

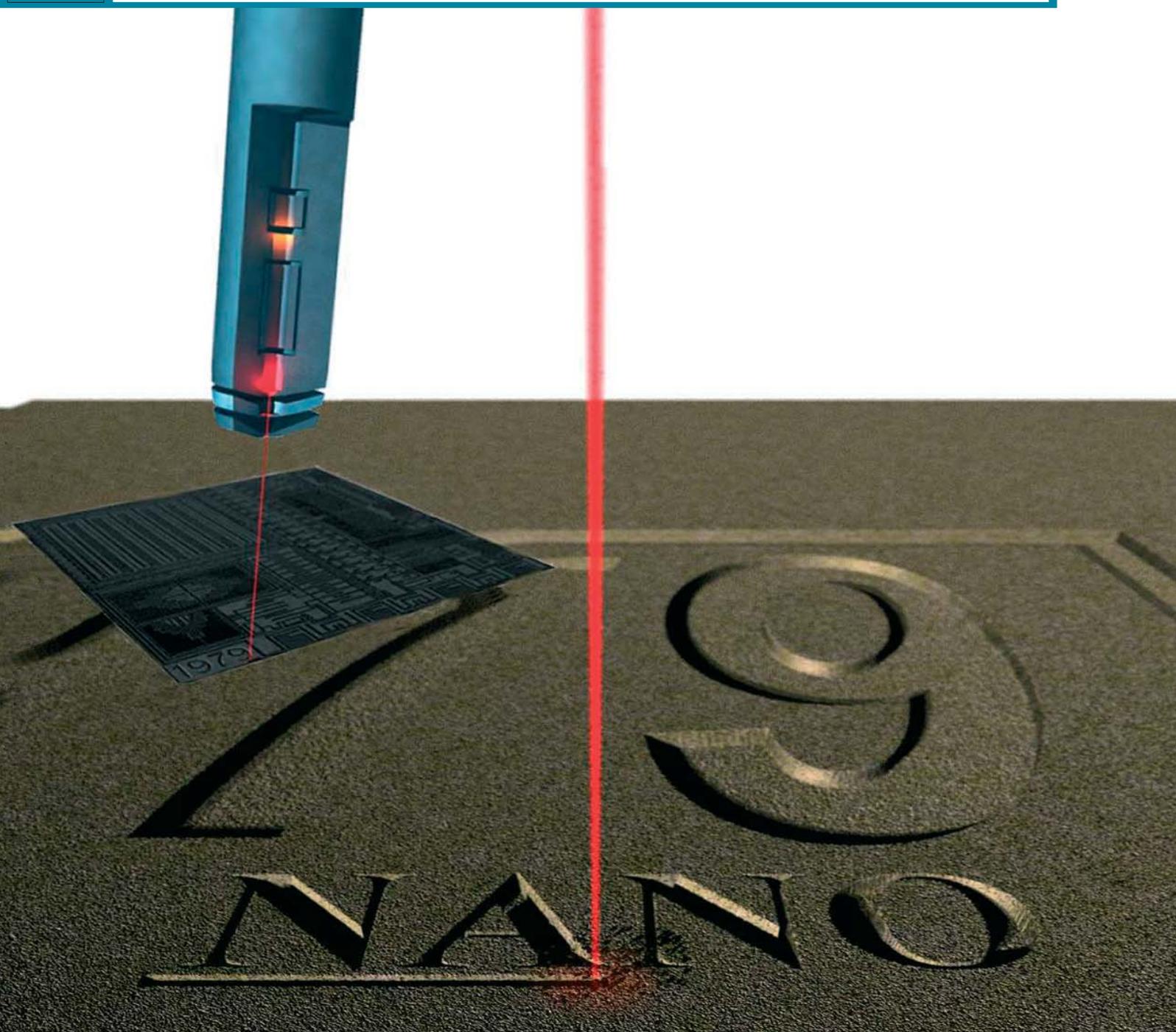


*nano*newsletter

n° 6

December 2006

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Exploring the Limits of Focused Ion Beam Technology

Pico-Inside Review on Non-Contact Atomic Force Microscopy (NC-AFM)



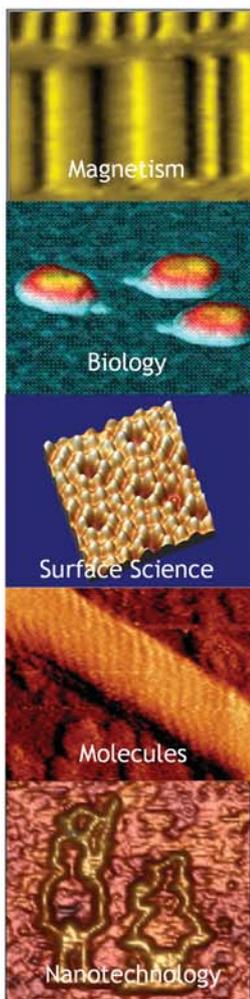
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Dear Readers:

This E-Nano newsletter issue is dedicated to present a review on Non-Contact AFM (NC-AFM) and therefore to provide a better understanding on how to image individual molecules, nanostructures, etc. on surfaces with this technique.

Patterning of materials on the nanoscale is currently one of the major challenges for Nanotechnology. The research report presents how Focused Ion Beam technology (FIB) overcomes some basic limitations of current nanofabrication techniques.

We would like to thank all the authors who contributed to this issue as well as the European Union (IST/FET/NANO) for its close collaboration. Finally, we would like to thank you, our readers, for your interest and support and wish you a Happy New Year 2007!

Dr. Antonio Correia

E *nano* newsletter Editor
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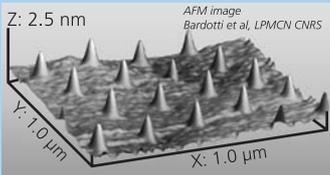
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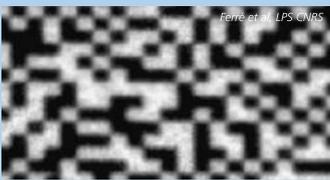
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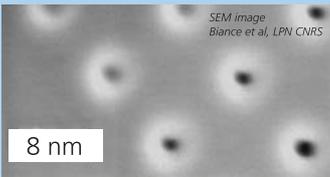
Ion beam lithography, nanofabrication and engineering workstation



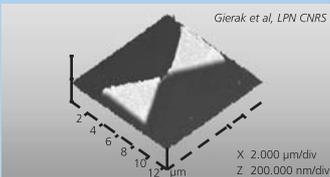
Functionalising surfaces



Thin film magnetism



Nano patterning



Gold cluster FIB patterning

a novel tool concept

ionLINE

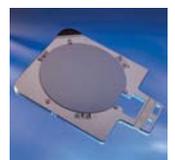
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Exploring the Limits of Focused Ion Beam Technology

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The development of tools, techniques and methods for the patterning of materials on the nanoscale is one of the major challenges for nanotechnology. In parallel with the need to develop technologies compatible with mass-production requirements, alternative tools capable to perform rapid prototyping of individual nanodevices are also required. In this article we report on our work aiming to explore the nano-structuring potential of a highly focussed pencil of gallium ions. We show that Focused Ion Beam technology (FIB) is capable of overcoming some basic limitations of current nanofabrication techniques and to allow innovative patterning schemes for nanosciences.

1. Introduction

The idea of focusing a pencil of energetic ions to define nanostructure was first mentioned by Feynman in his visionary speech "There is plenty of room at the bottom" [1]. This idea of using ions for nanofabrication was still considered as a pure dream only a few years ago and Focused Ion Beam technology was deemed unable to enter the ambitious "nano" application field. Today, thanks to the support of the European Commission through the FP5 project "NanoFIB" (G5RD-CT2000-00344) [2], we have demonstrated that a dedicated FIB system is able to compete with and even have better global performances than the most advanced top down nanofabrication techniques. This effort allowed us to demonstrate the possibility to use an ultra-sharp probe of particles to build nanostructures using a bottom-up philosophy; grow materials and structures from atomic and molecular building blocks at the level of a few atoms or single molecules.

2. The experimental set-up

2.a. Ion Source technology. Historically, the developments of Liquid Metal Ion Sources (LMIS) allowed the development of the FIB technology in the late 70's [3]. This punctual ion emitter has been shown to be a remarkably high brightness source for a large number of ionic species. But in spite of this brightness that can exceed $10^6 \text{ A cm}^{-2} \text{ sr}^{-1}$ [3], the gallium LMIS is certainly the weakest link of the chain when developing very high-resolution ion beams (spot size < 10 nm). Apart from this often-reported limitation arising from an important energy width for the emitted beam (> 4.5 eV), some more fundamental problems are apparent when this emitter is operated close to its limits. These limitations are the long-term spatial stability of the emitting point (a liquid metal jet) and electrohydrodynamic instabilities in the liquid supply. Experimentally, a LMIS has a minimum emission current, and this limits performance. A benefit of reducing the LMIS' emission current is that the energy distribution's full width at half maximum (FWHM) should fall rapidly, whilst the angular intensity

falls slowly. This was supported by the current dependence of the energy spread initially modelled with the Boersch effect. But in practice this is not verified [4].

To address these specifications we have developed and optimised a source geometry which is described elsewhere [5]. This emitter is extremely stable and allows long unattended nanofabrication processes, and allows increasing significantly the on-axis angular intensity of the emitted beam. As the beam current value I_p is proportional to the square of the beam half-angle α and to the on-axis beam angular intensity $dI/d\omega$. The final result is that this gallium LMIS' geometry allows on-axis angular intensity that may reach $80 \mu\text{A/sr}$ (compared to $20 \mu\text{A/sr}$ normally).

2.b. Ion optics design concept and properties. A dedicated FIB optics concept was developed to allow nanofabrication; i.e. patterning at a deep sub-10 nm scale. At this point it is necessary to stress out that even if such performance levels are already claimed for commercial

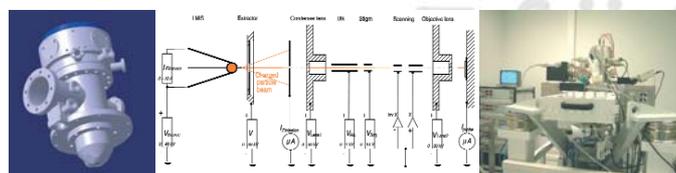


Figure 1: (a): Schematic CAD drawing of the NanoFIB column completely designed, engineered and fabricated at LPN-CNRS. (b) Internal structure and (c) View of the complete system in operation at LPN

instrument, for a non-specialist, these performances remain difficult to achieve routinely. Indeed, between the imaging mode where probe current below 1 pA are used and the patterning mode where probe current can be raised up to the nA regime, the performances seem to be quite different. The consequence is that even if high spatial imaging resolution close or below 5 nm may be obtained in the Scanning Ion Microscopy mode with extremely low probe currents, this one may remain inaccessible in the patterning mode which is the most desirable issue for any FIB user. In our case we developed an ion optics concept (Figure 1) using a patented optical architecture [7] allowing high-performance and routine generation of ion probes with FWHM diameters in the range 150 to sub-10 nm with a minimum as small as 5 nanometres. Concomitantly the rise distances in the ion probe current distribution were also kept around the same value to reduce the often-reported side defect generation (beam selectivity) [6]. A second specification was to maintain the transported current high enough by selecting a beam defining aperture size. Typically 5 μm apertures allow 5 pA in the probe (nanofabrication) and 100 μm apertures well above 600 pA (micromachining). In this FIB co-column the beam-defining aperture is placed in front of the first lens just at the entrance of the ion optics and can be selected amongst 4 to 7 different sizes. This concept allows an effective rejection of the accelerated ions having divergent or perturbed trajectories we consider as responsible for the tail generation. Here only emitted ions with paraxial trajectories can enter the optics and reach the tar-

get without loss. In this optics concept, the maximum ion energy may be increased up to 40 keV. For complete details on the performance of this high-resolution ion optics column the reader should refer to the companion paper from Hawkes and Lencová. The results are as follows: for a beam voltage $V = 40$ kV, an ion source size $d = 30$ nm, an energy spread of the beam $DV = 5$ eV, an acceptance angle $\alpha_0 = 0.1$ mrad (μm aperture) and a 6 mm working distance, we finally find a spot diameter d about 5 nm (FWHM). This performance level was verified in practice (Figure 2).

2.c. Dedicated “nano-writer” environment. We have based our instrument concept on a RAITH150 EBL writer specifically engineered to operate the UHV FIB column and a proprietary control-software suite for automated beam optimisation [7].

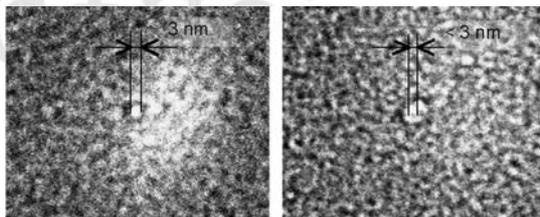


Figure 2: 50 nm x 50 nm transmission electron microscopy images showing nanometre-sized pores (bright) drilled in a 20 nm thick membrane for a same point dose $\sim 10^6$ ions.

This high performance “single beam / nano-writer” configuration was selected first to allow ultra short working distances and therefore strong demagnification mode for the ion optics. We may recall that this is a mandatory condition for any LMIS-based FIB to enter the deep sub-10nm patterning domain [8]. Second as we are aiming to fabricate objects having sizes and morphologies well below the Scanning Electron Microscope imaging threshold, we have implemented a highly accurate stage equipped with a 2-axis laser interferometer providing a physical positioning resolution of 2 nm. Using this metrology tool, navigation and pattern placement on either “as grown” or complex samples becomes extraordinary simple and reliable, allowing the system to be operated by non-experts in a true multi-user environment. In addition a three point piezo height-levelling system ensures fine control of the working distance and finally a high performance 10 MHz pattern generator governs the ion beam writing strategy allowing to complementary modes:

(i) Scanning Ion Microscopy mode and manual placement of edited patterns in single write fields for classical milling tasks (3D-nanofabrication, lamellae preparation, local deposition and etching,...) or calibration routines

(ii) Direct and automated GDSII-based exposure on chip and wafer level in single and multilayer modes. Here, dedicated auto-mark detection in combination with task-creation tools allow long run unattended batch processing fulfilling the nano-fabrication schemes that are described hereafter.

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Charged Particle Optics Theory

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The analysis of charged-particle optics systems requires accurate calculation of the potential and field distribution in the sources, lenses and other elements involved, followed by evaluation of the optical properties of these components. This second stage may be performed by exact numerical ray-tracing through the fields or by solution of a paraxial ray equation and evaluation of the aberration integrals, which are available for all the optical elements currently in use in electron and ion optics.

Such calculations provide guidance for electron and ion optical design, in which we attempt to find systems with specific properties, subject to constraints of various kinds: avoidance of high-voltage breakdown, ease of construction and alignment, accommodation of deflection system and detectors. In the NanoFIB project in particular we needed to know how to modify an earlier lens design in order to produce the smallest possible ion spot.

The critical element in a FIB instrument based on a liquid-metal ion source (Ga ions in the case of NanoFIB) is the ion gun; the improved design provided a significantly better performance yielding a beam with high brightness and small virtual source size. The source must operate at low emission current, so that its properties are not adversely affected by discrete Coulomb interactions. We have devoted much attention to the theoretical study of the properties of such sources (these are surveyed by Forbes, 2003) as well as to the simulation of discrete Coulomb interactions (Radlicka & Lencová, 2007); this work confirmed that most of the energy broadening occurs in the first few micrometres from the source, the emitting area of which is around 1 nm in radius, and furnished useful information about the trajectory displacement, which increases the virtual source size. Additional Coulomb interaction effects beyond the aperture placed above the entrance to the electrostatic lens by which the final probe current is limited do not cause any further deterioration of the probe properties, as confirmed by the well established program MONTEC/Interac (Kruit & Jansen, 1997).

In order to improve the ion-optical properties of the two-

lens system, first the program suite POCAD (van der Stam & Kruit, 1999) was used. The resulting design of the final (probe-forming) lens and the scanning system of NanoFIB were then analysed with the powerful program suites ELD and EMD (Lencová, 1997), developed in Brno and Delft. The programs for the computation of the fields are based on the finite-element method (FEM), using graded, topologically regular meshes, and they are very fast even for the large meshes needed for accurate computations. For estimation of the error in the field obtained and for identification of critical parts of the lens, a novel computation method was developed (Lencová, 2002). **Figure 1** shows the geometry of one of the probe-forming lenses investigated, the potential on the axis and several equipotentials. **Figure 2** shows the sources of any errors; these occur at mesh-points near the electrodes and are so small that their effect on the computed optical properties is negligible.

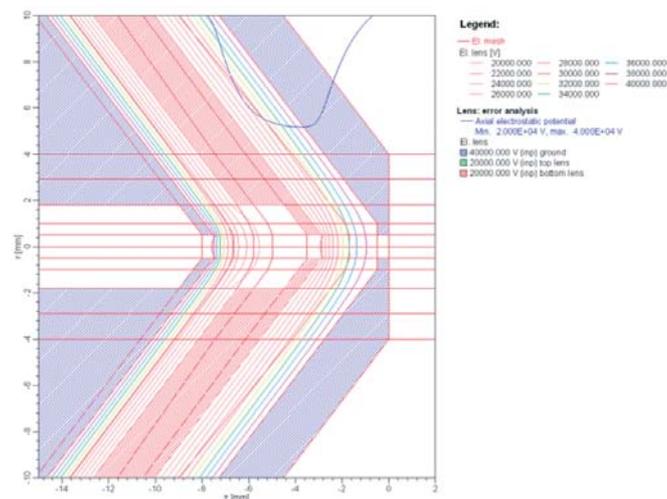


Figure 1: Geometry of the final lens, and potential on axis plus the equipotentials

The programs have recently been extensively reorganized and improved by integrating the field computation programs with Windows user interfaces and collecting the computations of optical properties into a single program, thus providing a single environment for electron and ion optical design. This new and efficient tool (EOD) was described at a recent congress on Charged-particle Optics (Lencová & Zlámál, 2006). **Figures 3 and 4** show the overall layout of the ion-optical system as a cutaway and schematically; the axial trajectories are scaled x500 and the deflected rays x50 to make them visible. From the theoretical point of view, elaborate formulae that show which elements of a multi-lens system contribute to the chromatic aberrations of electrostatic lenses were derived as part of the NanoFIB project (Lencová & Hawkes, 2004; Hawkes, 2004). Similar formulae for the geometrical aberrations had been derived earlier by Hawkes. Finally, spot diagrams, which provide a dramatic visual representation of the shape of the probe and the current distribution within it, were produced. These can be calculated very rapidly for both the

axial spot and a spot deflected away from the axis; the procedure for computing such diagrams, for the NanoFIB system in particular, was developed in Brno (Oral & Lencová, 2003).

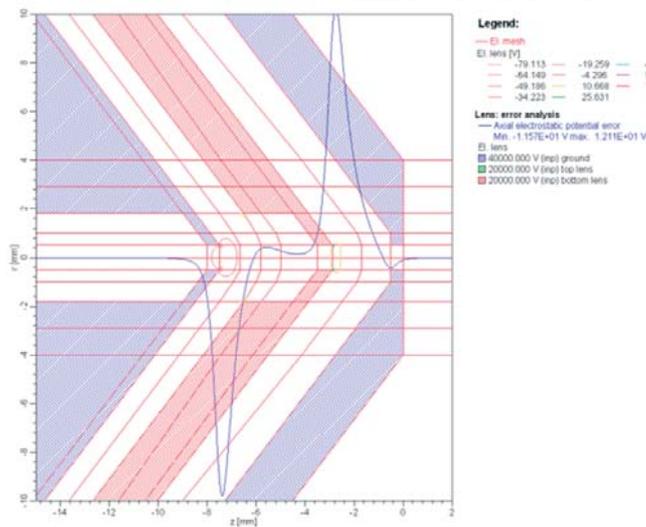


Figure 2: Error of potential – the sources of errors are at the mesh points error of this size has negligible effect on computed optical properties

Figure 5a shows the beam profile for typical values of the parameters of NanoFIB; **Figs 5b, 5c** show the current density in the beam deflected 100 μm away from the axis in the Gaussian image plane (b) and 50 μm above this plane (c). The ellipticity of the spot is a consequence of the chromatic aberration and cannot therefore be removed by focusing; nevertheless, the spot is not significantly enlarged for this deflection of more than 10000 beam diameters.

The size of the final spot d is determined by the demagnification M of the virtual spot size d_0 , the beam-semiangle α_0 , the chromatic aberration coefficient C_c , which depends on the position of the working distance (22 mm for 2 mm and 39 mm for 6 mm working distance, the value is related to the image plane), the relative energy width $\Delta V/V$, and to lesser extent on the spherical aberration (160 or 1340 mm) as

$$d^2 = M^2 d_0^2 + (C_c \alpha_0 \Delta V / V)^2 / M^2 + (C_s \alpha_0^3)^2 / M^6$$

For typical values of 30 nm for d_0 and 0.1 mrad for α_0 we find that the optimum magnification is 0.13 or 0.15, and the value of d is then 5.5 or 6.5 nm.

This value gives a somewhat pessimistic estimate of the probe size (not 3 nm diameter!); the ion distribution within the true spot must be obtained from a thorough analysis in which the spatial and angular current distribution in the source are taken into account.

The effect of spherical aberration is less important for the size estimate than the spot demagnification and the chromatic effects.

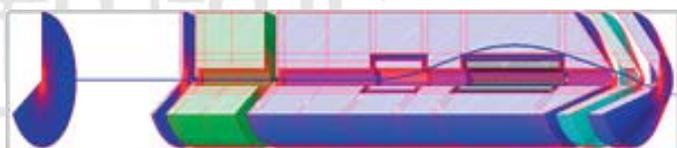


Figure 3: The overall layout of the ion optical system – with deflected trajectories of ions (increased 50x to make them visible).

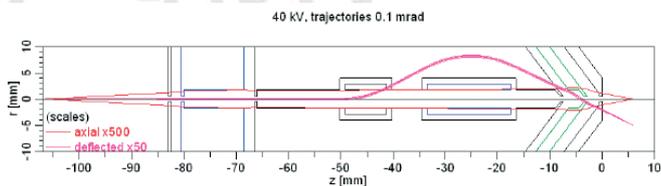


Figure 4: Plot as above – red trajectories (axial) are scaled 500x, deflected ones 50x to make them visible

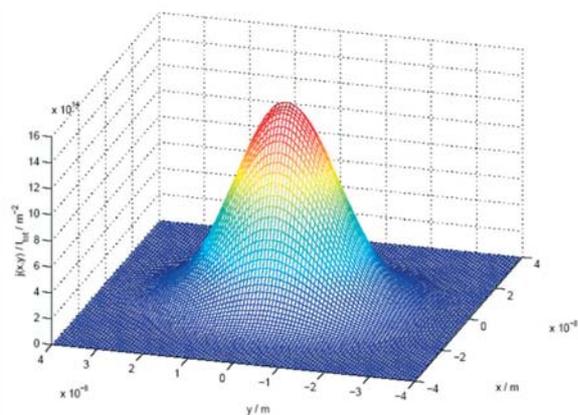
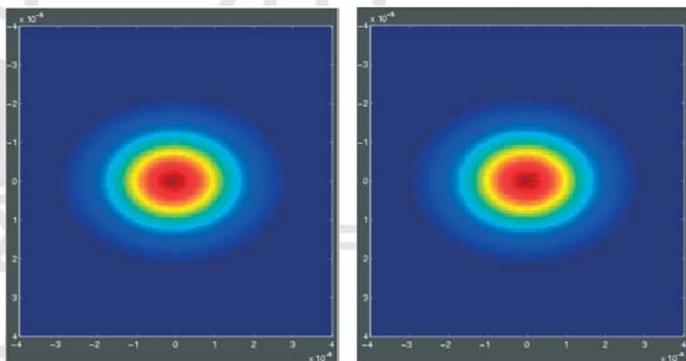


Figure 5a: Beam profile for typical parameters of NanoFIB



Figures 5b and 5c: Beam deflected to 100 micrometers away from axis – at the Gaussian image (left) and at 50 micrometers (right) above the image; the elliptical spot is a consequence of the chromatic error, so that it cannot be removed by focusing, but the spot is not significantly enlarged for this deflection by over 10000 beam diameters.

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Magnetic Nanopatterning by Focused Ion Beam Irradiation

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Introduction

Magnetic nanopatterning is a challenging area for recording and memory applications, but also for basic research on nanoelements that could stimulate future technological developments. The main goal of this note is to underline magnetic patterning with NanoFIB at low dose, that minimizes etching of the target.

Principle

Large changes of the magnetic properties of ultrathin magnetic layer structures may be driven by ion irradiation at low dose, especially with Ga ions [1]. For example, in the Pt/Co system, ion irradiation produces intermixing of Co and Pt ions at interfaces, which causes a dramatic reduction of the perpendicular magnetic anisotropy, coercivity and Curie temperature. To fix ideas, a virgin Pt/Co(1.4nm)/Pt film is a rather hard ferromagnet ($H_c \sim 300$ Oe). Under an irradiation dose, $D = 8 \times 10^{14}$ Ga ions/cm², it is transformed into a soft ferromagnet ($H_c \sim 20$ Oe), and finally becomes paramagnetic for $D > 1.5 \times 10^{15}$ Ga ions/cm² (Figure 1).

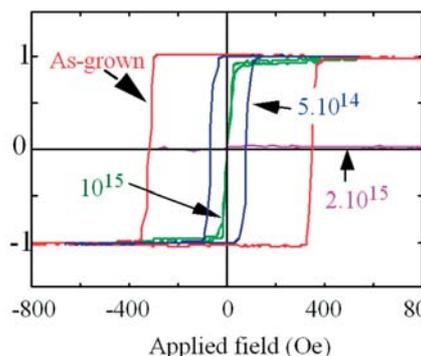


Figure 1: Pt/Co(1.4nm)/Pt film : Hysteresis loops for different uniform irradiation doses

A Co/Pt multilayer is even more sensitive to irradiation : it is ferromagnetic for $D < 10^{14}$ Ga ions/cm², its anisotropy falls in-plane for $10^{14} < D < 5 \times 10^{14}$ Ga ions/cm², and becomes paramagnetic above [2]. Magnetic nano-patterning is designed by scanning the focused Ga ion spot on the film (Figure 2). At low dose, the etching depth of the Co/Pt target by Ga is linear with D and calculated to be 1.3 nm for $D = 10^{15}$ Ga ions/cm². Even at that dose, the Co layer is not etched and changes of magnetic properties come only from the chemical ion intermixing at interfaces.

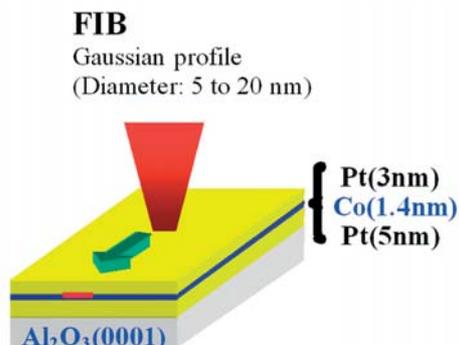


Figure 2: FIB scanning principle

Magneto-Optical Microscopy (MOM) as a tool for checking irradiation damage

As a test, the patterning of a Pt/Co/Pt film as an array of parallel NanoFIB lines can be rapidly checked by MOM (Figure 3) [3], even for very small doses ($D = 2 \times 10^{13}$ Ga ions/cm², i.e. ~ 20 ions/spot for a spot diameter of 10 nm), while scanning electron or atomic force microscopy observations fail. This comes from the fact that there is no etching nor the formation of nanobumps at extremely low dose. In counterpart, MOM check sensitively tiny intermixing effects.

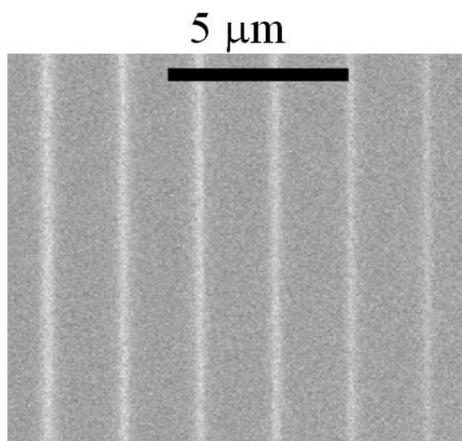


Figure 3: MOM image of an array of 50 nm wide NanoFIB lines with increasing dose (from right to left)

Results

Arrays of magnetic dots and tracks : the role of dipolar interactions

Magnetic arrays of separated narrow tracks or small dots have been designed by NanoFIB patterning at low dose. These squares or elongated nanoelements were respectively separated by a set of parallel or orthogonal NanoFIB irradiated lines with narrow width (5 to 20 nm). Typical demagnetized states are shown in Figures 4, 5.

The dynamics of the magnetization reversal in these dipolarly coupled elements has been extensively studied [4].

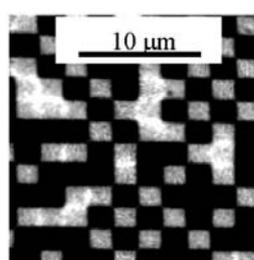


Figure 4: MOM image of a demagnetized Pt/Co (1.4nm)/Pt square dot array

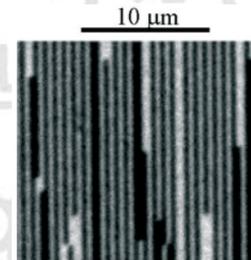


Figure 5: MOM image of a demagnetized Pt/Co(1.4nm)/Pt track array (370 nm)

Due to the sharp gaussian NanoFIB profile, the coercivity is reduced at the element's edges. As a consequence, nucleation takes place in these softer regions so that the magnetization switching in elements is controlled only by the internal domain wall propagation field H_p , known to be uniform in perfect films. Thus, NanoFIB stands as a unique tool to design large arrays of nanoelements with identical magnetic switching properties. This might have a beneficial impact on the design of future discrete recording media and memories. We demonstrated that Pt/Co(1.4nm)/Pt nanodots are still ferromagnetic for sizes down to 70 nm. This is encouraging to design ultra-high density recording media up to the superparamagnetic limit.

Competition between exchange and dipolar interactions

NanoFIB patterned lines at very low dose cannot fully isolate the designed magnetic nanodots. The magnetic interaction (from parallel to anti-parallel) can be manipulated by increasing the dose in lines, that induces a transition from a collective to a more individual behaviour (Figure 6) [5]. This can be applied to realize magnetic logic devices. Isolated type of elements (Figure 7) may be also designed for targeted fundamental research.

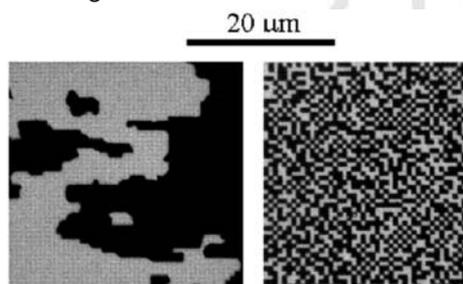


Figure 6: MOM images of demagnetized Pt/Co(1.4nm)/Pt square (1μm x 1μm) dot arrays when increasing the dose in nanoFIB lines (from left to right)

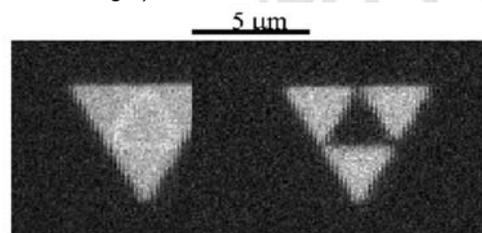


Figure 7: MOM images of the field-induced magnetic states of coupled nanostructures (NanoFIB lines separate grey and black states)

Towards magnetic nano-circuits

The realization of single magnetic narrow nanotracks may be obtained between two close parallel lines. So, information can be transmitted by fast wall motion in such nanotrack; this is favored thanks to an irradiation-induced wetting effect at track edges (**Figure 8**) [6].

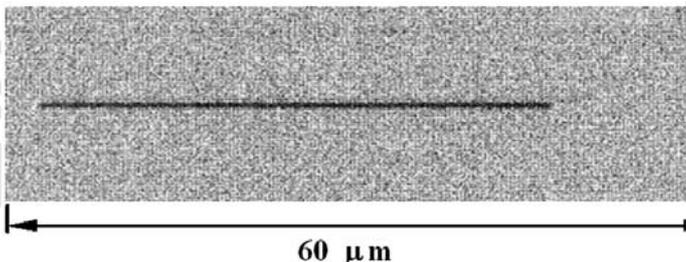


Figure 8: MOM snapshot of the domain wall motion in a narrow (450 nm wide) track designed by NanoFIB

Applications

Ultra-high density discrete magnetic recording. Design of magnetic recording heads. Fast nanomagnetic memories. Magnetic nanosensors. Nanomagnetic logic

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Mixing Top Down and Bottom up Approaches for the Fabrication of 2D Arrays of Nano-Sized CoPt-Magnetic Dots

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Introduction

The design and fabrication of quantum dots systems and the study of their properties, play an increasing role directly related to the large number of potential applications, such as for example high-density recording. To produce an ideal device that would consist of a periodic arrangement of monodispersed quantum dots on an appropriate substrate, we have developed a new approach based on surface defect generation by nanoFIB technology followed by the deposition of pre-formed nanometre-sized clusters on patterned surfaces. The originality of this approach lies in the fact that cluster formation and substrate functionalisation are completely independent.

Principle

Samples are prepared by depositing Co₅₀Pt₅₀ magnetic clusters (2 nm mean diameter) in a soft landing-regime using the LECBD-technique (Low Energy Cluster beam Deposition) [1], onto functionalized HOPG-substrates with

2D-organized arrays of nanodefects. Such defects act as nucleation centres for the deposited clusters which diffuse on the HOPG-surface leading to the formation of stable 2D-organized cluster-assembled dots [2]. Topographic images are obtained using tapping mode AFM (TMAFM) and scanning tunnelling microscopy (STM).

Results

NanoFIB patterning of the HOPG surface

The basic pattern design is a matrix of points (dots) defined with a fixed step size and a constant point dose. The dependence of FIB-defect morphologies on the ion dose has been extensively studied [2]. Nanocraters for high ion-dose ($\geq 5 \cdot 10^4$ ions/point) and nanobumps for low ion-dose ($\leq 10^4$ ions/point) are systematically observed.

Figure 1 exhibits the morphologies of defects for two different ion doses. **Figure 2** shows STM images of defects presented in **Fig. 1 a)** and **b)**.

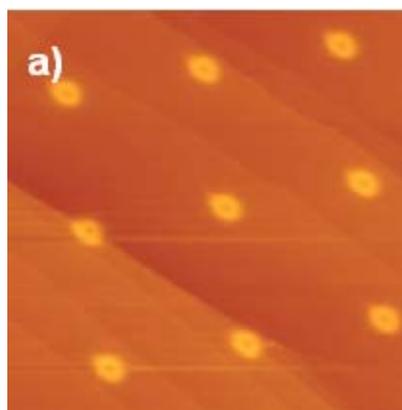


Figure 1a: 2,5 μm x 2,5 μm TMAFM image of FIB patterned nanocraters in HOPG (105 ions/point), step 1000 nm.

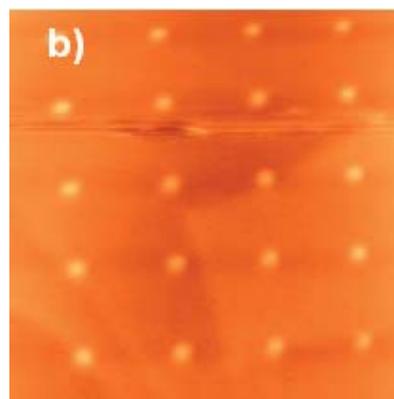


Figure 1b: 1,2 μm x 1,2 μm TMAFM image of FIB patterned nanobumps in HOPG (104 ions/point), step 300 nm.

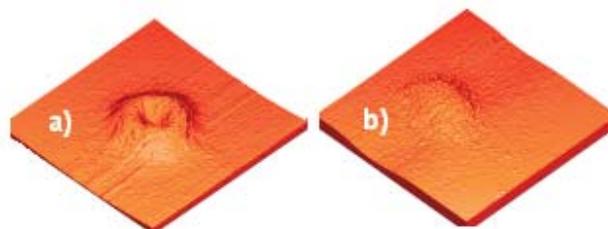


Figure 2: 3D STM images of defects a): nanocrater in HOPG (105 ions/point), ($z_{\text{max}}=5$ nm) b): nanobump in HOPG (104 ions/point), ($z_{\text{max}}=1$ nm).

Clusters organization

TMAFM-image of 0.2 nm thick $\text{Co}_{50}\text{Pt}_{50}$ clusters film deposited onto a FIB-patterned HOPG substrate (10^4 ions/point and step =300 nm) at 573 K is presented in **Figure 3a**. The diffusion of clusters and their sensitivity to ultra-small FIB-defects (nanoprotrusions) lead to the formation of 2D-organized arrays of cluster-assembled dots. To improve the quality of the organisation on the HOPG-surface, a post-annealing in UHV at 973 K is performed (fig.3b). Such a treatment increases the magnetic cluster anisotropy by inducing a phase transformation of the supported clusters from the disordered face-centered-cubic (fcc) structure to the L1_0 -ordered face-centered-tetragonal structure (fct) [3]. A positive effect of annealing on the organization is clearly visible **Figure 3b**.

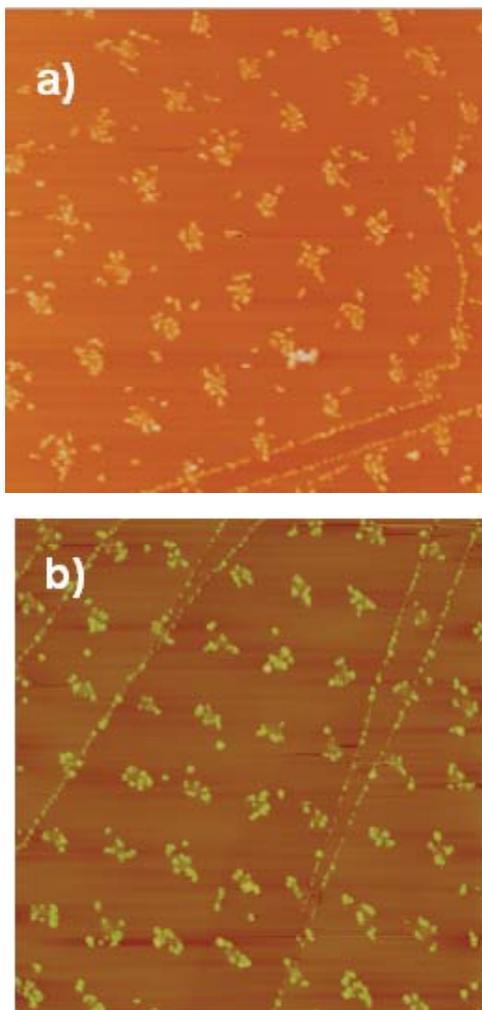


Figure 3: TMAFM images ($2\ \mu\text{m} \times 2\ \mu\text{m}$) of a 0.2 nm $\text{Co}_{50}\text{Pt}_{50}$ film deposited at 573 K on FIB-patterned HOPG surfaces presented in figure 1b.

a) before heat treatment
b) after annealing at 973 K for 2h.

Conclusion

These results show the promising direction of organisation process achieved using a technique based on the deposition of preformed clusters on functionalized patterned surfaces using a very high resolution focused gallium ion beam. We have shown that trapping effects of preformed clusters on point defects allow the possibility to organise

nanoparticles in arrays. This is of major interest for future applications to nanoelectronics, nanomagnetism or nanooptics.

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Selective Epitaxy of Patterned III-V Semiconductors Surfaces Using Raith ionLiNE's nano-FIB Technology

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Introduction

New photonic materials are key progress enablers in a wide area of optical communication, data-storage and processing, as well as in sensing, display and lighting technologies. The discrete nature of the electronic states of $\text{In}(\text{Ga})\text{As}/\text{GaAs}$ quantum dots (QD) is very promising for the realisation of high performance optoelectronic devices [1]. The control of the QD nucleation using FIB textured substrates could allow an increase of their density as well as a reduction of their size dispersion, and thus leading to a considerable improvement of the related laser performance. The control of the QD size dispersion could also facilitate the fabrication of broad band emitters or amplifiers for telecommunications applications.

Principle of operation

We used our nano-FIB technology of ionLiNE system to pattern nanostructures into a SiN_x thin film layer to allow subsequent localised epitaxial regrowth. This technique is of high interest since the feature size is very small and has the potential to allow the localised growth of quantum dots. The technique we have developed is similar to lithography with a thin SiN_x layer of 10 nm deposited on semi-insulating InP substrates.

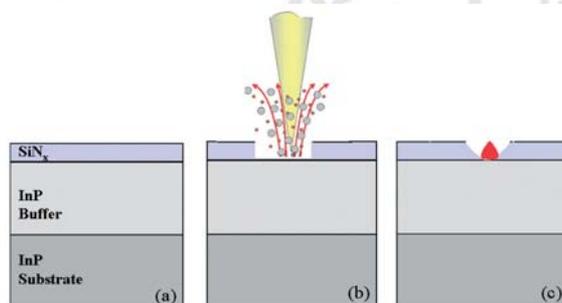


Figure 1: Schematic representation of the main processing steps used for the selective epitaxy experiments. (a) SiN_x mask deposition on the InP substrate, (b) nano-patterning of the SiN_x masking layer and (c) epitaxial re-growth using the patterned mask to localize the growth.

The SiN_x thin film used as a sacrificial mask was then patterned with our FIB system and the operating conditions are the following Ga⁺, 35 keV, probe size 8 nm FWHM, probe current 6 pA. The patterns were matrix of dots having different dose and feature geometries. **Figure 1** schematically shows the main processing steps.

We concentrated our investigations on the realisation of low dimension dots. After the FIB patterning Metal-Organic-Vapour-Phase-Epitaxy (MOCVD) using an EMCORE D125 reactor was used to regrow InAs within the patterned holes. The growth was carried out at a pressure of 60 Torr and at a temperature of around 500°C. The growth conditions – mainly the temperature and the flow of the precursors – have been carefully chosen to obtain a very low growth rate. **Figure 2** show the structures for a same periodicity and different doses leading to different hole sizes after the regrowth process within the nano-engraved circular openings.

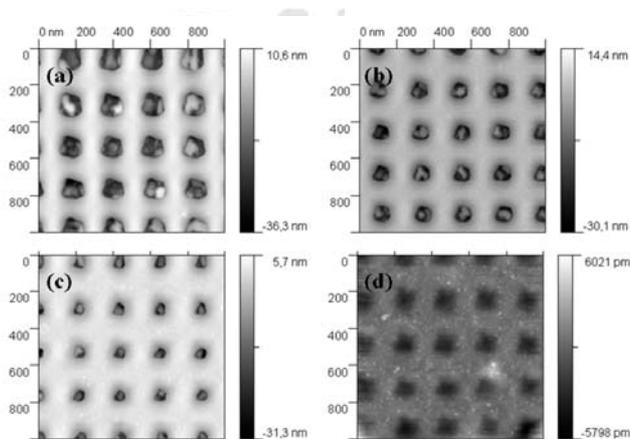


Figure 2: Atomic Force Microscopy images of grown structures for different nano-FIB-drilled holes (a) 50 ms/dot, Depth 29 nm, (b) 20 ms/dot, Depth 23 nm, (c) 30 ms/dot, Depth 29 nm, (d) 10 ms/dot, Depth 4 nm

On **Figures 3a & 3b** the SiN_x mask was removed by HF prior to the observation in a scanning electron microscope. A homogeneous growth within the patterned areas can be observed and those results are very promising in respect to the objective of growing localised quantum dots [2].

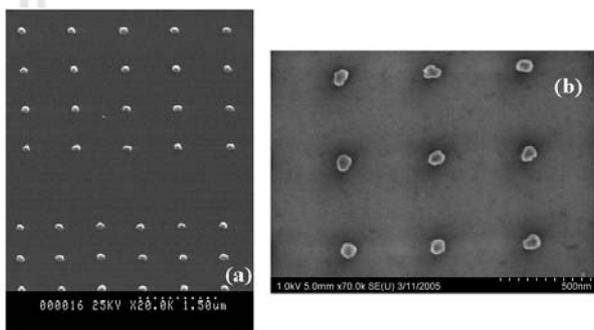


Figure 3: (a) Scanning electron micrograph of the nano-FIB patterned field with nanostructures grown and (b) close-up on a dense field of round features. Both close-ups show a homogeneous and well-localized growth.

Conclusions

We have demonstrated that direct FIB nano-patterning of highly crystalline III-V substrates allowed localised growth of InAs dots on either on GaAs or InP substrate. Small features organised in relatively large arrays were fabricated providing the first steps towards the creation of localised QDs.

FIB Technology Applied to the Improvement of the Crystal Quality of GaN and to the Fabrication of Organized Arrays of Quantum Dots.

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Introduction

The objective of this work is oriented towards the improvement of the ELO technology; Indeed a prerequisite of the improvement of nitride based laser diodes in life time and output power is the procurement of high quality substrates, with threading dislocation densities lower than 10⁶cm⁻². Actually, the epitaxial lateral overgrowth (ELO) has proven to be an efficient technology to reduce the density of TDs in the low 10⁷cm⁻² [3, 4]. Further reduction of TDs densities by ELO requires multistep processes and highly sophisticated mask designs.

Fabrication of organized arrays of QDs

The GaN/AlGaIn and GaN/GaN heterostructures have attracted considerable attention as candidate for novel quantum devices. The fabrication of microstructures as QW and QDs is technologically not straightforward. The selective epitaxial growth allows to fabricate such quantum objects. Basically the QW sot QDs are growth on the top of a truncated triangular stripes (QWs) or pyramids (QDs). However the size of the QWs or QDs needs to be further reduced for practical applications. It is expected that the FIB technology will provide a new generation of quantum devices.

We have demonstrate the possibility of achieving selective epitaxy and Epitaxial Lateral Overgrowth (ELO) using nanometric openings patterned with a Focused Ion Beam (FIB). The test structure we used in this work consists of an AlN buffer layer on 6H-SiC with very high crystal quality close to pseudomorphism. This substrate is first covered with a thin (~ 2 nm) silicon nitride mask deposited by CVD. Then our high resolution FIB system is used to create regular openings with a range of sizes (lowest 100 nm) and organisation.

Finally GaN was grown on the structure by Metal-Organic Vapour Phase Epitaxy MOVPE). The relatively low ion dose (2.7 10¹⁶ ions/cm²) required to remove the very thin surface layer allows reasonable patterning speeds (0.45 ms/pt) and minimises FIB induced defect extension in the vicinity of the patterned GaN structures. After this selective epitaxy of GaN pyramids selectively grown on AlN/6H-SiC was observed (**Figure 1**).

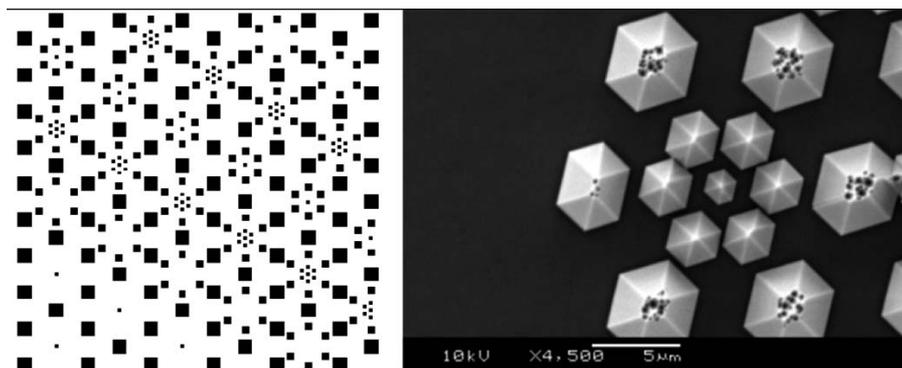


Figure 1: Schematic of a pattern and SEM picture of structures selectively epitaxially grown of GaN on AlN/6H-SiC on openings made on SiN₄ by FIB (Ga⁺ 35 keV). Note that for the largest opening, the selective epitaxy is not fully terminated.

Finally in order to check the quality of the selective growth process Transmission Electron Microscopy (TEM) investigations were performed on thinned pyramids. As expected the TEM images obtained (**Figure 2**) have revealed that, and even though the lateral regrowth occurred properly, the AlN layer underneath became somehow amorphous. This is most likely due to damages created by the incident Ga beam. Therefore, annealing need to be made to recover the AlN buffer crystallinity. However, it is worthwhile mentioning that even though the AlN layer underneath the overgrown region became amorphous, a fully coherent array of aligned pyramid was obtained [5].

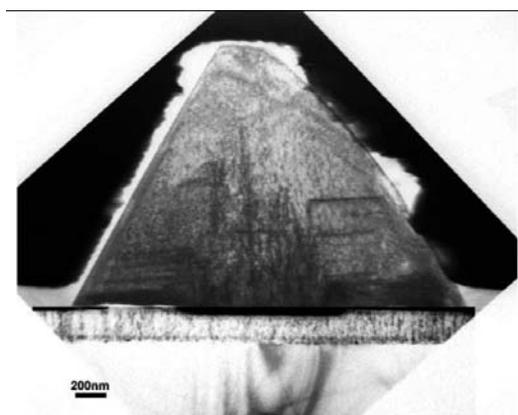


Figure 2: High Resolution Transmission Electron Microscopy image of a thinned pyramid showing the threading dislocations (TDs), from the AlN buffer, bend at 90° when they encounter the {11-22} growing facet (dark lines).

Conclusion

We have proposed and demonstrated a new method in Epitaxial Lateral Overgrowth (ELO) at the nanometric scale. This new ELO technology based on FIB processing has been demonstrated, which, however needs further research effort. This effort is currently continuing. This new method is very promising with reference to the improvement of the Epitaxial Lateral Overgrowth (ELO) technology and the fabrication of organised arrays of QDs.

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Nanoengraved SiC Membranes with FIB Technology

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Introduction

Nanoengraving of membranes as a template for nanopores and nano-masks fabrication is an application field of growing interest. Obviously there is a considerable application potential for such nano-sized holes or nano-pores. Indeed numerous applications were recently reported aiming to use such membranes as stencils or masks to grow or deposit nanostructures [1], to localise molecular scale electrical junctions, switches and nano-transistors or spin-injection devices [2, 3]. Our goal is to use such patterned membranes for molecular biophysical applications such as optimized filter for DNA separation or electrical sensors for single biomolecule electrical detection [4].

Principle of operation

FIB nanoengraving of a membrane is interesting if the membrane can be made homogeneous, conductive and thin enough; i.e. with a thickness comparable to the projected range of the incoming ions. We used home-fabricated silicon carbide membranes due to the excellent properties of this material that can be deposited as thin amorphous layers with good uniformity. This material allows the dissipation of electrical charges transported by the impinging ions. Under optimised conditions a very low surface roughness (typically some Å°) is obtained. In addition when the SiC film is under an optimised tensile stress, this allows the whole composite layer to remain flat and to be made self-supporting as depicted on **Figure 1**.

The optimised stress value brings us the ability to make the nano-machining of the membrane possible. A 300 μm x 300 μm opening is patterned with photolithography on the backside of the wafer allowing to etch the silicon from the back side in TMAH solution.

One limiting parameter in this experiment is when attempting to obtain a nano-pore is the membrane thickness itself. Only the use of a membrane around or below 20 nm in thickness allowed us to reduce the pore diameter below the 10 nm range. These membranes were drilled with a 35

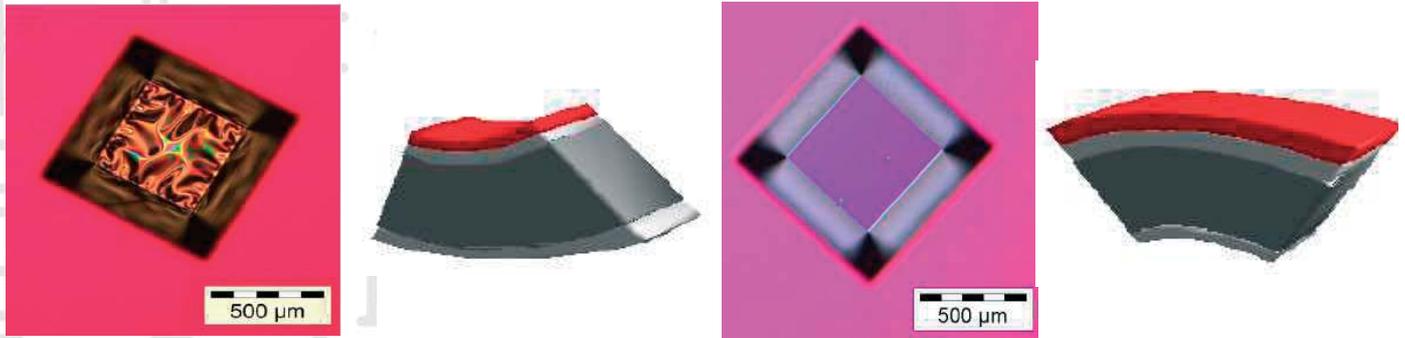


Figure 1: (a) View of a crumpled SiC membrane (central window) due to low stress (b) detail of a similar SiC membrane under correct tensile strength.

keV Ga⁺ beam, focused within a 5 nm FWHM probe that is transporting around 2 pA. The irradiation pattern was a matrix of dots with the point dose varying continuously between 10 ms to 200 ms within a single writing field, giving respectively point dose 2.5 · 10⁶ up to 5 · 10⁷ ions/point. After the FIB drilling process the membranes were observed by TEM (transmission electron microscopy - Philips CM20-200 kV). A specific methodology was developed (Figure 3) with the calibration of the critical dose allowing to obtain open nanopores (3a) for a given membrane batch. Then an array of nanopores was drilled with a dose varying +/- 10% around the previous calibrated dose (3b & 3c). Each nanopore was then inspected in a TEM and a quick localisation of these nanopores was made possible using the alignment cross we added around the nanopores (3d). These structures irradiated with a very low surface dose were found to modify only the surface roughness of the membrane.

The **Figure 4** shows some of the smallest nanopores we have defined. The dose necessary to drill such nanopores

stands around 1 · 10⁶ ions/point. The evidence of opening is made by the TEM observation of a white Fresnel diffraction annulus around the white spot (**Figure 4**).

Conclusions

In this work we have successfully demonstrated the possibility to apply FIB technology for the direct fabrication of nanopores having pore diameters as small as 3 nm. This result is defining a novel state-of-the-art for direct removal of material using a FIB probe as opposed to sculpting methods. We are currently planning to use these devices for electrical transport measurements and molecules trapping.

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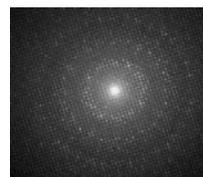
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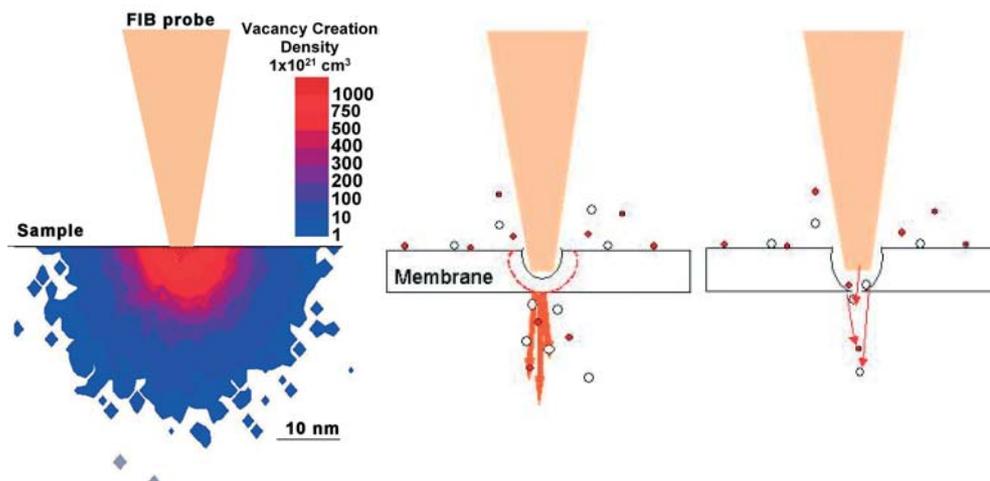


Figure 2: TRIM simulation of the interaction of a 35 keV Ga⁺ beam with a target showing the damage localisation. Schematic of the membrane engraving processes: entry side sputtering, rear side stamping and forward scattering along the wall of the nanopore.

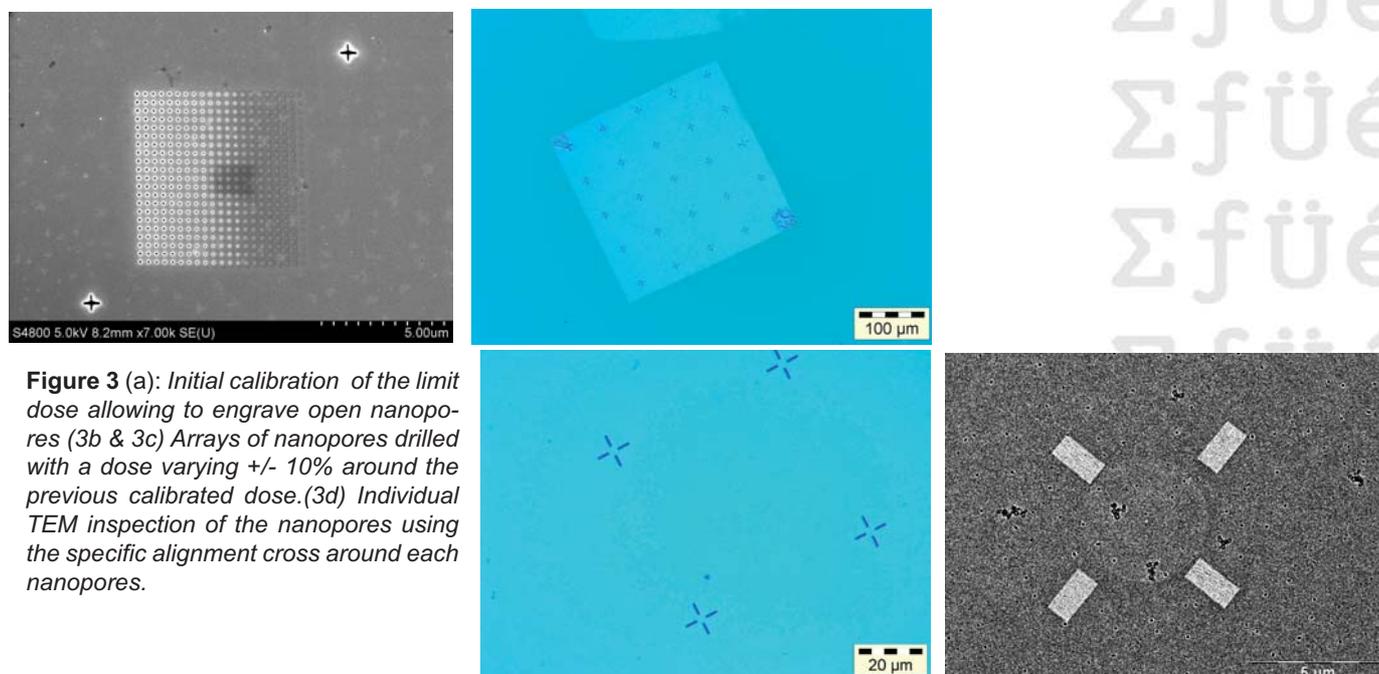


Figure 3 (a): Initial calibration of the limit dose allowing to engrave open nanopores (3b & 3c) Arrays of nanopores drilled with a dose varying +/- 10% around the previous calibrated dose. (3d) Individual TEM inspection of the nanopores using the specific alignment cross around each nanopores.

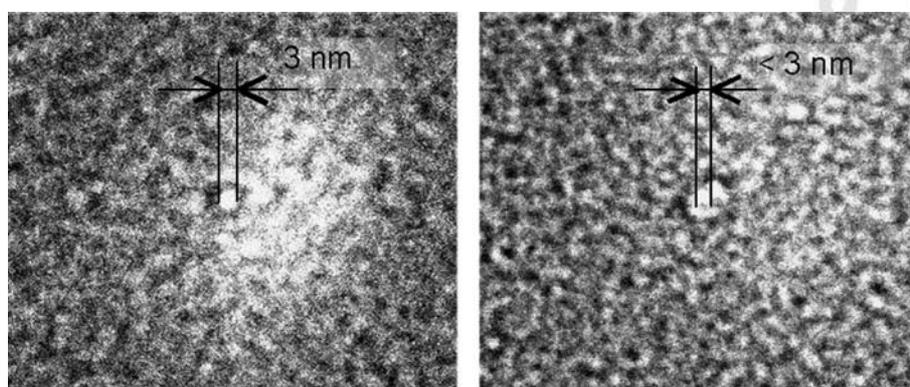


Figure 4: 50 nm x 50 nm transmission electron microscopy images showing nanometre-sized pores (bright) drilled in a 20 nm thick membrane for a same point dose $\sim 10^6$ ions.



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“Modelling for Nanotechnology” (M4nano) is a WEB-based initiative led by four Spanish Institutions: **Phantoms Foundation**, **Parque Científico de Madrid (PCM)**, **Universidad Autónoma de Madrid (UAM)** and **Universidad Complutense de Madrid** to maintain a systematic flow of information among research groups and therefore avoid that research efforts in Nanomodelling remain fragmented.

Networking is vital to any scientist and even more so for persons working in the inter-disciplinary field of Nanotechnology and in particular Nanomodelling. To fulfil this necessity, **M4nano** will provide a comprehensive guide -“who’s who”- of groups working in Modelling at the nanoscale listing their accomplishments, background, infrastructures, projects and publications. In this way, **M4Nano** completes the aim of both raising awareness of scientists in Nanotechnology modelling issues and aiding them in developing beneficial collaborations and employment opportunities.

Emerging research areas such as Molecular Electronics, Biotechnology, Nanophotonics, Nanofluidics or Quantum Computing could lead in the mid-term future to possible elements of nano-based devices. Modelling behaviour of these possible nanodevices is therefore becoming more and more important and should allow to: (i) Visualise what happens inside a device (ii) Optimise the devices under study (iii) Improve understanding of device properties (physical, chemical, etc.).

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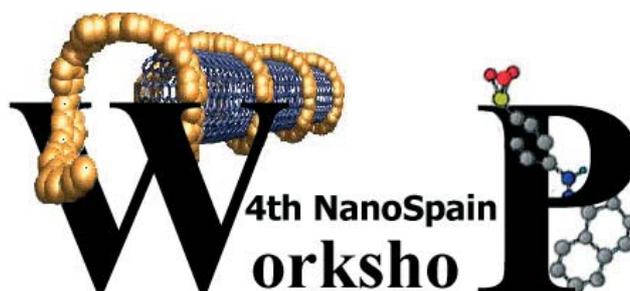
Information spreading will also be enhanced using mailing list alerts, press releases and flyers. Collaborations with similar initiatives such as the **NanoHub** (USA) or **Icode** (Italy) will also be set-up.



For more Information contact to:

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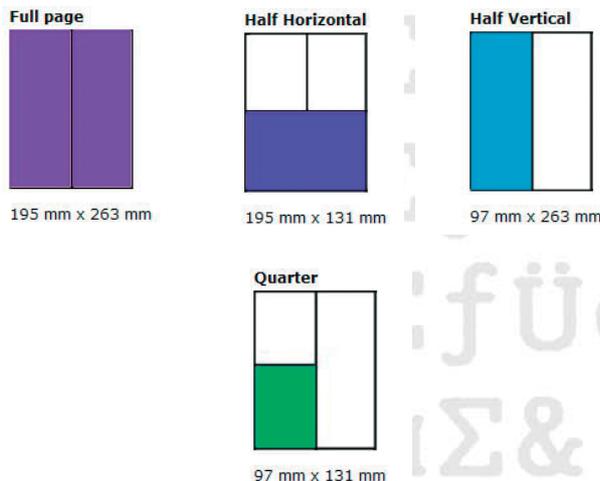
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Post-doctoral Position (CEA, Grenoble, France): "Porphyrin Chemistry"

In the framework of a project involving three groups of the CEA-Grenoble, a memory with redox active molecules as storage elements is under development. This project deals with the problems arising from the miniaturisation of micro-electronic components. For mid-term applications it seems that "hybrid electronics" devices could be promising. Such devices are still based on a CMOS technology but they are also associated to other concepts such as the use of nanowires or conducting polymers as channel.

Please send a letter or e-mail with full C.V. (including list of publications and contact information of two references) and a covering letter to:

Florence Duclairoir, DRFMC/SCIB/RI2C, CEA-Grenoble, 17 rue des martyrs, 38054 Grenoble cedex 9, France
florence.duclairoir@cea.fr

Post-doctoral Position (CEA-LETI, Grenoble, France): "Dynamic integrated near field tools for organic and molecular electronics"

The development of advanced forms of near field techniques, which combine under ultra-high vacuum (UHV) the functionalities of imaging, nano-structuring, addressing and electrical characterization, holds the promise of key-advances both for fundamental and technological research (studies of nano-objects physical properties in ultra-clean conditions, development of new nanocharacterization, nano-patterning and nano-manipulation tools). Such techniques are particularly suitable for research in the field of molecular and organic electronics. To probe the intrinsic electronic properties of organic materials [B. Grévin & P. Rannou, Nature Materials 3, 503 2004], the in situ electrical connection of nano-objects evaporated under UHV conditions is indeed highly desirable.

Further information about the position can be obtained from Dr M. Brun mickael.brun@cea.fr +33(0)4-3878-3505 or Dr B. Grévin, benjamin.grevin@cea.fr, +33(0)4-3878-4615, fax +33(0)4-3878-5113.

Ph.D. Position (DRFMC/SPrAM CEA-GRENOBLE, France): "Scanning probe microscopy (LC-STM/STS, FM-AFM/NC-AFM) investigations under UHV of model self-organized pi-conjugated molecular and macromolecular architectures"

This PhD, in the frame of molecular and organic electronics, will be devoted to study the local conformation, self-organization and electronic properties of pi-conjugated oligomers and polymers on reference surfaces by scanning tunnelling microscopy (STM), scanning tunnelling spectroscopy (STS) and non contact atomic force microscopy (NC-AFM) techniques under ultra-high vacuum (UHV). The materials investigated will be selected as "model systems", either for their self-organization and electronic properties. One of the main goals is to achieve sub-molecular resolution in both STM and NCAFM operation on self-organized monolayers of oligomers and polymers, or on pi-conjugated nanofibrils and nano-wires deposited or nano-structured on conducting, semi-conducting and insulating reference surfaces.

Further information about the position can be obtained from Dr B. Grévin, benjamin.grevin@cea.fr, +33(0)4-3878-4615, fax +33(0)4-3878-5113 or from Dr P. Rannou, patrice.rannou@cea.fr +33(0)4-3878-2749.

Post-doctoral Position (ENSCP - Paris, France): "New Er doped crystals for optical information processing"

Rare earth ion doped crystals are well adapted to optical information processing. Applications include optical processing of wideband radiofrequency signals and quantum information processing. Erbium is particularly interesting because of its optical transition in the telecom window and its long coherence time. Besides, its magnetic properties make it attractive quantum information manipulation.

New crystals are being investigated in order to optimize the properties required for the applications. The project includes the characterization of these new crystals using EPR optical coherent spectroscopy and their use in original optical processing experiments.

Applications should be e-mailed to: Dr. Philippe Goldner – E-mail: philippe-goldner@enscp.fr

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Ph.D. Position (Universidad de Granada - Granada, Spain): *"Research on synthesis and characterization of new bimetallic bifunctional nanoparticles"*

Preparation of ferritin-based metallic nanoparticles: Why ferritin?

- 1.- Size and shape control of the particles
- 2.- Protection against agglomeration
- 3.- Facilities for implementation: a).- direct applications, b).- as precursor of nanostructured materials

Contact: José M. Domínguez-Vera - Tel.: +33 958 248097; E-mail: josema@ugr.es

Post-doctoral Position (CEA/Saclay - Gif-Sur-Yvette, France): *"Innovative chemistry for the preparation of resonant tunneling molecular diodes connected by carbon nanotubes"*

Molecular electronics aims at using individual molecules or small groups of organized molecules as the active part of electronic devices. It takes advantage of the size, the diversity, the quantum properties and the self-organization properties of organic molecules. Two central issues of Molecular Electronics are the development of new efficient molecular devices and their connection to the macroscopic world. In this framework, the goal of the present project is to develop circuits based on molecular resonant tunneling diodes on silicon. To achieve such a goal, carbon nanotubes will be used as electrodes to locally connect small numbers of molecules organized on silicon. Indeed, nanotubes present several advantages for such an utilization, in particular, their small diameter (~ 1nm), high conductivity and exceptional mechanical properties.

Applications should include a detailed CV, a list of publications and the names and contact details of two referees.

Contact : Bruno Jousselve – E-mail: bruno.jousselve@cea.fr

Post-doctoral Position (ICN - Barcelona, Spain): *"Nano-optics"*

The nano-optics group at Institut Catala of Nanotechnology invites applications for one post-doctoral research position. Areas of interest include: nao-optical devices, subwavelength structures for light propagation and confinement, electronic interaction in nanostructures, and modification of the emission properties of nanostructures.

Please send your applications including CV, references and a statement of research plans to: icn@uab.es.

Ph.D. Position (CIDEMCO - San Sebastian, Spain): *"First-principles simulation of semiconductor nanocrystals - photovoltaic applications"*

The Energy Efficiency Area at CIDEMCO, Technological Research Centre is seeking for a PhD student focused on ab initio simulations of semiconductors for photovoltaic applications.

CV's should be sent to:

Dr. Maider Machado

Pol. Ind. Lasao, Área Anardi 5 - 20730 Azpetia (Spain)

maider.machado@cidemco.es

Researcher (LABEIN-Tecnalia - Derio, Spain): *"Nanotechnology researcher for the Center for Nanomaterials Applications in Construction"*

Tecnalia is seeking to recruit a Nanotechnology Researcher.

LABEIN-Tecnalia is a private, non-profit research centre and is part of the TECNALIA Corporation. It is located in the Basque Country, in Spain. The mission of LABEIN-Tecnalia is to support enterprises and administration bodies in their research and innovation needs by means of research, development and innovation projects as well as in technology transfer, technological services, training, and dissemination activities. For further information about LABEIN-Tecnalia see <http://www.labein.es>

Candidates should send the C.V. and salary expectations to:

LABEIN-Tecnalia, P.O. Box 1234, 48080 Bilbao (Spain), attn. RR.HH.

Or via the webpage: <http://www.labein.es> mentioning the reference: NAN (LABEIN-Tecnalia - Derio, Spain)

NANO Conferences - <http://www.phantomsnet.net/Resources/cc.php>

(January 2007)

▣ **The 1st European Topical Meeting on Nanophotonics and Metamaterials**

January 8-11, 2007. Seefeld ski resort, Tirol (Austria)

<http://www.nanometa.org/>

Nanophotonics, Theory & Modeling

▣ **Spanish Molecular Electronic Symposium**

January 19, 2007. CNRS - Delegation Midi - Pyrenees. Toulouse (France)

<http://www.phantomsnet.net/SMS2007>

Molecular Electronic



(February 2007)

▣ **Advances in Single-Molecule Research for Biology & Nanoscience**

February 2-5, 2007. Linz (Austria)

<http://afm.tm.agilent.com/linz2007.html>

Nanomedicine, Nanobiotechnology

▣ **Nanoelectronics - materials and technology**

February 14, 2007. London (UK)

http://www.iop.org/Conferences/Forthcoming_Institute_Conferences/event_6521.html

Nanoelectronics

▣ **International Nanotechnology Exhibition & Conference (Nano tech 2007)**

February 21-23, 2007. Tokyo (Japan)

<http://www.ics-inc.co.jp/nanotech/en/index.html>

Nanotechnologies, Nanotechnology Business

▣ **Trends in Nanoscience 2007**

February 24-28, 2007. Irsee (Germany)

<http://www.uni-ulm.de/sfb569/irsee/>

Molecular Electronics, Nanochemistry, Nanopatterning

(March 2007)

▣ **Nanotech Northern Europe 2007 (NTNE2007)**

March 27-29, 2007. Helsinki (Finland)

<http://www.nanotech.net/>

Nanotechnologies

▣ **Nano and Giga Challenges in Electronics and Photonics**

March 12-16, 2007 - Phoenix, Arizona (USA)

<http://www.atomicscaledesign.net/ngc2007/>

Molecular Electronics, NanoPhotonics & Nano-Optoelectronics, Theory & Modeling

▣ **4th NanoSpain Workshop**

March 12-15, 2007. Sevilla (Spain)

<http://www.nanospain.org/Workshop4/>

NanoMaterials, NanoBiotechnology, Nanotechnologies



▣ **The International Conference on Nanotechnology: Science and Applications**

March 10-17, 2007. Luxor (Egypt)

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Nanomaterials, Nanophotonics & Nano-Optoelectronics, Theory & Modeling

▣ **APS March meeting 2007**

March 05-09, 2007. Denver (USA)

<http://www.aps.org/meetings/march/index.cfmno>

Magnetism, NanoBiotechnology, Nanotechnologies

NANO News - <http://www.phantomsnet.net/Resources/news.php>**▣ Taking Nanolithography Beyond Semiconductors [14-12-2006]**

<http://www.science.psu.edu/alert/Weiss12-2006.htm>

A new process for chemical patterning combines molecular self-assembly with traditional lithography to create multi-functional surfaces in precise patterns at the molecular level.

Keywords: Nanofabrication, Nanopatterning, Self-Assembly

▣ Beyond silicon: MIT demonstrates new transistor technology [09-12-2006]

<http://www.nanotechwire.com/news.asp?nid=4117>

MIT engineers have demonstrated a technology that could introduce an important new phase of the microelectronics revolution. Del Alamo's group recently fabricated InGaAs transistors that can carry 2.5 times more current than state-of-the-art silicon devices.

Keywords: Nanofabrication, Nanosensors & Nanodevices

▣ Gold, silver and copper can produce single-walled carbon nanotubes [06-12-2006]

<http://www.nanowerk.com/spotlight/spotid=1104.php>

Researchers in Japan succeeded in developing a nanoparticle activation method that shows that even gold, silver, and copper act as efficient catalysts for SWCNT synthesis. These non-magnetic catalysts could provide new routes for controlling the growth of SWCNTs.

Keywords: Nanotubes

▣ How to Shrink a Carbon Nanotube [30-11-2006]

<http://www.physorg.com/news84116089.html>

A research group has devised a way to control the diameter of a carbon nanotube. This useful new ability may help carbon nanotubes become more easily incorporated into new technologies.

Keywords: Nanoelectronics, Nanotubes

▣ Focused ion beam milling of nanocavities could lead to photon-on-demand light sources [30-11-2006]

<http://www.nanowerk.com/spotlight/spotid=1081.php>

Artificial opals are gemstones that are of considerable scientific and technological interest as photonic crystals, as components of light sources, solar cells, and chemical sensors. They are conveniently made from periodic stackings of nanospheres.

Keywords: Nanophotonics & Nano-Optoelectronics, Nanofabrication

▣ Nanowires growth without gold [28-11-2006]

<http://www.physorg.com/news83935101.html>

Silicon nanowires can help to further reduce the size of microchips. Scientists at the Max Planck Institute for Microstructure Physics in Halle have for the first time developed single crystal silicon nanowires that fulfil the key criteria to this end. The researchers used aluminium as a catalyst to grow the nanowires.

Keywords: Nanoelectronics, Nanofabrication, Nanosensors & Nanodevices

▣ Getting the dope on a single atom of dopant [27-11-2006]

<http://www.rsc.org/chemistryworld/News/2006/November/27110602.asp>

Scientists in the Netherlands have successfully probed the electronic and quantum mechanical properties of a single atom of dopant in a silicon transistor.

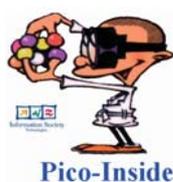
Keywords: Nanoelectronics, Molecular Electronics, Nanochemistry

▣ Cloning Nanotubes [14-12-2006]

<http://www.technologyreview.com/NanoTech/17899/page1/>

Researcher can now make exact copies of carbon nanotubes, potentially overcoming one of the greatest obstacles to widespread applications of the nanomaterials.

Keywords: Nanoelectronics, Nanotubes



Dielectric Substrates for Anchoring Organic Molecules with Computing Functionality

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Keywords: dielectric surface, insulator, organic molecules, immobilisation, NC-AFM

Electrical insulation is a key issue in the design of any electronic circuit. While there are plenty of trivial solutions available for insulation with synthetic materials for wiring in conventional circuits, insulation, or more precisely leakage currents, may become a technical challenge or an effective limitation in present and future integrated circuit design. It has been shown, for instance, that the thickness of the gate oxide that is a vital part of any field effect transistor in an integrated circuit based on Si-SiO₂ technology cannot be made thinner than 1.3 nm since otherwise the leakage current will prohibit proper operation [1]. According to the roadmap of the Semiconductor Industry Association [2], this would imply reaching the end of the silicon road in the year 2012 [1].

The problem of leakage current may impose also severe limitations when introducing single molecule electronics for computing. As molecules that are likely candidates for molecular electronics applications have a rather low conductivity compared to that of doped silicon, an effective insulation against a (semi)conducting substrate is of utmost importance. One concept that has been developed to facilitate the controlled contact of a molecule to a metallic structure while avoiding unwanted channels of current flow is to decouple the conducting molecule from the substrate by weakly conducting spacer legs [3]. While this approach has successfully been demonstrated in a proof-of-concept experiment [4], it is of limited value for practical applications as for molecular device operation, not only the molecule but also the conducting pad contacting the molecule has to be isolated from any conducting substrate. Therefore, a design integrating molecules and electrodes on a non-conducting substrate would be more desirable for molecular electronics devices.

The quest for suitable combinations of functional conducting molecules and electrically insulating substrates that will eventually allow molecular device fabrication is one of the central issues in the development of atomic scale technologies for the PicoInside project [5]. While techniques for the preparation and characterisation of suitable insulating substrates and the deposition of organic molecules are well developed, the big challenge is the fabrication of stable structures by their combination. In this

contribution, we introduce three candidates for dielectric substrates that are investigated in the framework of the PicoInside project, namely calciumdifluoride, titania and muscovite mica and discuss their suitability as substrate materials for molecular electronics. The main point here is a detailed analysis of the substrate surface structure and the mechanisms of interaction of molecules with the surface including physi- and chemisorption as well as anchoring by firm chemical bonds. The method of choice for such studies that is also utilised here is dynamic scanning force microscopy (SFM) operated in the so-called non-contact mode of operation (NC-AFM). This technique has been shown to be capable of revealing the details of surface structures on fluorides, oxides and other dielectric surfaces with a resolution ranging from the micrometer scale to atomic resolution [6].

Prototype materials that are principally suitable for our purpose are alkali halides and alkaline earth halides where surfaces can be prepared by cleaving bulk crystals [7] or by growing them on metallic substrates [8]. We have extensively investigated the CaF₂(111) surface that allows the preparation of large, atomically flat terraces by a simple cleavage technique typically yielding results as shown in Fig. 1. The step edges have a height of 0.31 nm corresponding to a F-Ca-F triple layer in the fluorite structure. They frequently appear to be slightly charged what is apparent in Fig. 1 by the small bright spots that are not found on flat terraces but localise at edge or kink positions. The precise origin of the charging features is not yet clear, however, we speculate that they are due to charges trapped by sub-surface structural defects. Dislocation defects may, for instance, easily be created in the vicinity of step edges by the cleavage process imposing considerable stress on the crystal. While it is possible to reduce the overall charge present on the crystal by heat treatment, it is most difficult to remove the charged spots like the ones shown in Fig. 1 as they are apparently tightly bound to the crystal. Charges may, of course, be detrimental for the controlled adsorption of molecules as the local electric field may strongly polarise, attract or repel a molecule and fundamentally change its adsorption properties.

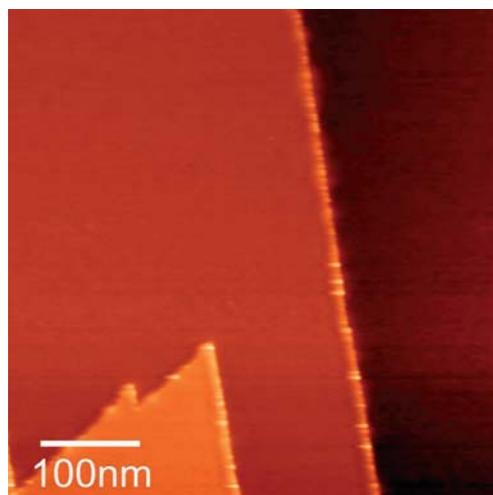


Figure 1. Face of a CaF₂ crystal cleaved along the (111) direction. The bright spots located at step edge and kink sites are due to charges.

The surface areas between step edges of a cleaved $\text{CaF}_2(111)$ surface are atomically flat and perfectly well ordered. The two fundamental contrast patterns obtained in atomic resolution imaging of the surface with a dynamic force microscope are reproduced in **Fig. 2** and are well understood [9]. Frames (a) and (b) represent the cationic and anionic sub-lattices, respectively. The apparent triangular contrast feature dominating frame (b) is the result of a weighted superposition of contributions from first and third layer fluorine ions as depicted schematically (white triangle) in frame (c). When imaging a large number of flat terraces, one can hardly find any atomic scale defect, although, one would expect intuitively that the process of cleavage should produce at least a primary population of surface vacancies or F centres. Vacancy related defects are not apparent in our measurements as they are too mobile at room temperature, however, there is indirect evidence that they exist [9, 10].

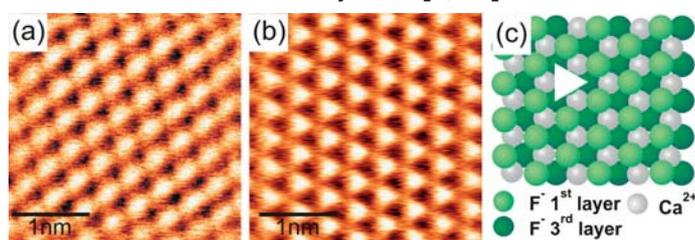


Figure 2. Imaging the cationic (a) and anionic (b) sub-lattices of the $\text{CaF}_2(111)$ surface on a flat terrace with atomic resolution dynamic scanning force microscopy. Frame (c) is a schematic representation of the topmost surface layers illustrating the formation of triangular contrast features that are characteristic for the anionic sub-lattice.

In a preliminary deposition experiment, we evaporated Perylene-3,4,9,10-tetracarboxylic-3,4,9,10-diimide (PTCDI, see **Fig. 3(a)**) further on referred to as S13 onto the surface to obtain a sub-monolayer coverage on $\text{CaF}_2(111)$. The S13 molecule is based on the aromatic board of the perylene molecule that is functionalised by carboxylic and aliphatic groups. The original intention of these measurements was to study SFM contrast formation of individual deposited molecules and explore properties of binding the molecule to a flat terrace. It turned out, however, that S13 cannot easily be attached to the $\text{CaF}_2(111)$ surface as molecules readily diffuse towards step edges where molecules cluster in packages with several nanometres side length. This phenomenon is illustrated in **Fig. 4** showing a string of molecular clusters positioned at the lower side of a step edge. The molecular clusters are apparently not well ordered and it is not possible to highly resolve these structures. At present stage it is not clear how the clusters are formed, however, we speculate that local charges may provide initial nucleation centres.

These measurements reveal a common problem faced when depositing organic molecules on well prepared alkali halide and alkaline-earth halide surfaces. The interaction between molecules and surface is rather weak and diffusion barriers so low that molecules readily diffuse across the surface to surface irregularities acting as trapping centres. This process often effectively suppresses the controlled adsorption of single molecules as well as the formation of well ordered structures by molecular self-orga-

nisation. While nucleation at step edges is detrimental for the controlled deposition on dielectric surfaces with large terraces, it can be utilised for creating molecular arrangements on nanostructured dielectric surfaces. Recently, nanometre sized shallow pits created by electron irradiation of $\text{KBr}(001)$ have successfully been used to trap SubPc molecules and to create regular molecular arrangements within the pits [11].

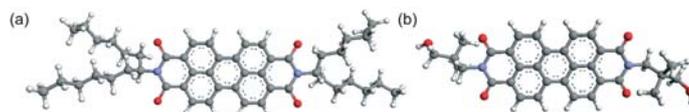


Figure 3. Structure of the two PTCDI compounds. (a) is termed S13 because of the side chain that contains 13 carbon atoms. The molecule in pane (b) is termed DMHP-PTCDI. It is smaller and lighter than S13 but has hydroxyl groups at the end of each side chain that may form additional bonds to a substrate.

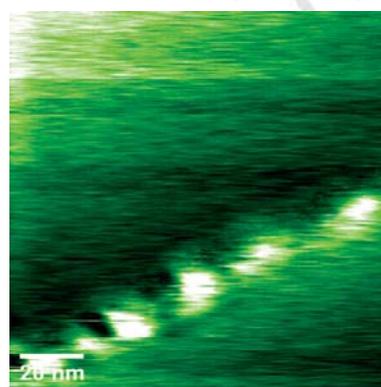


Figure 4. Step edge on a $\text{CaF}_2(111)$ surface decorated a string of clusters composed of S13 molecules.

Muscovite mica is a universal dielectric substrate that has extensively been used for the deposition of metallic clusters [12], organic [13] and especially bio-molecules [14]. Its most appealing feature as a substrate is the possibility to create extremely large atomically flat terraces by a very simple procedure of peeling off layers. There is much expert knowledge available about this mineral material and its applications, however, the vast amount of knowledge on surface and adsorption is based on studies performed in a liquid environment. This also applies to atomic scale studies of the surface that have recently been introduced by advanced SFM techniques facilitating atomic scale imaging in a liquid environment. In these studies it was possible to identify the hexagonal structure of surface SiO_2 units on mica (see **Fig. 5**) and to demonstrate the order and cleanliness of the surface present in aqueous solution [15].

Imaging the mica surface in an ultra-high vacuum environment with high resolution is a much more challenging task. As the material contains layers of K^+ ions that are exposed upon cleaving the material, the cleavage process normally produces a large amount of surface charge as the interface K^+ ions will never stick in equal parts to separated cleavage faces.

As mica is a very good bulk electrical insulator, these charges will prevail in an ultra-high environment and result in a very high and in homogeneously distributed surface

potential that strongly hampers and mostly prohibits high resolution force microscopy imaging. The charge can be removed by cleaving mica in air what results in a significant coverage of the surface with molecular adsorbates and surface reaction products. A typical result for such a surface investigated with dynamic force microscope is shown in **Fig. 6(a) to (c)**. It is evident from the images that the surface coverage introduces a sub-nanometre roughness and the overall result is a much less well defined surface than one would expect from an atomically flat substrate. In a preliminary study we deposited DMHP-PTCDI onto the surface and found that this molecule indeed sticks to the surface and condenses into irregularly shaped islands as shown in **Fig. 6(d) to (f)**. These islands appear to be rather flat on the surface, however, it has not yet been possible to image the molecular layer with high resolution and to test its structural order.

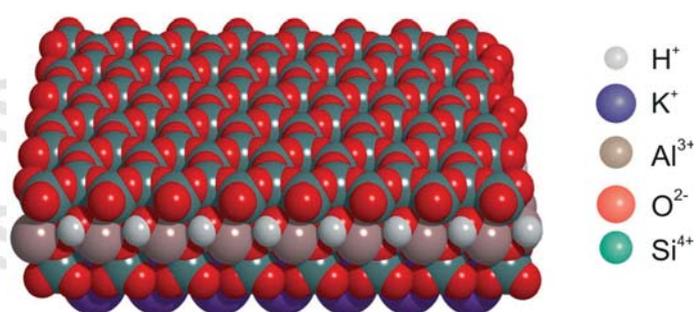


Figure 5. Structure model of a Muscovite mica surface layer.

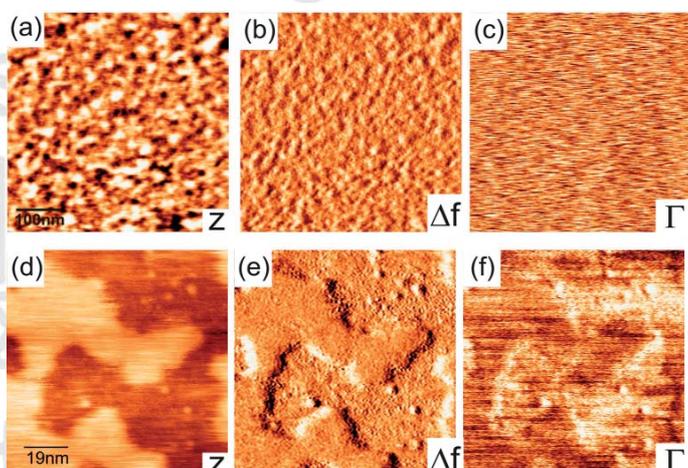


Figure 6. (a) to (c) Surface structure of muscovite mica cleaved in air as revealed by dynamic scanning force microscopy. (d) to (f) Deposited molecules of DMHP-PTCDI, condense into irregularly shaped flat island. The images display the (a, d) topography, (b, e) detuning and (c, f) dissipation signals, respectively.

The (110) surface of rutile TiO_2 provides another dielectric substrate that may be well suited for molecular adsorption studies in the context of the PicoInside project. Fixing small organic molecules on this surface by a carboxylic anchor has been demonstrated for molecules deposited under conditions of an ultra-high vacuum [16] as well as adsorption of larger molecules from solution [17]. The surface is prepared by mechanical polishing followed sputtering/annealing cycles in the ultra-high vacuum and reveals

a row structure as shown in **Fig. 7**. While frame (a) clearly exhibits atomic resolution on the rows, the dumbbell shaped apparent depressions found in frame (b) are the characteristic contrast feature of substitutional hydroxide on oxygen rows. The identification of vacancies and hydroxide species on the oxygen rows with SFM has recently been established [18] and previously scanning tunnelling microscopy (STM) studies had revealed the complex mechanisms of their formation [19]. Vacancies that are the result of surface preparation under slightly reducing conditions determine the anchoring of molecules on this surface. STM and SFM investigations clearly showed that vacancies are rapidly (within minutes) converted into hydroxide species in the presence of a water partial pressure in the 10^{-9} Pa region what is typical for ultra-high vacuum conditions. Therefore, processing under very clean conditions will be a central issue for molecular deposition on $\text{TiO}_2(110)$. It will then be possible to use vacancy sites for anchoring molecules functionalised with a carboxylic group.

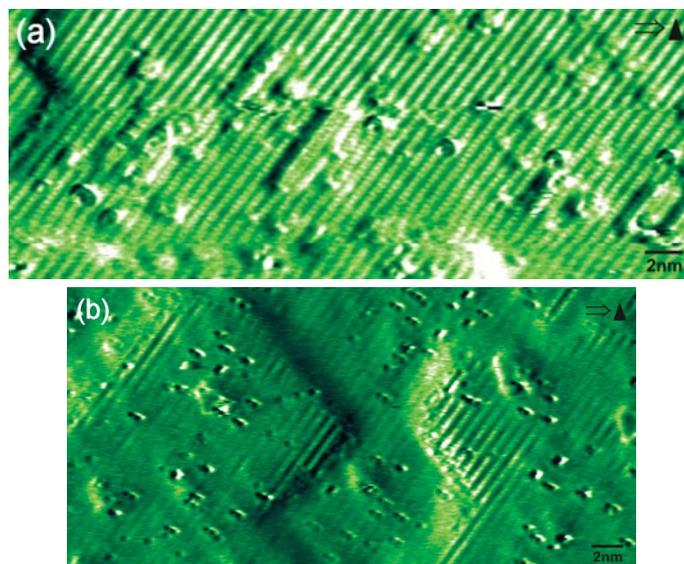


Figure 7. The (110) surface of TiO_2 imaged with dynamic force microscopy. (a) Reconstruction into rows along (001) that are partially contaminated. (b) Hydroxide defects resulting from dissociation of water at oxygen vacancies found on a slightly reduced surface.

In summary, we introduced three dielectric materials as potential substrates in the context of molecular computing applications. The $\text{TiO}_2(110)$ surface requires elaborate preparation in the ultra-high vacuum but facilitates anchoring via a carboxylic molecular functionality. However, it has to be explored, how anchoring sites can be prepared in a controlled way in a regular array or at specific sites. CaF_2 and muscovite mica allow the preparation of large atomically flat terraces by simple cleavage procedures. However, terraces on $\text{CaF}_2(111)$ do not naturally provide anchoring sites and also in this case it has to be explored how they can be prepared in a controlled way. Air cleaved mica readily facilitates molecular adsorption, however, the molecule-substrate interaction is mediated in this case by surface contaminants and the details of the resulting surface structure has to be characterised in detail. Further

investigation is needed to find the optimum substrate and surface treatment for a controlled deposition of single molecules and molecular layers with computation functionality.

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Non-contact AFM as Indispensable Tool for Investigation of Patterned Insulators

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Keywords: NC-AFM, insulators, patterning

Patterning insulating surfaces

Surface modifications induced by electronic transitions are quite important for nanofabrication [1,2]. For instance, modified insulating surfaces can be used as templates for adsorbing medium-sized (<100 atoms) functional organic molecules, which is extremely interesting in the development of hybrid molecular electronic devices [3, 4]. The mobility and aggregation of molecules as well as their eventual ordering on an insulating surface is determined by the weak balance between intermolecular and molecule-substrate interactions. The final arrangement of the molecules can be observed by non-contact force microscopy (NC-AFM) [5] often with single-molecule resolution [6].

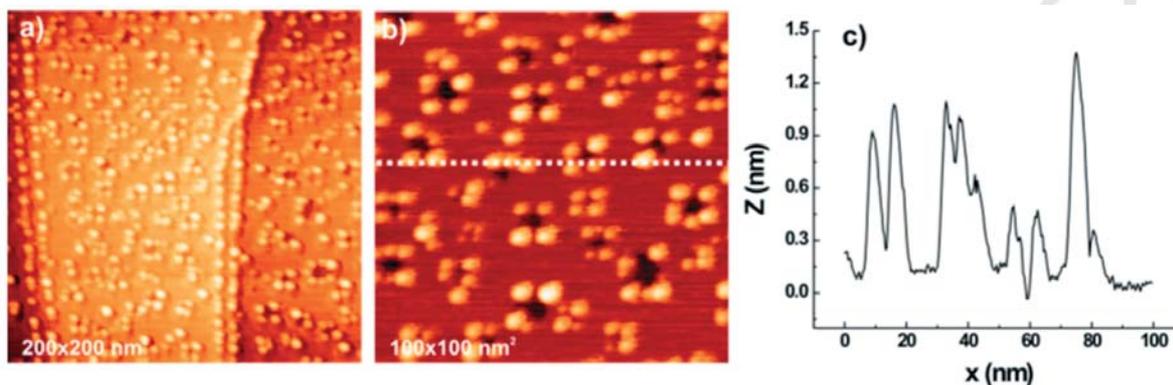


Fig1. a) NC-AFM topographical image of 200x200 nm² on KBr(100) with pits created by electron evaporation and 0.3ML gold. b) Zoom on 100x100nm² for a better view of how the gold clusters decorate the pit corners. c) Cross section of the topography over 100 nm proving that the gold formed indeed clusters (due to the large dimensions).

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When a ionic crystal is excited by electrons [1], the desorption of surface atoms is induced by the formation of Frenkel defects in the bulk (*F* and *H* centers), which diffuse and recombine with the surface. At the first stages of the typically layer-by-layer surface removal monatomic depth rectangular pits are formed, which are several nanometers in size. These pits can be successfully used as molecular traps. For instance, our group has succeeded in confining chloro-[subphthalocyaninato] boron- (III)

(SubPc) molecules on a modified KBr(001) surface in ultrahigh vacuum (UHV) at room temperature [3].

In order to realize a viable device based on an ordered array of few molecules, such arrangement has to be connected to metallic electrodes. Fortunately, nanopatterns can also serve to control the nucleation of metal nanostructures at surfaces [7]. **Figure 1** (page 25) shows almost 0.3 ML of gold evaporated on a KBr surface with pits at room temperature. The gold decorates the step edges and the pit edges from outside being located on the upper terrace edge. No gold atoms or clusters were observed at the bottom of the pits. In a recent study Matievsky et al. observed that metal nanoparticles on KBr can even be used to nucleate the growth of pits, resulting in pits with metal nanoparticles inside [8]. The same group was also able to finely tune the size of the pits by using an electron-beam evaporator with extremely small current densities. A minimum pit size of approx. 3 nm² was observed. This size corresponds to the removal of 28 atoms and was attributed to the impingement of single charged particles on the surface of the crystal. These pits should be small enough to immobilize and isolate individual large molecules, such as SubPc, which otherwise have long diffusion lengths on insulating surfaces [9].

An alternative method for getting patterned insulating surfaces is simply given by annealing [10]. Heating of ionic crystals in vacuum results in atomic diffusion from corner sites of steps.

This process smoothens cleavage steps and evolves spirals of steps around the intersection of dislocations with the surface. These spirals provide a regular array of monatomic or diatomic steps,

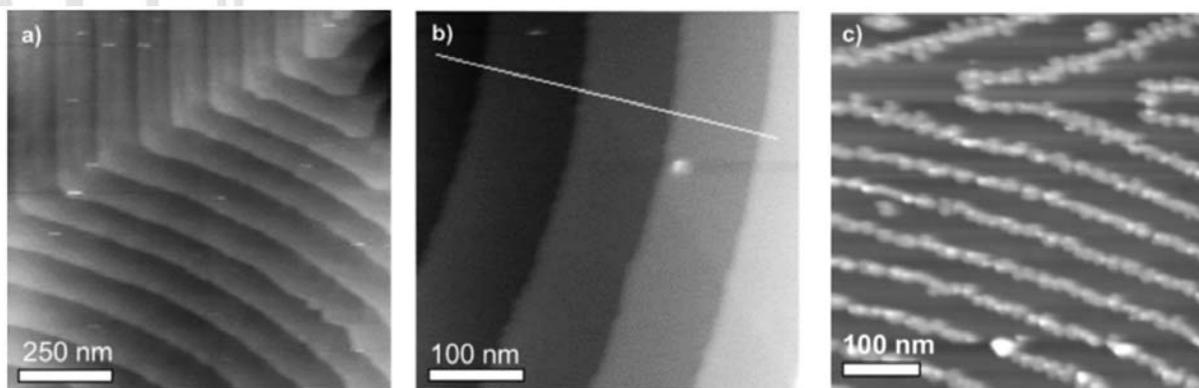


Fig. 2 a) Topographical image of the spirals consisting of parallel monoatomic steps separated by atomically flat terraces on KBr, b) a closer look at the created structure, c) sublimation of 0.5 ML of Cu-TBPP at room temperature on the spirals. The structure was obtained by heating the crystal at 380°C in ultra high vacuum for 20 minutes.

separating atomically flat terraces (**Fig. 2a,b**). The typical terrace width is between 50 and 150 nm [11]. The enhanced interaction at the steps of evaporation spirals makes them a candidate for guided growth of molecular adsorbates (**Fig. 2c**).

Virtual AFM: NC-AFM from Experiments to Simulations

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Since almost a decade, non-contact atomic force microscopy has proven capable of yielding images showing contrasts down to atomic scale on metals, semiconductors, as well as insulating ionic crystals, with or without metallic or adsorbate overlayers. For a better understanding of the non-contact imaging techniques the development of numerical simulations is mandatory.

Our group has recently introduced an atomic force microscope (AFM) simulator [12]. Our “virtual” AFM closely mimics the electronics of a real setup including the following components: 1) an oscillator representing the cantilever and its detected fundamental bending resonance,

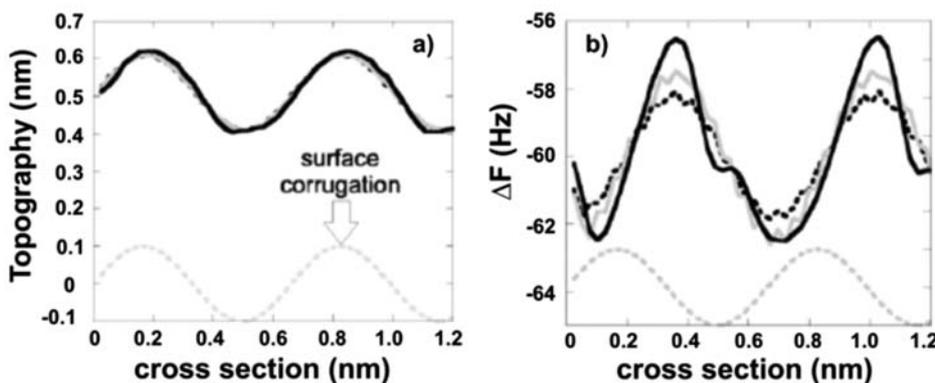


Fig. 3 Calculated cross section of a sinusoidally corrugated surface for various PLL gains.

a) Topography, b) Frequency shift.

2) an amplitude detector (rms to dc converter), 3) a phase-locked loop (PLL), 4) a phase shifter, 5) a proportional-integral (PI) controller to adjust the amplitude of the tip

oscillation and 6) another PI-controller to adjust the distance between tip and sample. Both a user-friendly LabView version and a faster implementation in C have been realized.

Several virtual experiments have been performed to test the reliability of the simulator and to compare it with the real setup. The interaction between tip and surface is modelled as the sum of Van der Waals long-range contribution and a short-range Morse potential. Simulated scan lines along a sinusoidally corrugated surface and across a monatomic step are compared with the assumed profile (see Fig. 3 - page 26). The computed "damping" and topography variations systematically change with the time constants of the controllers and the scan speed. The influence of various adjustments is studied: Working off phase, working with small and large oscillation amplitudes, in the presence of noise. The detailed analysis of these experiments allows one to determine optimum adjustments of the individual electronic blocks and their joint performance in real experiments.

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Molecular-Resolved Imaging With Non-Contact Atomic Force Microscopy (NC-AFM)

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Summary

We have studied initial stages of growth of the organic molecular semiconductor - 3,4,9,10 - perylene-tetracarboxylic-dianhydride (PTCDA) on reconstructed InSb (001) and GaAs (001) surfaces in ultrahigh vacuum. Using non-contact atomic force microscopy (NC-AFM) it was found that on InSb surface the growth is determined by preferential diffusion of molecules along surface reconstruction rows, leading to the formation of long molecular chains. In contrast, on GaAs surface the growth proceeds in the "sticks-where-it-hits" mode. In most cases the PTCDA molecules supported on InSb surface are imaged by NC-AFM as dark features, despite the fact that they constitute elevated topographic forms. This indicates that imaging occurred through repulsive interactions.

Surface and molecular imaging with NC-AFM

One of the goals of Pico-Inside Project is to learn how to image individual molecules, metallic mesa nanostructures, and self-assembled molecular structures on surfaces of wide band gap materials with a NC-AFM microscope, and then to develop manipulation strategies for single molecules.

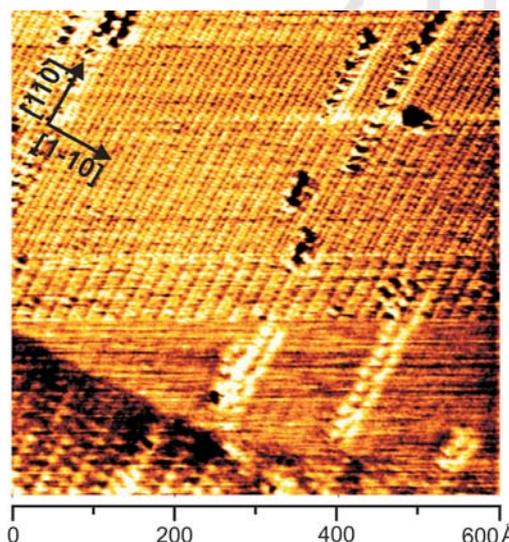


Fig. 1. A high resolution NC-AFM image of 0.07 ML PTCDA molecules adsorbed on $c(8 \times 2)\text{InSb}(001)$ at 450 K. The atomic structure of the substrate is resolved with superimposed chains of adsorbed molecules. Various structural patterns are seen as the stripes along the x -axis of the figure due to changes in chemical composition of the tip apex.

Studies of topography and molecular electronic effects are complemented by exploring the stability, rigidity and conformational flexibility of the entities with force spectroscopy and methods of force controlled molecular manipulation.

There is a large number of substrates on which ordered growth of planar organic molecules have been observed. Ionic insulators, layered materials and metals are among those most often studied. As far as semiconductor surfaces are concerned, however, the growth is difficult due to dangling bonds present on the surface leading to strong chemisorption.

That strong binding of molecules to a substrate makes the diffusion impossible and prevents them from creating well ordered structures. In order to omit that obstacle semiconductor surface passivation is necessary. In such conditions, the growth of ordered films of 3,4,9,10-perylene-tetracarboxylic-dianhydride (PTCDA) on Si [1-4] and GaAs [1, 5-10] was reported. PTCDA molecules are archetypal molecular semiconductors widely used in organic layer growth experiments. Recently, it has been shown that it is actually possible to create ordered molecular films on selected non-passivated surfaces of group III-V semiconductors, Indium rich surfaces are most of all among those on which such a growth is possible [11-13]. Substrate symmetry is crucial for the growing structure morphology due to a strong molecule-substrate interaction. Surfaces of (001) group III-V compounds are usually highly reconstructed and anisotropic, with structure depending on details of their preparation. That gives the unique opportunity of controlling grown structures. Most of the existing data concerning growth of molecular assemblies on semiconductors are based on LEED and XPS experiments, only few present some real space STM images of the adlayers [14,15]. High resolution NC-AFM images for such systems are very seldom

Atomic resolution combined with chemical sensitivity by NC-AFM

InSb(001) $c(8 \times 2)$ surface has been chosen as a model substrate for investigation of anchoring behaviour of PTCDA molecules on III-V compounds. A large effort has been put in the past into defining the correct structural model of the surface. At present, the widely accepted model describes that surface as covered by rows of In atoms running in [110] direction and predicts dimerisation occurring in the second bilayer and giving $c(8 \times 2)$ symmetry [16,17]. The system of parallel atomic rows forms a highly anisotropic template for diffusion of adsorbates.

A close up NC-AFM image (see Fig.1) reveals the atomic structure of the surface

with superimposed chains of adsorbed PTCDA molecules. The structure of the substrate is strongly anisotropic and appears as rows of atoms running along the [110] direction. However, atomically resolved NC-AFM images of $c(8 \times 2)$ InSb (001) do not show the same pattern every time; instead the patterns change abruptly during scanning in an uncontrolled way (see the stripes in Fig. 1 parallel to x-axis). This phenomenon could only be explained after analyzing the image formation more deeply. In NC-AFM the tip is oscillating at its resonant frequency over the surface. Although the oscillation amplitudes used are ~ 10 nm, at its closest approach the tip is in a close vicinity to the sample. In that range of distances, various forces act on the tip that alter the oscillation frequency. Van der Waals and electrostatic forces can be assumed as varying slowly within the atomic scale. In contrast, chemical forces are extremely short ranged and vanish within a few tenths of nanometer from the surface [17]. Certainly, for a surface of two component materials, the chemical interaction changes substantially for each constituent. As a result, an image of such a sur-

face is usually quite different from the topographic relief. The chemical composition of the tip apex is the key to understanding image formation. It is virtually impossible to avoid contaminating the tip with the sample material. In the case of InSb the tip is likely terminated with either In, or Sb atom. As a result, an Sb sublattice is imaged as a set of protrusions with a In-terminated tip, whereas a In sublattice is imaged as set of protrusions for an Sb-terminated tip. It is essential to understand that those protrusions do not necessarily mark the geometric positions of atoms. Therefore, in principle, various sublattices could be imaged depending on tip chemical composition. In general, three distinct patterns were found on $c(8 \times 2)$ InSb (001) surfaces (only two of them could be distinguished in Fig. 1) corresponding to the surface geometry of the indium sublattice, antimony sublattice and the sum of both sublattices [17,18]. Consequently, it was demonstrated that the tip apex composition dependent NC-AFM possesses the ability to resolve various chemical species present on the surface. This chemical sensitivity of the NC-AFM probe has been explored for high resolution imaging of PTCDA molecular assemblies on AIII-BV surfaces.

PTCDA molecule imaging on AIII-BV surfaces

In effort to investigate anchoring of organic molecules on semiconductor surface PTCDA molecules and $c(8 \times 2)$ InSb(001) and $c(8 \times 2)/(4 \times 6)$ GaAs (001) surface were chosen. The substrates are particularly interesting due to the fact that the reconstructed surfaces are strongly anisotropic and additionally, indium compounds in contrast to gallium compounds, do not require passivation in order to allow assembling of molecules into ordered structures. Therefore, quite different organization of molecules on those two substrates is expected despite their similarities in surface atomic structure.

The structure of 0.07 ML of PTCDA molecules evaporated onto InSb (001) surface at 450 K is presented in Fig. 2.

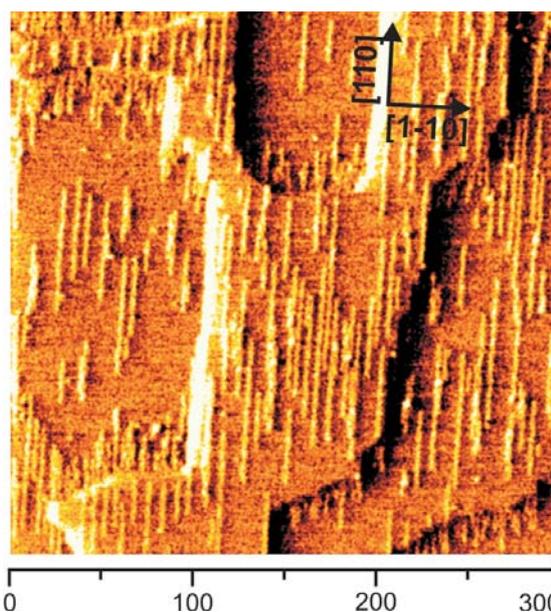


Fig. 2. NC-AFM image of 0.07 ML of PTCDA on $c(8 \times 2)$ InSb(001) deposited at 450 K.

This large scale image of the surface shows chains of molecules running parallel to [110] direction (i.e. in the direc-

tion of atomic rows on the InSb surface), often bunched by into groups of two or three chains together. The LEED pattern (not shown in the figure) indicates that the structure of the surface remains intact, while molecular adsorbates are reflected only as faint streaks in $[-110]$ direction. At terrace edges the chains are shorter and more dense.

Overall structure of the adsorbate do not change in a large temperature window (from RT to 550 K) apart from the fact that the chains tend to be shorter at lower temperatures.

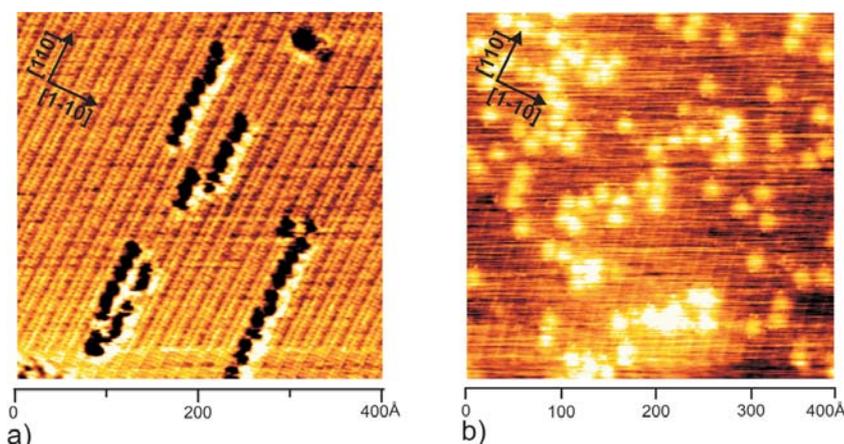


Fig. 3. Different PTCDA assembling modes for 0.07 ML adsorption at 450 K on $c(8 \times 2)$ InSb (001) surface (a), and on $c(8 \times 2)/(4 \times 6)$ GaAs (001) (b).

More detailed analysis on PTCDA molecular chain organization could be based on high resolution images where atomic/molecular structure of the system is seen for both the molecules and the substrate as demonstrated in **Fig. 3a** for InSb and **3b** for GaAs. In case of InSb substrate the molecules lie within a chain in two equivalent positions shifted by approx. 4 Å perpendicularly to the chain while their spacing along $[110]$ direction is close to triplicate base surface lattice unit (i.e. 3×4.58 Å). As expected,

PTCDA molecules deposited on GaAs(001) are adsorbed in “sticks-where-it-hits” mode, although behavior similar to the one seen on InSb might occur for higher temperatures. Inspection of individual molecule images obtained still at higher magnification (see **Fig. 4b**) reveals that even for GaAs substrate PTCDA molecules are adsorbed at well defined sites between the rows of topmost Ga atoms on the reconstructed surface.

Appearance of the molecules in NC-AFM image depends on used detuning – at large tip-molecule distances attracting interaction dominates and molecules are depicted as bright features (see **Figs. 4a and b**). However, upon approaching the molecule repulsion prevails and the molecule appearance changes to depression. That is repetitive and non-destructing behavior. Using scanning probe techniques it is usually very difficult task to measure interaction over specific point on the lattice, because of mechanical noise giving the height uncertainty and

thermal drifts as well as piezo-scanner creeps giving large uncertainties both for the in-plane tip position and for the height. To overcome this difficulties, for specific system studied here where only rare protruding PTCDA chains are present, we used a following scheme: we control the scanner by rather weak feedback constant to achieve effective constant height mode. Therefore the tip-surface distance is determined by the average detuning value Δf_{aver} . Since only slow mechanical vibrations (below 1 Hz) are present in our system, and the scanning frequency is 3 Hz, the tip always “follows” the surface when the stage is bent due to slow vibration. Using these settings we scan the surface and gradually

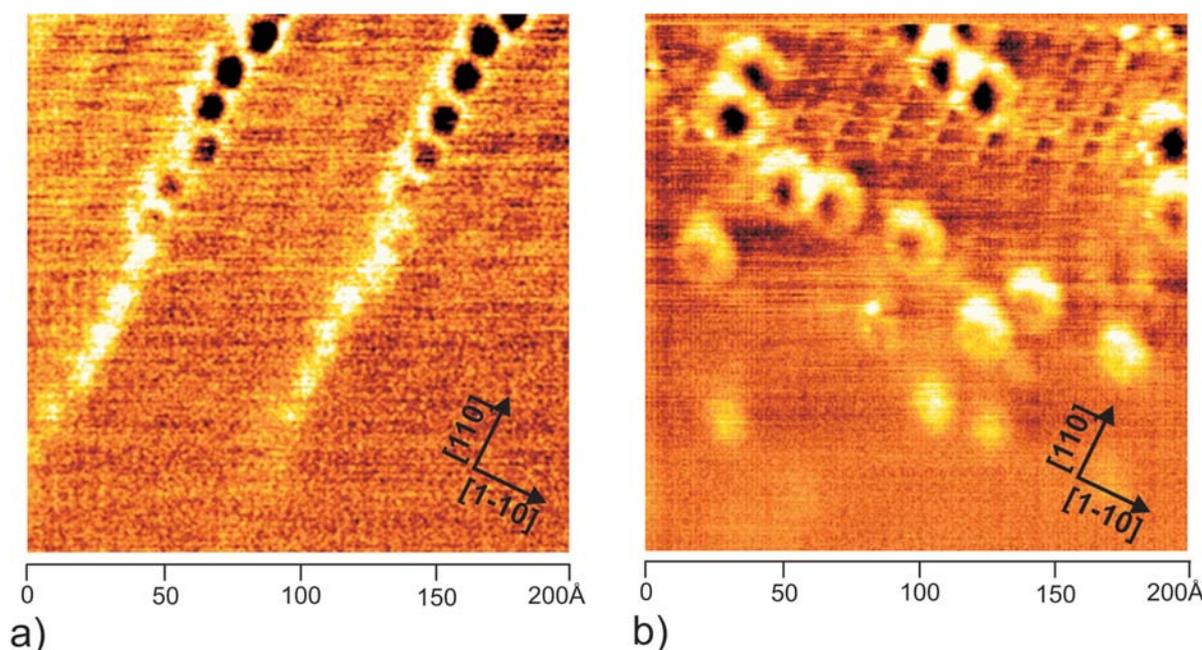


Fig. 4. The Δf image recorded with minimal z-feedback in order to maintain constant tip-surface separation as determined by the average detuning. Δf was gradually increased from right to left in 1 Hz intervals, at the end it was set back to initial value to correct for the vertical drift. (a) the image taken for PTCDA/InSb(001), and (b) PTCDA/GaAs(001) at corresponding experimental conditions.

increase the detuning by 1 Hz steps and at the end we set the initial detuning value. The results are shown in **Figs. 4a** for InSb substrate and **4b** for GaAs. For large tip-surface separations only smooth background is seen, by reasonable assumption, due to mesoscopic van der Waals interactions.

Upon increasing D_{faver} PTCDA molecules appear as bright features (imaging occurs via attractive interactions). Further increasing of D_{faver} the molecules are imaged as dark cores surrounded by bright rims. Since the vdW background gives the zero reference level we may conclude that the imaging process occurs now via repulsive and attractive short range interactions.

Conclusions

We have studied PTCDA molecules deposited on InSb (001) $c(8 \times 2)$ and GaAs (001) $c(8 \times 2)/(4 \times 6)$ surfaces by means of a frequency modulated non-contact atomic force microscopy. The adsorption of PTCDA on InSb (001) $c(8 \times 2)$ is dominated by one-dimensional diffusion resulting in long PTCDA chains assembled parallel to $[110]$ crystallographic direction and with the intramolecular distance close to threefold substrate lattice parameter. PTCDA on GaAs (001) $c(8 \times 2)/(4 \times 6)$ grows in "sticks-where-it-hits" mode. The PTCDA molecules imaging mechanism is dominated by repulsive interactions being either due to electrostatic forces or due to the core-core repulsion.

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Atomic resolution AFM on NaCl at 5 K using the QPlus sensor

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Over many years, low temperature STM has been established as an advanced imaging and spectroscopy tool in various scientific fields.

A large number of self-built as well as commercial instruments are working on a high performance level with a z-stability in the picometer range and allow for applications such as molecule spectroscopy (including inelastic tunnelling spectroscopy, IETS), atom and molecule manipulation, spin-polarised STM/STS and many others. One reason for the widely spread use of this technique is the relative technical simplicity of a scanning tunnelling microscope, even under the stringent experimental conditions when working at liquid Helium temperatures. However, in the field atomic force microscopy the situation is different. A comparatively limited number of self-built or commercial low temperature instruments are successfully working on a high performance level, which is basically resulting from the technical complexity of an optical beam deflection or interferometric AFM detection. Nevertheless, applications drive the development of a easy to handle, reliable and high performance AFM technique.

A prominent example can be found within the PicoInside project: A molecule based computing circuit requires a single molecule to be locally manipulated and electronically de-coupled from the underlying substrate. Current approaches use wide band gap semiconductors for decoupling or thin insulation layers which would still allow for an STM based manipulation process.

On the other hand, the use of an insulating substrate would ensure perfect electronic de-coupling of the molecule but requires AFM based manipulation. The establishment of a reproducible experimental route for this process represents a major milestone of the PicoInside project.

Aiming at a low level of technical complexity, purely electrical AFM sensors are favourable. Among a small number of approaches, the piezo-resistive (PR) detection scheme is one of the few that has proven atomic resolution but never been established as a reliable and routine AFM technology at a high performance level. One reason is that the first stage PR signal detection is highly integrated into the cantilever itself, thus requires a high level of cantilever process technology and basically depends on the sensor availability from commercial suppliers. In addition, the simultaneous use in high performance STM and STS mode is limited since cantilever probes need metal coating for conductivity reasons.

An alternative (also purely electrical) approach is based on a quartz tuning fork and uses of the piezo-electric effect of quartz. In general, tuning forks are mass products (e.g. used in wristwatches) and comparably cheap. Although the commercial availability for a complete sensor is still very limited, it is accessible for the scientific community since probe tips can easily be mounted (glued) onto a tuning fork. In the late 90's Franz Giessibl presented a new approach that makes use of a quartz tuning fork, commonly called the "QPlus sensor", which showed excellent atomic resolution on (conducting) Si(111) [1,2]. One prong of the tuning fork is fixed while the SPM probe tip is mounted to the (free oscillating) second prong. It thus acts as a quartz lever transforming its oscillation (f_{Res} typ. 20-25 kHz, depending on tip mass) into an electrical signal as a result of the piezo-electric effect. The sensor can either be electrically excited by an AC voltage or mechanically.

The used feedback signal is based on frequency shift originating from tip-sample force interaction. However, for true force detection in non-contact AFM operation mode on conducting samples, the role of the tunnelling current cross-talk needs careful consideration.

The basic motivation of the QPlus sensor is to improve AFM resolution for short range forces by the high spring constant of the sensor (approx. 1800 N/m, cantilever typ.

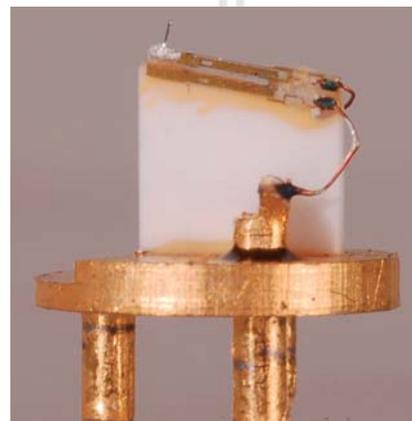


Figure 1: Image of a QPlus sensor mounted to the LT STM sensor carrier. Tungsten tips are glued onto the oscillating prong of the tuning fork. The use of W enables the QPlus sensor to be used simultaneously or alternatively in STM mode.

a few ten N/m) and small oscillation amplitudes in the range of 1nm or below (cantilever typ. 10 nm), which more precisely matches the range of the involved (chemical) forces. From a technical point of view, the integration of such a sensor is straight forward in terms of instrument design. Due to the high sensor spring constant and close distance between tip and sample, smallest external (mechanical) perturbations will have strong impact since

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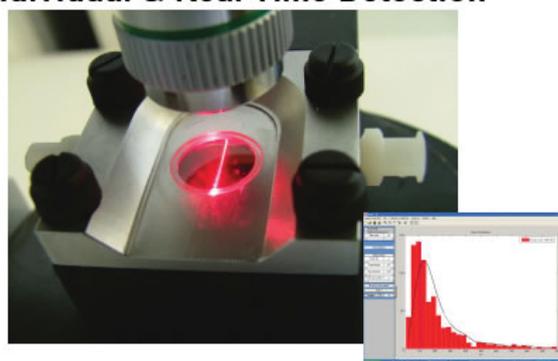
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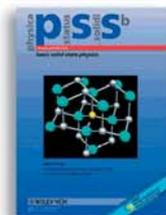
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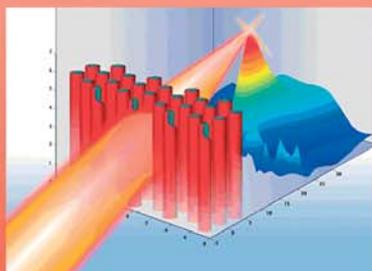
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Focusing slabs made of negative index materials based on inhomogeneous dielectric rods (V. V. Sergentu, V. V. Ursaki, I. M. Tiginyanu, E. Foca, H. Föll, and Robert W. Boyd, p. R48)

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involved forces a nearly 3 orders of magnitude larger and may lead contrast changes (e.g. formation of chemical bonds, etc.) and instable imaging.

This requires an extremely stable SPM platform with a z-stability in the range of a few picometer and is the reason why the well established "Omicron Low Temperature STM" (LT STM), has been chosen (up to date built nearly 100 times). A relevant design change is the implementation of three electrical contacts at the sensor carrier in conjunction with a fully UHV compatible transfer concept. Also here, well proven and existing technology from the "Omicron VT SPM" has been employed and two sensor carriers can alternatively be used (STM only, QPlus), while a QPlus sensor can also be operated simultaneously in STM mode. As an important requirement, the technical realisation ensures the known STM/STM performance

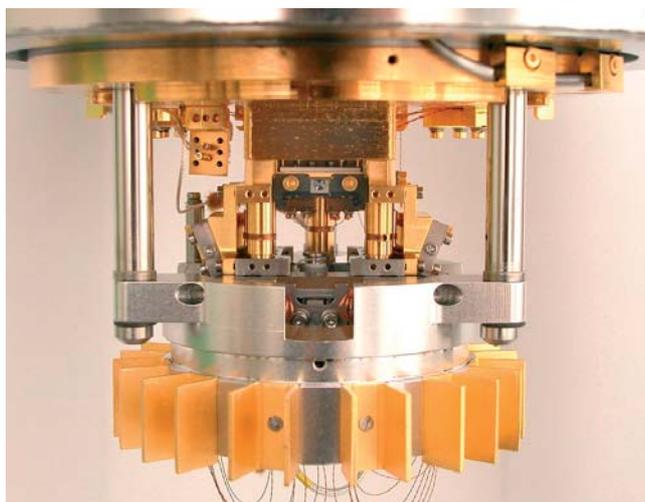


Figure 2: The "Omicron LT STM" microscope stage (version with in-UHV lens arrangement for optical detection and evaporation from below)

level of the instrument.

The major technical challenge lies in the pre-amplification technique. As already previously developed for RT instruments, the detection circuit scheme guarantees a distance control based on the vibrational signal with clear separation of the tunnelling current. This is especially important for quantitative AFM on conducting samples, since a tunnelling current peak at the oscillation reversal point of the lever can easily dominate the force feedback signal.

Moreover, piezo-electrically generated signals are very small and therefore the first stage detection circuit needs to be close to the sensor in order to ensure sufficient S/N ratio. In fact, this required the development of a low temperature ($T < 5$ K) pre-amplifier, fully UHV compatible and bakeable. In addition, the design ensures continuous operation over the full temperature range up to room temperature without re-calibration of the pre-amplifier. The use of a low temperature pre-amplification circuit requires careful consideration of the absolute temperature of the sample-tip system. The sample temperature can be precisely measured by a thermally well coupled Si diode.

In contrast, the temperature of the sensor tip is not directly accessible, since the wiring of a temperature sensor would change the thermal situation completely. Therefore,

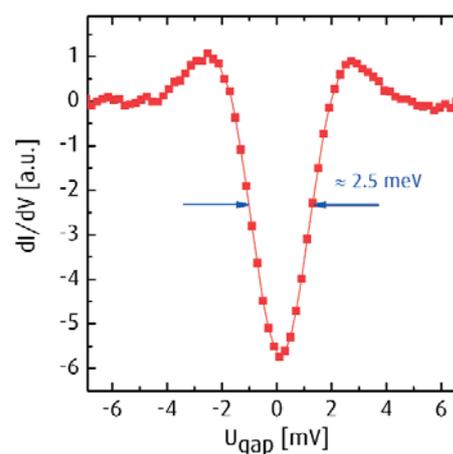


Figure 3: STS reveals the superconducting gap of a Niobium STM tip, sample: (Au(111)).

we utilised a superconducting Niobium ($T_C \sim 9.3$ K, $2D_{T=5K} = 2.53$ eV) STM tip for modulation spectroscopy on an Au(111) surface. The width of the superconducting gap is directly determined by the absolute temperature of the tunnelling gap and thus represents the most direct access to the sensor temperature. The temperature is deduced from energy gap width $2D$ using BCS theory (uncertainty roughly 1K). With active AFM pre-amplifier, the measured width of $2D \sim 2.5$ eV ($f_{Mod} = 1967$ Hz, $V_{Mod} = 150$ μ V_{pp}) corresponds well to what has been measured by S. H. Pan et al. in 1998[3]. Moreover, this measurement proves that the STM/STS performance of the system is not altered and the energy broadening is exclusively determined by temperature and modulation amplitude.

So far, QPlus sensors are not commercially available from sensor suppliers. We thus established a reliable manufacturing process for sensor assembling on dedicated LT STM carriers. The best image performance was achieved using conventionally wet-chemically etched tungsten tips, glued onto the tuning fork.

This approach is very simple and does not involve a sophisticated process technology. In addition, the use of tungsten tips allows for highest performance in simultaneous or alternative STM/STS operation.

Measurements on Si(111) 7x7 show that tunnelling current and vibrational signal are clearly separated. In addition, benchmark measurements on NaCl with a typical corrugation of approx. 10pm prove that resolution on insulation samples is competitive to best cantilever based AFM results. Measurements with comparable quality have been performed at LN₂- and room temperature to prove the performance of the detection circuit at elevated temperatures. In addition, a spring suspension compensation mechanism allows for constant vibrational de-coupling without re-adjustment.

In summary, a low temperature QPlus AFM detection has successfully been developed on a well proven experimental platform without compromising on the original STM/STS performance. Especially the combination of both QPlus AFM and STM modes on an extremely high performance level has a great potential for various applications, including AFM based molecule manipulation on

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insulating surfaces representing a key application of the PicoInside project.

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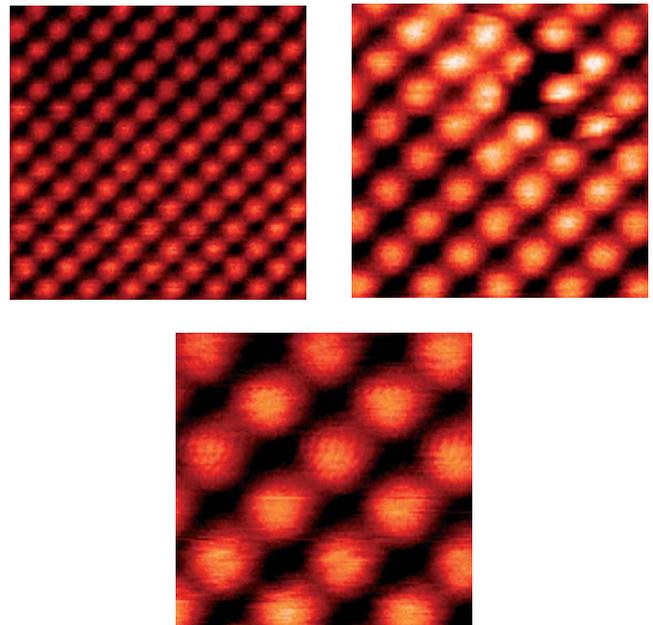
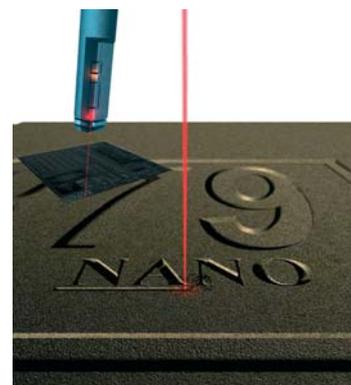


Figure 4: Atomic resolution on NaCl(100) using QPlus force detection at $T = 5$ K. No FFT filtering applied. Measurement in df feedback (not constant height). NaCl lattice constant 5.65 \AA (fcc), typical corrugation 10 pm . Measurement parameters: $df = -2 \text{ Hz}$, oscillation amplitude $\sim 1.5 \text{ nm}_{pp}$, $f_{res} \sim 25 \text{ kHz}$, $Q = 36671$. Image size from left to right: $10 \times 10 \text{ nm}^2$, $6 \times 6 \text{ nm}^2$, $3.5 \times 3.5 \text{ nm}^2$.



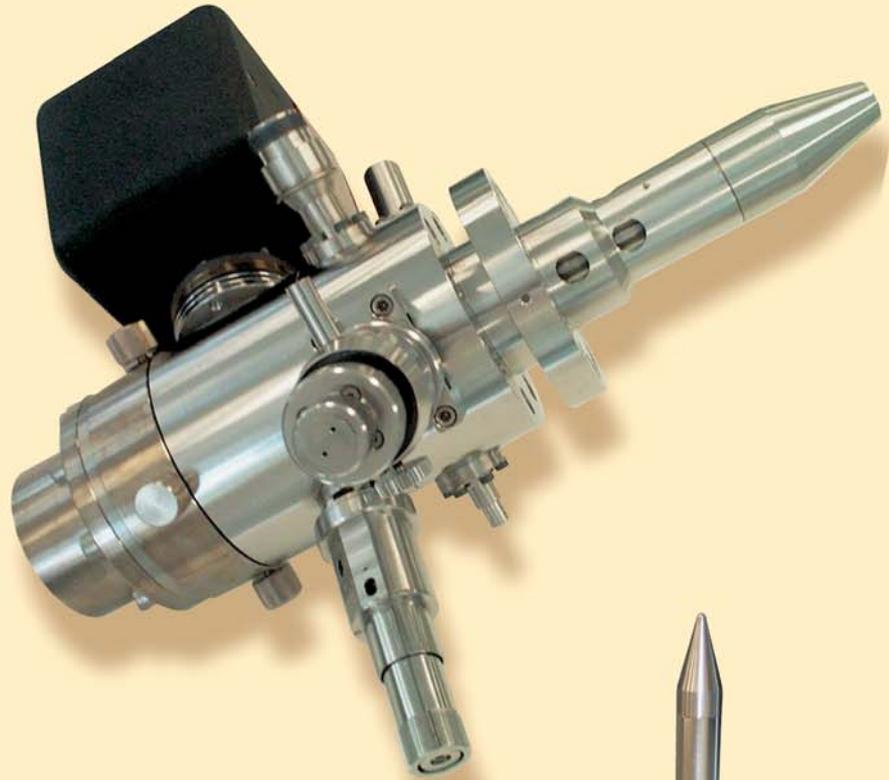
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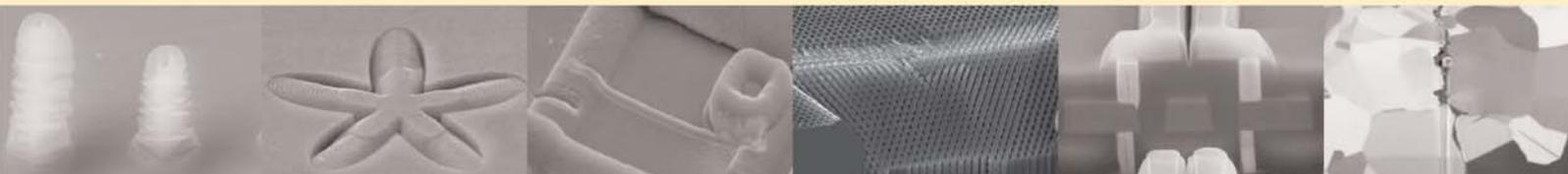
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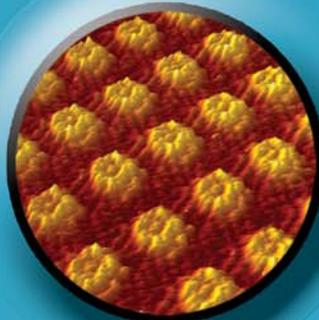
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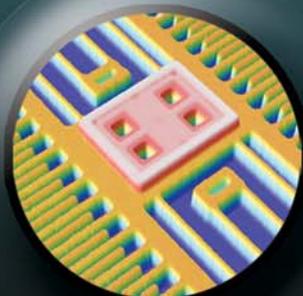
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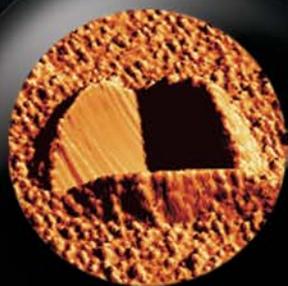
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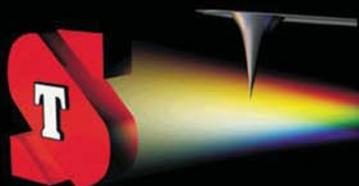
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