

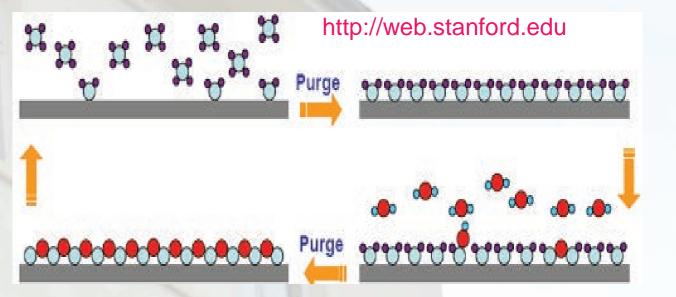




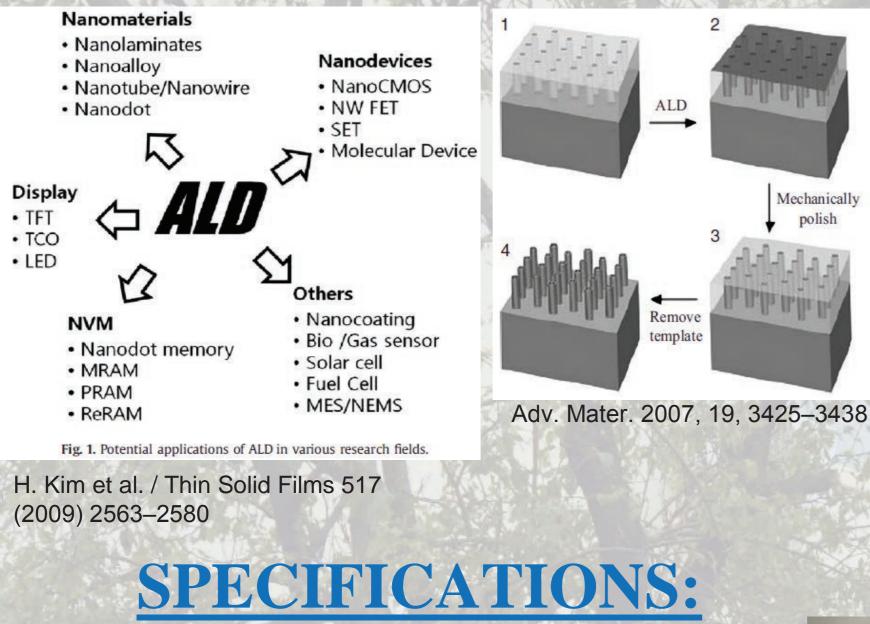
Plasma Enhanced Atomic Layer Deposition BENEQ TFS-200 D.Z. Dimitrov¹, V.B. Mehandzhiev², B.S. Blagoev³, J. Leclercq⁴, P.K. Sveshtarov⁵ ¹dzdimitrov@issp.bas.bg, ²vmehandzhiev@issp.bas.bg, ³blago@issp.bas.bg, ⁴jerome@issp.bas.bg, ⁵peter.sveshtarov@issp.bas.bg **Clean Room**

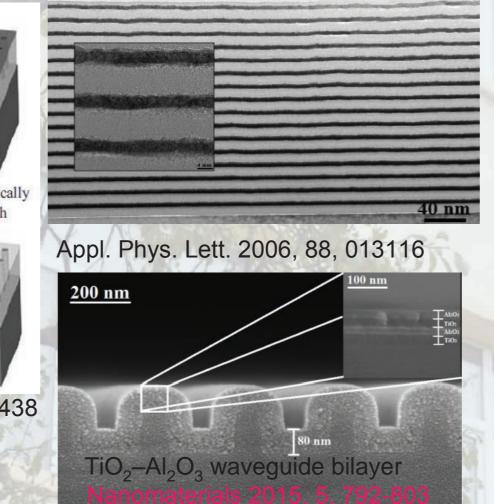


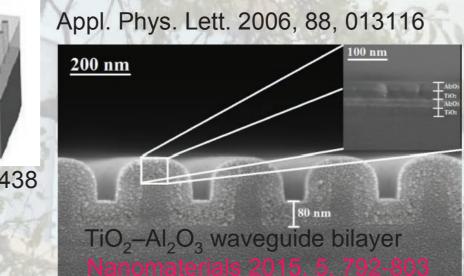
ALD is a thin film deposition technique that is based on the sequential use of a gas phase chemical process. In contrast to CVD, ALD is a 2 step process where in the 1st step one reactant has to form a self limiting monolayer on the surface. Once a monolayer of the 1st reactant is formed then the introduction of a 2nd reactant simply converts the 1st reactant to a layer of some desired solid material.



APPLICATIONS:











TRAINING:

99/2

DEZ_/TMA_pulse ratio

98/3

Films deposited on quartz substrate

97/4

- Thermal and Plasma-enhanced modes, temperature rating up to 350 °C
- 4 liquid precursors: H₂O; DEZ; TMA; BTBAS and 3 solid precursors: NiCp2; CoCp2; FeCp2
- 6 gas lines (including ozone)
- sample chamber: Ø200 × 5 mm

1.0 1.3 1.5 1.8 2.0 X[um]

0.0 0.3 0.5

0.8

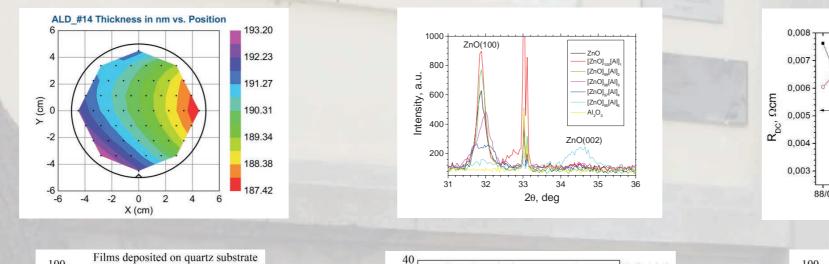
Films deposited on glass substrat

films: Al₂O₃; ZnO; ZnO:Al₂O₃; SiO₂; NiO; Ni; CoO; Co; FeO; Fe; and superlattices from the upper films.





RESULTS:



Deposition of ZnO:Al₂O₃ thin films by ALD

Composition	a [Å]	c [Å]	Crystallite Size [nm]
ZnO	3.259(3)	5.192(7)	41±3
[ZnO] ₁₀₀ [Al ₂ O ₃] ₁	3.249(2)	5.181(8)	54±6
[ZnO] ₉₉ [Al ₂ O ₃] ₂	3.246(1)	5.184(6)	46±3
[ZnO] ₉₈ [Al ₂ O ₃] ₃	3.255(3)	5.21(1)	23±2
[ZnO] ₉₇ [Al ₂ O ₃] ₄	3.269(5)	5.24(1)	15±1
[ZnO] ₉₅ [Al ₂ O ₃] ₆	3.289(7)	5.28(1)	15±1

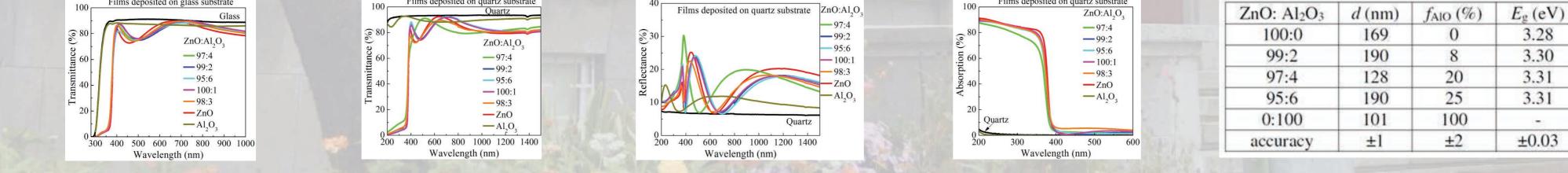
3.28

3.30

3.31

3.31

-



Joint studies with INERA partner

Partner Institutions: Institute of Physics at Polish Academy of Sciences (IP PAS); International Laboratory for High Magnetic Fields and Low Temperature Physics, Poland (ILHMFLT); Department of Engineering Sciences, Uppsala University, Sweden (DES UU); Fachbereich Technik, Bielefeld University of Applied Sciences, Germany (FT BUAS); The Liquid Crystal Laboratory at National Research Council (CNR) and the University of Calabria, Italy (UNICAL)



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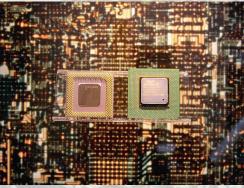




communication



information





REGPOT-2012-2013-1 NMP htpp://www.inera.org/



Femtosecond Laser System Dr. Ekaterina Iordanova, Dr. Georgi Yankov Department "LASER, ATOMIC, MOLECULAR AND PLASMA PHYSICS"

The femtosecond laser system consists of several units- amplifier, oscillator, pumping laser and optical parametric amplifier. The Ti:Sapphire regenerative amplifier is Spitfire Ace model with pulse width ~35 fs and pulse energy ~6 mJ. The Spitfire Ace performs optimally when seeded with Mai Tai® SP oscillator. The Spitfire Ace is pumped with Empower® 45 laser, which delivers over 45 W of average power in a compact package. The enhanced stability makes the Spitfire Ace ideal for pumping multiple optical parametric amplifier (OPA) systems.

The TOPAS Prime automated OPA is an instrument for wavelength extension of the Spitfire® Ace[™] Ti:Sapphire amplifier system. Wavelengths can be generated from the deep UV range through the infrared (240–2600 nm).



MODULES AND SPECIFICATIONS:

Ultrafast Amplifier - Spitfire Ace Pulse width <35 fs Ultrafast oscillator - Mai TaiAverage power>400 mW

Pulse energy	>6 mJ
Repetition Rate	1 kHz
Beam diameter	10 mm
Polarization	Linear, Horizontal

28 mJ

3 mm

527 nm

Energy per pulse

Beam diameter

Wavelength

Tuning range	780–820 nm
Peak power	> 95 kW
Beam diameter	1.5 mm
Polarization	Horizontal

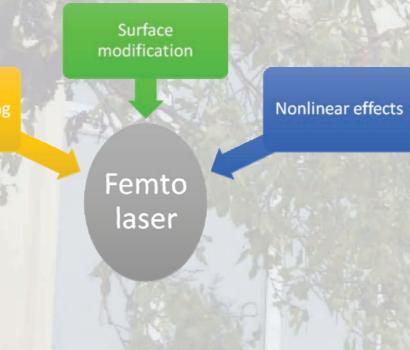
Wavelength Converter - TOPAS P

240 – 2600 nm
11 mm
Linear, Horizontal

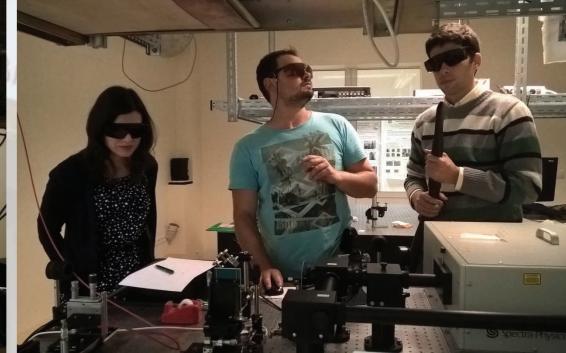
TRAINING:

APPLICATIONS:

- Scientific Research
- Micromachining
- Medical
- Surface modification
- Bio-imaging
- Non linear effects
- Biomedical devices
- Thin film ablation



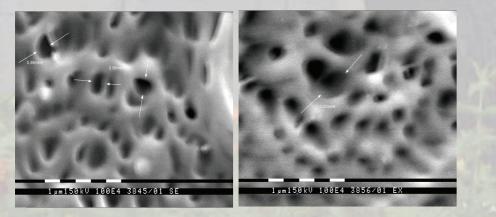




RESULTS:

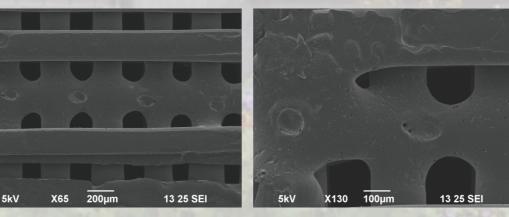
The femtosecond laser treatment of materials offers advantages over chemical and other physical methods. Precise **modification** of certain **surfaces**, that are difficult to be treated with conventional methods, is enable. In tissue engineering, the three – dimensional (3D) scaffolds fabricated from polymers are used to provide structural support and guidance of cells for creation of new tissue generation. The consecution of experiments demonstrated the possibility on creation of surface micro formations, applying different laser fluences, at 1 kHz repetition rate for fixed time of exposure 1 sec at 800 nm central wavelength. The main advantages of femtosecond laser **micromachining** are the microsized structure creation, no material property change, significant reduction or complete removal of heat-affected zone, clean process look. Some of the main applications can be seen in surface structuring, photonic devices, biomedical devices, thin film ablation, material milling, grid cutting, hole drilling.

Surface modification of gelatin thin film



SEM image of the surface topography of thin gelatin film, laser irradiation = 35 fs, λ = 800 nm Energy – 630 – 230 µJ

Surface modification of polycaprolactone scaffolds



pulse -35 fs , λ = 800 nm, energy = 100 μJ Line distance ~ 150 μ

Micromachining of different materials



pulse -35 fs , λ = 800 nm, cutting speed 10 mm/s

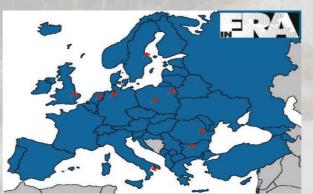
Joint studies with INERA partners:

Laser-plasma matter interaction:

National Institute for Lasers, Plasma and Radiations Physics, Romania; Eindhoven University of Technology, The Netherlands.



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Portable Potentiostat and Galvanostat SP-200 Dr. M. Iliev, Dr. Y. Marinov **DIVISION SOFT MATTER PHYSICS**

The modular potentiostat/ galvanostat SP-200 incorporating the latest technology has excellent specifications and can be applied as universal measurement device with main field of application in electrochemistry research. SP-200 offers floating mode, analog filtering, built-in calibration board and excellent electronic stability for better cell control. The electrochemical workstation has been equipped with an ultralow current probe, which extends the current range down to 1 pA; the upper current range is 500 mA. Electrochemical impedance spectroscopy measurements in the frequency range from 10 µHz to 3 MHz are an additional option of the purchased instrument. The device is suitable for studying electrical and dielectrical properties of multifunctional nanostructures. As well as by dielectric permittivity and dielectric loss measurements performance of electric devices can be evaluated.



MODULES:

- Work station
- Ultralow current probe
- **Portable computer**



APPLICATIONS:

Compact and powerful electrochemical workstation SP-200, **Bio-Logic** instruments is designed for a variety of domains and applications:

Materials testing

SPECIFICATIONS:

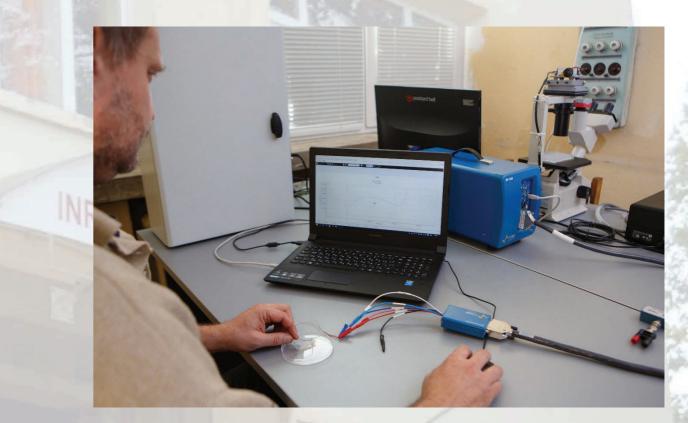
- Compliance voltage ±10 V
- EIS capability 10 µHz to 3 MHz
- Current ranges with low current option 1 pA to 500 mA (with gain)
- Current resolution 76 aA
- Input impedance 100 T Ω (//6 pF)

• Faradey cage



- Electrophysiology
- photovoltaics
- fuel cells

TRAINING:



RESULTS:

Results on impedance spectroscopy study of ZnO-Al₂O₃ thin films deposited on silicon substrates by Atomic Layer Deposition (ALD) system, purchased in the frame of the Project INERA, are presented.

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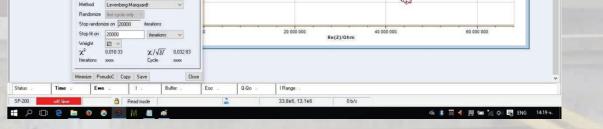


Impedance spectra of ALD Al₂O₃ layer.



Impedance spectra of ALD ZnO:AI layer.

Joint studies with INERA partner:



Impedance spectra of nanostructured liquid crystal gel

Materials studied: Metal oxides (Al₂O₃, ZnO) and nanostructured liquid crystals films. Partner Institutions: Dipartimento di Fisica, Università della Calabria, Via Pietro Bucci, Cubo 31C, 87036 Rende (CS), Italy



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MAXIMEM Membrane Filtration Unit Dr. D.Mitev, Dr. D.Peshev

WG4 – Nanomembrane and liquid crystal nanostructures: research and applications

MAXIMEM laboratory membrane filtration system (Prozesstechnik GmbH) with its compact and flexible construction gives possibilities for performing all pressure driven membrane processes. The system is made of stainless steel frame, frequency controlled pump, temperature controller for the jacketed feed vessel, flexible membrane modules and electronic display. The experimental conditions for operating pressure, pump throughput, temperature, permeate flux, etc. are precisely controlled and recorded for further analysis. The modern filtration set-up delivers a variety of possible application as extraction and concentration of herbal substances for pharmaceutical usage, purification and desalting of valuable products, bigger scale membrane filtration, etc. The capabilities for separation of thermally and chemically unstable substances make the equipment unique for the purpose.



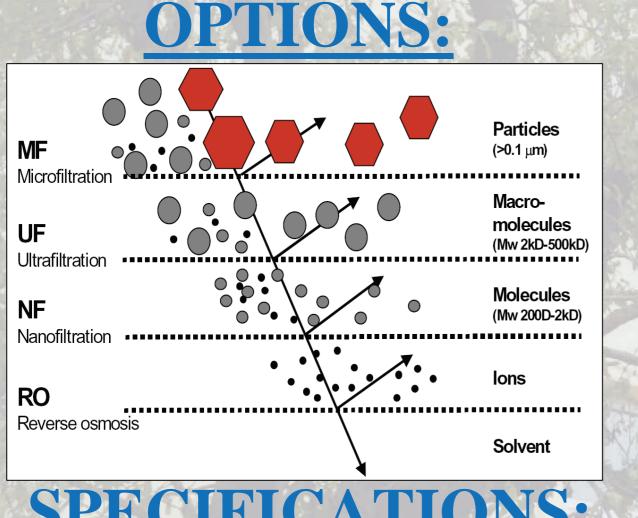
MODULES:

Control Unit for automatic diafiltration
and continuous operation. Main functions:
Automatic diafiltration at constant level
Convert the basic membrane unit from batch unit into a continuously operating

Other purposes: Simple metering pump unit to pump liquids at

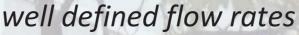
APPLICATIONS:

- High reverse osmosis, nanofiltration, ultrafiltration, microfiltration
- Concentration, diafiltration, loop operation, batch and continuous
- Membrane filtration with larger amounts of product



SPECIFICATIONS:

- Operating pressure: up to 60 bar
- Temperatures: within 0°- 60°C



 Approximate permeate flow meter (accuracy within +/-5% of the measured value) Concentration, purification and desalting of valuable products, waste stream treatment and recycling

TRAINING:



RESULTS:

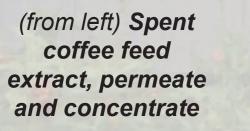
Spent coffee extracts recovery: using dead-end nanofiltration, with a feed of 600 mg L⁻¹ GAE*, we managed to concentrate to 2075 L⁻¹ GAE with a single process (fresh short espresso 4400 mg L⁻¹). Recovery 0.88 according to Folin-Ciocalteu analytic method for antioxidants / reducers. <u>PECVD-assisted membrane modification</u>: Low-temperature plasma processes rendered possible an application of several type of coatings: siloxane (dense and well differentiable; they exhibit dual-mode mechanism of the deposition combined with hetero-inclusions within the layers). On the contrary, films derived by aliphatic hydrocarbons are rather loose, but uniform polymer deposits. Although having some specifics, toluene-based films show close proximity with the pentane-derived ones. *GAE: Gallic acid equivalents



Pentane

Toluene





membrane modification PECVD setup Plain polymer

Contact angles for plain substrate and the plasma-deposited films type H (20 min), P (5 min) and T (20 min deposition)

HMDSO

Joint studies with INERA partner:

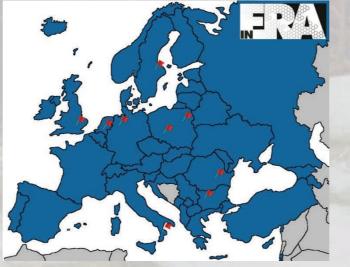
Plain polymer substrate (left), and plasma coated (Pentane 5 and 20 min; right). The thicknesses of the coatings are 12 and 85 nm (quartz resonator). Magnification 100.00k

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Materials studied:Membrane processes for spent coffee treatment. Membranes modification through PECVD approaches.Partner Institutions:Department of Chemical Engineering and Chemical Technology, Imperial College, UK (Prof Andrew Livingston & Dr Ludmila Peeva)



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Automated Microfluidic Platform CellASIC® ONIX, Millipore Merck Dr. Julia

Dr. Yordan Marinov; <u>e-mail</u>: ymarinov@issp.bas.bg

Dr. Julia Genova; e-mail: julia.genova@issp.bas.bg

LABORATORY BIOMOLECULAR LAYERS

An automated microfluidic platform CellASIC® ONIX, Millipore Merck for dynamic cell culture analysis and environmental has been delivered and installed. The CellASICTM ONIX Microfluidic Platform with its novel beneficial features gives multiple opportunities for long-term live cell analysis experiments by combining the highest precision controls, maximum functionality, and simplified user operation. Cutting edge microfluidics technology provides an improved cell culture microenvironment control, exceptional quality for high magnification microscopy, and superior media switching capabilities. The newly delivered system allows a variety of possible applications as 3D cell culture, migration in reponse to chemogradient, cell response to changing media conditions, neural stem cell analysis, host-pathogen interactions, bacteria and yeast single cell response, hyperic conditions to mimic tumor environments.



MODULES:

Microfluidic platform
 Microincubator controller

APPLICATIONS:

- Cell response over time
- Interrogating 3D cell culture
 Chemotaxis/migration
 Drug dose/response
 Neural stem cell analysis
 Host-Pathogen interactions
 Hypoxic conditions to
 mimic tumor environments
 Bacteria single cell analysis
 Yeast single cell analysis

Tri-gas mixer Microfluidic Plates

er		10-1-11			
		Function		Minimum Volume (µL)	Maximun Volume (
In	let 1	Inlet for driven perfu	0 /	10	350
In	let 2	Inlet for establishme	-	50	350
DUTLETS IN	let 3	Inlet for establishme	0	50	350
In In	let 4	Inlet for establishme	0	50	350
ln	let 5	Inlet for establishme	0	50	350
In	let 6	Cell inlet for cells into chamber	0	10	350
100	utlets 7 nd 8	Accept flow from chamber		50	900

s for pressure driven flow

Mammalian M04 Yeast Y04

Bacteria B04

SPECIFICATIONS

Microfluidic System

Outputs Number of outputs Pressure range Pressure accuracy Pressure stabilization time Optional inputs Pressure supply

Vacuum

Gas mixture Cell Culture Region Gas Environment Accuracy For gas flow at 3 mL/min For gas flow at 20 mL/min

8 0–0.7 bar (0–10 psi) 10.3 mbar (± 0.25 psi) < 5 seconds

Clean, dry air, nitrogen (N_2) , or argon regulated between 2.1 and 6.9 bar (30 and 100 psi). Steady vacuum regulated between 711 millibar (21 in. Hg) and perfect vacuum Flow rate of 3-100 mL/min

< 10% deviation from delivered gas < 2% deviation from delivered \sim



Microincubator controller

Temperature Control Temperature control range Control method Set method Achievable stability

Rise time (25 °C to 37 °C) Cooling time (37 °C to 25 °C) Calibration plate accuracy Optional gas flow Flow rate Premixed gas input Room temperature to 40 °C Bi-directional PID Up and down keys, increment 0.1 °C ± 0.2 °C

< 10 minutes < 15 minutes ± 0.1 °C Pawel Kroliczak, technical manager at Merck Millipore performed the installation of the microfluidic platform and demonstrated some of its possible applications. Four young specialists from ISSP-BAS, namely Dr. Janez Pavlic, Dr. Victoria Vitkova, Dr. Julia Genova and Denitza Mitkova have been trained to operate the system and ave received certificates.

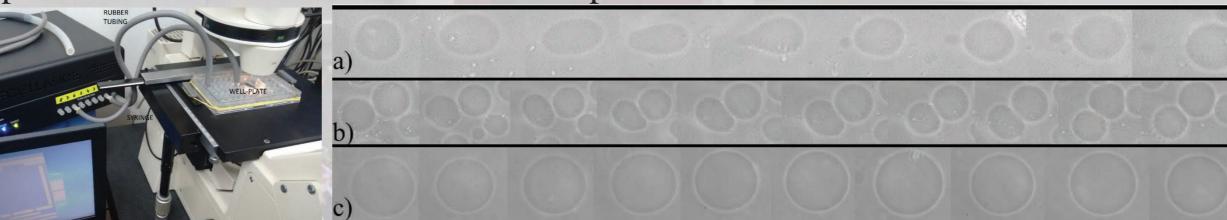




RESULTS: Study of the morphology of lipid vesicles in presence of Amphotericin B via modified microfluidic platform

3.0 mL/min typical, ± 0.5 mL/min Clean, dry, premixed gas containing air, CO₂, N₂, and oxygen (O₂) up to 25%, regulated to 3.1–3.8 bar (45–55 psi)

Cell Culture Region Gas Environment Accuracy For gas flow at 3 mL/min For gas flow at 20 < 10% deviation from delivered gas < 2%



Tri-gas mixer

General Gas supply requirements

Gas supply consumption

Ouputs Mixed gas Flow meter accuracy Flow meter stability Flow meter drift Compressed air, N₂, and CO₂ Clean and dry, > 99.5% pure Regulated to 2.1–6.9 bar (30–100 psi) Air, 0–100 mL/min N₂, 0–120 mL/min CO₂, 0–10 mL/min

0% or 1–21% 02 0% or 1–15% CO₂ $\pm 2\%$ of full scale at 21 °C $\pm 0.20\%$ CO₂ per 1 °C* $\pm 0.1\%$ CO₂ per day* Morphology of lipid vesicles in addition of a) amphotericin B in concentration 10^{-5} g/l in the outer vesicle environment; b) amphotericin B in concentration 10^{-3} g/l in the outer vesicle environment; c) control with addition of glucose solution.

J. Genova, M. Dencheva-Zarkova, J. I. Pavlič, Morphological study of lipid vesicles in presence of amphotericin B via modification of the microfluidic CellASIC platform and LED illumination microscopy, Journal of Physics: Conference Series 682 (2016) 012029.



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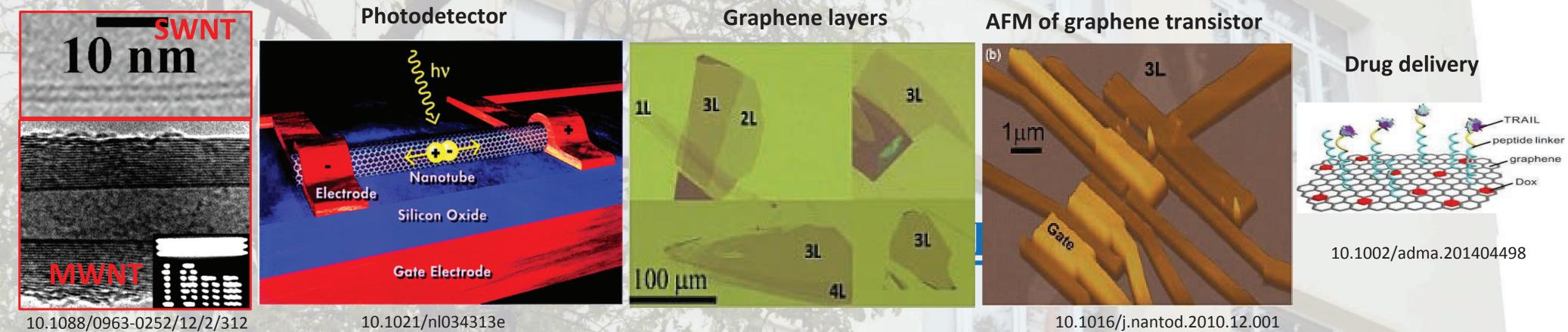


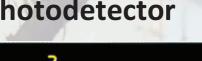
Plasma Enhanced Chemical Vapor Deposition reactor Oxford Instruments Plasmalab 100 Dr. P. Sveshtarov, Dr. J. Leclercq, Dr. V. Mehandzhiev, Dr. B. Blagoev and Dr. D. Dimitrov **CLEAN ROOM**



Plasma-enhanced chemical vapor deposition (PECVD) is a process used to deposit thin films from a gas state (vapor) to a solid state on a substrate. The Oxford Instruments PlasmaLab 100 PECVD reactor can work like PECVD or LPCVD (Low pressure chemical vapor deposition). It is used to grow graphene and carbon nanotubes at Nanocenter ISSP-BAS

APPLICATIONS:





SPECIFICATIONS:

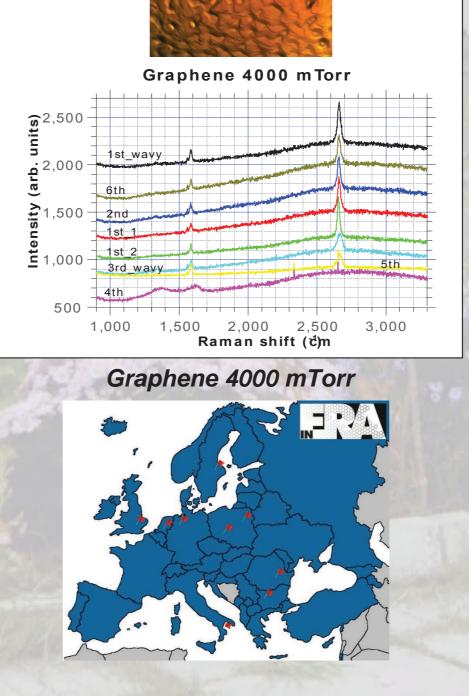
•Temperature range from room temperature up to 1200 °C .Low (200kHz) and High (13.56MHz) frequency plasma Automatic load power control (0-300W) .Continuous or pulsed plasma mode .Vacuum loadlock with dry pump •7 gas lines : Ar and H₂ as carrier gas, N₂, C₂H₂, NH₃, CH₄, O₂ and H₂ as reactive gases . Substrate size up to 200 mm

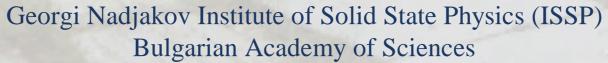
RESULTS:

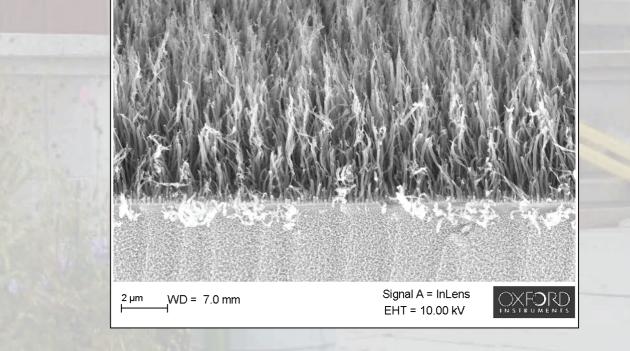
TRAININ

Graphene was grown on copper substrates at different low pressures by thermal CVD and characterized by Raman spectroscopy and SEM. The graphene samples show good quality and are consistently mono-layered, confirmed by the Raman spectra. Carbon nanotubes were grown by PECVD using RF plasma precursor activation and deposition steps. Curled tangled nanotubes, straight well aligned nanotubes and 3 µm long nanotubes were obtained. Both processes are being refined and optimized to ensure reproducibility and quality.









Long Carbon Nanotubes









Spectroscopic Ellipsometer Woollam M2000D

Dr. A. Szekeres, Dr. P. Terziyska and Dr. T. Hristova-Vasileva

DIVISION NANOPHYSICS

M2000D ellipsometer, working with rotating compensator, is designed for optical characterization of thin dielectric, semiconductor, organic layers and multilayer structures and liquid samples. The spectral range of 195-1000 nm is especially suitable for determining the optical constants and thickness of semiconductor and oxide films. Measurement at short wavelengths increases the sensitivity of the device to ultrathin films with thicknesses less than 0.1 nm. Besides film thickness and optical constants, other properties as composition, crystallinity, anisotropy, surface and interface roughness, etc. can be determined, as well as lateral inhomogeneity can be registered by automatic sample mapping.



MODULES:

 Instex Heat Stage (Temperature range for ellipsometric measurements: approximately - 80° to 600°C; Angle of incidence range: 45° - 90°

• 500 µl Horizontal Liquid Cell



APPLICATIONS:

High accuracy and precision in a wide spectral range and fast data recording make M2000D a powerful tool for in-situ monitoring and process control, largearea uniformity mapping and thin film characterization.

SPECIFICATIONS:

- Spectral Range: 193 to 1000 nm
- Angle of Incidence Range: 45° 90°

OPTIONS:

- Horizontal Auto translation stage (100 mm X-Y)
- Focusing Optics (300 µm beam size)



 Computer controlled sample positioning system and automated z-height adjustment



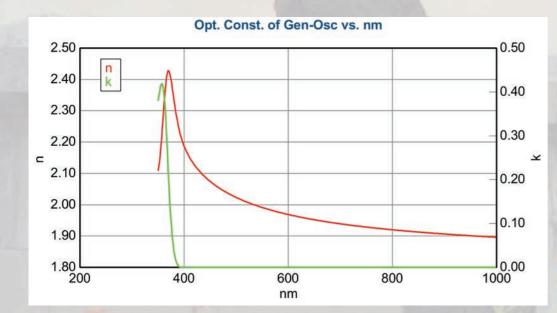
Automated 300x300 mm XY mapping

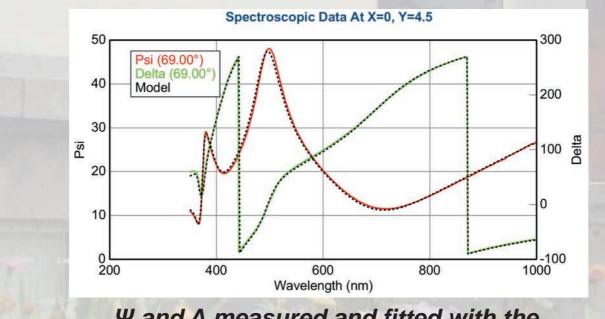


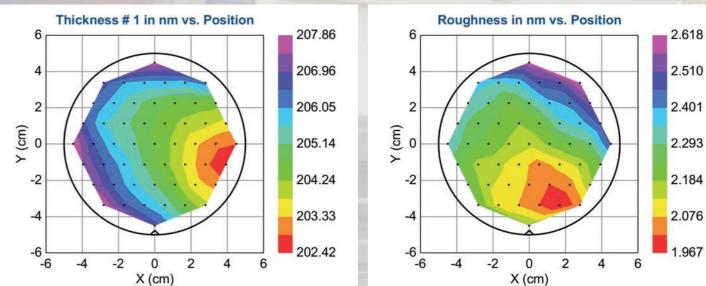


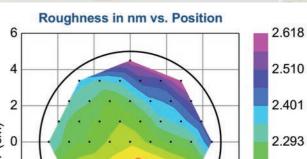
RESULTS:

Results on spectroscopic ellipsometry study of ZnO-Al₂O₃ thin films deposited on silicon substrates by Atomic Layer Deposition (ALD) system, purchased in the frame of the Project INERA, are presented. The wavelength dispersion curves of the refractive index n and the extinction coefficient k of the ZnO layers are determined from the General Oscillator model of the experimental ψ and Δ data. The 2D maps of the ZnO films deposited on 4 inch Si wafer show good thickness homogeneity and low surface roughness.









Refractive index and extinction coefficient dispersion of ALD ZnO:Al layer.

 Ψ and Δ measured and fitted with the GenOsc model.

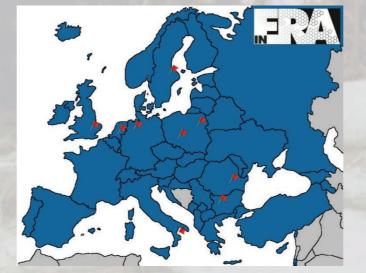
Thickness variation map and roughness variation map of the ZnO:Al layer deposited on a 4 inch Si wafer.

Joint studies with INERA partner:

Materials studied: Metal oxides (Al₂O₃, ZnO, TiO₂, CoO, FeO, NiO, ZnMgO, WO₃-MoO₃) and nitrides (AlN, GaN) and DLC films. Partner Institutions: Institute of Physics at Polish Academy of Sciences (IP PAS); National Institute for Lasers, Plasma and Radiation Physics, Romania (NILPRP); Department of Engineering Sciences, Uppsala University, Sweden (DES UU)



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Georgi Nadjakov Institute of Solid State Physics (ISSP) **Bulgarian** Academy of Sciences





EUROPEAN REGIONAL DEVELOPMENT FUND

EUROPEAN UNION

INVESTING IN YOUR FUTURE

Bulgarian Academy of Sciences Georgi Nadjakov Institute of Solid State Physics

72, Tzarigradsko Chaussee Blvd., 1784 Sofia, BULGARIA



LABORATORY OF OPTICS AND SPECTROSCOPY

Assoc. prof. T. Tenev, Assoc. prof. R. Peyeva, Assoc. prof. K. Antonova, eng., Ph.D. I. Miloushev, Assist. prof., Ph.D. Ts. Ivanov, Assist. Prof., Ph.D. L. Yankova

EQUIPMENT: High vacuum technology system for thin optical films deposition Symphony 9 (Tecport Optics)



APPLICATIONS:







Mirrors

Interference filters



AR coatings



Beamsplitters

SPECIFICATIONS:

pass filters

- Chamber size 960mm diameter x 1100mm height
- Ultimate pressure 10⁻⁷ Torr
- Operation pressure 10⁻⁶ Torr in 20 min

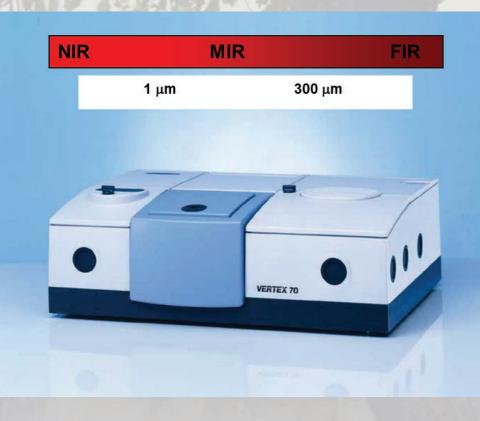
• E-beam and Resistance evaporation

FEATURES:

- Ion-assistant deposition
- Possibility for co-deposition
- Dual-rotation planetary and single-rotation dome systems
- Thin film deposition monitoring:
- Quartz crystal monitor
- Direct and indirect (Intellementics) optical thickness control
- Powerful software packages:
- Symphony OPUS Operating Software
- Film maker and Film Director for optical monitoring

SPECTROSCOPY INSTRUMENTS:

FT-IR spectrophotometer BRUKER - Vertex 70









Spec. eflection

under 13°- 83°



range



Attenuated total reflection

pol. degree 2-36 µM 95.8-99.7% **20–1000 μм 93%**

FEATURIES:

- Transmission and Reflection spectrophotometry
- of solids and liquids
- Time-resolved spectrophotometry
- Electro spectrophotometry

SPECIFICATIONS:

- Spectral range: 10 000–30 см⁻¹(1–300 µm)
- Spectral resolution: 0.16 СМ⁻¹
- Spectral accuracy: 0.005 см⁻¹
- Photometric accuracy: 0.1 %

Polarizers 0° – 360°

Signal-to-noise ratio: 10 000:1 pp

UV-VIS-NIR spectrophotometer Perkin Elmer - Lambda 1050 MODULES:





URA-reflection under 8°- 65° **InGaAs sphere**

150mm integration Polarizer drive

FEATURIES:

 Wave-lenght spectrophotometry Time-drive spectrophotometry Polarization spectrophotometry **SPECIFICATIONS:**

 Spectral range: 175 – 3300 nm UV/Vis spectral resolution: ≤ 0.05 nm NIR spectral resolution: ≤ 0.20 nm Photometric range: 8 A



http://www.issp.bas.bg

Проект BG161PO003-1.2.04-0027-C0001 "Обновявяне на технологично оборудване и апаратура за иновативни научноприложни разработки на многослойни оптични стректури" с финансовата подкрепа на Оперативна програма "Развитие на конкурентоспособността на българската икономика" 2007-2013, съфинансирана от Европейския съюз чрез Европейския фонд за регионално развитие. Цялата отговорност за съдържанието се носи от Института по физика на твърдото тяло "Акад. Георги Наджаков" и при никакви обстоятелства не може да се приема, че отразява официалното становище на Европейския съюз и Договарящия орган.

