



### Sommaire

- RENATECH strengthens European scientific infrastructure landscape
- Specific technological tools
- Recent technological realisations
- Events
- Realise your project at RENATECH network



### RENATECH strengthens European scientific infrastructure landscape

"With an interdisciplinary field like nanoscience, whose success depends on a dialogue between diverse scientific approaches and an intense circulation of ideas, lowering the barriers to transnational collaboration is a must-do to keep Europe ahead of the competition." ( <http://cordis.europa.eu/fp7/coordination/pdf/nanosci-eplus.pdf> )

Countries all over the world are aware of the strategic importance of nanoscience and nanotechnologies, and strong and dedicated research programs have been launched as a result. In Europe, strong national commitments exist to fund nanotechnology and nanoscience research.

In accordance with the FP7 ambition to catalyse and to leverage research infrastructures of international relevance, RENATECH is playing a determining and central role within French initiative and is showing the strategic relevance of sustainable efficient research infrastructure. As an excellent example can be cited Transpyrenees Action on Advanced

Infrastructures for nanoscience and nanotechnologies (TRAIN<sup>2</sup>) where RENATECH network, represented by LAAS, is successfully participating together with its Spanish and Portuguese partners on the construction of a framework for the development and cooperation of European infrastructures and centers of research in nanosciences and nanotechnologies. In addition, the added value of collaboration between the partners is to increase the competitiveness of scientific and technological transfer in targeted emerging fields, with a significant impact for the industries in electronics, for the environment, health and energy.

Due to its RENATECH experience, LAAS platform, the leader of Networking work package, has disseminated Renatech best practices: creation of an interactive process capabilities table, exchanges of people from technical staffs, technical workshops, and editing of a "handbook" about expertises and equipment available through the TRAIN<sup>2</sup> partner's platform. Thanks to the fruitful exchanges and successful results within this project which is finishing by the end of 2012, TRAIN<sup>2</sup> partners in connection with others European platforms, work on a proposal for future calls on integrating infrastructures in the frame of the Horizon 2020 program. RENATECH, through the LAAS platform, is in charge of coordination of the first draft of this proposal.



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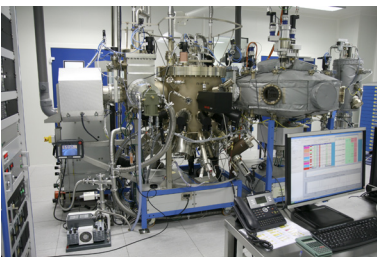
Web: <http://train2.eu/inicio.action>



## Specific technological tools

LAAS

### Molecular Beam Epitaxy system Operational date: September 2012



The equipment, a Riber MBE412, is a Molecular Beam Epitaxy (MBE) system.

It consists on a MBE chamber connected to an ultra-high vacuum cluster tool chamber. This automated tool also distributes a loading chamber with 10 platens capability, a park station with 5 places, and a high temperature degassing chamber (up to 800°C).

The MBE412 chamber is able to grow on 2", 3x2", 3" and 4" wafers. It is devoted to the growth of III/V As-based epitaxial layers.

The twelve cells ports of this chamber are equipped with: two arsenic cracker cells, two gallium, two indium, and two aluminium effusion cells, an antimony cell and a nitrogen valved

plasma cell. N-type doping is provided by a silicon cell, whereas p-type doping comes from a dual line CBr<sub>4</sub> injector.

For in situ control of growth, the MBE chamber is equipped with - a 12kV High Energy Electron Diffraction (RHEED) system with a KSA400 camera and its analysing software.

- a pyrometer

- a band gap shift measurement system allowing the precise and absolute determination of the growing substrate temperature (KSA BandiT)

The system is fully automatized, and the growth on a batch of wafers is possible. The Riber Crystal software controls all the cells parameters and is interfaced with the robot.

This machine is primarily dedicated to the growth of photonic devices.

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SCIENTES &  
TECHNOLOGIES

### New equipment for low temperature ICPECVD deposition at FEMTO-ST technological facility



High-density plasma technology is becoming increasingly attractive for the deposition of dielectric films such as silicon nitride (SiN<sub>x</sub>), silicon dioxide (SiO<sub>x</sub>), silicon oxynitride (SiON<sub>x</sub>) and hydrogenated amorphous silicon (a-Si:H). In particular, inductively-coupled plasma chemical vapor deposition (ICPECVD) offers a great advantage for low temperature processing over plasma-enhanced chemical vapor deposition (PECVD) for a range of devices including compound semiconductors (InP, GaAs ...). The material properties of these films have been investigated as a function of ICP source power, RF chuck power, chamber pressure, gas chemistry, and temperature.

The ICPECVD films will be compared to PECVD films in terms

of roughness, homogeneity, refractive index, density and other film characteristics. Compare to the traditional high temperature technology, the fabrication of MOEMS-MEMS/ NEMS and semi-conductors at lower temperature have many advantages (i.e. the co-integration).

The SI 500 D plasma enhanced deposition tool is configured to deposit SiO<sub>x</sub>, SiN<sub>x</sub>, SiON<sub>x</sub>, and a-Si:H films in a temperature range from room temperature up to 350°C with lower hydrogen content. The large range of thin dielectric properties also allows addressing optical applications. A brief insight into the possibilities of this kind of gases mixtures, on thin layers properties with different stoichiometries, has been investigated. A database is available at FEMTO-ST for the applications in Optoelectronics, Microelectronics and Microsystems.

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LTM

PTA

### Atomic layer deposition system

The equipment is a FijiTM from CambridgeNanoTech. Fiji plasma atomic layer deposition (ALD) system is designed for optimal performance and versatility for plasma ALD deposition. The Fiji system is designed for optimal uniformity for all deposition materials. Indeed the reactor and chuck are optimized to get laminar flow that increases deposition uniformity while minimizing precursor consumption and cycle times. Different deposition modes allow precise control of deposition process: continuous Mode™ for rapid growth of conformal films, and plasma mode™ for difficult nitrides and metals.

Our Fiji system includes 6 independent precursor lines for solid, liquid, or gas chemistries and 3 plasma gas lines for maximum utility (N<sub>2</sub>, O<sub>2</sub> and H<sub>2</sub>). The interactive software and highly customi-

zable experimental parameters allow rapid setup and execution of experiments with easy analysis of resulting data. A load lock system (single wafer up to 8 inches) is connected to the deposition chamber allowing a wafer transfer under secondary vacuum with turbo molecular pump. The precursors available on our Fiji system are Hf, Ti, Al and Pt for HfO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Pt, respectively.

Cambridge NanoTech also provides many other ALD precursor for oxides (La<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, ZnO, ZrO<sub>2</sub>, Ta<sub>2</sub>O<sub>5</sub>, In<sub>2</sub>O<sub>3</sub>, SnO<sub>2</sub>, ITO, Fe<sub>2</sub>O<sub>3</sub>, MnO<sub>x</sub>, Nb<sub>2</sub>O<sub>5</sub>, MgO, Er<sub>2</sub>O<sub>3</sub>, Nitrides (WN, Hf<sub>3</sub>N<sub>4</sub>, Zr<sub>3</sub>N<sub>4</sub>, AlN, TiN, NbN<sub>x</sub>) and Metals (Ru, W, Ni, Fe, Co).

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## Recent technological realizations

LTM

PTA

### Direct self-assembly of PS-b-PDMS block copolymers

Nowadays in microelectronics, devices are fabricated by projection UV photolithography ( $\lambda = 193$  nm). In order to reach future needs in resolution, new alternatives lithography technics with high throughput and low cost has to be developed. Directed self-assembly (DSA) is a very promising method that has been proposed in the ITRS lithography road map.

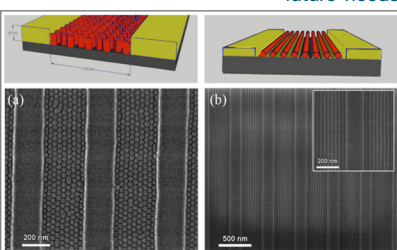
One approach that is studied at LTM in the LAMAND European framework program is to realize DSA of PS-b-PDMS block copolymers onto tunable surface chemistry substrate. Indeed, PS-b-PDMS (which present a PDMS block which is more robust to etching) has been preferred to PS-b-PMMA commonly used now in microelectronic because of its ability to enhance resolution to sub 10 nm half pitches. However, the very low surface energy makes the PDMS blocks orientation difficult.

In order to make the orientation possible, we fabricate substrates by soft UV nano-imprint lithography. According to the resin that is used for substrate fabrication, it is possible to get PDMS cylinders orientation along the 3 possible directions has presented on the figure above (the 3rd orientation, i.e. across the grooves, is not shown).

Another approach investigated by our group is to realize self-ordering in multilevel scale by combining previous method with direct nano-imprint lithography of block copolymer. Thus, directed self-assembly could be obtained during imprint process with the mold. In these conditions, multi-level and multi-orientation are achievable.

The other research path that our laboratory is following is on the synthesis of new block copolymer that could reach ultimate resolution below 5 nm half pitch. Collaboration with CERMAV lab is on the way to realize vegetal based block copolymers.

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## Electrical study of ferroelectric thin films



image of Pt pads on PZT thin film (optical microscope)

The process described in the following offers the possibility to study electrical properties of ferroelectric thin films i.e.  $\text{PbZrTi}_{1-x}\text{O}_3$  (PZT) thin films. Indeed, to assess the leakage currents, conduction mechanism and ferroelectric properties, top and bottom conductive electrodes are needed for electrical contacts. The top contacts are made by lithography or by the use of a shadow mask during the deposition of the electrode material. The bottom electrode is the substrate or a conductive layer between the ferroelectric film and the substrate.

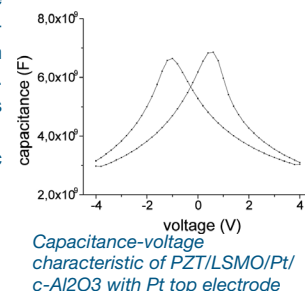
In this work, the conductive layer is an epitaxial Platinum (Pt) (111) film deposited by MBE on sapphire substrate. An area of Pt is masked during the deposition of the buffer layer ( $\text{LaSr}_x\text{Mn}_{1-x}\text{O}_3$  layer (LSMO)) and the PZT layer in order to make the bottom contact. Buffer layer is used to provide PZT nucleation. LSMO and PZT layers are deposited by PLD technique.

By a UV lithography process, metallic top electrodes can

be deposited on the ferroelectric surface. First, a reversible photoresist (AZ5214) is deposited by spin coating and is baked. Then, the stack is illuminated with UV light through a mask patterned with rectangles ( $250\mu\text{m} \times 220\mu\text{m}$ ). Then, a second baking is needed to make the photoresist negative. The stack is submitted to a last UV illumination without masking the sample surface. After the development step using a solution AZ400K, Pt is deposited on the sample by DC sputtering. The resist is then removed by acetone (lift-off process). The Pt pads are  $240\text{nm}$  thick and  $250 \times 220\mu\text{m}$  (figures 1 and 2). Typical capacitance-voltage curve is shown in figure 3.

This work was supported by Eniac "Nanocom" project.

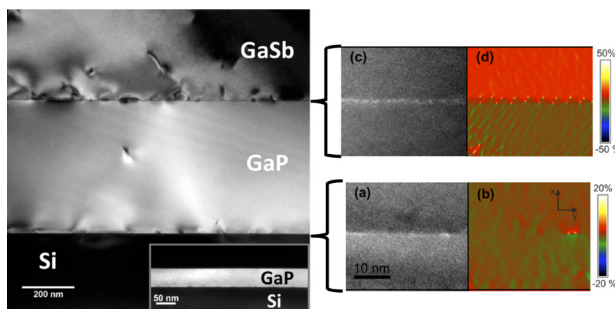
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## Monolithic integration of InAs-based heterostructure on exact (001) Silicon

As the CMOS silicon technology faces a power consumption crisis, the interest in III-V low electron effective mass materials increases considerably for low voltage operation transistor devices. However, the monolithic integration of such III-V devices in the silicon technology requires the development of a buffer layer accommodating the lattice mismatch (about 12% between InAs and Si) and solving the problem of anti-phase domain (APD) formation.

At IEMN, we have recently demonstrated a two-step solution addressing first the problem of APDs during the epitaxial growth of a pseudomorphic GaP interfacial layer on a (001) exactly oriented silicon substrate [1]. The mismatch is then accommodated through a GaSb buffer layer



002 dark field TEM and HRTEM cross sections of the GaSb/GaP/Si template. The inset shows the GaP nucleation layer prior to the MBE deposition. Images b and d: yy component of the strain tensor

grown on the GaP/Si template. The APD free GaP/Si templates from NAsP III/V GmbH in Marburg have been grown by MOVPE on a 300 mm exactly oriented (001) p-doped Si wafer and used for the subsequent Molecular Beam Epitaxy (MBE) of the GaSb/GaP accommodation layer prior to the growth of an AlSb/

InAs high electron mobility heterostructure. Thanks to the formation of a regular nano-periodic array of misfit dislocations confined at the interface between GaP and GaSb, a  $0.6\mu\text{m}$  thick buffer layer was sufficient to achieve a high quality InAs channel exhibiting an electron mobility as high as  $28\,000\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  and  $110\,000\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  at 300K and 77K respectively. This result paves the way towards the integration of low electron effective mass III-V materials into Silicon

technology.

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## Optical Extinction in a Single Layer of Nanorods

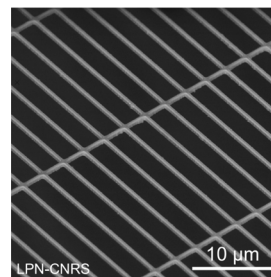
Can a transparent material with holes in it block light? Normally the answer would be no. However, LPN has shown that a web-like structure made up of an array of evenly spaced nanorods appears to block almost 100% of a specific light wavelength. This new finding confirms a theoretical prediction made several years ago: that a structure resembling a type of diffraction grating made of nanorods regularly ordered in a 1D or 2D array would perfectly reflect light of a specific wavelength.

This array, which is fairly challenging to make, consists of transparent freestanding dielectric silicon nitride nanorods around  $500\text{ nm}$  thick that are lined up in a single layer of rows  $3\mu\text{m}$  apart (see figure). It is realized with a combination of numerous deposition techniques (dielectric and metallic), wet and dry etching, e-beam and UV lithography. The stress in the free-standing structure is carefully controlled by annealing. The rods only cover 15% of the surface area and the rest of the structure is empty space.

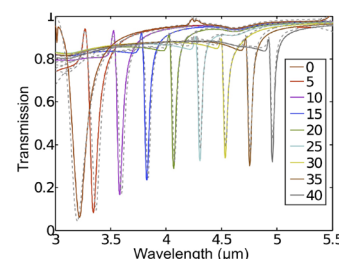
This «photonic nanoweb» behaves like a transparent material which can selectively reflect or trap (absorb) photons in a narrow wavelength range. The sharp spectral opacity window, in the form of a characteristic Fano resonance, arises from the coherent

multiple scattering in the array. The narrow resonance can be spectrally tuned with the angle of incidence, and leads to huge electric-field intensity enhancements in the rod. We also show that nanorods made of absorbing material exhibit a 25-fold absorption enhancement per unit volume compared to unstructured thin film. The counterintuitive «photonic nanoweb» might be used to make a new generation of optical devices, including light filters and sensors.

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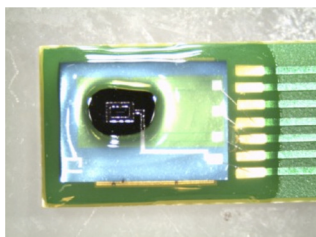


SEM image of a free-standing nanorod array.



Transmission spectra measured for different angles of incidence, showing the spectral tunability of the resonance. Nearly-perfect optical extinction is demonstrated at normal incidence.





Integration of ElecFET microsenors using silicon-based microtechnologies

PH-metry is a key technique for bio-analysis and should be integrated accordingly. In order to do so, potentiometric detection techniques were developed through the study of chemical field effect transistors (ChemFET). In the frame of biodetection, works aimed to the realization of enzymatic field effect transistors (EnFET), leading to the detection of different biochemical species such as urea and creatinine. Nevertheless, pH-EnFET microsenors are often limited to the hydrogenases enzymatic family, whose main mechanism is related to the amine NH<sub>2</sub> function hydrolysis. As a result, in order to extend the application field of pH-EnFET-metry for biochemical analysis, it is necessary to develop new detection/transduction approach.

An original microsenor, named ElecFET for electrochemical field effect transistor, was therefore developed. This microdevice is associated to the combination of amperometric and potentiometric techniques at the microscale. It results from the functional integration of an electrochemical microelectrode with a pH-ChemFET microdevice on a single chip. Its detection

principles are related to the microelectrode polarization to trigger electrochemical reactions in solution, and to the monitoring of the pH variations obtained around the pH-sensitive area. Thus, electrolysis phenomena and pH measurement are closely embedded at the microscale, enabling new electrochemical detection potentialities. First experiments were focused on water electrolysis, allowing to monitor impulsional pH variations in buffered microvolumes. Then, the ElecFET concept was applied to the hydrogen peroxide H<sub>2</sub>O<sub>2</sub> oxido-reduction phenomena, enabling the [H<sub>2</sub>O<sub>2</sub>] potentiometric detection on the [10 – 100 mM] range (sensitivity 1.25 mV/mM sensitivity). Finally, by integrating enzymatic layers based on glucose, lactate or glutamate oxidases known to be responsible for the production of hydrogen peroxide H<sub>2</sub>O<sub>2</sub>, the detection of biochemical species such as glucose as well as lactate or glutamate ions were demonstrated.

In conclusion, thanks to the ElecFET detection potentialities at the microscale, the pH-EnFET-metry technique was extended to the oxidases enzymatic family, enabling the analysis of different biochemical species in the frame of health and/or agribusiness applications.

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## Events

French Symposium on Emerging Technologies for micronanofabrication/

Journées Nationales sur les Technologies Emergentes en micro-nanofabrication

May 21-23, 2013

Palais des Festivités, Evian, France

The main objective is to bring together on an interdisciplinary basis all the major actors of the scientific community involved in the development of emerging technologies for micro-nanofabrication, with applications in the domains of optics and photonics, physics of nanostructures, electronics, chemistry, biology. The symposium will focus on emerging technologies for micro-nanofabrication, from fundamentals to complex integration techniques.

**THE 1ST RENATECH TECHNOLOGY PRIZE WILL BE AWARDED AT THE JNTE 2013 SYMPOSIUM.**

The RENATECH Technology prize will reward an outstanding work in micro-nanofabrication accomplished by a PhD student during his thesis work.

For any inquiry, please contact: [jnte13@lpn.cnrs.fr](mailto:jnte13@lpn.cnrs.fr)



## Realize your project with RENATECH network

1.Contact RENATECH network via:

common entrance point: [renatech-accueil@cnrs-dir.fr](mailto:renatech-accueil@cnrs-dir.fr)

or contact directly one of RENATECH facilities to discuss your application:



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2.The application will be worked through and evaluated by the reception team at each facility.

3. Realize your project

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