RENATECH

French national nanofabrication network

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A - Renatech technology prize

During the JNTE (French Symposium on Emerging Technologies for micronanofabrication) which took place in November in Lyon, Renatech awarded the second Renatech technology prize to

> Komla NOMENYO from Charles Delaunay Institute (Troyes) for his works on "Photonique UV: structuration top-down du ZnO" and

> Fabrice STEHLIN from the Institute of Materials Science of Mulhouse for his work on "DUV photolithography of metal-oxo clusters."

The prize rewards any outstanding work in micro and nanofabrication accomplished by a PhD student during his thesis work. Technological achievements and their impact for the community are specifically considered.



K.Nomenyo and F. Stehlin's representative: O. Soppera





ZnO based UV photonics: Enhanced emission and energy transfer through top-down micro and nanostructuring

Nanotechnology and Optical Instrumentation, Charles Delaunay Institute, UMR 6281, CNRS/University of Technology of Troyes

ZnO is a promising II-VI semiconductor for UV applications although p-type ZnO is not yet available. Nevertheless it remains an alternative material for GaN and its alloy InGaN. Indeed, threading dislocations density is higher in GaN films due to the lattice mismatch between the semiconductor and the substrate on which it's grown. To obtain a high quality GaN, one needs thick films of the order of a few microns. Unfortunately, thicker films involve multimodal components and are difficult to apply for photonic solutions with ultimate control of light where lower optical mode density is preferred. Hence there is a growing interest for ZnO as a photonic material. Not only does ZnO exist in epilayer form, but it also has great optical properties. For example, the exciton binding energy of ZnO (60 meV) is higher than that of GaN (21 meV). This allows ZnO to emit light at ambient temperature and interestingly, it increases the device brightness.

In this work, we achieved ZnO micro and nanostructuring for fabrication of compact and efficient devices. There were two main challenges: first, to achieve structures with size of less than 100nm and second major problem is ZnO hardness. It is easy to dissolve ZnO by using acids and chemicals, however, it is very difficult to etch ZnO by dry etching (plasma) which is preferred as it is more reliable and provides uniform patterns with vertical sidewalls. Here, we have used Electron beam lithography (EBL) with a lift-off process in combination with inductively coupled plasma (ICP) reactive ion etching (RIE). Nickel (Ni) has been used as a mask in order to have a high selectivity in the presence of C_2F_6 and O_2 ionized gases. The etching rate used was 26nm/s in order to avoid roughness. Figure 1 shows SEM images of ZnO photonic structures. We also developed a specific EBL writing method to etch holes in ZnO by using Ni as a mask. Holes cannot be easily obtained by standard EBL technique combined with lift-off; neither when an electropositive resist nor when an electronegative resist is used. More interesting, the process is 4 times faster.

ZnO thin films were grown by pulsed lased deposition (PLD) method. They exhibit high structural and optical properties. These films were structured to control both spontaneous and stimulated emissions (figure 2) [1][2]. These structured films are now being implemented for the development of confinement of light in the UV range. The applications of this newly growing field are tremendous for ex. in lighting (2nd generation white LEDs), photodetection (solar-blind photodetectors), transparent photovoltaics, decontamination (photocatalysis) or in the much longer term, for extremely high speed telecommunications. It is worth noting that sub 100 nm fully controlled nanostructured ZnO films are of interest for other applications such as ultrasensitive chemosensing and energy scavenging due to the multifunctional properties of ZnO.



Figure 1: Photonic ZnO (138nm thick) micro and nanostructures obtained by EBL combined with RIE/ICP: (a) and (b) are microdisks with respectively 6µm and 1.5µm diameter; (c) nanorod photonic crystal with 75nm radius and 375nm pitch. (d) Ni mask design by a so call electron beam shift lithography to etch hole photonic crystal with 40nm radius and 120nm pitch.



Figure 2: (a) Photo of lasing-mode in ZnO thin film grown by PLD in c axis. The sample is 1 cm \times 0.5 cm and 560nm thick of ZnO on sapphire: optical gain = 1369 cm-1 and losses = 6.2 cm-1. (b) ZnO microdisk structuring used to increase, 3 times more, the electron-hole plasma emission in ZnO after the population inversion. It also decreases the laser threshold.

K. Nomenyo, A.-S. Gadallah, S. Kostcheev, D. J. Rogers and G. Lerondel, Appl. Phys. Lett. 104, 181104 (2014)
 A.-S. Gadallah, K. Nomenyo, C. Couteau, D. J. Rogers, and G. Lerondel, Appl. Phys. Lett. 102, 171105 (2013)



DUV photolithography of metal-oxo clusters. From photochemical processes to the applications in nanofabrication.

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The main purpose of this thesis was to provide a precursor of metal oxides (ZrO₂, TiO₂, HfO₂) compatible with DUV interference photolithography technique. Transition metal oxoclusters (MOC) obtained by complexation of an organic ligand and a partial hydrolysis have been proposed as building blocks. DUV irradiation (193 nm) allows a direct excitation of the MOC, which leads a photo-induced crosslinking and gives to the material a negative photoresist character. A detailed spectroscopic study allowed proposing a mechanism of photocrosslinking. The nano-structuring was performed by interferometric DUV lithography at 193 nm, chosen for its potential to write nanostructures on relatively large areas, and could be extended to 2-photon stereolithography and e-beam lithography. Furthermore, in the case of TiOC, the nanostructures can be fully mineralised at room temperature by an additionnal photochemical treatment. For ZrOC and HfOC, an additional thermal annealing step allows to obtain a crystalline structure MO2.

This work has also been extended to materials with semiconducting properties that can be direct-written by laser.





B - MNE 2015 - The Hague - 21-24 september

MNE (Micro and Nano Engineering) 2015 is the 41st international conference on microand nanofabrication and manufacturing using lithography and related techniques. The conference brings together engineers and scientists from all over the world to discuss recent progress and future trends in the fabrication and application of micro- and nanostructures and devices. Applications in electronics, photonics, electromechanics, environment, life sciences and biology are also discussed.

Strengthening its effort in openness to the socioeconomic world, RENATECH engineers used to participate and present their results. Renatech's network was also present at the exhibition.



FIB fabrication of large arrays of zero-mode waveguides for fluorescently labeled biomolecules detection

Keywords: FIB, zero-mode waveguides, fluorescence, single-molecule

Zero-mode waveguides (ZMWs) are optical nanostructures fabricated in a thin metallic film capable of confining the excitation volume to the range of zeptoliters [1]. This small volume of confinement allows single-molecule fluorescence experiments to be performed at concentrations of fluorescently labeled biomolecules that are physiologically relevant (Figure 1). The ZMWs can be fabricated using electron-beam lithography, deep-UV lithography, nanoimprint lithography and focused ion beam (FIB) technology [2,3]. In this presentation we will detail our efforts aiming at fabricating such devices using a dedicated high-resolution FIB nanowriter [4] capable of prototyping large arrays of such devices with pre-defined geometries, ultra-high precision and highest reproducibility. In this work we have specifically addressed the question of sputtered material re-deposition and of local electrical charge removal for these particular devices combining a thin metal layer (AI, Au) deposited on a glass substrate. For the first calibration experiments, multiple arrays of 400 nanoholes were milled in 100 nm thick metal layers (Figure 2) using a classical milling approach with ion doses ranging from 5.10¹⁶ ions/cm² to 5.10¹⁷ ions/cm² and diameters ranging from 50nm to 130nm. Patterned nanowells dimensions were measured with SEM and AFM and the corresponding volumes deduced. An optimum was evidenced from the measurements which showed that the FIB fabricated nanowells for the selected diameters and depths were found to be compatible with ZMWs experiments.

Then, using a solution of fluorescently labeled DNA at μ M concentration, the efficiency of these ZMWs was analyzed and showed a high heterogeneity level which highly decreases the number of potential usable devices. Indeed, particle re-deposition has been found to pollute the floor of neighboring ZMWs when milling the nanowells in the metal layer covering the surface of fused-silica substrate, impairing their optical performance and their ability to immobilize biomolecules. These measurements were shown to correlate with the FIB patterning strategies that have been applied during the initial experiments (ZMWs milled last presented no metallic re-deposition) thus requiring further improvements.

Therefore, an additional optimization of the FIB milling strategy was developed to allow a significant reduction of material re-deposition and to ultimately gain nanometer control of the depth of the wells. This strategy leading to a more homogeneous fluorescence background distribution and improved uniformity over the entire array (Figure 3) will be described in detail. Using this procedure, fluorescence signals corresponding to single molecules specifically bound to the floor of ZMWs were measured showing signal to noise ratios improved up to a factor of 3.

Finally, this FIB milling strategy was applied to fabricate large arrays of 4489 ZMWs (100µm x 100µm). The transmitted light was measured and showed coefficient of variations as low as 1.9% assessing a better homogeneity of FIB-processed ZMWs (Figure 4).

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DNA-CV5 Figure 1. Principle of biomoof Cy5-labelled DNA is added metal cladding the floor of the ZMW are glass excited by the evanescent field and emit fluorescence. Fluorescence laser Fluorescence excitation Figure 2. SEM image of ZMWs arrays with a Figure 3. Fluorescence zoom on a 90nm circular nanowell (insert) background distribution for 400 ZMWs after FIB patterning Figure 4. Transmitted light image of a 100x100µm array of 4489 ZMWs showing a coefficient of variation of 1.9%

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Plasmonic Substrates by soft UV Nanoimprint Lithography for Bimodal Biochemical Detection

Keywords: Soft UV-NIL, Nanoimprint Lithography, Plasmonics, SERS, SPRI.

The fabrication of bimodal plasmonic substrates, which are obtained by UV-NIL and reactive ion etching (RIE), is optimized in order to obtain large arrays (some mm²) of gold nanodisks on gold film for industrial applications. This specific design of gold nanodisks on gold film allows two modes of optical characterization: the Surface Plasmon Resonance Imaging (SPRI) and the Surface-Enhanced Raman Scattering (SERS) effect. The first step of fabrication is the realization of the silicon master mold designed by electron beam lithography. RIE processes have been optimized to take care of the verticality of the hole walls in Si master mold, which has an effect on the fabrication of the PDMS stamps. The next step is the imprint process through the AMONIL resist with PDMS stamps (Figure 1). Next, the etching of the residual thickness of AMONIL is stopped at the level of the gold film. Then, an Au layer (30 nm) is evaporated on this gold film and the remaining layer is removed via a lift-off process. Gold nanodisks arrays on a gold film were obtained with diameters (D) varying from 100 to 500 nm by step of 50 nm with a periodicity (P) of 600 nm. To validate our fabrication process, we identified the characteristic peaks of thiophenol molecule by SERS measurements (Figure 2.a) by using a Xplora spectrometer from Horiba Scientific (^Alaser = 660 nm, power = 288 μ W). Nanostructures are also characterized by SPRI (Figure 2.b) in Kretschmann Configuration with Bovin Albumin Serum.



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Hydrophobicity and anti-icing performance of nanoimprinted fluoropolymers films under overcooled temperature Keywords: Superhydrophobic, nanopatterning, anti-



Figure 1 : a) Dots imprinted in a FEP foil; b) FEP surface roughened by plasma etching

Figure 2 : water contact angle and CA hysteresis on roughened films

03



Keywords: Superhydrophobic, nanopatterning, anti-icing, fluoropolymers films, surface roughness

Abstract :

The formation of ice on cold solid surfaces can cause serious problems on facilities, electrical networks, transportation... The usual techniques used for deicing are active techniques such as heating, mechanical device or spraying deicing fluid. They have negative consequences due to the environmental degradation and energy consumption. A passive solution, environmentally friendly, consists on the use of superhydrophobic surfaces with intrinsic anti-icing performance. Superhydrophobicity can be obtained by Nanoimprint Lithography (NIL) and/or by roughening induced by plasma etching as shown on figure 1 on FEP (Fluorinated ethylene propylene) foils. The hydrophobic and icephobic performances have been characterized by Caw and CA hysteresis measurements at room temperature and under overcooled conditions. We have demonstrated that a water contact angle higher than 150° and a CA hysteresis lower than 10° can be obtained on these surfaces, which correspond to a superhydrophobic behavior. Moreover, the delay time of a water droplet freezing has been determined and it has been shown that some droplets are still liquid on surfaces cooled at -15°C after more than 20 minutes. This icephobic behavior is very interesting for anti-icing applications.

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Multiplexed microfluidic stamp inking for automated micro-contact printing process



Keywords: soft lithography, µCP, microfluidics, multiplexing, magnetic clamping, microarray

Micro-contact printing is a versatile technique for patterning biomolecules on a large range of substrate materials at lab scale. Nevertheless, it is not yet widespread at an industrial level due to the difficulty in the automation of the various steps of the technique. Among these issues, the control of the inking of the stamp in an automated and multiplexed format still requires dedicated investigations.

Here, we propose to achieve this step through a new microfluidic approach that enables both integration in an automated commercial system and possible patterning with different molecular species (multiplexing). Compared to other inking methods based on direct incubation or droplet deposition, microfluidics provides many advantages regarding the control of the flow rates, injection time and reduction of the sample volumes. Overall it offers a gain in reproducibility that is highly desirable in most bioanalytical processes. Indeed the quantity of molecules deposited inside each feature turns out to be more controlled as compared to conventional manual inking and printing [1].

However, for this specific application, the fluidic micro-channels need to be removed after inking to allow the subsequent contact of the stamp surface with the substrate to be patterned. This imposes a technical constraint related to the sealing of the microfluidic channels. Most technological approaches rely on irreversible sealing methods based on covalent bonding which is not compatible with the present application where a microfluidic system reversibly sealed on the stamp is preferable for the inking step [2]. To overcome this limitation we propose an innovative approach where magnetic forces are involved to hold the microfluidic channels and the stamp together without leakage.

The inking system is fabricated in PDMS by soft lithography. The stamp was made in two steps: a molded PDMS layer and a backplane obtained from a mixture of PDMS and iron particles. The microfluidic channels were obtained by PDMS casting on micro machined aluminum molds. The microfluidic channels and the stamp were reversibly sealed using the magnetic attraction between the magnetic layer of the stamp and the magnetic socket of the micro contact printer Innostamp40 [3], [4] (figure 1).

The inking process was entirely integrated in a commercial the micro-contact printing automate. A peristaltic pump connected to a mobile pipetting system was used to load the ink from a titration plate and to inject it through the stamp into the microfluidic channels. The pipetting system was connected to both the inlet and outlet of the microfluidic device through disposable pipetting cones. This method provides a direct control of the flow rate in the microfluidic channels and a low pressure in the device as the pump is used to simultaneously inject and suck the sample.

The inking process relies on i) ink injection, ii) air aspiration, iii) PBS washing. After the microfluidic inking step the stamp was dried and printed on glass slide surface as described in [3].

As a proof of concept, the microfluidic part has been designed with 6 independent channels making the multiplexed inking possible as shown in figure 2. Inking solutions were sequentially injected in each microfluidic channel. This procedure was repeated for each channel.

Different fluorescently-labelled streptavidin solutions have been successfully deposited and transferred by this automated microcontact printing method. Quantification of the fluorescence intensity of the printed surfaces is illustrated figure 4. The very low coefficients of variation demonstrate the homogeneity and reproducibility of the process in comparison with more conventional inking approaches. Investigation of the multiplexing scalability though miniaturization is currently under progress to provide more versatility for diagnostic or bioanalytical applications.

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[4] http://www.innopsys.com



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C - JNTE 2015 - Lyon - 18-20 November



The main objective of JNTE (French Symposium on Emerging Technologies for micro-nanofabrication) 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. This year, JNTE hosted the French-Japanese Workshop on

Emerging Nanotechnologies, organized by Renatech and the Japanese Nanofabrication Platform. The aims the workshop is to to tighten the links between both networks. The workshop focused on technological specificities of each laboratory.

Glass micro-optics and MEMS: application to array-type Mirau micro-interferometer

Interferometric techniques are fundamental for optical metrology, due to their unique capability for non-destructive and high-accuracy measurement of a broad range of quantities.

Miniaturization of interferometry systems has become an important issue in response to an increasing demand for the reduction of system size, lower manufacturing cost, as well as new metrology challenges such as in vivo imaging. The combination of glass micro-optics and MEMS technologies allows a cost-efficient fabrication of mm-size "active" microinterferometers that integrate micro-optical components with silicon microactuators for the scanning of light beams or for precise generation of an optical phase shift. The use of very stable borosilicate glass as an optical material enables vertical integration of all components by multi wafer bonding, offering a high alignment accuracy and wafer-level encapsulation. We demonstrate the potential of this approach on the case of 4x4 array-type Mirau microinterferometer, composed of glass lenses $(\forall = 1.9 \text{ mm})$, comb-drive vertical microscanner with arrays of suspended reference micromirrors and planar TiO₂/ glass beamsplitters. The device is a key part of a portable SS-OCT system for early cancer diagnosis.

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Mach-Zehnder interferometers based on Ge-rich SiGe waveguides for optical interconnects





On-Chip optical interconnects is a promising option to overcome the limitations of electrical interconnects such as the high power consumption and limited bandwidth. In this context Ge-rich SiGe on graded buffer waveguides are a promising platform for photonic integration, because of the possibility to couple them with efficient active devices such as compact Ge/SiGe quantum wells optical modulators or photodetectors. We recently demonstrated at a wavelength of 1550nm, low-loss bends with radii as low as 12 μ m. Multimode Interferometer beam splitters based on Ge-rich SiGe waveguide on graded buffer were designed, fabricated and characterized. Mach-Zehnder interferometers have also been investigated as a fundamental building block for a new photonic platform. A Mach-Zehnder interferometer exhibiting a contrast of more than 10 dB has been demonstrated. These results pave the way toward monolithic integration of Ge-based active devices with efficient and compact passive Ge-rich SiGe optical circuitry on bulk silicon wafers.

Low energy plasma enhanced chemical vapor deposition (LEPECVD) was used for the growth of the SiGe stack (at L-NESS lab in Como). The device fabrication was then performed at the nanocenter CTU-IEF-Minerve. The fabrication of the device is based on 2 etching steps. A hard mask is first patterned using deep UV lithography followed by reactive ion etching (RIE).

Waveguides were then etched using Inductively Coupled Plasma (ICP) etching. To reduce the curvature radius of bent waveguides, a deeper etch of the waveguides was performed to increase the mode confinement. The hard mask was thus used to self-align the second etching step with the patterned waveguides.

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III-V Growth and Technology for Quantum Optics in Microcavities



Single indistinguishable photons appear as key elements for quantum technologies. Scaling quantum technologies will thus require efficient and on demand single sources of indistinguishable photons.

Indistinguishable photons are usually generated at low rate and/ or with poor indistinguishability. Strong efforts have been done in the semiconductor community to overcome these limitations. III-V epitaxial quantum dots (QDs) have been demonstrated as excellent sources of quantum light. Over the past 20 years, great progress in the realization of ultra-bright sources of single photons have been made in the Laboratory for Photonics and Nanostructures of the CNRS, located in Marcoussis, in the south of Paris.

We work with InGaAs QDs grown by Molecular Beam Epitaxy (MBE). Two of the main issues with this system are the poor photon collection efficiency and the charge noise around the QDs. To circumvent these problems, we insert InGaAs QDs in micropillars to take advantage of the Purcell effect. The micropillar is obtained by etching down a planar microcavity surrounded by GaAs/Al0.9Ga0.1As distributed Bragg reflectors. The sample is doped to get an effective n-i-p diode structure where the Fermi level around the QD is well defined while the free carrier losses are minimized in the mirrors.

The challenge is to have a full control of the QD-cavity mode coupling, in terms of the spectral matching (same energy for the QD than for the cavity mode) and spatial matching (QD at the center of the micropillar).

Pascale Senellart's group has developed an advanced in-situ lithography technique allowing to position the QD within 50 nm of the pillar center and to spectrally adjust the cavity resonance to the QD transition with 0.5 nm spectral accuracy.

At the LPN, we have fabricated a single photon source at the state of the art. Under resonant excitation, indistinguishability of 0.9956 ± 0.0045 is evidenced with photon extraction of 65% and measured brightness of 0.154 ± 0.015 . These results make this source 20 times brighter than any source of equal quality.







Image 2 : Photoluminescence map of a QD located as the center of an electrically connected micropillar.

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Dense nanopatterning and suspension of silicon structures for energy harvesting and strainengineering applications



Silicon is remarkable for its electronic properties that place it at the heart of CMOS technologies. Silicon is also a prefered material for MEMS devices thanks to its mechanical properties. We study silicon nano-objects from the rather unconventional points of view of (i) thermoelectrics and (ii) extreme mechanical strain regime (>3 %).

Phononic crystal structures has been fabricated to make a thermoelectric material based on Si reducing the lattice thermal conductivity (κ_1), without affecting the electrical one (κ_F).

IEMN cleanroom facilities have the required equipments to fully process and characterize suspended MEMS Si structures. The picture below presents the main process steps.



We have fabricated integrated thermal metrology platforms thanks to selective vapour phase etching such as XeF2 gaz and vapour phase HF.

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Fig 1 : High

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Renatech dry etching capabilities

Si (LAAS)



Picture is suspended Si structure



Si (IEF)

Fig 2 : Nanowires

of Si



As shown of the picture above, heat conduction in Si could be reduced in order to boost its thermoelectric efficiency by means of phononic engineered nanolatticesand also by use of strain relaxation of silicon nitride beams, we were able to apply extreme strain on silicon nanowires, up to 5%, and to characterize it thanks to micro-Raman spectroscopy.Thermal conductivity goes from 55 W/m/K on a film structure to 12.5 W/m/K for a nanopatterned structure.



In Renatech facilities the fabrication of devices in micro and nanoscale use microelectronics materials for dry etching such as silicon (fig1), and III-V compounds (fig2-3) for nanosciences.

For these materials the Renatech facilities use mainly fluorine, chlorine based chemistries with continuous, switched and cryogenic ICP plasma. For shallow, low aspect ratio etching can typically be achieved with standard RIE equipment.

However, other challenging materials (fig4) that are not commonly used in microelectronics are dry etched.

Also, with the emergence of new semiconductor devices and architectures, there is a real need to limit plasma induced damage. One potential solution to minimize ion-induced damage is to pulse the plasma (fig5) for increasing selectivity and solving patterning-related issues.



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Scaling of semiconductor devices implies constant lithography improvement. While reducing dimensions towards the 10 nm node, photolithography is becoming a very expensive solution and Extreme UltraViolet lithography (EUVL) is not mature yet. In the frame of low-cost lithography resolution improvement, another promising solution is the use of diblock copolymers as a patterning mask, which allows defining sub-20nm patterns. The block copolymer self-assembling properties can be used to make a dense array of horizontal and vertical structures using graphoepitaxy (figure 1).

The challenge of this method is to pattern through a thin polymer mask (a few tens nanometers thick) by plasma etching. The choice of masking strategies (bi or tri layer approach) is the key point. As example, Si pillars of 40 nm in diameter can be achieved using PS-b-PDMS diblock copolymer and conventional 193 nm lithography stacks as masking strategy (figure2).

Marc Zelsman, Sophie Bohme, Cecile Girardot, Jerome Garnier, Javier Arias-Zapata, M. Delalande, G. Cunge, T. Chevolleau Figure1 : Lines and dot of PDMS with PS-b-PDMS block copolymer using the graphoepitaxy technique



Figure 2 : Nanopilars of Si with PS-b-PDMS block copolymer using a conventional 193 nm lithography as mask strategy



PESM – Grenoble (France) – May 9-10th



This 9th workshop is devoted to plasma etch and strip processes for micro and nanotechnologies. This year's workshop also addresses new and challenging topics in the field of emerging nanoscale devices such as MEMS/NEMS, optronics devices and 3D Integration. The workshop will also be open to new topics like plasma for biotechnology.

The objective is to provide a forum for open discussions between science and industrial application. It is dedicated to scientists, process engineers and Ph-D students. Topics include both fundamental and applied research, as well as issues related to introduction into manufacturing, with the progressive downscaling of device dimensions and the simultaneous demand for more functionality.

▶ JNMO – Les Issambres (France) – May 29th to June 1st



Nano, Micro and Optoelectronic days bring together every two years the majors actors of the scientific community involved in elaboration, physics and integration of components and nanodevices with main application in microelectronics or optoelectronic.



3 The application will be worked REALIZE Registrer on through www.renatech.org/projet YOUR and evaluated PROJECT by the reception team

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