Challenges and Opportunities for Focused Ion Beam Processing at the Nano-Scale

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Introduction

There is a solid consensus that new methods of structure fabrication, placement, and organization within the sub-10-nm resolution gap are urgently required to meet existing challenges in condensed matter, semiconductors, and biotechnologies. Standard top-down methods such as resist-based lithographies even used at the shortest available wavelengths have clearly identified limitations. On the other hand, bottom-up approaches, like scanning probe manipulation techniques, remain challenging when trying to generate reproducible, functional, and addressable nano-structures. Therefore, at the laboratory level new routes must be explored.

The patterning of samples using Focused Ion Beams (FIB) is a very popular technique in the field of inspection of integrated circuits and electronic devices. This is the case mainly for prototyping devices. The FIB technique allows 3D patterning of target materials using a finely focused pencil of ions having speeds of several hundreds of km/second at impact. Although most metals can be used in FIB technology as pure elements or in the form of alloys, gallium (the Ga^+ ion) is preferred in most cases. Practically, FIB patterning can be achieved either by local surface defect generation, by ion implantation, or by local sputtering. These adjustments are obtained easily by varying the locally deposited ion fluence with reference to the sensitivity of the target and to the selected FIB processing method.

High-Resolution Nano-Writing Instrument Using Ions

A few years ago we decided to develop a FIB system having global specifications compatible with nano-fabrication experiments [1], that is, a deep sub-10 nm nano-device fabrication capability. In particular we were aiming at focusing on a target—a FIB spot capable of fabricating directly and reproducibly nano-devices on materials as diverse as III-V semiconductors, metallic layers, thin- or ultra-thin films, or even atomically thin suspended graphene sheets [2].

The ion emitter. The Liquid Metal Ion Source (LMIS) is a simple, compact, and quasi-perfect point-type ion source. Therefore, we have examined several kinds of LMIS and decided to optimise a gallium ion emitter. This optimisation allowed us to obtain a record on-axis angular intensity of up to 80 μA/str and an extreme stability of the emission process (current variations less than 0.5 percent per hour). At this stage, long unattended nano-fabrication processes without the need of thermal heating cycles to recover adequate emission characteristics can be envisioned, opening the route to practical and reproducible nano-patterning applications.

The ion optics. Our FIB column concept was built on a patented concept where the source region is physically separated from the focusing and transport optics via a beam-defining aperture. The lens designs, shapes, and operation modes have been evaluated and selected against FWHM probe diameters

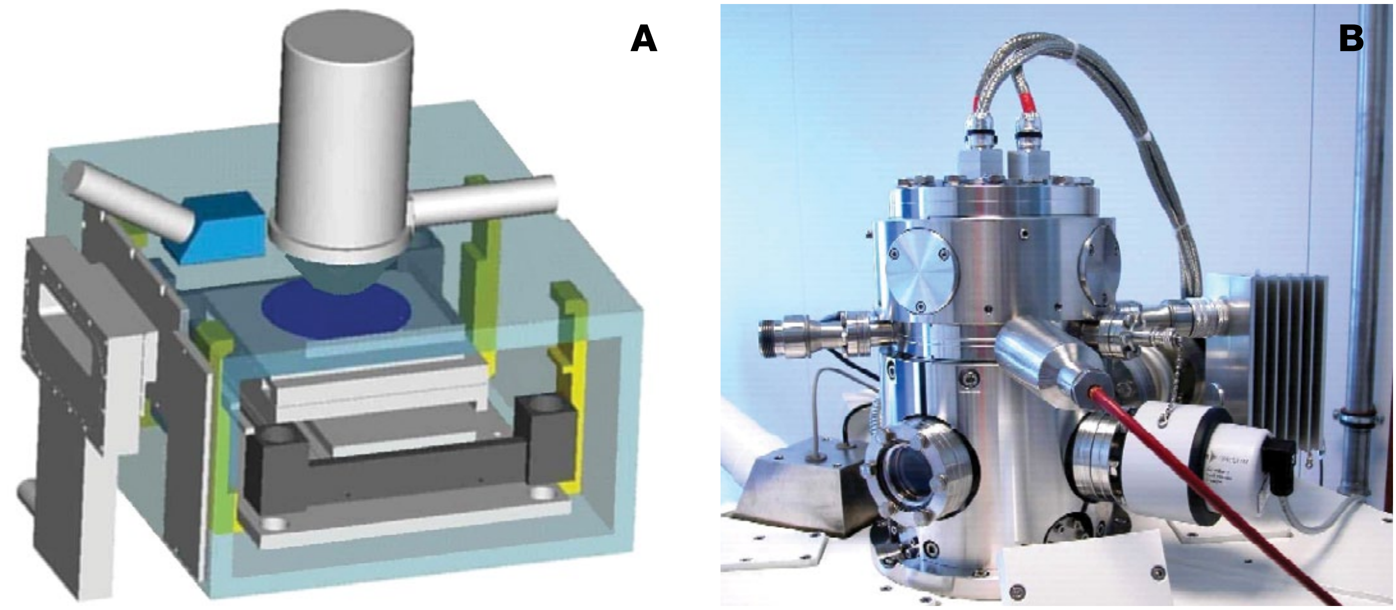

Figure 1: (A) Schematic view of the high-resolution FIB nano-writer. A compact single beam ion column is placed perpendicularly to a laser interferometer controlled stage (2 nm accuracy). (B) Picture of the instrument in our laboratory facilities.

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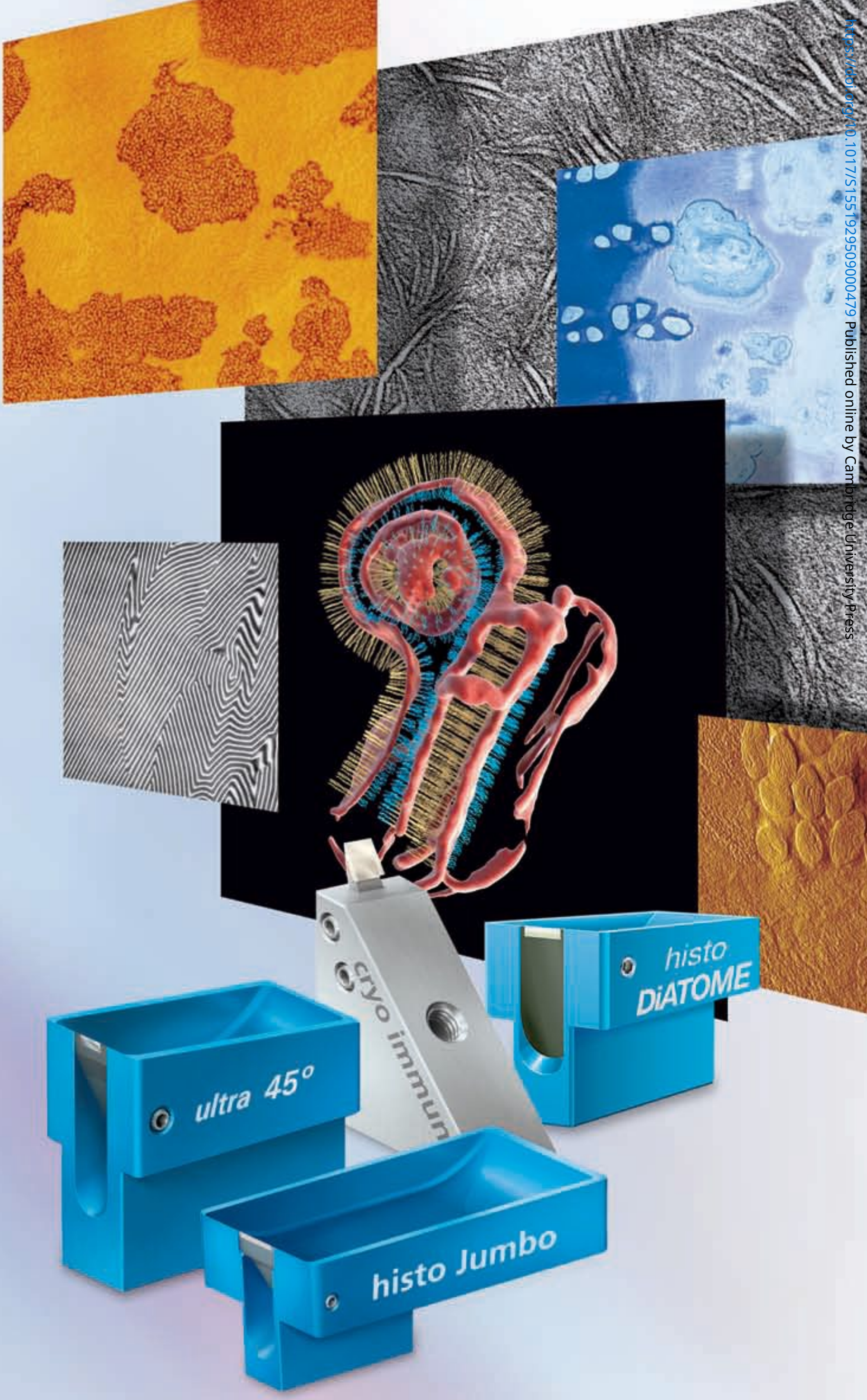

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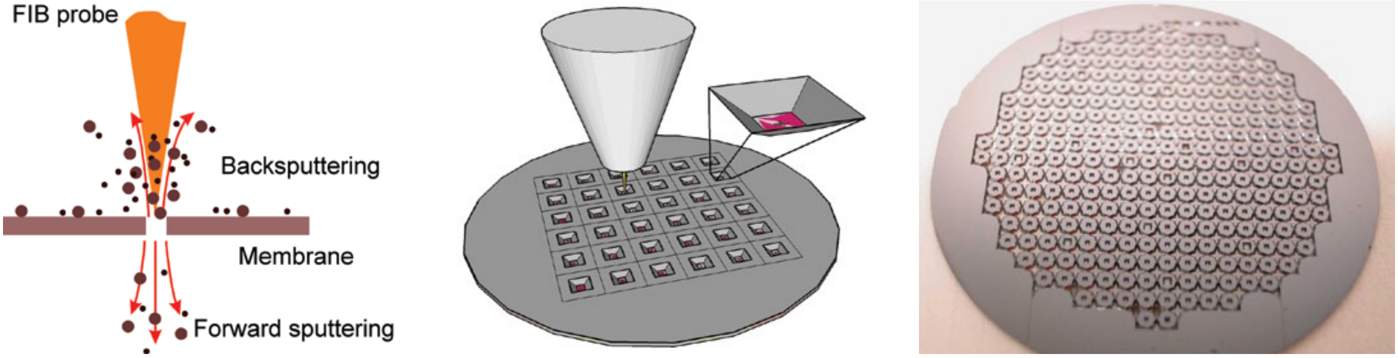

Figure 2: (A) Schematic of the membrane FIB sculpting process with back and forward sputtering. (B) Batch patterning of a complete wafer of individual nano-pores. (C) Picture of a two-inch wafer containing 284 identical devices patterned in a one-shot FIB experiment.

(5-nm calculated resolution [2]), and, owing to the strongly collimated mode, an efficient rejection of off-axis emitted ions is achieved, leading to minimum probe current tails. In practice our system is capable of delivering a deep sub-10-nm ion probe, transporting a probe current of several picoamperes within a probe current distribution that follows a pure Gaussian or "bell type" distribution.

The FIB nano-writer architecture. The FIB system we have developed (Figure 1) is based on a nano-writer architecture concept, that is, a single FIB column positioned over a high-accuracy sample stage and operated with short working distances (WD) to achieve a strong geometrical source demagnification. In our "single beam" configuration we have "sacrificed" the *in-situ* Scanning Electron Microscopy (SEM) imaging capability for the following reasons: (a) Many of the nm-sized structures we aim at fabricating (surface defects with no SE contrast, sub-surface ion beam mixing, etc.) are difficult or even impossible to monitor using a SEM; (b) *in-situ* electron bombardment and the resulting energy deposition may be a source for perturbation when attempting to pattern nanometer-scaled devices (enhanced defect diffusion in sensitive III-V semiconductors) or for material reconstruction through surface diffusion (structure clogging); (c) because our FIB optimum WD is only a few mm, *in-situ* SEM imaging capability would be too limited.

Thus, our experimental approach does not require an *in-situ* SEM imaging capability. In our case, the extreme positioning accuracy in reading (using the Scanning Ion Microscopy imaging mode) or defining alignment marks and the possibility of achieving extended automated patterning tasks (Figure 2) without operator control are key advantages.

FIB Patterning at the Nano-Scale: The Resolution Limiting Factors

Prior to considering FIB nano-fabrication of structures on a substrate [2], it is important to keep in mind that this process encounters several kinds of limitations that are independent of the ion optics itself. They can be summarized as follows:

Sample characteristics. As FIB machining is a direct writing process, a first limitation in fabrication of features originates from the physical characteristics of the target (composition, hardness, electrical conductivity) and its geometrical features (surface roughness, homogeneity). Indeed target characteristics have a huge effect both on etching speeds and the resulting structure geometry.

Spatial extension of the defects induced by FIB irradiation. These effects may originate primarily from a lack of selectivity of the FIB probe. Ultimately they are caused by the scattering of the implanted ions inside the target material and by the radiation enhanced diffusion (RED) effect, taking place only during the ion bombardment process.

Redeposition of sputtered materials. Scanning an energetic ion beam over a substrate allows etching patterns of arbitrary shape as a result of physical sputtering. This sputter

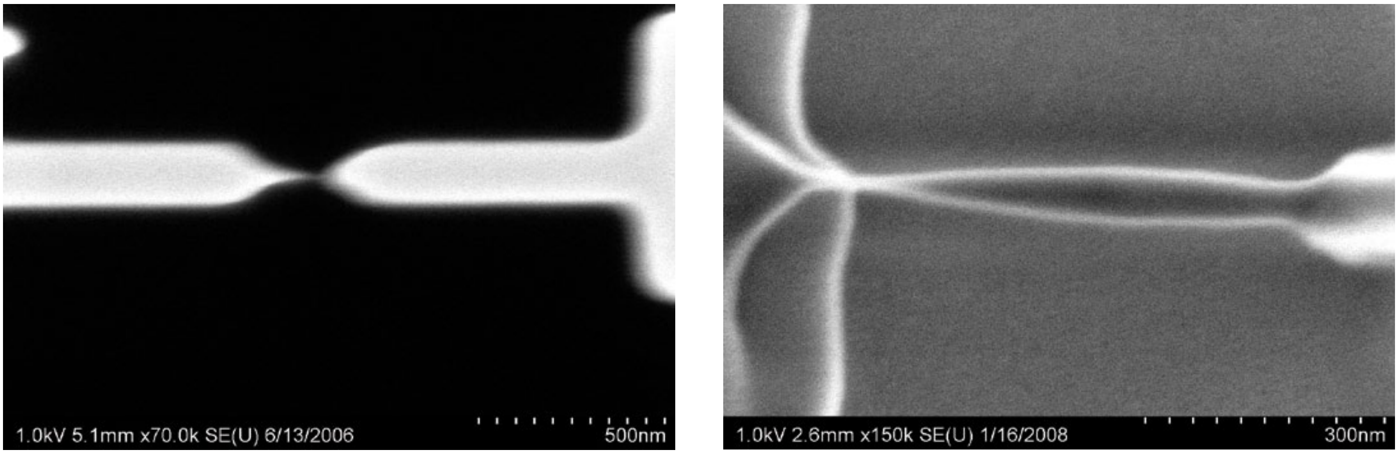

Figure 3: (A) Scanning electron microscopy image of a nano-wire sculpted within a SiC thin membrane. Minimum width is less than 7 nm. (B) SEM image of a nano-wire sculpted in a 2-3 nm thick graphene sheet. Note that the nano-wire seems to roll on.

rate Y (Y ~ sputtered atoms/primary ion) is found to depend on both the sample and primary ion species.

Local material fluence and swelling. It has been observed that low-dose spot impacts exhibit reproducible bumps or volcano shapes. This effect of swelling originates from structural changes at the surface due to ion beam-induced damage and the addition of implanted ions.

Ultra-Thin Membranes as an Ideal Template for FIB Nano-Processing FIB nano-engraving of a membrane is interesting if the membrane can be made homogeneous, conductive, and thin enough, that is, with a thickness comparable to the projected range of the incoming ions. Such thin targets allow a better ion-deposited energy localization and a reduction of straggling effects with a membrane thickness between 10 nm and a few tens of nanometers. In addition, FIB processing of ultra-thin membranes allows some interesting technical features because there is a clear separation between the upper face impacted by the incoming ions and the lower face that remains preserved. These advantages are illustrated in Figure 2A.

Most of the energy deposited by incoming ions and collision cascades remains located at the upper side of the patterned membrane, thus preserving the backside. Moreover, scattering effects of the incoming ions allow efficient guiding effects, that is, auto-focusing through the nano-pore itself. Finally, the engraved material is efficiently ejected, thus allowing very sharp edges to be obtained. In addition, there is almost no possibility for sputtered material to redeposit on the backside of the foil because these particles are ejected via a scattering phenomenon with highly directive transfer of kinetic energy. All these advantages can be used to pattern thin self-supported membranes (SiN or SiC) within patterns as diverse as pores or nano-wires.

1 - Nano-Wires Direct Fabrication

We have demonstrated the possibility of fabricating nano-wires within thin SiC membranes (Figure 3A), but a new effort is aimed at assessing the promise of FIB direct engraving of graphene sheets using our finely focused pencil of gallium ions. We have shown that graphene deposited within atomic-thin layers could be engraved within structures such as rings or nano-wires down to a few nanometers (Figure 3B) while preserving the interesting physical characteristics of the materials. In light of our experiments, it now appears possible to achieve good quality and reproducible fabrication of free-standing graphene structures using an ultra-sharp pencil of ions. Furthermore, the potential of this technique may be considerable for many applications in the field of electromechanical device fabrication.

2 - Nano-Pore Containing Devices

Nano-pores in thin solid-state membranes, individual or as arrays, have found a growing number of applications, for example as stencils or masks to grow or deposit nano-structures or to fabricate single-molecule electronic detectors or sensors. The latter is probably the more important and consists of using the membrane as a dividing wall in an electrolytic cell and drawing charged molecules by an electric field through the pore. The resulting current blockage signal reveals information about the passing molecule so that, for example, DNA or proteins can be manipulated and studied at a single-molecule level in native conditions.

We have successfully demonstrated the possibility of applying FIB technology for the direct fabrication of devices with drilled pores having diameters down to a few nanometers (Figure 4) while reaching very interesting throughput capabilities. Indeed identical devices requiring individual FIB processing times around 100 ms can be fabricated with quantities compatible with Lab-on-a-Chip device research and commercialization requirements.

Conclusion

The examples here suggest a major paradigm shift for FIB processing because before these achievements, nano-fabrication with scanned focused ion beams, as a sequential process, was not expected to provide a mass-production capability of devices. The success of our FIB approach in this application is due to the very high performance of our instrument, its capability to work "in-line" with standard lithography techniques, and to the high added value of the final device. Owing to these performance characteristics, there is now a promising future for Focused Ion Beam processing at the nano-scale. MT

References

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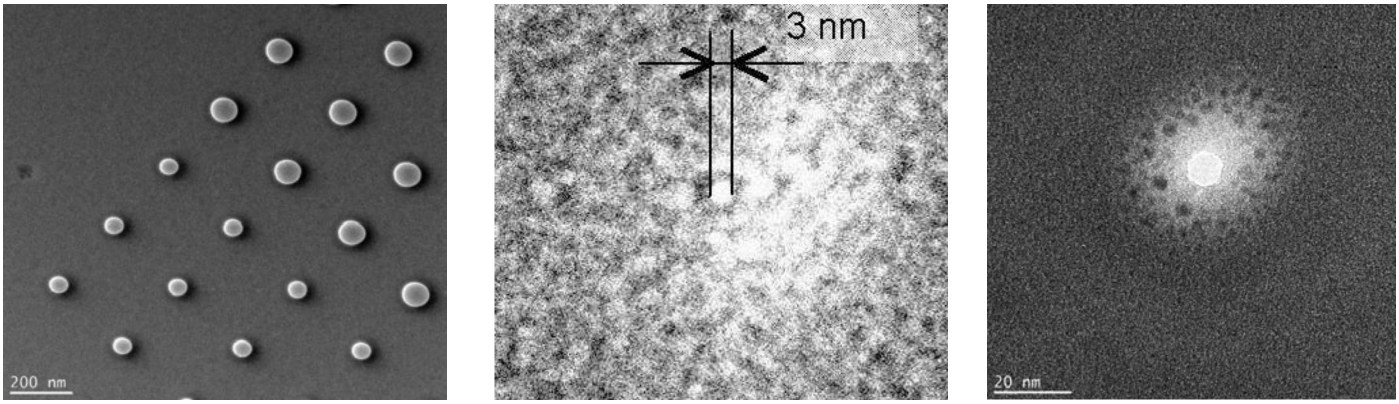

Figure 4: (A) TEM image of an array of perfectly circular pores drilled in a 50 nm thick Si_xN_y membrane. (B-C) TEM images of pores of different sizes drilled respectively in a 20 nm thick SiC and 50 nm thick Si_xN_y with a dose ~10^6 ions/pt. The smaller pores have a similar aspect ratio length/diameter ~ 7.