studying the electronic structure of solid surfaces.

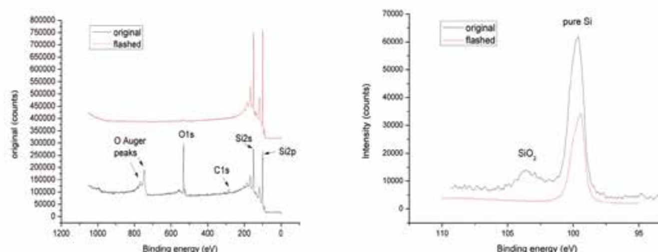
PRINCIPLE XPS



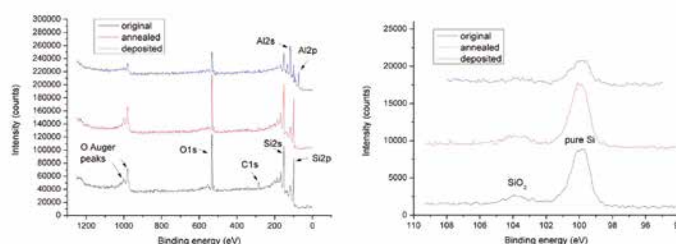


RESULTS XPS

Cleaning of a Si substrate by flash annealing: A silicon substrate surface is covered by an ultra-thin layer of silicon dioxide. One usable method to remove this layer and to get a pure silicon surface is the flash annealing, when the sample is repeatedly and shortly annealed at the temperature of 1250 °C. Since an e-beam heater is not able to provide such a rapid annealing, the sample is flash annealed in a separate Preparation Chamber by the direct resistive heating method and afterwards transferred to the PES chamber for an XPS analysis. The XPS spectra reveals a disappearance of oxygen, carbon and SiO_2.



Deposition of Al on SiO_2:
Sometimes it is not necessary to remove the silicon dioxide layer from a silicon substrate. A sample annealing at the temperature of 600 °C for a few hours removes carbon contaminations from the silicon substrate, but the silicon dioxide remains undamaged. The example shows an XPS spectra of the original sample, after annealing at 600 °C and after deposition of alumina in the Custom Deposition Chamber.

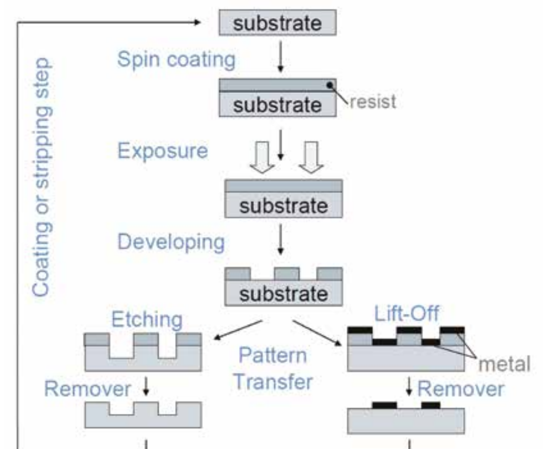


Heidelberg DWL66fs

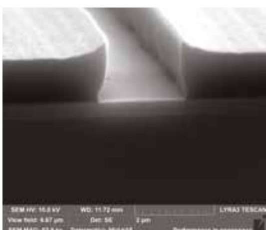
UV Direct-Write Laser (DWL)

DESCRIPTION

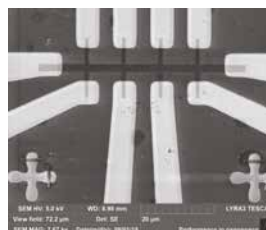
DWL66FS is a patterning tool capable of writing structures with a dimension down to 1 μm on basically any flat substrate. It can be used in various research areas for fabrication of MEMS, BioMEMS, Microoptics, ASICs, Microfluidics, Sensors, CGHs, and all other applications that require microstructures. The instrument writes directly on the substrate (e.g. wafer), so no photomask is needed. The photomask can be fabricated using this instrument, and then used for mask aligner. The writing is done by exposing the photosensitive film on top of the substrate using a UV diode laser of 405 nm wavelength. At the end the laser passes through the writing head with a focal length of 4 mm, determining the instrument resolution of 1 μm. During operation a high resolution interferometer controls the position of the sample stage with high accuracy. This allows the performance of very precise overlay exposures. The DWL features two CCD cameras used for metrology and alignment purposes. The whole system is placed in a laminar air flow box providing constant environmental conditions crucial for the exposure.



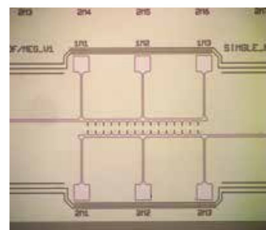
RESULTS



SEM image of AZ5214E resist profile after development.



Contacts for measurement of transport properties of shaped 2D materials - SEM image. Author: Miloš Hrabovský



Part of the single nanowire gas sensor array - optical micrograph. Author: Ondrej Chmela

PRINCIPLE

The DWL66FS is a UV-optical lithography tool. In the process, a substrate is first coated with photosensitive polymer film called photoresist. This is done on a dedicated machine usually with the spin-coating or spray-coating method. After thermal treatment, such a substrate is put into, e.g., the DWL system, where the photoresist film is exposed to UV light in a controlled manner. In the following step, known as development, exposed areas are either removed from the film (positive photoresist used) or left in the film (negative photoresist used). The result is a resist mask on the substrate, which then undergoes etching, deposition, or doping, as required. A photoresist film is finally removed (stripped). These steps can be repeated many times to produce the final device, e.g. microchip.

SPECIFICATIONS

Substrates	From 25×25 mm up to 200×200 mm
Structures resolution	Down to 1 μm
Address grid	Down to 50 nm
Write speed	10 mm²/min
Exposure mode	Raster, Special – 3D
Layout alignment	Camera system
Alignment accuracy	250 nm
Chamber	Climate
Auto focus	Air-gauge
Data input formats	DXF, CIF, GDS, Gerber, BMP, ASCII, STL

MORE INFO

Guarantor: Erik Pálesch (Erik.Palesch@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/uv-direct-write-laser-system-heidelberg-instruments-dwl-66-fs/

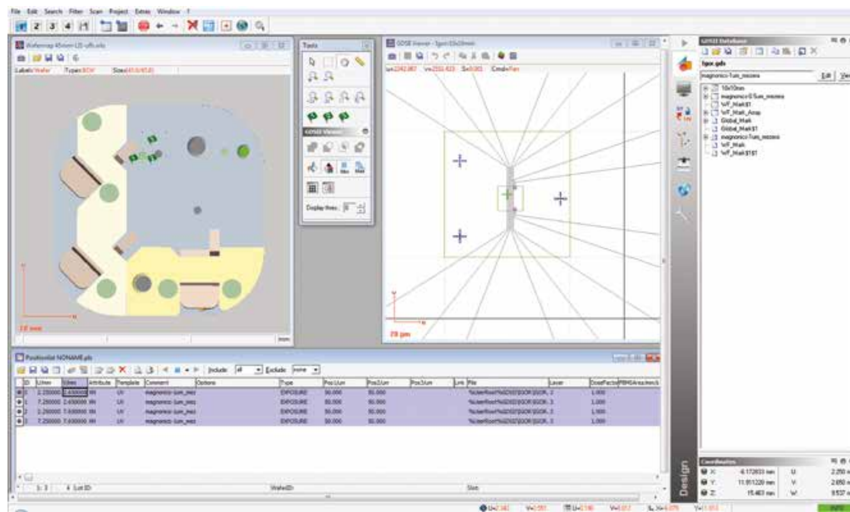
TESCAN MIRA 3 & RAITH LIS

SEM/E-beam writer TESCAN MIRA3

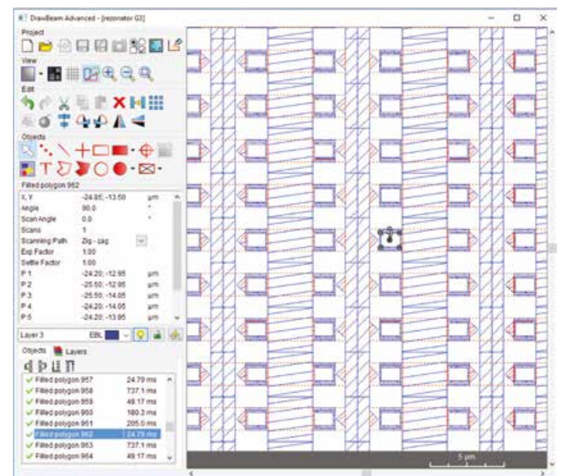
DESCRIPTION

The Scanning Electron Microscope (SEM) in the Nanofabrication laboratory is preferentially used for ebeam lithography (EBL), where the resist-coated sample is selectively exposed to the focused electron beam by means of electrostatic beam blanker and the sophisticated nanolithography attachment, allowing the preparation of very small patterns (< 50 nm) on the resist surface. The stage accuracy of a common SEM is the key limitation for most lithography patterning. Therefore, the instrument is equipped with the laser interferometer stage (LIS) to allow for ultra-high resolution structuring of areas of a millimeter in size.

RAITH - ELPHY Plus

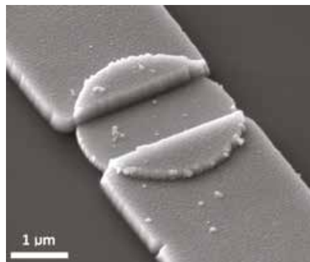


TESCAN - DrawBeam



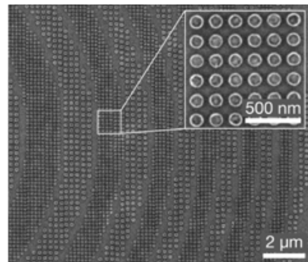
RESULTS

Two-step Lithography



A NiFe disc was fabricated in the first step and golden contacts were added in the second EBL step. The contacted disc was prepared for anisotropic magnetoresistance measurement of magnetic vortex which could nucleate on such structures. (With permission of Marek Vanatka)

Zone Plate Fabrication



SEM micrograph of the zone plate made of silver plasmonic nanodiscs, which were created for the measurement using a coherence-controlled holographic microscope (CCHM). (With permission of Jiri Babocky)

SPECIFICATIONS

SEM	
Product name	MIRA 3 XMH
Emitter	Schottky cathode
Acceleration voltage	200 V–30 kV
Probe current	2 pA–200 nA
Detectors/ Resolution	SE/ 1.2 nm @ 30kV In Beam SE / 1.0 nm @ 30kV
Chamber vacuum	< 9e^{-3} Pa (<5e^{-4} Pa reachable)
Sample size	Up to 2″ wafer size

LIS	
Patterning area	45 mm × 45 mm
Z movement	25 mm
Resolution	2 nm
Stitching accuracy	≤ 100 nm
Overlay Accuracy	≤ 100 nm
Lithography Software	
DrawBeam (Tescan)	
Elphy (Raith)	

MORE INFO

Guarantor: Vojtěch Švarc (vojtech.svarc@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/scanning-electron-microscope-e-beam-writer-tescan-mira3/

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Electron beam lithography and imaging

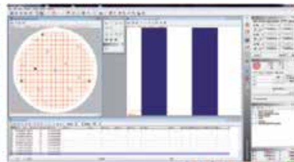
Raith150 Two

PRINCIPLE

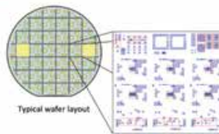
Electron-beam lithography is the practice of scanning a focused beam of electrons to draw custom shapes on a surface covered with an electron sensitive film called a resist (“exposing”). The electron beam changes the solubility of the resist, enabling selective removal of either the exposed or non-exposed regions of the resist by immersing it in a solvent (“developing”). The purpose is to create very small structures in the resist that can subsequently be transferred to the substrate material, by etching or thin film deposition.

FEATURES

Fully automated 8 inch loadlock



Raith NANOSUITE - convenient exposure setup

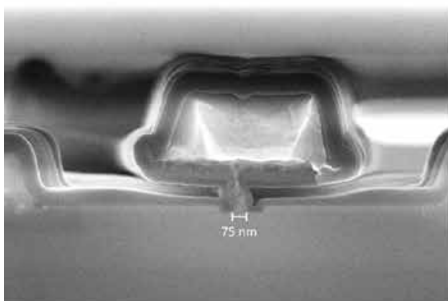


Chip layout with Writing Fields aligned according to gate positions

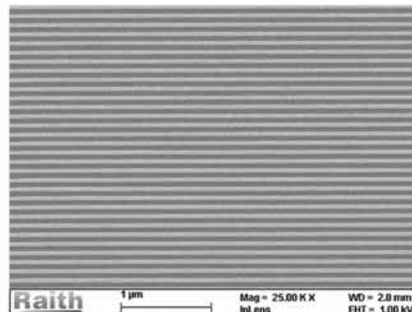
SPECIFICATIONS

Beam current range	5 pA – 20 nA
Beam energy	20 eV – 30 eV
Stage travel range	150×150×20 mm
Current density	≥ 7500 A / cm²
Current stability	≤ 0.5 % / 8 hours
Minimum line width	< 8 nm guaranteed
Stitching accuracy	≤ 35 nm (mean +3σ)
Overlay accuracy	≤ 35 nm (mean +3σ)

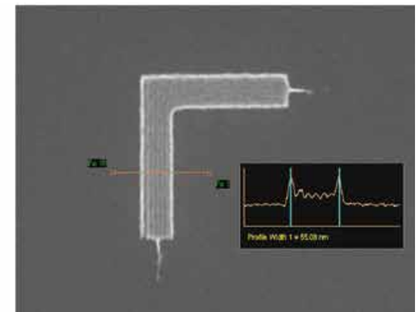
EXAMPLES OF APPLICATIONS



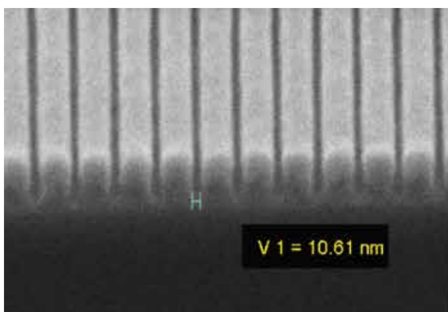
High electron mobility transistor (HEMT) with 75nm T-gate



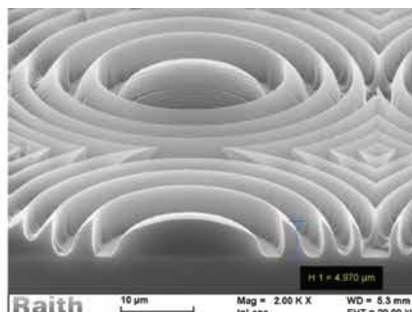
60nm pitch grating in PMMA e-beam resist



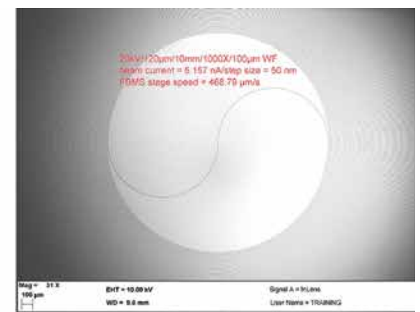
4.5nm lines and spaces in HSQ e-beam resist



Accurate 11nm lines in PMMA (crossection) e-beam resist



3D-Fresnel lens array in 5µm thick e-beam resist



Optical delay waveguide written with traxx-a stitch error free writing mode

MORE INFO

Guarantor: **Robert Doczy** (robert.doczy@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/e-beam-writer-raith150-two/

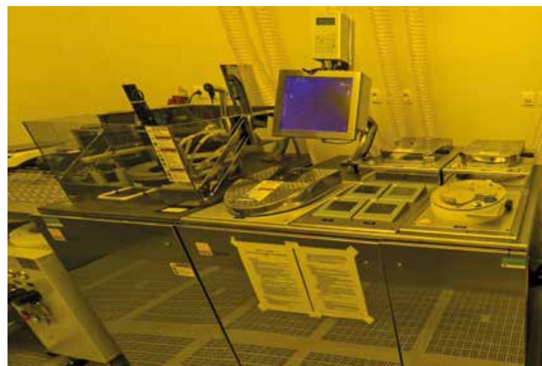
CEITEC | BRNO UNIVERSITY OF TECHNOLOGY | UNIVERSITAS MASARYKIANA BRUNENSIS

Resist coating and development system

SUSS MicroTec RCD8

DESCRIPTION

The SÜSS MicroTec system consists of RCD8 semi-automatic Resist Coat and Develop Platform, manual LabSpin6 spin coater platform, two manual hot plates HP8 and vapor primer VP8. The RCD8 is a tool which can be converted from a spin coater with the GYRSET® closed cover coating technology without backside contamination to a developer within a few minutes. Furthermore, square substrates and pieces can be coated all the way to the corners with a homogeneous thickness of resist. Spin coating is the process of evenly coating a spinning substrate with a solution. The solution (lithographic resist) is dispensed at the center of the wafer. Subsequent acceleration as well as the rotation speed and the time allotted to the individual steps ensure that a homogeneous thickness of layer remains after excess resist is spun off. Alongside the process parameters, the physical properties of the solution or resist determine the thickness of the applied film. Developing is the process of selective removing of resist after exposure.



RCD8 Platform

Hot plate HP8

Spin coater LabSpin6

Vapor primer VP8

Dispense arm

LabSpin6 platform is manual coater system that has been designed for a variety of lithographic chemicals. The speed of LabSpin6 is 100–8 000 rpm with acceleration up to 4 000 rpm/sec. The process bowl is made of polypropylene and the bowl cover is made of safety glass.

For a uniform and stable vapor priming of the substrates, dehydration of the surface by high temperature baking (up to 200 °C) is needed. Applying HMDS by Vapor primer to dehydrated wafers is one common method for achieving the surface hydrophobicity.

SPECIFICATIONS

General	
Substrate size	2" to 8" (200 mm) round, 2" to 6" square
Substrate handling	manual, lift pins
User interface	SUSS MMC Tool Control on Windows 7, PC with touch screen control
Max. # of recipes	unlimited
Max. # of process steps	50
Utilities	400 V, 16 A, 50 Hz, vacuum not needed, produced internally by N_2
Module: Open Bowl coater	
Spin speed max	10 000 rpm ± 1 rpm (with safety hood)
Spin acceleration	1–7 000 rpm/s
Module: Gyrset® Coater	
Spin speed max	3 000 rpm ± 1 rpm with GYRSET®
Spin acceleration	1–3 000 rpm/s
Module: Puddle Developer	
Waste	individual drain connection
Bowl material	polyethylene
Nozzles	optional dispense arm with 3 lines
Module: Hotplate (HP8)	
Controller	via MMC Tool Control of RCD8 or separate controller
Temp. Range	60–250 °C (± 0.5 °C < 120 °C; ± 1 % ≥ 120 °C)
Options	nitrogen purge

MORE INFO

Guarantor: **Erik Pálesch** (erik.palesch@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/resist-coating-and-development-system-suss-microtec-rcd8/

Mask aligner

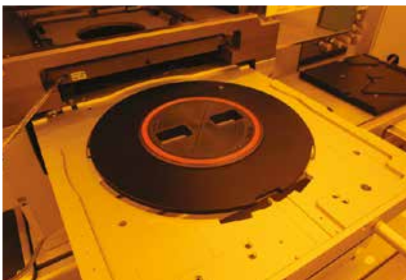
SUSS MicroTec MA8

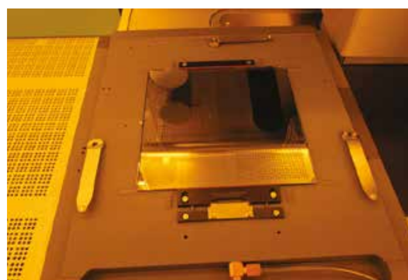
DESCRIPTION

Süss MicroTec MA8 is a standard UV litography tool for exposing wafers through a mask. A photoresist-coated wafer is illuminated by UV light in the range of 350–450 nm wavelength, which is produced by a 1000 W Hg lamp. Such power of the UV-lamp ensures high work-flow. This tool is equipped with high performance MO Exposure Optics® to give uniform illumination over the surface of an up to 6 inch wafer. Exposure can be carried out in proximity mode or in contact mode. In the first case there is a defined distance between the mask and wafer during exposure, while in the other case the wafer and mask are brought into direct contact. Depending on the force which pushes the wafer to the mask, the tool works in the so-called soft contact, hard contact, or vacuum contact mode. Vacuum contact offers highest resolution of pattern transfer, but also the highest mask wear. MA8 is equipped with top side optical microscopes and bottom side microscopes, so alignment is possible from both sides of the wafer.

PRINCIPLE

MA8 is a UV-optical lithography tool. In the process, a flat substrate coated with photosensitive polymer film (called photoresist) is exposed to UV light in a controlled manner. Exposed areas are subsequently either removed from the film (positive photoresist used) or left in the film (negative photoresist used) during the development procedure. The result is a mask created on the substrate, which then undergoes etching, deposition, or doping, as required. The photoresist film is finally removed (stripped). These steps can be repeated many times to produce a final functional device, e.g. microchip.





SPECIFICATIONS

Mask and Wafer/Substrate	
Wafer size	1" – 200 mm
Max. substrate size	200×200 mm
Min. Pieces	5×5 mm
Wafer thickness	max. 10 mm
Mask Size	standard 2"×2" up to 9"×9" (SEMI)
Exposure Modes	
Contact	soft, hard, vacuum
Proximity	exposure gap 1–300 mm
Gap setting accuracy	1 µm
Modes	constant power, constant dose
Options	Flood exposure
Exposure Optics	
Resolution	1.5 µm (vacuum); 2 µm (hard); 3 µm (soft); 3.5 µm (proximity 20 µm)
Wavelength range	UV400 350–450 nm
Exposure source	Hg lamp 1000 W
Intensity uniformity	less than 3.5 % (200 mm)
Alignment methods	
Top side alignment (TSA)	accuracy less than 0,5 µm (with assisted alignment & SUSS MicroTec recommended wafer targets)
Bottom side alignment (BSA)	accuracy less than 1 µm

MORE INFO

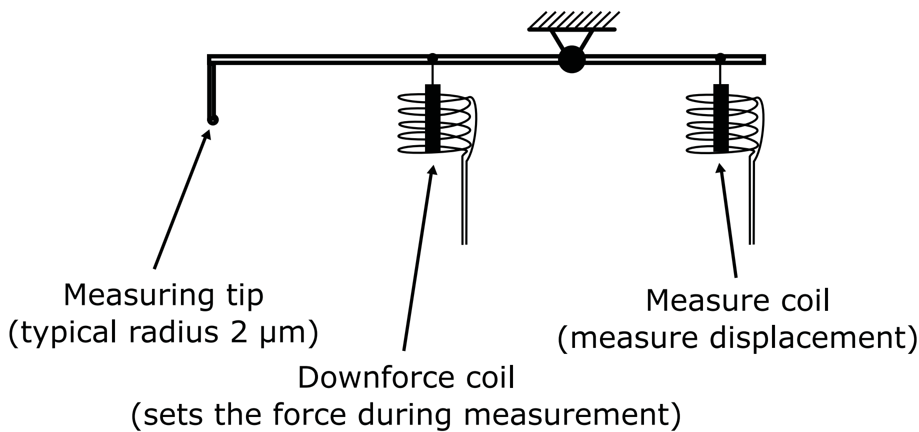
Guarantor: **Erik Pálesch** (erik.palesch@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/mask-aligner-nanoimprint-lithography-suss-microtec-ma8-ba8-gen3/

Mechanical Profilometer

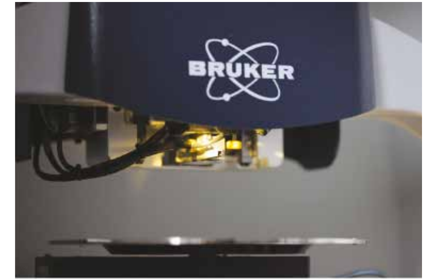
Bruker DektakXT

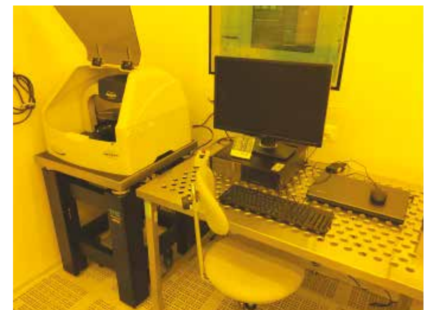
PRINCIPLE

The DektakXT stylus surface profiler is an advanced thin and thick film step height measurement tool. In addition to profiling surface topography and waviness, the DektakXT system measures roughness in the nanometer range. Available with a standard manual sample-positioning stage or an optional automatic X-Y or theta stage, it provides a step-height repeatability of 5Å (<0.6 nm). In addition to taking two-dimensional surface profile measurements, the DektakXT system can produce three-dimensional measurements and analyses when equipped with the 3D Mapping Option.



RESULTS





SPECIFICATIONS

Stage	diameter of 200 mm
Max. points	120
Repeatability	< 5 Å
Vertical resolution	1 Å
Pulse Freq.	1 - 200 kHz
Resolution	0.5 μm
Repeatability	1 μm
Vertical Range	56 mm

MORE INFO

Guarantor: Dalibor Šulc (dalibor.sulc@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/laser-dicer-oxford-lasers-a-series/

Fume hoods

DESCRIPTION

The fume hoods located in the labs of ISO 5 cleaness are specially designed for lithographic and wet etching purposes. We have two fume hoods made of stainless steel for work with organic solvents and six fume hoods which are used for work with corrosive liquids (acids/bases for etching and development). These are made of polypropylene (PP). Three of these PP fume hoods can only be used for clean processes of at least MOS grade. This involves use of special clean chemicals and only specific plastic labware. For example, one fume hood is reserved for RCA wafer cleaning process.

Generally each fume hood contains a set of process baths with specific functions (heating, ultrasound agitation, etc.). When used in combination with dynamic rinsing, one is able to utilize a complete and accurate wet process for a bundle of substrates. Still, there is enough room to work easily with just small samples. Functions of the fume hood are conveniently controlled by the user software accessible by the touchscreen.

With the help of this wet process unit we can put the substrate through the complete nanofabrication process – from pre-cleaning, followed by lithography, then deposition/etching/doping, ending with final substrate cleaning.

PRINCIPLE

Resist is a cover material which protects parts of a substrate that won't be attacked by aggressive etching agents. These are mostly corrosive/dangerous liquids, including hydrofluoric acid, TMAH, or perchloric acid. Often etching mixtures are used to provide a more controllable etching process. This means better selectivity, or slower etching. Wet etching is in most cases isotropic – etches with the same speed in all directions. An exception might be KOH etching of silicon, which is anisotropic – different etching speed along different crystal planes. Wet etching might be superior to more modern plasma dry etching in cases when we want to work with more substrates at the same time.

FEATURES

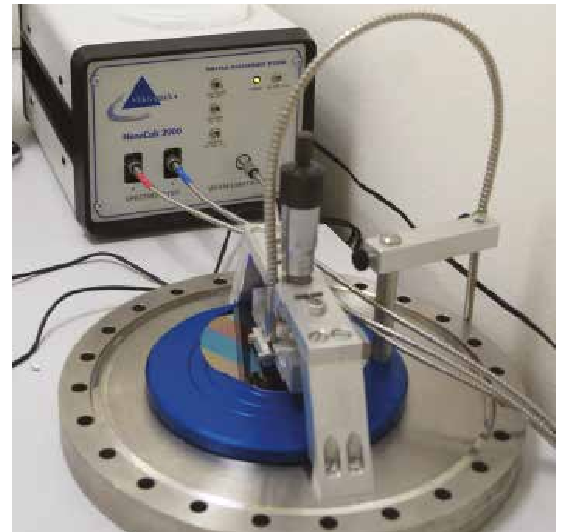
- Specific process baths for various etching & cleaning processes
- Built-in ultrasound & megasound agitation available
- Heating, cooling function
- Dynamic rinsing function
- Water shower & nitrogen gun for quick drying
- Built-in canisters for handling hazardous chemical waste Intuitive user friendly software

MORE INFO

Guarantor: **Erik Pálesch** (erik.palesch@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/fumehoods/

Spectroscopic reflectometer

NanoCalc 2000



DESCRIPTION

NanoCalc is a reflectometry thin film measurement system. It is based on spectroscopic reflectometry to accurately determine optical or non-optical thin film thickness in the range of a few nanometers up to hundreds of micrometers with a resolution of 0.1 – 1 nm. It is suitable for applications in a variety of semiconductor applications, such as resist thickness and oxide film thickness measurement. The NanoCalc system measures anti-reflective coatings, anti-scratch coatings and rough layers on substrates such as steel, aluminum, brass, copper, ceramics and plastics. A big advantage of this type of measurement is the fast observation of the thickness of these films.

FEATURES

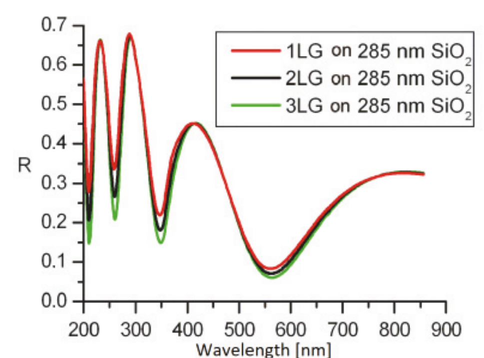
- UV/VIS/NIR and high resolution configurations
- accuracy to 1nm, resolution to 0.1nm
- measure up to 10 layer stacks
- measurement of transparent metallic layers down to 1 nm thick
- adaptors for complex geometries and accessories for thickness mapping
- sophisticated algorithms enable defect and roughness tolerant measurements
- large database ensures accuracy over a broad range of materials

SPECIFICATIONS

Spectral range	250 – 1100 nm
Total thickness range	10 nm – 100 um
Resolution	0.1 nm
Repeatability	0.3 nm
Absolute accuracy	< 1 % (100 nm – 10 um)
Number of layers	up to 10 layers
Distance with fiber	1 – 5 mm
Distance with optic	5 – 100 mm
Angle with optic	90° (nominal incidence)
Spot size	400 um

APPLICATIONS

Measuring software



Comparison of reflectivity measurement on a single, double, and triple layer of Graphene on SiO_2. [with permission of Zuzana Lišková]

MORE INFO

Guarantor: **Vojtěch Švarc** (vojtech.svarc@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/spectroscopic-reflectometer-ocean-optics-nanocalc-2000/

Atomic Layer Deposition (ALD)

Ultratech-Cambridge Nanotech Fiji 200

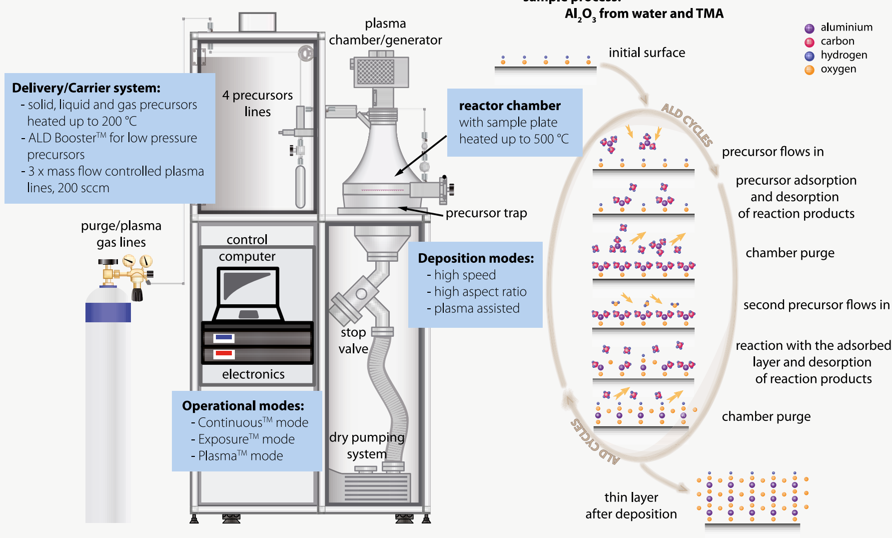
DESCRIPTION

Atomic Layer Deposition is a deposition technique for very thin layers with the thickness control down to a single atomic layer. It belongs to the CVD techniques family. The thickness precision is achieved by pulsed deposition, where first a metal-containing precursor is introduced into the chamber and after a short time (allowing for a monolayer adsorption) the chamber is pumped down. The following step is exposure to the oxidizing precursor (for oxides) or nitrogen containing precursor (for nitrides). Thus, a monolayer of target material is grown. The metal-containing precursors are usually organometallic ones, for oxidation a water or oxygen plasma can be used, nitrida-tion is done using water or nitrogen plasma. To achieve the deposition in the ALD mode, the sample is heated up to a certain temperature, for most processes being in the range 150–300 °C.

FEATURES

ALD system for up to 8“ samples, equipped with plasma generator.
Standard materials: Al_2O_3, AlN, HfO_2, HfN, TiO_2, TiN, SiO_2, SiN, other materials on request.

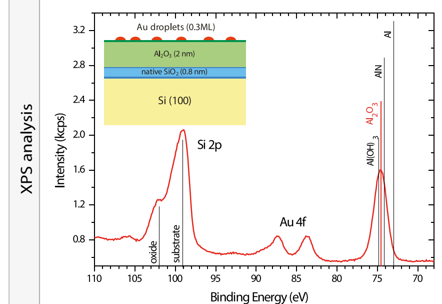
- thermal deposition within range RT-500 °C
- 4 precursor lines, with possible upgrade to 6
- plasma-enhanced deposition (3 plasma gas lines)
- expo mode for homogeneous deposition on high-aspect-ratio nanostructures
- controlling software allows preparation/modification/storage individual recipes
- fully automatic programmable operation



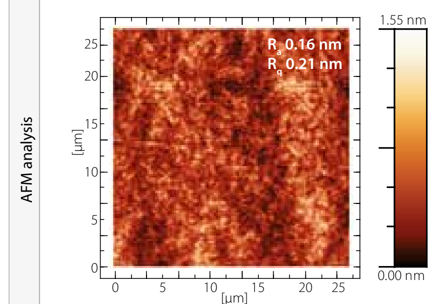
APPLICATIONS

Al_2O_3 diffusion barrier deposition

XPS analysis



AFM analysis



MORE INFO

Guarantor: **Marek Eliáš** (marek.elias@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/atomic-layer-deposition-system-ultratech-cambridgenanotech-fiji-200/

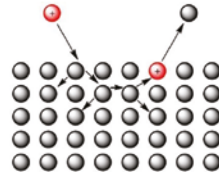
Ion beam etching Scia Systems Coat 200

DESCRIPTION

Ion beam etching (IBE) removes material from the etch target by bombardment with directed and precisely controlled ion energies. IBE is also referred to as "ion beam milling". The IBE source generates plasma from a noble gas, typically argon. A set of electrically biased grids establish the ion beam energy and angular divergence of ions within the beam. The ion beam strikes the substrate, removing material by physical sputtering.
Ion beam etching provides directional flexibility that is not available in other plasma processes. While the etch rate with IBE is typically lower than for reactive ion etching (RIE), IBE offers high precision (high anisotropism) for applications that demand exacting profile control. Also, ion beam etching can be used to remove materials where RIE may not be successful. Ion beam can etch alloys and composite materials that are not compatible with RIE.
Ion beam etching has many applications, including nano-machining of magnetic transducers, MEMS devices, and trimming of surface acoustic wave (SAW) and bulk acoustic wave (BAW) filters. A newer application is fabricating high-performance non-volatile memory, specifically "spin transfer torque" MRAM (magnetoresistive random-access memory).

Ion Beam Sputtering

- Atoms of target material can be ejected by bombardment of energetic ions
- An atom can be ejected, when the kinetic energy of the recoil atom exceeds the surface binding energy
- Momentum exchange between incident ions and atoms of the target material due to collision cascades
- Deposition on the substrate surface and build a thin film of the target material
- Almost any material can be deposited, stoichiometric deposition of compounds
- Low process pressure (10^{-4} mbar) and temperature (<100°C)

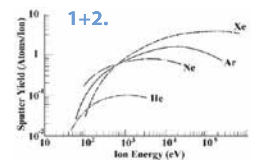


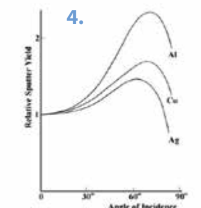
Sputter Yield

Sputter Yield is the number of removed target atoms for each incoming primary ion.

$$Y = \frac{n\ (sputtered\ atoms)}{n\ (primary\ ions)}$$

1. Energy of incoming ion
2. Mass ratio between incoming ion and sputtered atoms
3. Material to be sputtered (binding energy)
4. Angle of incidence of ion flux



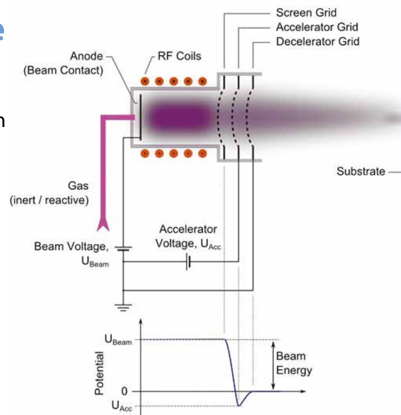


RF source as example

- Inductively coupled RF Plasma
- Grid system

→ Extraction and optics of ion beam

- MW power 0–800 W (operation range 250W–350W)
- Beam Voltage 0.05V–2 kV
- Accelerator Voltage 0.1 V –1 kV



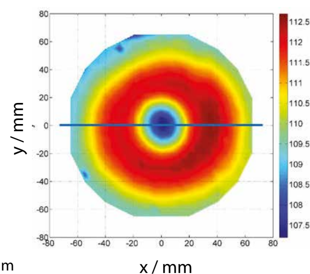
Properties of a microwave ion beam source

- High density plasma
- Long-time stability due to inner coupling instead of ICP source
- Less maintenance of the plasma cup required
- Wavelength in the range of the hardware size: standing wave effects
- Temperature and recombination effects are critical
- Tuning affects not only reflected power but also plasma density distribution in the source

Homogeneity of etching

SiO_2 Ion Beam Milling in Argon Process

- Energy 600 eV / Current 245 mA / Rotation 20 rpm / Perpendicular incidence
- Process time 300 s at 22 nm/min
- Helium cooling applied

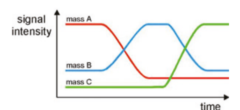


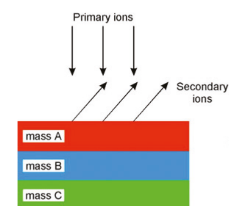
mean = 110.8 nm / min = 107.2 nm / max = = 112.2 nm (min–max)/(2*mean) = 2.5% / sigma = 1.1 nm

Sigma = 1.0 % for 150 mm wafer

End point detection by Secondary Ion Mass Spectroscopy

- End point analysis during ion etching by secondary ion signal detection
- Hardware integration and threshold level software integration
- Additional Residual Gas Analysis (RGA) and leak detection functionalities





SPECIFICATIONS

circular microwave broad ion beam source at frequency 2.54 GHz
ion energy 50–2,000 keV
sample size up to 6" wafer
He backside cooling
Loadlock
endpoint detection system SIMS HAL IMP 301/3F with accuracy 1 nm
Ar+ sputtering
reactive ion beam etching CHF3, O2, SF6

MORE INFO

Guarantor: **Marek Eliáš** (Marek.Elias@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/ion-beam-etching-scia-systems-coat-200/

Electron beam evaporator

BESTEC

PRINCIPLE

In the evaporation process, vapors are produced from a material located in a source (positively charged anode) which is heated by an electron beam (given off by a charged tungsten filament). The process is carried out in high vacuum (10^{-7} to 10^{-8} mbar) so that the evaporated atoms undergo an essentially collisionless line-of-sight transport prior to condensation on the substrate. The substrate is usually at ground potential (i.e., not biased).

Although E-Beam Evaporation is used in a wide variety of applications, it is particularly efficient in transferring pure and precise metal coatings that require high melting temperatures to substrates on the atomic and molecular level.

Several different layers of coating from different target materials can be applied with a multiple crucible E-Beam evaporator without breaking the vacuum making it adapt easily to a variety of lift-off masking techniques.

Electron beam gun

The impact of a high energy electron beam into a metallic sample offers a clean and high energy density method of heating (compared to resistive and inductive type of evaporators). In the electron gun, the electrons are produced by the hot electron emission from a tungsten cathode (thermionic gun) and are formed into a beam. At the impact point of the sample, most of the electron energy is given out as heat. Thermionic guns have the limitation of a minimum operating gas pressure of about 1.3×10^{-3} mbar [1].

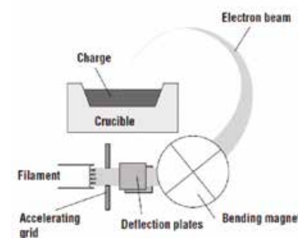
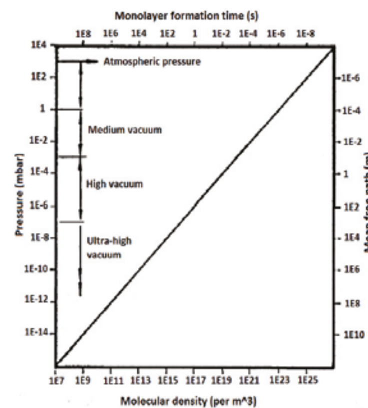


Fig. A popular e-beam evaporative source using a strong magnetic field which bends the beam through 270°. The beam can be rastered across the material to melt a significant fraction of the surface [2].

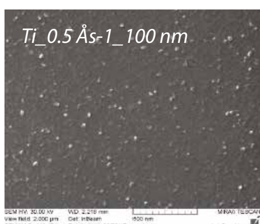


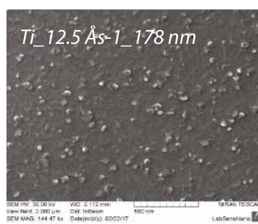
Values of molecular density, mean free path and time to form a monolayer, as a break of pressure, for air at 25°C [3] (1 mbar = 100 Pa).

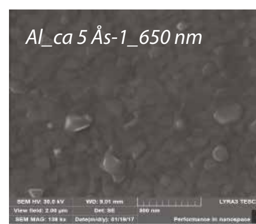
RESULTS

Thin film microstructure depends on:

- nature of the substrate
- temperature of the substrate during deposition (heating up the substrate increases the incident atom mobility and the step coverage of the coating)
- rate of deposition
- deposit thickness
- angle of incident of the vapor stream
- pressure and nature of the ambient gas phase [1]







SEM micrographs of some test depositions made by the e-beam evaporator at the room temperature and different deposition rates (the chamber pressure might vary as well). The deposit thickness is a more crucial parameter than the deposition rate - proven only on Ti layers.

SPECIFICATIONS

Accelerating voltage	up to 10 kV
Power source	up to 10 kW
Sample size	7×7″
Substrate temp.	RT–900 °C
Standard deposition materials	Au, Ag, Ti, Cr, Cr_2O_3, Co, Al, Cu, Ni, Fe, CrNi, NiFe

MORE INFO

Guarantor: **Marek Eliáš** (marek.elias@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/electron-beam-evaporator-bestec/

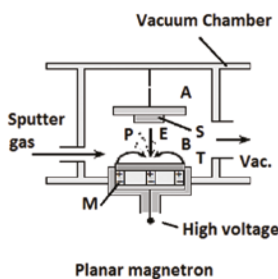
PUBLICATIONS

[1] R.F. Bushah: Handbook of Deposition Technologies for Films and Coatings, Noyes Publication, New Jersey, 1994
[2] S.A. Campbell: Fabrication Engineering at the Micro– and Nanoscale, Oxford University Press, Oxford, 2008
[3] N.S. Harris: Modern Vacuum Practice, McGraw-Hill, London, 1989

Magnetron sputtering system

PRINCIPLE

- sputter deposition of thin layers by an ion bombardment of a solid substrate (negativly charged target – cathode)
- using glow discharge of a process gas (Ar, O_2, N_2, etc.) in a magnetic field
- universal process – large range of applications
- standard (Ar) or reactive sputtering (O_2, N_2)
- good for layer by layer or alloy depositions
- relatively high deposition rates
- reduced substrate heating
- DC generators – used to sputter only conducting targets (charge accumulation on nonconducting targets)
- RF generators – conductors, semiconductors and insulator sputtering
- improved step coverage – higher impact energy and mobility of incident atoms compared to evaporation
- deposition conditions are generally determined empirically – i.e.: deposition rate, target voltage, working gas species and pressure, and the substrate temperature and plasma bombardment conditions
- targets can be formed by casting or by hot pressing powders. In addition, composite targets can be formed by placing wires, strips, or discs of one material over a target of another material.



Planar magnetron sputtering system using fixed bar magnets T: target, P: plasma, M: magnet, E: electric field, B: magnetic field (after Wasa and Hayakawa) [1].

- magnetron = sputtering source with magnetic plasma confinement
- magnetic field is induced on the cathode side to trap the electrone current
- electrons spiral around the magnetic fiel lines which increases their collision probability with neutral gas atoms and creation of ions
- higher ion density leads to higher io bombardment rate of the target
- allows plasma formation at lower pressure (10^{-5} to 10^{-3} torr)
- eliminates substrate heating by electro bombardment

Comparison of evaporation and sputtering

EVAPORATION	SPUTTERING
low energy atoms	higher energy atoms
high vacuum path • few collisions • line of sight deposition • little gas in film	low vacuum, plasma path • many collisions • less line of sight deposition • gas in film
large grain size	smaller grain size
fewer grain orientations	many grain orientations
poorer adhesion	better adhesion

Planar magnetron target using permanent magnets to supply the magnetic field (after Wasa and Hayakawa) [1].

SPECIFICATIONS

Eight 2" magnetron sputter sources (targets) in confocal sputter up configuration
3 DC source, power up to 500 W
1 RF source, power up to 500 W
Substrate temperature RT – 900°C
Rotation of substrate
Sample size up to 4"
Process pressure 2×10^{-4} to 7×10^{-2} mbar
Gas line for reactive deposition O_2 or N_2
Targets, e.g. Pt, Au, Ti, Ta, Gd, Ru, Si, Co, NiFe, FeRh, SiO2

PUBLICATIONS

[1] S.A. Campbell: Fabrication Engineering at the Micro- and Nanoscale, Oxford University Press, Oxford, 2008

[2] W. H. Class: Deposition and Characterization of Magnetron Sputtered Aluminum and Aluminum Alloy Films, Solid State Technol. 22: 61, 1979

CEITEC publications

Mozalev, A. et al. Formation and gas-sensing properties of a porous-alumina-assisted 3-D niobium-oxide nanofilm. *Sensors Actuators, B Chem.* **229**, 587–598 (2016).

P. Gallina, Fabrication of Graphene Mid-Infrared Biosensor, Brno University of Technology, 2016.

MORE INFO

Guarantor: **Marek Eliáš** (marek.elias@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/high-vacuum-magnetron-sputteringbestec/

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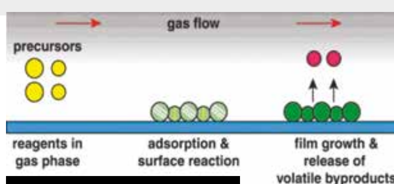
Metal Organic

Chemical Vapor Deposition

DESCRIPTION

This system processes a single horizontally oriented wafer (100 mm) placed on a heated substrate holder inside a cylindrical stainless steel reactor (wall temperature is kept at a lower value). The essential part is the showerhead through which the system delivers process gasses and precursor vapors at a reduced pressure. Metal-organic precursors decompose at the wafer surface, leaving the metal component to form the required layer, while the gaseous organic products escape.



Gas delivery system

PRECURSORS

For selected metals precurors are available in various configurations, solid or liquid forms and different levels of toxicity

- Bis(cyclopentadienyl)**zirconium** dichloride [Zirconocene dichloride] white *powder*, **respiratory, eye or skin irritation**
- Bis(tetramethyl-heptanedionato)barium hydrate [Ba(TMHD)2] white odourless powder, melting at 200°C, **poison**
- **Iron**(III) acetylacetonate, melting at 183 °C, **skin irritation**
- **Titanium**(IV) iso-propoxide, light yellow liquid, boiling at 58 °C, **highly flamable, toxic**

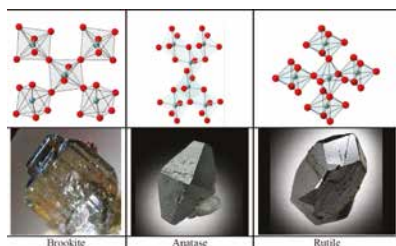
SPECIFICATIONS

Temperature control
from room temp. up to 800 °C, precision 1 °C
Precursor control
3 gas lines with mass flow controllers
carrier gasses (N2, Ar) + process gas (O2)
3 liquid precursor delivery lines with indep. control of carrier gas flow
2 thermally controled evaporators (CEM) + bubbler (10-50 °C with precision 0.2 °C)
Uniformity of the deposition
nonhomogeneity < 3%
improved by holder rotation (up to 200 rpm)

PRODUCTS

Titanium Dioxide TiO_2

UV protection films, sensors, biomedicine; inorganic **nanotubes** and **nanoribbons** can be produced from anatase phase by hydrothermal synthesis or anodization.



Iron(III) oxide Fe_2O_3

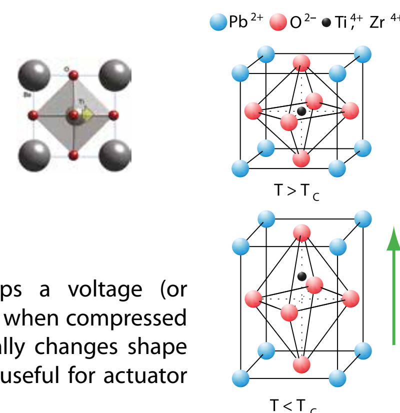
The most common ferromagnetic particle used in all types of magnetic storage and recording media small particles (<10 nm) of gamma phase (cubic) are superparamagnetic

Hafnium dioxide HfO_2

Optical coatings, **high-k dielectric** in MOS devices

Barium Titanate $BaTiO_3$

Capable of the **photorefractive effect:** nonlinear optical effect seen in certain crystals and other materials that respond to light by altering their refractive index. The effect can be used to store temporary, erasable holograms and is useful for *holographic data storage*. It can also be used to create a phase-conjugate mirror or an optical spatial soliton.



Lead zirconate titanate $Pb[Zr_xTi_{1-x}]O_3$

Piezoelectric material like PZT develops a voltage (or potential difference) across two of its faces when compressed (useful for sensor applications), or physically changes shape when an external electric field is applied (useful for actuator applications).

- pyroelectric
- ferroelectric (spontaneous electric dipole)

MORE INFO

Guarantor: **Filip Münz** (munz@physics.muni.cz)
Web: http://nano.ceitec.cz/metal-organic-chemical-vapor-deposition/

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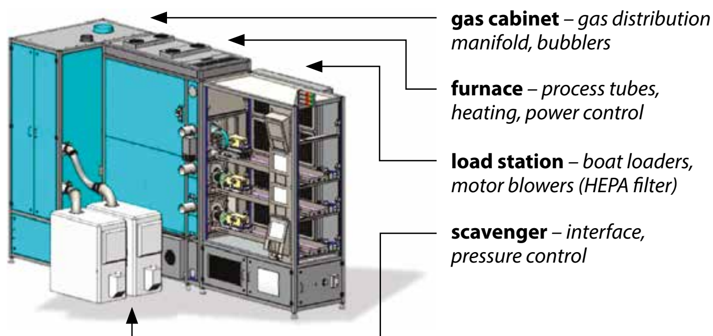
UNIVERSITAS MASARYKIANA BRUNENSIS

Low Pressure

Chemical Vapor Deposition

DESCRIPTION

The equipment is designed for thermal processing of wafers at reduced pressure (LPCVD) and with the addition of **process gases**. Wafers are placed on the inside of cylindrically shaped, open ended (LPCVD) quartz process tubes. Stainless steel flanges are attached to each open end of the process tube – they are used for evacuation of the process tube and for injection of process gases.
Heating is performed by a cylindrically shaped, thermally insulated heating element divided into three independent zones.
Wafers are placed perpendicularly to the tube axis on **quartz wafer boats** that are moved in and out using an SiC paddle on a motorized loader.



Up to 50 wafers of 100 mm can be placed in one quartz boat (single side deposition is possible by placing the wafers back-to-back). The atmospheric tube is equipped with a soft load port (the paddle places the boat in the center of the quartz tube and leaves the oven, the opening is sealed by an insulating door)

Proces gasses
N_2
O_2
N_2O
NH_3
H_2
SiH_4 silane
SiH_2Cl_2 dichlorosilane

Dopant diffusion
Phosphorus
from $PoCl_3$ in bubbler

Boron
from BBr_3 in bubbler

Cleaning
DCE dichloroethylene
from bubbler

SPECIFICATIONS

Currently 3 positions (of 4 available) are used for:

» **silicon nitride** (SixNy - variable stechiometry) growth
low pressure process from SiH_2Cl_2 and NH_3 prec.
up to 500nm thick layers in temp. range 600–800 °C

» **polycrystaline silicon** growth
from SiH_4 decomposition at low pressure
rate about 20 nm/min at 600 °C

» **atmospheric** pressure **oxidation** process
- dry oxidation - up to 4 nm/min at 900–1050 °C
- wet oxidation - up to 5 nm/min [using external burner creating hot water vapour from $H_2 + O_2$ reaction]
highest quality oxides are obtained with simultaneous DCE etching of spurious depositions

this position allows also for N and P-type doping (using phosphorus and boron) down to 40 Ohm/square resist.

silicon oxide/oxinitride growth at low pressure is possible in original design (high temp. from SiH_2Cl_2 and N_2O, low temp. process from SiH_4 and O_2), currently these are not used (oxides of similar quality are available from PECVD reactors)

- homogeneity required in all processes across a single wafer is better than 3%, less than 8% for the whole batch

MORE INFO

Guarantor: **Filip Münz** (Filip.Munz@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/atmospheric-pressure-chemical-vapor-deposition/

Cryogen-free measurement system

Mini Cryogen-Free Magnet System

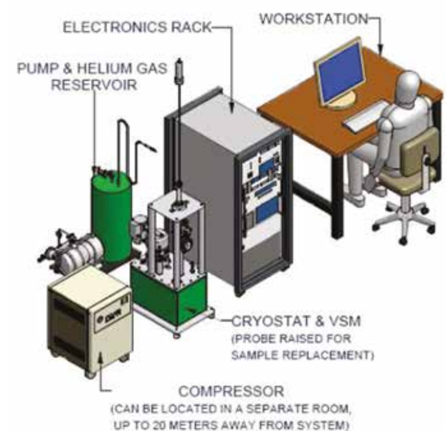
DESCRIPTION

The Mini Cryogen-Free Magnet System is a physical device used for the production of a strong magnetic field created by a superconducting coil. The system combines the latest cryogen-free technology with sophisticated measurement techniques providing a versatile, powerful investigative device achieving low temperatures and high magnetic fields without the direct use of liquid helium or nitrogen. A closedloop system uses helium gas for refrigeration of the superconducting magnet and the sample. The instrument is designed for the measurement of magnetic properties using VSM (Moment and AC Susceptibility) and electrical properties (Resistivity and Hall effect). The measurement range for the magnetic field is from – 9 T to 9 T and for temperature from 1.6 K to 400 K.

INSTRUMENT SETUP

The instrument is comprised of the following components:

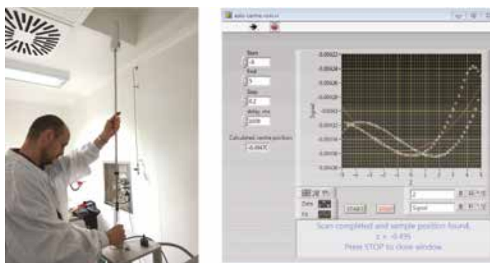
- A cryostat incorporating a cryocooler, superconducting magnet and a variable temperature sample space.
- A rack incorporating electronics for control and monitoring of the cryostat and any measurement options.
- Measurement system software.
- Sample probes.
- Measurement options (VSM, Resistivity).



Mounting of the sample

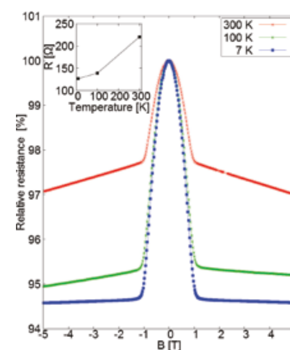
Samples with a maximum size of 10 mm are mounted on the special holders (VSM or Resistivity measurements).

The holders are fitted to a probe rod which is inserted into the device. The correct position of the sample is found both manually and using the autocentre utility



Resistivity measurements

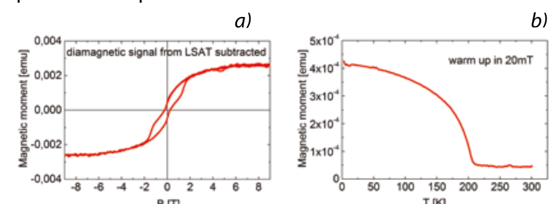
The Electrical Transport module provides the capability to perform DC resistance measurements and Hall voltage measurements in samples with resistance in the range from 1 μΩ to 1 MΩ with variable temperature (from 1.6 K to 400 K) and magnetic field (from – 9 T to 9 T)



Electrical transport measurements of NiFe wire with dimensions 1 μm × 100 nm × 100 μm at three different temperatures.

VSM measurements

The Cryogenic VSM (Vibrating Sample Magnetometer) is designed to measure DC magnetic moment. It allows the measurements of magnetic hysteresis loops or temperature dependencies (T = 1.6 K - 400 K, B = – 9 T - 9 T). Analysed samples can be in the form of bulk, thin films, powders or liquids.



a) hysteresis loop and b) magnetization dependence on temperature for 30nm thin ferromagnetic layer $La_{0.7}Sr_{0.3}CoO_3$ on LSAT substrate.

MORE INFO

Guarantor: **Jiří Liška** (Jiri.Liska@ceitec.vutbr.cz), **Adam Dubroka** (Adam.Dubroka@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/mini-cryogen-free-magnet-system-cryogenic-limited/

Semi-automated four-probe system

Summit 12000

DESCRIPTION

The Summit series semi-automated four-probe system allows access of the full range of test instruments for wafers up to 200 mm. Application includes RF/ microwave, device characterization, wafer level reliability, e-tests and modelling or yield enhancement. The system allows the automatic measurement of whole wafers once it is set by the user. Four-probe sensing is an electrical impedance measuring technique that uses separate pairs of current carrying and voltage sensing electrodes to make more accurate measurements than simpler and more common two-probe sensing. Separation of current and voltage electrodes eliminates the lead and contact resistance from the measurement. This is an advantage for the precise measurement of low resistance and capacitance values.

FEATURES

- Type of the measurement depends on the measuring instrument connected to the probes
- Capable of measuring VA, CV (high voltage, low frequencies, quasistatic) characteristics, pulse measurements and more
- Excellent EMI shielding for low noise measurements
- Wide range of temperature options from -60 to 300°C
- Precision 4-axis semiautomatic stage for accurate positioning temperature compensation
- Automated XYZ and theta correction for enhanced position accuracy

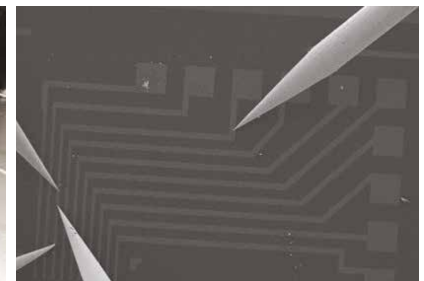
SPECIFICATIONS

Stage travel range	203×203×5 mm
Stage accuracy	≤ 2,5 µm
Stage resolution **Theta stage travel**	1 µm ± 5°
Probe force capability **Probe force deflection**	20 kg maximum ≤ 0,0015 µm

APPLICATIONS

Loading chamber for wafers up to 200 mm

Detail of the probes with tips inserted inside the chamber



Probes tips connected to the chip

MORE INFO

Guarantor: **Robert Doczy** (robert.doczy@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/semi-automated-4-probe-system-cascade-microtech-summit-12000/

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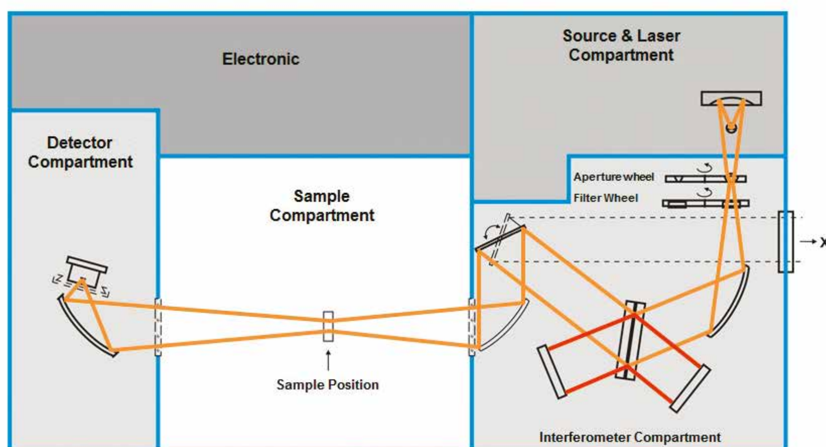
Infrared spectrometer

Vacuum FTIR Vertex 80v with microscope Hyperion 3000 KIT

DESCRIPTION

This instrument is a combination of a Fourier infrared spectrometer with an infrared microscope. With the spectrometer, one can perform transmission and reflection experiments on samples with a dimension of 1 cm^2 or larger. The microscope is used for measurements of either small or spatially inhomogeneous samples. The stage of the microscope allows localization of the place of interest on the sample or to perform mapping

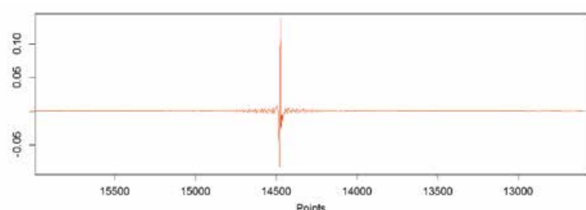
Beampath of the FT-IR-Spectrometer



SPECIFICATIONS

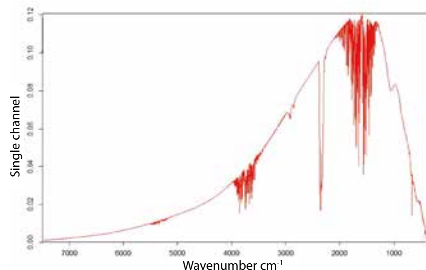
Vertex 80v	Range (50–14 000) cm^{-1}
Microscope Hyperion 3000 Detector MCT	Range (650–7500) cm^{-1}
Detector FPA	Range (900–4000) cm^{-1} 128×128 pixels measured simultaneously
Motorized XYZ stage for automated sample mapping	up to 10×10 cm
Option	Polarizers, VIS camera

Interferogram

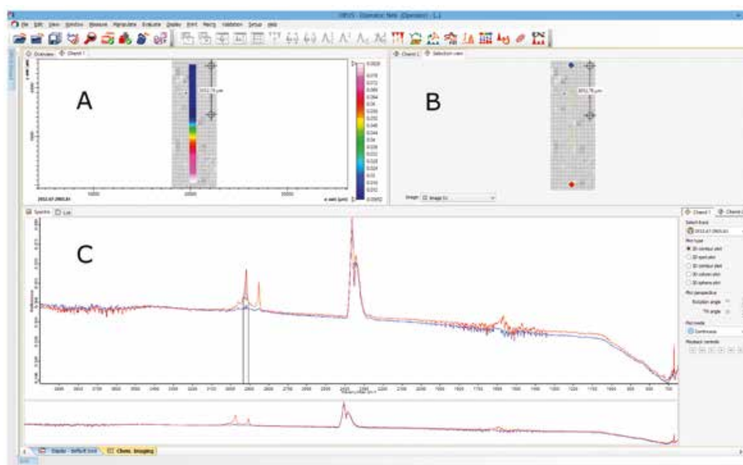


Fourier Transformation

Single channel spectrum



RESULTS



Infrared spectra Langmuiler Blodget film of stearic acid on Si
A - Peak Intensity at 2918 cm^{-1}
B - measurement point
C - first and last reflection spectrum

MORE INFO

Guarantor: **Alois Nebojsa** (Alois.Nebojsa@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/vacuum-ftir-vertex80v-microscope-hyperion-3000-kit-polarizors-vis-detector-bruker-vertex80v-hyperion-3000/

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FT-IR spectroscopic ellipsometer IR-VASE

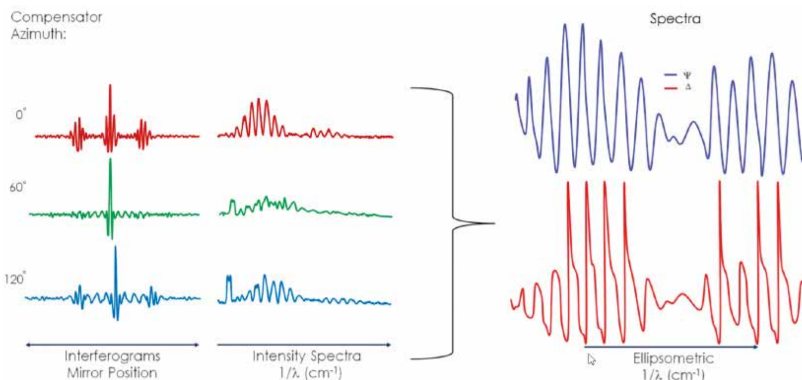
Woolam IR

DESCRIPTION

The IR-VASE extends the ellipsometric technique to longer wavelengths from 1.7 to 30 microns (333 to 5900 cm^{-1}) using the Fourier Transform technique. The infrared region provides very useful vibrational characteristics related to chemical composition or phonon modes (in the case of crystaline structures). The free-carrier peak near zero frequency reflects the doping level of the material or special modes of superconductors.

PRINCIPLE

The Fourier Transform (FT) technique is based on controlled movement of a mirror on one arm of the Michelson interferometer – a fast detector measures changes of light intensity (after interaction with the sample) related to the mirror position (interferogram). With FT this signal is converted into a spectrum – repeated measurements under a changing compensator orientation give access to all ellipsometric parameters (Psi, Delta, depolarization).

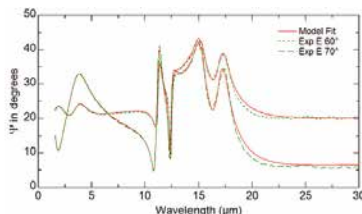


SPECIFICATIONS

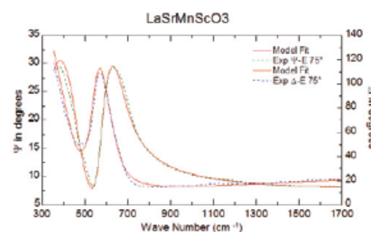
Spectral range	1.7 – 30 µm, 330 – 5900 1/cm
Angle of incidence	26° – 90°
Measured spot size	5 mm
Sample thickness	20 mm max.
Precision	0.1° in Psi, 0.8° in Delta
Techniques	Reflection and Transmission Ellipsometry Reflection alignement setup

Cryogenic measurements – Cold Edge Helium Closed-cycle Low-vibration Cryostat (ShoreLake) reaching temperatures down to 7 K; reducing vibrations is essential for elipsometric measurements

RESULTS



Measurements of several phonon vibrational modes for AlGaN layer on Si substrate with AlN interface



Bulk response of LSMSO modeled using a general oscillator model with a Drude term, 2 Lorentzian and 3 Gaussian peaks

MORE INFO

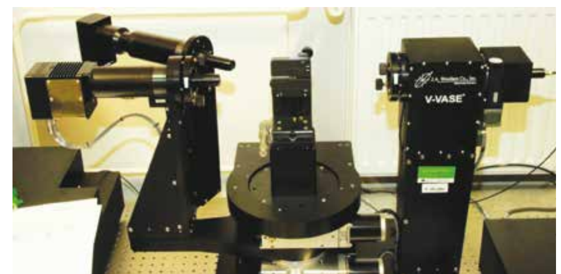
Guarantor: Alois Nebojsa (nebojsa@physics.muni.cz), **Filip Münz** (munz@physics.muni.cz)
Web: http://nano.ceitec.cz/mir-spectroscopic-ellipsometer-j-a-woollam-ir-vase/

NIR-UV spectroscopic ellipsometer V-VASE

Woolam VIS

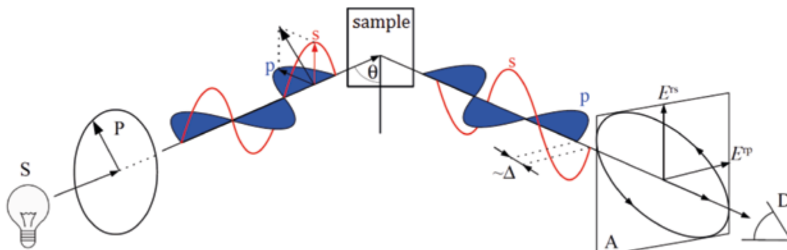
DESCRIPTION

The V-VASE is the most accurate and versatile ellipsometer for research on all types of materials: semiconductors, dielectrics, polymers, metals, multi-layers, and others. It combines high accuracy and precision with a wide spectral range from UV to NIR, precisely 190 to 2000 nm. The variable angle of incidence allows disentangling correlated parameters like optical constants and thicknesses.

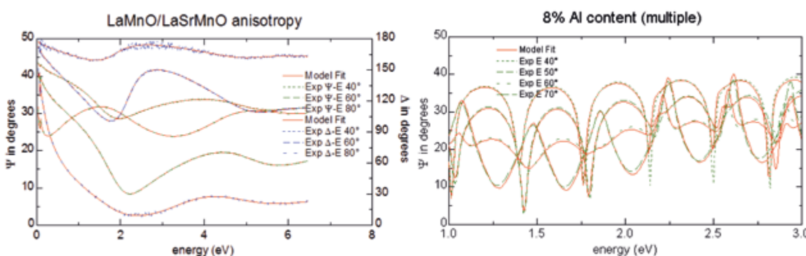


PRINCIPLE

Ellipsometry measures the change of polarization state typically upon reflection (transmission measurements are also possible) under high impact angles, allowing extraction of both real and imaginary parts of dielectric function (related to index of refraction and extinction coeficient) as well as thicknesses of multiple layers (when measured in several incidence angles). With the help of the rotating compensator (retarder plate) it can also estimate the level of depolarization.



RESULTS



Example of uniaxialy anisotropic bulk material (extraord. axis perpendicular to the surface) with both elips. angles fitted

Measurements of interferences in transparent region for AlGaN layer on Si substrate with AlN interface layer

SPECIFICATIONS

Spectral range	190–2000 nm, 0.62–6.42 eV
Angle of incidence	35° – 90°
Measured spot size	5 mm, with focusing probes 0.2 mm
Sample size	Ø 5–100 mm (vertically placed)
Precision	0.03° in Psi, 0.2° in Delta

Techniques	Reflection and Transmission Ellipsometry
	General ellipsometry (depolarizing samples)
	Scatterometry
	Cross-polarized R/T
	Total internal reflection ellipsometry (TIRE)
	measurement of surface plasmonic modes in interaction with deposited substance
	can detect very low concentrations of chemicals in solution or patogens using selective antibodies

MORE INFO

Guarantor: **Alois Nebojsa** (nebojsa@physics.muni.cz), **Filip Münz** (munz@physics.muni.cz)
Web: http://nano.ceitec.cz/nir-uv-spectroscopic-ellipsometer-j-a-woollam-v-vase/

Vacuum Ultraviolet Analytical Spectrometer

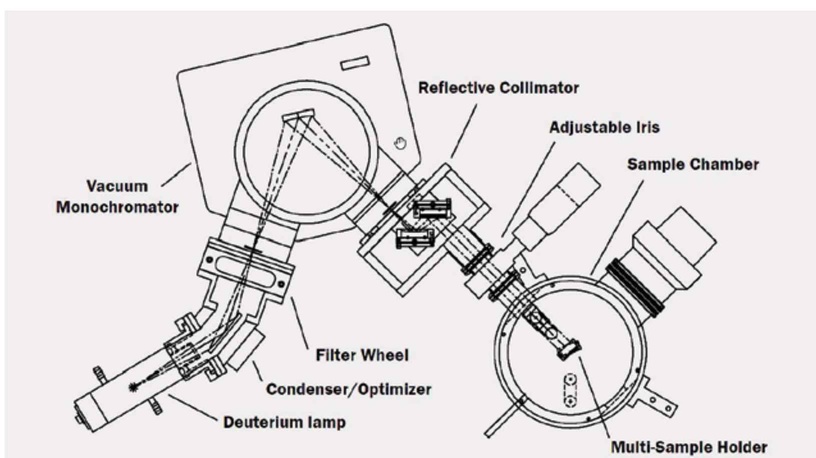
VUVAS 1000

DESCRIPTION

The VUVAS allows for precise direct measurements of reflection and transmission of samples in 120–350 nm range. It operates in moderate vacuum, independently pumped from a monochromator and sample chamber. Allows measurements under various angles of incidence, with three positions in the sample holder (up to 1inch samples). Optionally, users can use a cuvette holder and/or a polarizer with a motorized retarder for polarization or dichroism measurements.

PRINCIPLE

The spectrometer uses an air-cooled Deuterium lamp as a light source – its spectrum peaks at 160nm. An entrance and exit slit define the spectral resolution of the monochromator while the colimator and exit diaphragm create a parallel light beam of defined size that enters the sample chamber. After reflection (angle of incidence is set manually) or transmission through the sample the light enters a fibre ending at the photocathode of an photomultiplier located below the chamber. A high voltage controlling PM gain has to be chosen not to surpass the limiting current reached where the incident light reaches its peak intensity around 160 nm (7.7 eV). Low energy range (210–350 nm) is measured with a long pass filter.



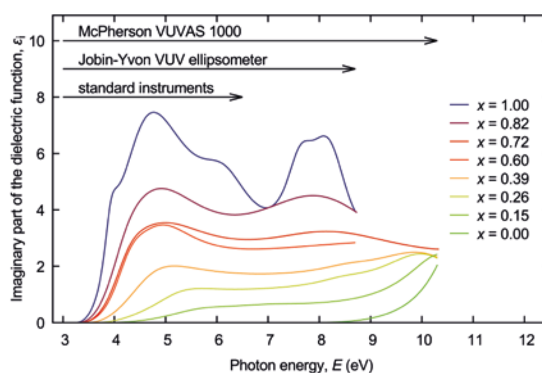
Schematic of Key Components in the VUVAS-1000 System

SPECIFICATIONS

Spectral range	115–400 nm, 3.1–10.5 eV
Angle of incidence	10° – 90° (transmission)
Measured spot size	8mm, can be reduced with iris
Sample size	Ø 5–25 mm (vertically placed), 2 positions + reference
Precision	<0.5% RMS (0.25 below 155 nm) 0.1 nm (for 10 µm slit), repeatability 0.05 nm
Stability	drift <1% /hour (no chopper/BMS included)

Techniques	Reflection and Transmission Spectroscopy
	Polarization measurements
	Circular Dichroism
	Light Scattering

RESULTS



Dielectric function reconstruction of different samples of $Ti_xSi_{1-x}O_2$ solid solutions (from low pres. plasma deposition) used for comparision with direct DFT (density functional theory) calculations. See: Pavel Ondračka et al. Physical Rev. B (2017) for more details.

MORE INFO

Guarantor: **Alois Nebojsa** (nebojsa@physics.muni.cz), **Filip Münz** (munz@physics.muni.cz)

Web: http://nano.ceitec.cz/vacuum-ultraviolet-spectrometer-mcpherson-vuvas-1000

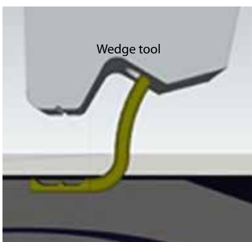
Wire bonding machine

Wire bonder TPT HB16

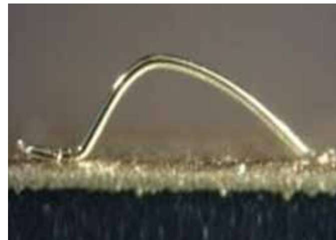
DESCRIPTION

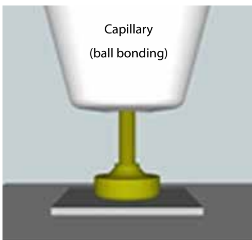
Wire bonding is a micro-welding technique for electrical interconnection of the sample substrate structures and semiconductor chip thin metallic layers. Contact between the sample surface and pure gold, alloyed aluminium or copper wire is provided by three main methods: ultrasonic, thermocompression and thermosonic bonding. The welding process is achieved by wire attached to the substrate with a bonding tool at the end of an ultrasonic transducer, which moves closer to a certain distance from the sample surface. To achieve enhanced welding capability, a sample is heated up to a certain temperature in the range of 20 °C to 250 °C.

WEDGE BONDING VS BALL BONDING



- *Bonding on very small bond pads*
- *Flat and long loops possible*
- *Gold and aluminium wire*
- *Aluminium wire without heating*



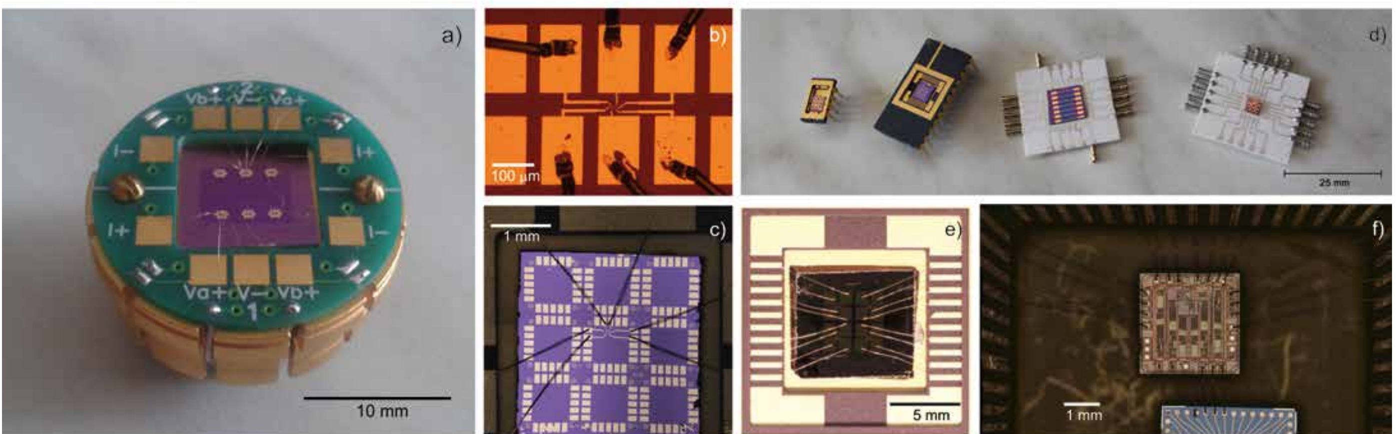


- *First bond connection more stable*
- *Bonding may be softer*
- *Wire moves vertically (for big height step)*
- *Gold and copper wire possible*

SPECIFICATIONS

Ultrasonic, thermocompression and thermosonic capability
Sample size up to 100×150 mm
Ultrasonic transducer (62 khz, up to 2 w power)
Sample (chip) holder heated up to 250 °c
Bonding tool heatable
Gold or alsi wires with diameters from 17 µm to 75 µm
Ribbon compatible up to 25 µm × 250 µm
Adjustable wire loop between first and second bond
Motorized holder movement in y and z axis
Software allows possibility to store up to 100 recipes

APPLICATIONS



Hall bar structures bonded into the puck holder (a) for low temperature measurement and DIP packages (b, c) for resistivity measurements. Samples with graphene (d), nanowires (e) and sensors (f) bonded through gold wires to the variety of packages and chip expanders .

MORE INFO

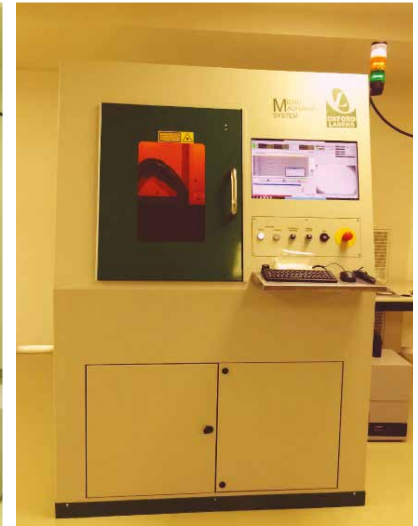
Guarantor: **Robert Doczy** (robert.doczy@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/wire-bonder-tpt-hb-16

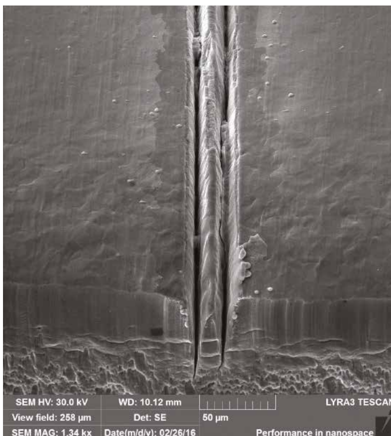
Oxford Lasers A series

Laser Dicer

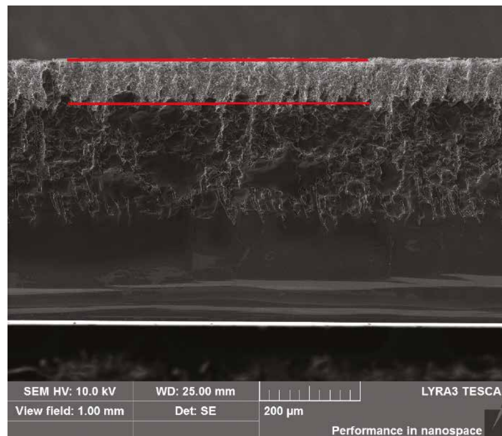
OVERVIEW

- 12 W Diode-pumped solid state Nd:YAG
- Universal aluminium chuck – suitable for sheets up to 1 mm thickness
- Cuts into various type of materials:
  - metals – ferrous, non-ferrous
  - silicon wafer
  - glass
  - plastic





cut in Molybdenum

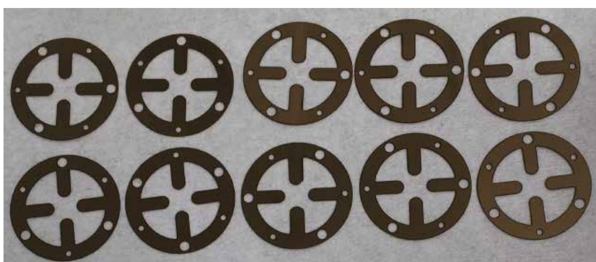


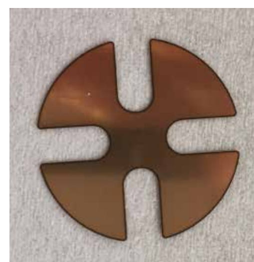
Silicon Cross Section

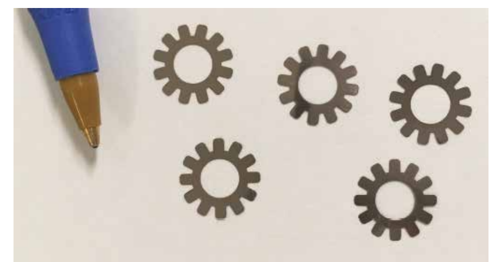
SPECIFICATIONS

Cutting range	200 mm × 200 mm
Laser	Diode-pumped Solid State
Wavelength	532 nm
Output	10 W @ 40 kHz
Pulse Freq.	1 – 200 kHz
Resolution	0.5 µm
Repeatability	1 µm
Vertical range	56 mm
Fume extractor	yes

EXAMPLES



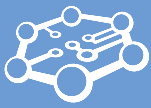




Various sheet metal parts.

MORE INFO

Guarantor: **Dalibor Šulc** (dalibor.sulc@ceitec.vutbr.cz)
Web: http://nano.ceitec.cz/laser-dicer-oxford-lasers-a-series/



Semiautomatic dicing saw for up to 6“ wafer

ESEC 8003 DICING SAW

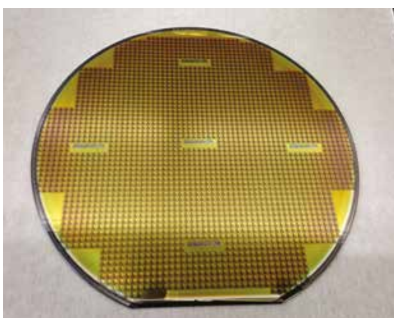
DESCRIPTION

A precision machine is used to cut semiconductor wafers into individual chips or dice. A programmable spindle speed and variable feed rate allow maximum dicing control and repeatability. Wafers are held to the chuck table by means of a vacuum and the chuck can accommodate circular and rectangular wafers. An extremely thin diamond blade is used to dice, cut, or groove semiconductor wafers, silicon, glass, ceramic, crystal, and many other types of material.

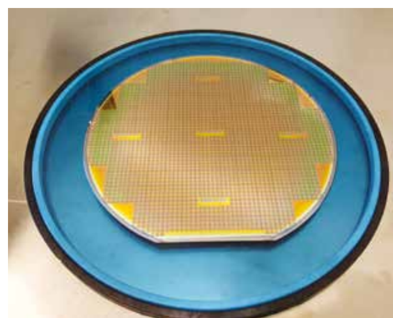
SPECIFICATIONS

Wafer diameter	25.4 mm – 152.4 mm (1“–6“)	**Y-axis (index)**	165 mm
		Index step	0.02–100 mm
Wafer thickness	0.01–5.0 mm	**Z-axis (verticle)**	10 mm
Min. cut-width	50 µm for Si wafer	**Rotation angle**	0.01–165 degrees
X-axis (feed)	240 mm	**Spindle speed**	15,000–50,000 rpm

EXAMPLES

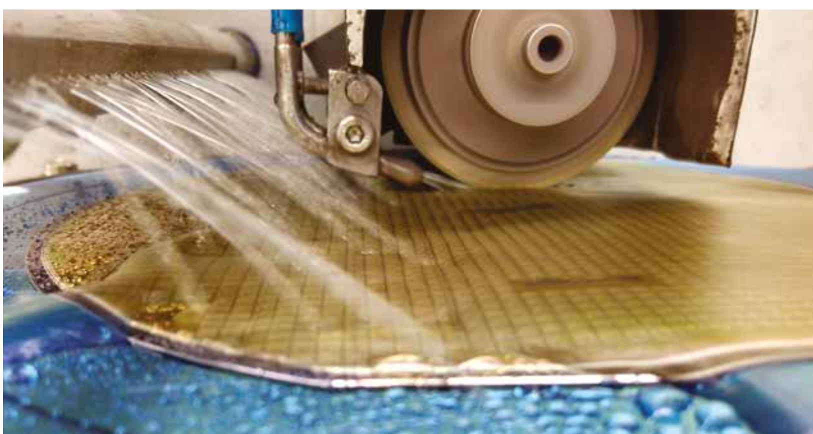


6“ wafer with structures before cutting



6“ wafer fixed on the tack adhesive

Screen view for sample before cutting



6“ wafer in cutting process

Screen view after cutting – the cut-width is about 50 µm

MORE INFO

Guarantor: Radovan Klusák (radovan.klusak@ceitec.vutbr.cz), **Jan Pekárek** (pekarek@feec.vutbr.cz)
Web: http://nano.ceitec.cz/semiautomatic-dicing-saw-esec-8003/