





# Materials and Processes for Micro & Nano Technologies Labs





## MATERIALS & MICROSYSTEMS LABORATORY



## Physics and Electronics Depts of Politecnico di Torino

http://www.polito.it/micronanotech



## <u>Mission</u>

- fundamental research on materials and processes for MEMS and NEMS
- design and realization of MEMS and NEMS
- technological transfer
- education



## <u>Staff</u>

- 5 Professors
- 5 Permanent Researchers
- 8 TD Researcher
- 9 Fellowships / Post Doc
- 10 PhD students
- 3 Technicians
- 1 Administrative



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#### Collaborations with Universities & Research Institutes

UC Berkeley – USA UCLA- USA University of Wisconsin - USA Linkoping University - Sweden Minatec - France Université Paris - Diderot - France **EPFL** – Switzerland loffe Physico-Technical Institute St. Petersburg – Russia University of Mining and Metallurgy of Krakow - Poland Instituto Superior Técnico - Portugal Instituto de Engenharia de Sistemas e Computadores -Portugal Istituto di Chimica dei plasmi C.N.R. in Bari – Italy CEFSA C.N.R. Institute in Trento - Italy C.N.R. Institute in Bologna - Italy C.N.R. Institute in Parma - Italy **INRIM** in Torino – Italy Environment Park, Torino, Italy Bruno Kessler Foundation, Trento, Italy Istituto per la Ricerca Contro il Cancro (IRCC) of Candiolo (Turin) - Italy **Telethon Foundation** Universities of Napoli, Catania, Bologna, Brescia, Trento, Verona – Italy University Cattolica of Sacro Cuore, Piacenza - Italy

#### Collaborations with industries

Vishay Semiconductor Italiana Olivetti i-jet S.p.A. **STMicroelectronics** LPE S.p.A. Varian, Vacuum Technologies Avago Technologies Fuiitsu Lab. Mitsui-Carbon Nano Technology **Brewer Science** Grinp S.r.l. Elettrorava S.p.A. Zaniboni AOM Cyanagen S.r.l. Biodiversity S.p.A. Euroclone S.p.A. Tecnobiomedica S.p.A. Papiro S.r.I.











## Metal growth

- [CRV **Thermal** evaporator ٠ system]
- E-beam evaporator [ULVAC EBX-14D system]

Thin film growth

#### •r.f. magnetron sputtering

- •TeO<sub>2</sub>, Silica:Ge, Silica:Sn, CeO<sub>2</sub> and YSZ.
- •3 RF magnetron sources, UHV chamber;
- •thermal control during deposition up to 1000 °C.







## Thin film growth



#### Low pressure CVD

- poly crystalline 3C-SiC, c-Si
- Pressure ~ 200-300 mT
- Temperature up to 2000 °C (heater) → 1600 °C (substrate)
- 4" maximum substrate diameter
- 3 process gas lines (SiH<sub>4</sub>, C<sub>3</sub>H<sub>8</sub>, H<sub>2</sub>, [NH<sub>3</sub>])





# Thin film growth

- •13.56 MHz  $\underline{Plasma \ Enhanced \ CVD}$  amorph. and mc Si based alloys (SiC\_X, SiN\_X, SiO\_X)
- •2.45 GHz Electron Cyclotron Resonance CVD poly-cryst. Si-based materials,  $a-SiN_{X'}$ ,  $a-SiO_X$
- Load-Lock chamber
- •several gas lines (SiH<sub>4</sub>, C<sub>3</sub>H<sub>8</sub>, NH<sub>3</sub>, H<sub>2</sub>, CO<sub>2</sub>, N<sub>2</sub>)







## Furnace system for oxidation and annealing

Thin film growth

#### Tempress System

- 5 zones temperature control, 1100 mm at constant temperature
- Digital Temperature Controller (DTC) for high accuracy temperature control.
- Process temperature up to 1200 °C







## Electroplating

- IKoClassic Standard Model from ECSI ElectroChemical Systems (Inc. 136 Chestnut St. Midland Park, NJ 07432).
- bench-top tool designed for fabricating MEMS, NEMS and High Density Interconnects
- range of feature size from nanometers to several millimeters
- It is able to electroplate any metal or alloy that can be electroplated in aqueous solutions including Copper, Nickel, Palladium, Gold, Platinum, Tin, Lead, Iron, Silver, Permalloy, Palladium-Nickel and Solder
- It is equipped to electroplate 4" wafer size and/or wafer sections with the appropriate set of wafer holder and anode. programmable operation.
- IKO with the FIBRotools Technology allows high control on the uniformity of the deposition by minimizing peeks of current density at the edges of the patterns and compressing and levelling the diffusion layer by the motion of the reciprocating anode.







Photolithography set-up  $\Rightarrow$  actual resolution ~ 2 µm...

- Wafer size: 4" max.
- Wafer/substrate thickness: 0 ÷2.5 mm.
- Exposure wavelength: 365 nm
- Exposure source 200 watt high pressure mercury arc lamp





...now upgraded with front-back double alignment



Substrate

- 1. Pattern Design (CAD)
- 2. Pattern Definition (PMMA exposure to e-beam)
- 3. Pattern Transfer (Etching [wet or dry])





#### Operation:

- contact mode
- non-contact mode
- lateral force microscopy
- tapping mode
- lithography

#### **Technical features:**

- X-Y high resolution flexure scanner
- Integrated capacitive feedback sensors.
- Resolution < 1.6 nm in High Voltage mode. < 0.16 nm in Low Voltage (LV)
- Position accuracy: 10 nm
- Z Scanner
  - Range 10  $\mu m$  (HV)/1  $\mu m$  (LV) .
  - Resolution < 0.16 nm in HV mode.

#### Facilities of Nanolithography Tool:

- Local anodic oxidation on metal films and semiconductor substrate

- Nano-scratching and Nano-indentation on polymeric films.
- -Current driven lithography on PMMA layers.
- -Local anodic oxidation on a n-type Si wafer:

## AFM Lithography

#### AFM imaging:

Cobalt dots realized by optical lithography, thermal evaporation of Cobalt and a lift-off step (1µm diameter, 2µm step)

diameter, 2µm step)



## Local anodic oxidation on metal films and semiconductor substrate.

Line length 950-1000 nm, line width 90-130 nm; dimensions depending on applied voltage. Local oxide height as a function of the applied voltage.







## **STS 320PC Reactive Ion Etching**

The STS 320PC Reactive Ion Etching is a turbo-pumped system, with a PC-based process controller and an integrated opticalemission end-point detector. It is surmounted by a glove box for a secure insertion and extraction of samples from the process chamber. It has 5 process gases ( $N_2$ ,  $H_2$ ,  $O_2$ ,  $SF_6$  and  $CHF_3$  currently) regulated by MFCs.

#### Plasma-etching Plasmafab 508

- Radio frequency (13.56 MHz) capacitive discharge
- 3 process gases regulated by MFCs (SF<sub>6</sub>,  $CF_4$ ,  $O_2$ )
- Max power 500W







## **Powder Blasting**

Powder blasting, or abrasive jet machining (AJM), is a technique in which a particle jet is directed towards a target for mechanical material removal. It is a fast, cheap and accurate directional etch technique for brittle materials such as glass, silicon and ceramics. The particle jet can be optimized for etching, while an eventual mask (according to the geometries to be patterned) defines the small and complex structures. It is a fast process; the time to etch through a 1.5 mm thick quartz wafer with one nozzle is approximately 60 secs in our set-up based on a Model HME II Unit from S.S. White Technologies, Inc. (151 Old New Brunswick Road, Piscataway, NJ08854). The particles are accelerated towards the target with a high-pressure. The airflow is mixed with the particles by a vibrating feeder. The mixture is directed through a circular tungsten nozzle (with a diameter variable from 0.35 to 0.8 mm) at the end of the tube. The particles hit the target with a speed, depending on the air pressure, in a separate box. Particles are made of alumina with a grain size variable from 23 to 95 mm





## Hot Embossing



### HOT EMBOSSING Jenoptik Jena Mikrotechnik - HEX01

Hot embossing of polymers is the cutting edge technology for the manufacturing of smart micro-components. Our equipment is the HEX01 hot embossing solution from JENOPTIK Mikrotechnik designed for applications in the emerging fields of micro-optics, microfluidics, smart materials and electronics subcomponents. It ensures highly precise moulding of any structures in polymers, especially microstructures with aggressive aspect ratio (up to 150:1). This is the easiest way to replicate a nanostructured and expensive mould into a more useful and cheaper polymeric material.

Nano-embossing can rapidly pattern large areas with feature sizes that previously required far slower and more costly electron-beam or ion-beam patterning. These technologies now may pattern the nano-scale features on the master die. The master die then replicates the features onto whole wafers, if only a single level of patterning is needed, or at chip size when several layers with an accurate overlay are required.







#### **Structural Characterizations**

X Ray Diffractometer (BB grazing angle and μbeam) Micro-Raman Spectrometer μFT-IR Spectrometer Micro-Photolum. Spectrometer (vis and UV excit.) Optical, AFM and STM microscopies

#### **Profilometer**

SNOM FESEM + EDX analysis Contact angle

#### **Optical Characterizations**

UV-Vis Spectrometer Photothermal deflection Spectrometer (PDS)

#### **Electrical Caracterizations**

Set-up for conductivity measurement vs temperature Set-up for I-V and C-V measurements Set-up for photoelectric characterizations



The profiler is a computerized, highly sensitive surface profiler that measures roughness, waviness, step height, and other surface characteristics in a variety of applications over a full 205 mm scan.

The built-in PC computing power offers precise, automatic measurement capability with the convenience and ease of use of Microsoft windows-based software control and data analysis.

•Vertical range = 160  $\mu$ m and 1 Å vertical data resolution.

•Measurement 205 mm (8 in.) substrates.







#### **Structural Characterizations**

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#### **Optical**, AFM and STM microscopies •

Profilometer SNOM FESEM + EDX analysis Contact angle

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- 5x, 10x, 20x and 100x optics
- up to 1000x real magnification (with CCD contribution)
- PC interfaced
- X-Y motorized stage for automated analysis (10 µm resolution)
- Z manual stage (0.1 µm resolution)





**Emission Scanning Electron** 

**Microscope (FESEM)** 

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- **FESEM + EDX analysis**

Contact angle

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SUPRA 40

- •Nominal resolution: 1.5 nm at 10 KV and WD= 2mm
- •Acceleration voltage: 0.1 30 KV
- •Probe current: 4 pA-10 nA
- •Magnification: 12 900,000 X

•Working distance: it ranges from 1 to 50 mm, depending on the operating conditions. The sample holder can host nine stubs. Each stub can contain samples of about  $1 \text{ cm}^2$ .





#### **Structural Characterizations**

X Ray Diffractometer (BB grazing angle and µbeam) Micro-Raman Spectrometer µFT-IR Spectrometer Micro-Photolum. Spectrometer (vis and UV excit.)

#### Optical, **AFM and STM microscopies**

Profilometer SNOM FESEM + EDX analysis Contact angle

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## DME DS -200 system

- ✓ Scanning area 200 µm x 200 µm
- ✓ vertical resolution <5 Å
- ✓ lateral resolution 3 Å
- $\checkmark$  contact and non contact mode and STM

#### **INFORMATION ON**

- $\checkmark$  surface morphology
- $\checkmark$  high resolution defects profile





#### **Structural Characterizations**

X Ray Diffractometer (BB grazing angle and µbeam)

#### **Micro-Raman Spectrometer**

μFT-IR Spectrometer Micro-Photolum. Spectrometer (vis and UV excit.) Optical, AFM and STM microscopies Profilometer SNOM FESEM + EDX analysis Contact angle

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The MicroRaman system is a Renishaw apparatus equipped with an Argon Laser with excitation at 514.5 nm. The most important features are:

<b>A</b>	
•Wavelengths range	400-1000 nm
<ul> <li>Spectral resolution</li> </ul>	1 cm^-1
<ul> <li>Spatial resolution</li> </ul>	1 μm (objective 100x)
•Depth	2 μm (objective 100x)
•Dimensions	650 mm x 400 mm x 650 mm
•Detector	CCD camera
<ul> <li>Available power</li> </ul>	with line 514.5 nm up to 200mW

Recently the system was upgraded with the acquisition of an Hot Cell (up to 1500°C) with which is possible to measure in-situ annealing process. This powerful tecnique is used to study quality of materials growth with our systems with the great advantage to delete the contribute of the substrate where the film is growth.

#### **INFORMATION ON**

✓ material structure

 $\checkmark$  dopant concentration





#### **Structural Characterizations**

X Ray Diffractometer (BB grazing angle and μbeam) Micro-Raman Spectrometer μFT-IR Spectrometer **Micro-Photolum. Spectrometer (vis and UV excit.**)

Optical, AFM and STM microscopies Profilometer SNOM FESEM + EDX analysis Contact angle

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#### **INFORMATION ON**

- $\checkmark$  material impurities
- $\checkmark$  dopant identification
- ✓ defects
- $\checkmark$  recombination centers on surface and in bulk







# Structural Characterizations X Ray Diffractometer (BB grazing angle and μbeam) Micro-Raman Spectrometer μFT-IR Spectrometer Micro-Photolum. Spectrometer (vis and UV excit.) Optical, AFM and STM microscopies Profilometer SNOM



FESEM + EDX analysis Contact angle

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FESEM + EDX analysis

**Contact angle** 

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## **RESEARCH LINES**



- Micro e Nanotechnologies for:
  - biotech applications
  - photonic structures
  - microfluidics
  - vacuum technologies
  - sensor development
  - high temp/power electronics
  - environmental monitoring & energy production
- Micro & Nanoscale characterizations
- Surfaces and biointerfaces technology
- Sano-bio-photonics
- Development of Nano-lithographies
- Nano-imaging and nanoparticles applications
- Ab-initio and FEM simulations
- Carbon NanoTubes technology
- Spintronics
- Laser marking & machining



















## LAboratorio di Tecnologie Elettrobiochimiche Miniaturizzate per l'Analisi e la Ricerca (LATEMAR)





## WHAT IS LATEMAR ?



LAboratorio di Tecnologie Elettrobiochimiche Miniaturizzate per l'Analisi e la Ricerca (LATEMAR)

Centre of Excellence funded by MIUR (through FIRB 2003-2004 grants) for public and private research structures operating in strategic fields for the Nation

## DIFFUSED LABORATORY

merges and coordinates Centres of Excellence in the basic research related to the development of micro and nanotechnologies and biotech together with R&D centres of extremely qualified Companies

<u>Coordination of the Laboratory</u> → Politecnico di Torino















# DEVELOPMENT OF POLYMERIC LAB-ON-A-CHIF FOR THE DNA ANALYSIS

- SU-8 LI THOGRAPHY
- COPPER ELECTROPLATING (both on Cu seed-layer and on Cu or AI bulk plates)
- HOT EMBOSSING on COC, PMMA, PC, PEHD, PS or PDMS casting in situ
- BONDING (thermoplastic, plasma activation, stick & stamp)
- DICING
- INTERCONNECTIONS fabrication & test



## DEVELOPMENT OF CANTILEVERS ARRAYS FOR GENOMIC AND PROTEOMIC DETECTION LATEMAR





## DEVELOPMENT OF CANTILEVERS ARRAYS FOR GENOMIC AND PROTEOMIC DETECTION LATEMAR

✓ Functionalization of cantilevers through silanization of silicon oxide covered surfaces or gold / thiols interfaces



## DEVELOPMENT OF CANTILEVERS ARRAYS FOR GENOMIC AND PROTEOMIC DETECTION LATEMAR




#### APPLIED MICROFLUIDICS → CANTARRAY based LAB-ON-CHIPs LATEMAR









HIGH SENSITIVITY AND RESOLUTION MICROSCOPIES AND SPECTROSCOPIES FOR BIO DETECTION **ATEMAR** 

- **Planar multilayers for** microcavities and 1D photonic crystals
- **Nanostructures for Surface Enhanced Raman Scattering Spectroscopy (SERS)**
- **Surface Plasmon and Polaritons**

**Near-field optical techniques for** Raman spectroscopy



0.5

λ=1530 nm

50

θ [deg.]

55

45

40

s-pol.

p-pol.

65

60





#### SURFACE WAVES IN 1D PHOTONIC CRYSTALS FOR SENSING APPLICATIONS





Multilayer exposed to organic vapors flow Diffusion into the porous matrix Real-time monitoring at  $\lambda_{RES}$ ,  $\theta_{RES}$ Silicon Nitride or porous Silicon material







#### Micro-Raman/SERS spectroscopy of Horseradish Peroxidase (HRP) molecules on p-Si/Ag

10  $\mu$ L of the analyte solution at several molar concentrations







### NEAR-FIELD MICROSCOPY FOR SUPER-RESOLVED RAMAN DETECTION







#### **2D-PHOTONIC CRYSTALS**





- Two-dimensional Silicon based structures
- Photonic waveguides
- Photonic structures in ridge waveguides





Collab. With Univ. of Catanzaro



#### 3D-PHOTONIC CRYSTALS: DIRECT & INVERTED OPALS





Polystyrene and silica nanospheres self-assembled in fcc structures (artificial direct opals)

Inverted opals (Si infiltration by













# PLASMA ASSISTED SURFACE MODIFICATIONS



# Surface modifications for dental prostheses and implants

#### **Partners**

• University of Turin, Dept. of Biomedical Sciences and Human Oncology

### **Objectives**

- improve the performance of dental prostheses
- barrier diffusion on metal implants (Ti, CrCo, ...)
- accelerate ostheointegration of implants

### **Methods**

- plasma-assisted thin film growth (PECVD, PVD)
- plasma-assisted surface modification
- surface characterisation (SEM, EDX, AFM)

### **Results**

- improvement of biocompatibility of composite materilas
- reduced bacterial adhesion on composite and ceramics



#### $a-SiO_x$ coated Ti implant



Bacterial adhesion on vergin (left) and a-SiO<sub>x</sub> coated (right) dental material



# PLASMA ASSISTED SURFACE MODIFICATIONS



### Diffusion barrier coatings for food packaging

#### **Partners**

• Politechnic of Turin, Dept. of Material Science and Chemical Eng.

#### **Objectives**

improve protection againts O<sub>2</sub>/CO<sub>2</sub>/H<sub>2</sub>O diffusion in food packaging

#### **Methods**

- plasma-assisted thin film growth (PECVD, PVD)
- plasma-assisted surface modification (etching)
- surface characterisation(SEM, AFM)

#### **Results**

- $a-SiO_x$  thin films for diffusion barrier
- characterisation of nanofilled PA6 films



SEM image of PA6 film with embedded nanostructures



# PLASMA ASSISTED SURFACE MODIFICATIONS



## Protective coatings for textile applications

#### **Partners**

• LameLedal s.p.a.

#### **Objectives**

• protection of metallic yarns during aggressive textile treatments

#### **Methods**

- plasma-assited coating deposition (CVD, PVD)
- chemical etching tests
- surface characterisations

### **Results**

protection layers and structures



## PLASMA DRIVEN CATALYSIS OF TOXIC EFFLUENTS



#### **Partners**

- Politechnic of Turin, Dept. of Material Science and Chemical Eng.
- MI s.a.s

### **Objectives**

Reduction of toxic emission from some industrial processes

### **Methods**

- plasma-driven catalysis (PDC)
- measurment of pollutant concentration (IR cell)

#### **Results**

• reduction of toluene by PDC



Example of PDC reactor



# ATOMISTIC SIMULATIONS OF SURFACES AND NANOSTRUCTURES



Structural and mechanical properties of functionalized surfaces:

- Alkyl terminated Si(001).
- Thiol functionalized Au(111).





Structural and Electronic properties of nanowires (InN, ZnO, CdSe) for photovoltaics applications.

Section of nanowires grown in the [0001] direction.

Molecular Dynamics simulations of Solid/liquid interfaces.

















#### 4H-Silicon Carbide wafers characterization (Wafer supplier Cree Inc.)

Detailed Structural and Morphological wafer surface characterization for defects identification and their influence in power electronics devices behavior









## **SPINTRONICS**



#### Patterned magnetic materials and multilayers

- Realization of micro and submicro patterns of pillars and antipillars (square and hex lattice) of Ni, Co, NdFeB and multilayered materials (both through optical lithography and EBL);
- > AGFM/Kerr magnetic characterization;
- ➢ FESEM/AFM/MFM imaging;
- numerical contrast enhancement algorithms;
- ➤ magnetoresistive measurements.











# **Other activities**

> Synthesis and characterization of polymeric matrix composite materials (PMCs) containing dispersed antiferromagnetic nanoparticles (NiO) or dispersed ferromagnetic carbon nanotubes; ➤ implementation of finite element method and genetic algorithm simulations of magnetic MEMS.







4.2



#### **EDUCATION**



#### http://www.master-nanotech.com/



Diplôme d'ingénieur en Micro et Nano Technologies pour les Systèmes Intégrés

Laurea Specialistica Micro e Nano Tecnologie per System Integrati



Didactic activities followed by the staff belonging to the Lab are set in the framework of the courses of different level present at the Polytechnic of Turin (degree courses, master courses, PhD courses). Among them we can cite: courses about basic Physics, Structure of Matter, Solid State Physics, Materials for Optics and Photonics, Physics of Surfaces, Physics of the Electronic Materials, Integrated Systems, Physics of the Processes for Microtechnologies, Devices & Technologies for Microsystems, Applications of Microsystems, Microsystems for medical applications.

Since september 2004, the staff of the Lab is managing a new Degree program "Nanotechnologies for ICT" in collaboration with the universities of Grenoble (INPG) and Lausanne (EPFL). Students are following courses in english, planned along three semesters in the three Institutes. The staff of the Lab gives lectures about Physics of Technological Processes, Microsystems Basics, CAD for Microelectronics, Design of Microsystems and CAD for MEMS.









#### MICROLA S.r.I. Spin Off of Politecnico di Torino





## Products

**QDPSSL** ( $\lambda = 1064$ nm)

- 10W
- 20W
- 40W

 $\begin{array}{l} \text{ODPSSL} (\lambda = 532 \text{nm}) \\ \text{5W} \end{array}$ 

MLP ( $\lambda = 640 \div 1064$ nm)

- Up to 100W





Fibre coupled high power Diode Laser



End pumped Q-switched resonator

