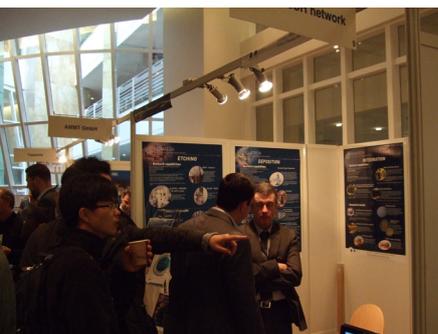


### Sommaire

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### RENATECH: where science and industry meet



One glance at modern life shows: progress is impossible without micro- and nanotechnologies! Starting with research laboratories and universities, equipment and material manufacturers right up to the finished chip, these technologies must always be state of the art, with availability and accessibility for academic scientists and high-tech companies at national and international scale.

In this context RENATECH continues to strengthen its relations with scientific academic and industrial community by participating at main European nanotechnological meeting points and by offering an access to its facilities to industrial users.

For the second time RENATECH was present at SEMICON Europa in Dresden in October 2011 - the leading forum for semiconductor and microelectronics manufacturing in Europe. Besides the booth at French pavilion, RENATECH also presented its technological expertise and core process capabilities at TechArena, an effective forum for intensive exchange and discussions between exhibitors for new equipment, process engineers, suppliers and potential research partners.

RENATECH started 2012 with another important international event - the 25th international conference on Micro Electro Mechanical Systems (MEMS) held in Paris. MEMS is one of the premier annual events reporting research results on every aspect of microsystems technology.

Both manifestations proved to be very successful for French national nanofabrication network and showed the increasing international interest in its research activities and technological achievements.

As a remarkable example of RENATECH openness to industrial users can be cited an industrial production line for piezoelectric components at Mimento technological facility (FEMTO-ST, Besançon).

Mimento Technology Centre has acquired industrial facilities dedicated to the micro-fabrication of piezoelectric components such as Surface Acoustic Waves devices. The originality of the project is to share these industrial facilities between research and industry. This technological platform is composed of fully automatic equipments distributed in three class-100 areas

enabling to process up to 25 four-inch wafers in one batch. At the moment, we have developed processes to fabricate SAW devices on quartz (fig.1a) and LiNbO<sub>3</sub> substrates operating at frequencies ranging from a few hundred MHz to 1.5 GHz (fig.1b).

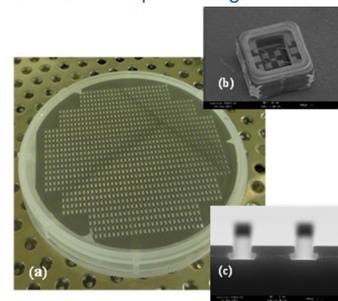
The first step of the process consists of cleaning and preparing each substrate by means of a wet (BOE7) or dry etch. To produce SAW devices with small dimensions (line or gap width < 1 μm), we have developed a dedicated bi-layer lift-off process combining the use of LOR3A and SPR505 resists. These resists are spin-coated using our SVG tracks and the exposure is then performed by our Nikon NSR I9 stepper (UV 365nm) whose ultimate line resolution is 0.35μm. For the time, our bi-layer method has allowed for achieving critical dimensions down to 0.43 μm. Other processes are currently being developed to address a large range of device fabrication. To complete the fabrication of SAW devices, we deposit the metal layer using an evaporating machine whose thickness accuracy is better than ±3%. After the lift-off step, acoustic measurements of several devices manufactured on our industrial line have been performed and the results have shown expected responses (as shown in figures 1 and 2).

An industrial activity dedicated to the global exploitation of the fabrication line is being currently developed in the business incubator of Franche-Comté. This future start-up emerging from FEMTO-ST research activities will answer any demand for technological fabrication according its specific capabilities (4-inches wafer processes, highly controlled metal deposition, high resolution lithography, standard / non standard wafer processing, full SAW device process including design and characterization) and will manage the evolution of its production range toward thin film devices and composite wafer fabrication and processing.

For further information contact:

S. Ballandras,  
[sylvain.ballandras@femto-st.fr](mailto:sylvain.ballandras@femto-st.fr)  
 E. Courjon,  
[emilie.courjon@femto-st.fr](mailto:emilie.courjon@femto-st.fr)

SAW devices batch-processed on the new fabrication line (a) 4 inches quartz wafer with several hundred operational devices (b) one of these devices operating at 1.5 GHz diced and packaged for effective insertion in an oscillator loop (c) Technological detail on the two-resist lift-off process





## Specific technological tools

LAAS

### New equipment for material annealing at LAAS technological facility

Two new furnaces have just been installed in the thermal processes area of the LAAS clean room. Those furnaces have been purchased at AET Company. The technology used in such furnaces is standard.

Each stack has 2 tubes.

Their main characteristics are the following:

Flat Zone Length: 50 cm

Maximum operating temperature for 3 tubes: 800°C

Maximum operating temperature for 1 tube: 1100°C

The maximum wafer diameter that can be processed is 150mm and small samples can be also treated with appropriated quartz boats.



The available gases are N2 and N2/H2.

The processes of those 4 tubes are controlled by one OS.

This equipment is dedicated for the annealing of different materials taking care of the contamination problem.

In fact, if we look at the development of the micro system technology, "new" materials with different crystalline states appear with the development of new deposition techniques like inkjet, atomic layer deposition system.....

In general, after the deposition, the material must be annealed in order to get the appropriated electrical, mechanical, optical...., characteristics.

Consequently, our new furnaces start to be used for Bismuth, Zinc oxide, Lead Zirconate Titanate, (PZT), Ferrites...anneals.

Contact : [rousset@laas.fr](mailto:rousset@laas.fr)

femto

### Equipment for MEMS characterization at FEMTO-ST facility

The MEMS Analyzer is a measurement system for micro and nano-devices like MEMS, MOEMS and NEMS, which detects the vibration amplitude and the resonance frequencies modes with many Scan Points in a short time. Amplitude, phase and impedance of the structure vibration oscillation can be measured and presented as a function of frequency. Moreover, the mechanical behaviors of the different strain modes can be shown by the deflection Shapes 3D animation

Initially, people were more interested in working on vertical resonators because they were technologically easier to manufacture. The majority of recent demonstrator devices use lateral technologies mainly because of the design flexibility: squares, disks, beams etc. These resonators can be classed by the vibrating direction: Out-of-plane (vertical resonators) and In plane (lateral resonators). So, in our equipment, we have two dynamic measurements in one:



In-plane and Out-of-plane. We know that the resonant frequency increases when the beam stiffness increases, so smaller resonator sizes induce a resonant frequency increase. The dynamic measurement In-Plane uses the Laser Doppler Vibrometry up to 20 MHz while the Out-of-plane uses the Stroboscopic Video Microscopy up to 2 MHz. In this case, video camera captures images that are used to determine the movement of the microstructure like Comb-drives. It is well known that this displacement is in the nanometer range (nearly 10–20 nm for the beam, 1–5 nm for the disk). In our system, the resolution is picometer range and nanometer range respectively for In plane and Out of plane measurements. Therefore, this machine is an ideal tool for accurate, rapid analysis and 3D dynamic characterization of MEMS, MOEMS and others microstructures.

Contact:

Etienne Herth: [etienne.herth@femto-st.fr](mailto:etienne.herth@femto-st.fr)

FMNT PTA

INAC  
INSTITUT NANOSCIENCES  
ET NANOTECHNOLOGIES

### ODUCAT EITRE 6 Nano Imprint Lithography Tool at PTA

The system allows the replication of patterns in the micro- and nanometer range on up to 6 inch wafers for applications including LED/Opto electronics, MemS/Nems, optical/magnetic storage, Bio devices, displays or PV. It can realize nano-imprints with a large scale of tunable parameters. A heater allows to anneal samples from 30°C to 250°C, and the Soft Press technology allows to apply a pressure from 10 bar to 80 bar on the whole surface. Moreover, a watercooling system is mounted on the equipment allowing quick decreases of temperature and the option of controlling temperature gradient. It offers the possibilities to adapt NIL conditions for keep substrates safe.

It is designed for the use of the classical thermal NIL, UV NIL, and the combined UV and thermal NIL.

The main technological interest of this equipment comes from the combination of the Soft Press technology and the use of an Intermediate Polymer Stamp, IPS. With Soft Press® technology, pressure is applied to the stamp and substrate using compressed

air, ensuring pressure uniformity over the entire imprint area. This allows stamp and substrate to conform to each other, eliminating negative effects from thickness variations, bow or waviness in stamp or substrate. Soft Press® enables thin and uniform residual layer over large areas, which is critical for high-resolution printing and pattern transfer fidelity. The Intermediate Polymer Stamp, IPS®, enables contamination control and increases the stamp lifetime through a two-step process where the contact between the hard master stamp and the hard substrate is avoided. Instead, the master stamp is replicated into a soft intermediate polymer stamp that transfers the structures into the target substrate. The IPS® technology therefore greatly impacts the overall costs associated with NIL, making the Obducat solution very cost efficient.

Contact:

Corinne PERRET FMNT-PTA [corinne.perret@cea.fr](mailto:corinne.perret@cea.fr),  
Cécile GOURGON FMNT-LTM [cecile.gourgon@cea.fr](mailto:cecile.gourgon@cea.fr)



## Recent technological realizations

iemn

### Gold nanodot arrays for nanofabrication

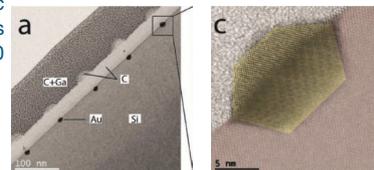
A large array of sub-10-nm single-grain gold nanodots for use in nanotechnology is described by Nicolas Clément and colleagues at the Institut d'Electronique Microélectronique et Nanotechnologie, CNRS, University of Lille, France [Small 7(18):2607-2613, 2011]

A uniform array of single-grain Au nanodots, as small as 5–8 nm, was formed on silicon using e-beam lithography. The as-fabricated nanodots are amorphous, and thermal annealing converted them to pure Au single crystals covered with a thin SiO2 layer. These findings were based on physical measurements by AFM, atomic-resolution STEM, and chemical techniques using energy dispersive X-ray spectroscopy. The authors demonstrated the formation by e-beam lithography of sub-10 nm Au dots with small dispersion

and perfect alignment. Such precise formation of small dots enabled them to identify the critical size that determines whether a dot is composed of single or multiple crystal domains. Moreover, they showed that annealing at moderate temperature can convert Au dots from amorphous to single-crystalline, and then they were covered with a thin SiO2 layer. After easy removal of the SiO2 (dilute HF etching), these nanodots can be used as electrodes for the characterization of organic self-assembled monolayers (SAMs) with less than 200 molecules.

Contact:

[nicolas.clement@iemn.univ-lille1.fr](mailto:nicolas.clement@iemn.univ-lille1.fr)



(a) STEM image showing the bulk silicon (Si), five annealed dots (Au), carbon layer (C), and platinum layers. (c) Coloured STEM image of a single annealed nanodot (260°C, 2h).

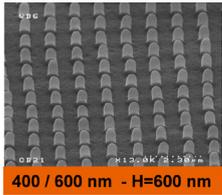


RENATECH  
French national nanofabrication network

[www.renatech.org](http://www.renatech.org)

## Nanoimprint on flexible polymer films - IPS technology

The aim of the ANR N-POEM project is to design, fabricate and characterize emerging periodic nanostructures for three electromagnetic and optical functionalities: microwave regime absorption, filtering and antireflection. These materials are based on strongly coupled periodic structures composed of sub-micrometric patterns. The different partners composing the project group expertise in nanofabrication, nanosciences including design and simulation. The Nanofabrication process is based on nanoimprint on flexible polymer films (IPS technology). These processes lead to the fabrication of arrays of nanostructures. Their interest is a reduction of technological steps, resulting in a decrease of production cost and time. The aim of this project is to develop technological processes for industrial applications. It will lead to a high maturity level of nanoimprint technique, compatible with industrial requirements in new domains such as

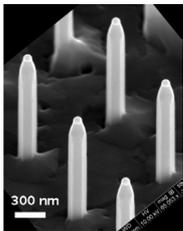


Principle of an agile MEMS-controlled reflectarray antenna



## Fabrication of original III-V heterostructures based on nanowire growth

Nanowires (NWs) are defined as structures that have an unconstrained longitudinal size while their lateral size is restricted to tens of nm typically. As such, they are often referred to as one-dimensional (1D) materials. Their formation by bottom-up growth offers a very large flexibility to fabricate original heterostructures. At LPN, self-standing NWs of III-V materials are elaborated by molecular beam epitaxy. Two examples of hetero-structured NWs are illustrated below. These objects could become important building blocks for future electronics, opto-electronics, or electromechanical devices.



1. Organized array of GaAs/Ga<sub>1-x</sub>Al<sub>x</sub>As core-shell heterostructures.

LPN has fabricated organized arrays of NWs by patterning a thin film of Au catalyst. The NW structure consists of an undoped GaAs core surrounded by an AlGaAs shell including a Si  $\delta$ -doping. Because of the charge transfer from the doping impurities to the GaAs core, a 1D electronic system is formed. The NWs are finally buried in a planar epitaxial layer which guarantees stable and reproducible transport characteristics. We observed weak localization of electrons in low-temperature magneto-transport measurements. We extract a phase coherence length of electrons in the 1D channel at 4K, larger than 0.5  $\mu$ m [1].

## Single-step, high-throughput biofunctionalization of nanoelectromechanical systems by nanocontact printing method

One of the most promising applications of nanoelectromechanical systems (NEMS) is foreseen in the field of ultrasensitive mechanical biosensing. So far, the issue of freestanding nanostructures functionalization has been seldom addressed because of the absence of generic techniques allowing large-scale molecular delivery at the nanoscale. The method developed at LAAS relies on the use of a modified nanocontact printing (nCP) process where one monolayer of antibodies is delivered onto a chip containing up to 106 nanostructures/cm<sup>2</sup> from the high-parts (grooves) of a polymer stamp while its base sits on the nanocantilevers' chip, thus providing mechanical stability. The presence of antibodies is validated by fluorescent microscopy and measurement of the nanocantilever resonance frequency shifts provoked by the added biological mass demonstrates that the cantilevers retain their mechanical integrity. First, arrays of nanocantilevers were fabricated by sacrificial release using silicon-on-insulator (SOI, 318nm P-type Si / 1 $\mu$ m SiO<sub>2</sub> / 525 $\mu$ m Si) substrates. A UV stepper photo repeater (1 Line CANON FPA) was used to pattern the shape of the nanocantilevers via a 600nm thick positive photoresist layer (Figure

1). The nanocantilevers were functionalized by a modified nCP technique that uses the grooves of a polydimethylsiloxane (PDMS)

devices for aircraft manufacturing, photovoltaic, information and communication technologies... Demonstrators corresponding to microwave absorption and infrared filtering are obtained by a coating of adequate material on nanopatterned polymer film. Industrial applications require large surface and light-weighted devices, especially for aircraft manufacturing. Thus, the substrates have to be flexible, thin and larger than 10x10 cm<sup>2</sup>. Moreover fabrication processes have to be robust and low cost. This project is running for one year, and technological processes are performed on the FMNT PTA platform in Grenoble, by LTM researchers. The figure presents a SEM picture of patterns imprinted on a plastic film.

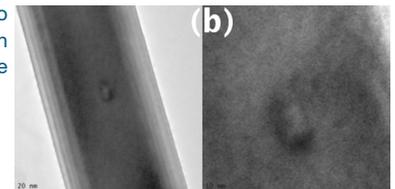
Contact :

Corinne PERRET FMNT-PTA, [corinne.perret@cea.fr](mailto:corinne.perret@cea.fr)  
Cécile GOURGON FMNT-LTM, [cecile.gourgon@cea.fr](mailto:cecile.gourgon@cea.fr)

For the development of photonic devices, we have fabricated InP NWs containing InP<sub>1-x</sub>As<sub>x</sub> quantum dots (QDs), as well as core-shell structures of these materials [2]. Different growth conditions are necessary to form the core and then the shell. Fig. 2b shows a single QD which is perfectly positioned on the axis of a photonic wire. The QD diameter is fixed by the size of the catalyst particle and the QD height is adjustable with the growth time. The final shape of the wire is optimized to funnel the QD emission into the optical mode guided by the wire (Fig.a). The tapered end favors a good external collection of photons by reducing the divergence of the output beam. By adjusting the InP<sub>1-x</sub>As<sub>x</sub> composition, the emission is easily tuned in the telecommunication wavelengths range. These objects, obtained in a single growth run, open a new route to the fabrication of efficient single photon sources.

Contact :

jean-christophe.  
[harmand@lpn.cnrs.fr](mailto:harmand@lpn.cnrs.fr)



2. (a) InP photonic wire with a single InP<sub>1-x</sub>As<sub>x</sub> QD inside. (b) TEM observation of the InP<sub>1-x</sub>As<sub>x</sub> QD.

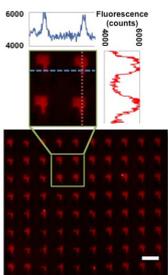
stamp to transfer the biomolecules. Stamps were fabricated by injecting a PDMS mixture on a specific master mold into a dedicated stainless steel set up and cured at 60°C for 4 hours. This procedure enabled creating precise composite stamps consisting of a patterned 1.5cmx1.5cmx400 $\mu$ m PDMS film glued to a 1.5cmx1.5cmx1mm thick glass slide, thus preventing any distortion or deformation of stamp when withdrawing it from the master mold.

For biofunctionalization purpose, we used the printing process enabling the molecular transfer to occur from the grooves of the stamps. The stamp was inked by incubating for 2mins in a 400 $\mu$ g/mL Alexa Fluor® 660 donkey anti-goat antibody solution. Immediately after inking, the stamp was aligned with the 1.5cmx1.5cm NEMS chip, using a microscope and an automated chuck. Backpressure was applied onto the stamp in order to deform the grooves and print the antibodies on the nanocantilevers. Fluorescence images of functionalized NEMS are shown in Figure 2.

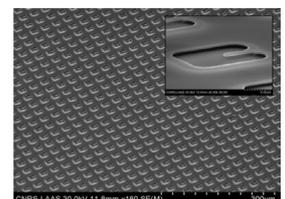
In conclusion we developed a method for biofunctionalizing a large-scale array of nanocantilevers using a modified nCP process. The success is confirmed by fluorescence observations showing that all cantilevers retain their mechanical integrity and their sensing capability.

Contact :

Liviu Nicu [nicu@laas.fr](mailto:nicu@laas.fr)



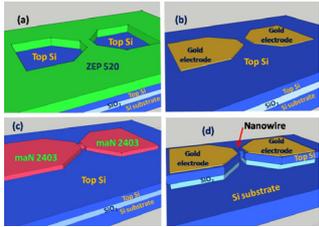
Fluorescence images of the nanocantilevers after printing the donkey anti-goat antibodies (scale bar 30 $\mu$ m). The inset shows a zoom of the cantilever array with corresponding raw levels of fluorescence plotted along two axes of the array.



Scanning electron microscopy pictures of fabricated silicon nanocantilevers



## Mechanical study of top-down fabricated silicon nanowires

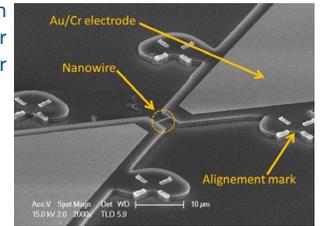


Silicon nanowire device fabrication steps

Single crystal silicon nanowires with a width down to 25 nm and a length to width (L/w) aspect ratio up to 150 were fabricated by surface micromachining of thin SOI wafers. Starting SOI wafers had a 1 µm-thick buried oxide and a 340 nm-thick top Si layer with a boron doping level of  $7.5 \times 10^{14} \text{ cm}^{-3}$ . The top layer was thinned down to 220 nm by successive dry thermal oxidation and BHF chemical etching steps. Some devices also include Au/Cr electrodes patterned by lift-off for further electrical measurements. First, a positive resist (ZEP 520) was spin coated onto the top Si layer. For each device, this resist was patterned with a 20 keV Raith 150 electron-beam lithography system. A 20 nm layer of chromium and a 150 nm layer of gold were deposited by e-beam evaporation and patterned by lift-off. Then, Si nanolines along the <100> direction were patterned by e-beam lithography using maN 2403 negative photoresist. The unprotected top silicon layer was etched at 5 oC by reactive ion etching in a SF<sub>6</sub>/O<sub>2</sub> plasma. SEM observations have shown that the nanolines were straight whatever their size. Finally, the nanowires were released by HF vapor etching of the buried silicon oxide. It was demonstrated

that such top-down fabricated clamped-clamped nanowires are laterally buckled when L/w is larger than 36. This is attributed to an unexpected high compressive residual stress estimated to be in the -270 MPa to -335 MPa range from a simple post-buckling model. Estimations of stress generation by amorphization during etching, by air surface oxidation, by thermomechanical effects and by die bending could not account for this high value. Occurrence of a large stress in silicon nanowires fabricated by the top-down approach must obviously be considered for future investigations of transport phenomena in these devices and of their properties as well as for their applications in sensors.

Contacts:  
[pierre.allain@u-psud.fr](mailto:pierre.allain@u-psud.fr),  
[alain.bosseboeuf@u-psud.fr](mailto:alain.bosseboeuf@u-psud.fr),  
[xavier.leroux@u-psud.fr](mailto:xavier.leroux@u-psud.fr)



SEM-FEG image of a Si nanowire device with alignment marks and contact electrodes



## Events

### RENATECH users' information day

Monday 19 march 2011, 10h – 16h

The annual users' information day has become the traditional event for the academic and industrial micro- and nano-technological community.

This year manifestation will embrace the whole French territory and will be organised in the regions, in the laboratories of the large technological facilities. Thus the participants will have an opportunity to know better the network facilities which are located in their regions and to visit the clean rooms. Close interactions with the local actors of the large technological facilities will provide an excellent opportunity to establish new contacts and to learn more about the procedures of project submission and its realisation in the network.

IEF and LPN facilities will welcome the participants at IEF laboratory in Orsay, IEMN - in Villeneuve-d'Ascq, FEMTO-ST – in Besançon, LAAS – in Toulouse, PTA and CEA LETI – in Grenoble. To participate, please contact directly the laboratory of your choice.



## 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:



IEMN technological facility  
 Contact: [plateforme@iemn.univ-lille1.fr](mailto:plateforme@iemn.univ-lille1.fr)



FEMTO-ST technological facility  
 Contact: [mimento@femto-st.fr](mailto:mimento@femto-st.fr)



IEF technological facility  
 Contact: [ctu@ief.u-psud.fr](mailto:ctu@ief.u-psud.fr)



LAAS technological facility  
 Contact: [plateformertb@laas.fr](mailto:plateformertb@laas.fr)



LPN technological facility  
 Contact: [centrale-techno@lpn.cnrs.fr](mailto:centrale-techno@lpn.cnrs.fr)



PTA technological facility  
 Contact: [accueil@ptagrenoble.com](mailto:accueil@ptagrenoble.com)



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

3. Realize your project

For further information concerning RENATECH newsletter contact: [Elena.Hoffert@cnrs-dir.fr](mailto:Elena.Hoffert@cnrs-dir.fr)

