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APPLICATION NOTE

3D Nanoprototyping with a DualBeam instrument

Rapidly design, create and inspect micro- and nano-scale functional prototype devices

Devices and structures made on a nanoscale now have a number of real world applications, and the potential for further development and uptake is still growing. However, converting the latest ideas and designs into something real for research, testing and prototyping can pose a significant technical and financial barrier.

Thermo Fisher Scientific offers a smart and efficient way of turning nanoscale designs into reality. Using finely focused particle beams along with state-of-the-art patterning engines and precision stages integrated into our latest focused ion beam (FIB), scanning electron microscopes (SEM) and DualBeam™ instruments (combined FIB and SEM) offer an efficient way of turning your nanoscale designs into reality.

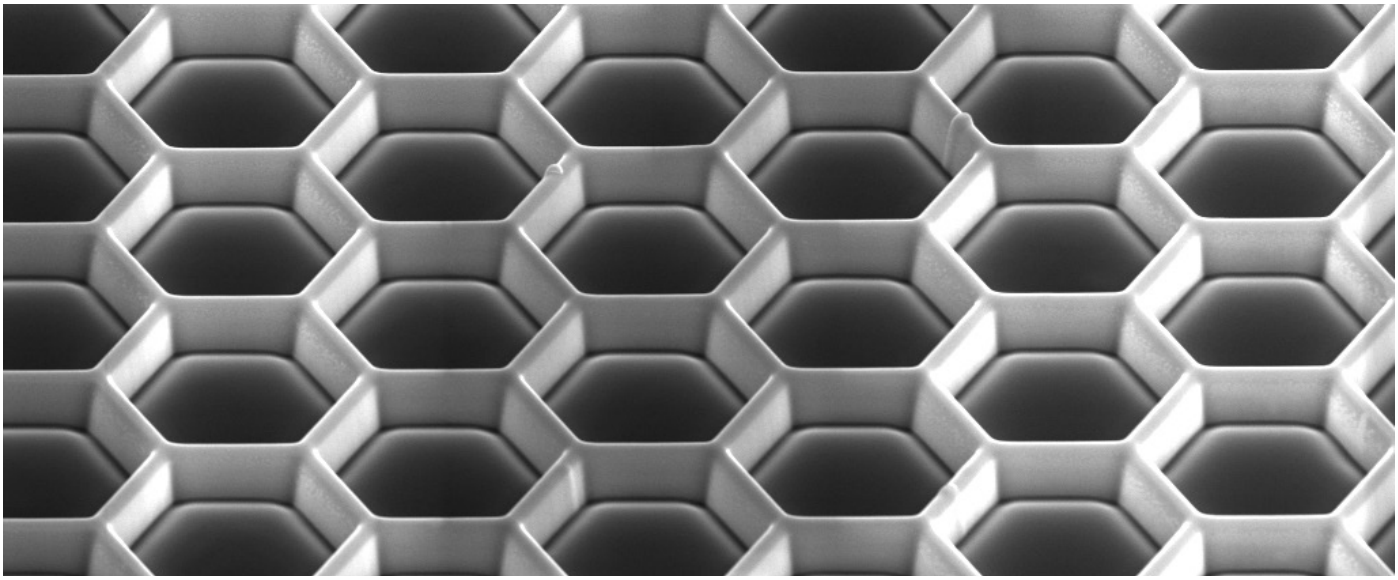

Figure 1. Secondary electron image of a honeycomb structure milled into a silicon substrate using the FIB on a Thermo Scientific™ DualBeam instrument. The hexagonal shapes are accurately formed using the Thermo Scientific integrated patterning engine designed for advanced patterning.

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About DualBeams

The DualBeam combines a focused ion beam (FIB) with a high resolution scanning electron microscope (SEM). Both FIB and SEM look at exactly the same point in space, giving the ability to directly mill nanostructures and immediately observe them non-destructively. Prototype nanostructures can be designed and created extremely quickly. Besides providing non-destructive imaging, the electron beam itself can be used for lithography and depositions on a nanoscale. Overall, the DualBeam, with fully integrated and automated FIB and SEM columns, a 16-bit patterning engine, gas injectors (GIS) and high-precision Piezo driven stage, offers a whole range of nanomachining, characterization and sample preparation options for various techniques.

Nano-prototyping with a DualBeam

Prototyping with a DualBeam describes the creation of any nanoscale or microscale (functional) device using the FIB, the electron beam and/or the beam chemistries.

A focused ion beam (usually gallium ions), accelerated to the keV energy range, will directly sputter away the material it impacts. By focusing the ions to a fine beam and by accurately steering and switching the beam, the sputtering process can be controlled to produce complex, even three-dimensional shapes, down to a nanoscale. In order to achieve the best results from ion beam milling, some additional factors need to be accounted for, such as optimum milling angle, beam shape and most importantly, redeposition. During the ion milling process, the very small amounts of sputtered material are ejected in a plume away from the impact site. Much of it is ejected at a high angle, that is, back in the same general direction that the ion beam came from, and is eventually pumped away. However, some of the material is ejected at a lower angle and can build up, or redeposit, in the small local features created by the FIB, which could cause problems. In order to prevent unwanted artifacts such as redeposition, a milling strategy is used. The simplest, and most commonly used, method is to allow the FIB to mill the same pattern many times in quick successions rather than just once. An example of the difference between these two milling methods, a nano-fluidic trench, is shown in the images of Figure 3. Obviously, the clear "U" shaped channel is the best result in this case. Multiple passes of the ion beam over the pattern are the best strategy for generating this type of structure.

Figure 2. Inside a DualBeam chamber.

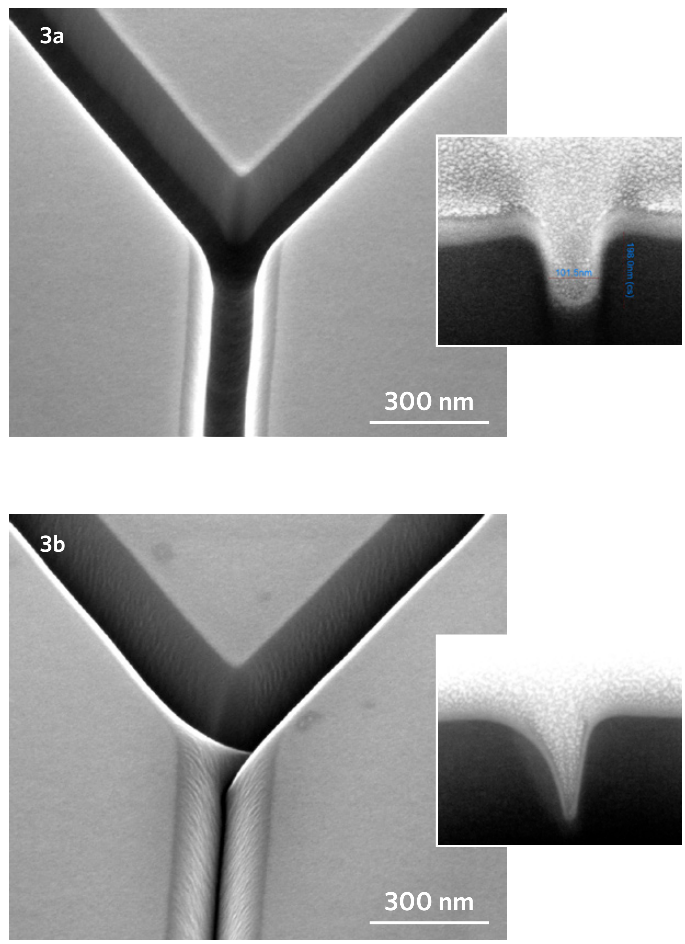


Figure 3. Detail of a nanofluidic trench (the insert shows a FIB cross section of the trench imaged in a TEM). Optimized FIB patterning strategies (3a) yield good profile control and clean, vertical sidewalls. Typical e-beam lithography patterning strategies (3b) do no account for the specificities of the ion beam milling process, therefore yielding redeposition and/or unwanted milling.

FIB milling strategies

Milling difficult charging samples

Although the ion beam can mill any material, including diamond, not all materials mill in a completely straight forward way. Soft polymer materials may deform under the beam and require freezing or the use of beam chemistry to mill them successfully. The varying grain structure of other materials, such as copper, may make the milling uneven so that beam chemistry is required to prevent some grains milling faster than others. Another common problem is charging of a non-conducting sample while it is being milled with the ion beam, which causes the beam to drift, distorting the shape to be milled. This is illustrated in Figure 4. The charging problem arises from the build-up of positive charge from sample surface ions, which eventually makes a large enough field to deflect the ion beam. A very simple solution is to coat the sample with a conducting layer. This can even be done *in situ* with use of beam chemistry. However, some samples must not be coated and some are just too big or too topographic to be coated. The most elegant and general way of enabling the milling of non-conducting samples is to use a flood of low energy electrons to neutralize the ion beam's charge on the surface. This method of using charge neutralization is shown in Figures 5-6.

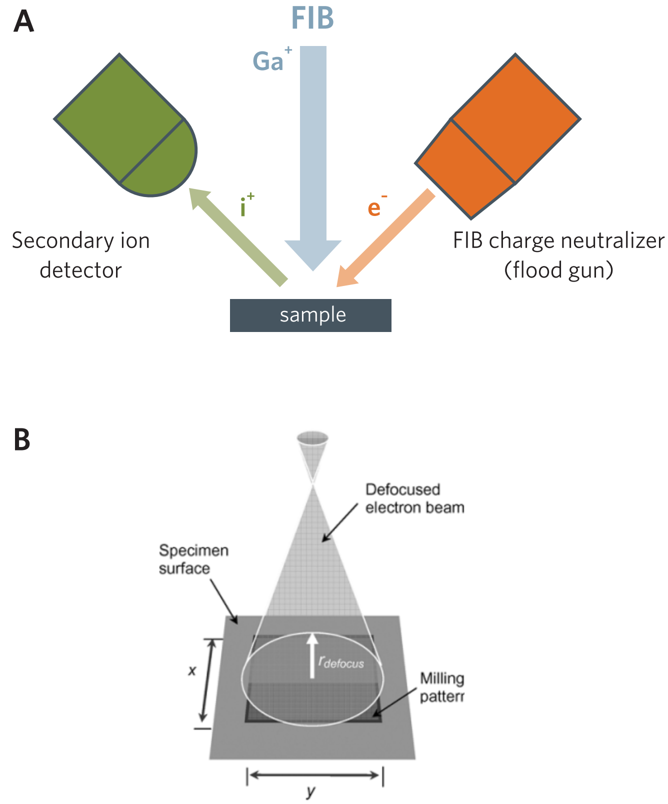


Figure 4. Solutions for efficiently neutralize positive charge build-up resulting from FIB milling on non conducting materials, such as a dedicated low energy electron flood gun (6a), or a defocused SEM beam irradiating the surface during FIB milling (6b) can be used in DualBeams.

D.J. Stokes et al, J. Phys. D: Appl. Phys. 40 (2007) 874–877.

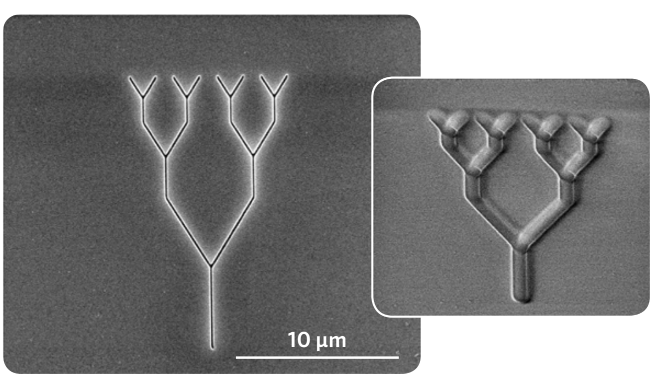


Figure 5. A flood of low energy electrons is used to neutralize the charge built up on the surface by scanning with the positively charged gallium ion. A nano-fluidic channel can be cut perfectly even in a quartz substrate. Without charge neutralization (see image insert) the ion beam is deflected during milling and results in an inaccurate cut.

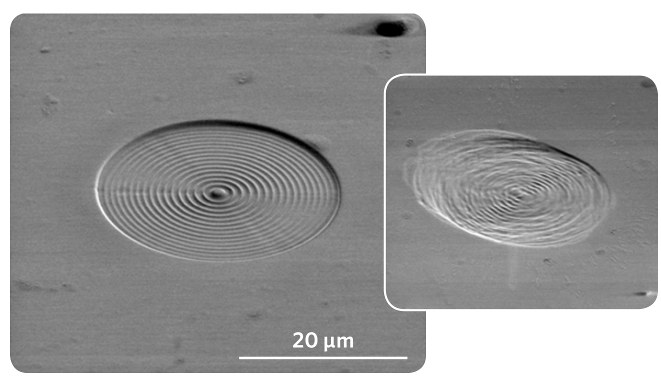


Figure 6. Using charge neutralization a circular pattern can be perfectly milled in glass. When the charge neutralization is turned off (see image insert) the ion beam moves erratically during milling and results in poor milling.

FIB prototyping in photonics

A focused ion beam is a remarkable tool as it can mill any material to a very fine scale. Polishing an edge with an ion beam can leave it almost atomically smooth. When the ion beam is integrated into a modern high-precision computer controlled system, these techniques can be combined, enabling nanoscale feature to be created on demand. Small features that guide and interfere with light need to be made with a high degree of accuracy at the nanoscale. Even more critically, many of these features have to be replicated precisely and placed at exactly the correct position to achieve the required effect. In most cases these photonic structures have been made using lithographic techniques, which can be a slow process, is often expensive, and restricts the selection of materials that can be used to make the structures. With a FIB, on the other hand, a structure can be conceived, designed, and made by one person using a range of materials. Although FIB will likely never compete with the commercial production of photonic materials, it is still the fastest way to prototype these structures.

The rapid prototyping of a variety of photonics devices has been demonstrated, including: split-ring oscillators (Figure 7), microlenses (Figure 8), nanoantennas (Figure 9), waveguides (Figure 10) and more.

A DualBeam not only allows writing of the prototype devices, it can also immediately perform a quality control of the shapes. This can be done via top down inspection (Figure 7a), 3D FIB cross-section (Figure 7b), or Slice and View Software applied through the structure. In the case of the split-ring oscillator (Figure 7), the aspect ratio of the the milled trenches is large, which will give the pattern good optical performance. However, not all the trenches are consistent. This is due to preferential milling of different grain orientations within the gold. These problems may be overcome either by reducing the preferential milling with beam chemistry, or simply by milling longer (until the difference in trench depths is no longer significant).

DualBeams are also able to create patterns over a very large range of scales, very accurately. Structures with a critical dimension below 10 nm can either be milled or directly deposited. Using advanced patterning tools like the Thermo Scientific™ NanoBuilder™ Software (see pages 10-11), photonics nanodevices can be accurately written over hundreds of microns.

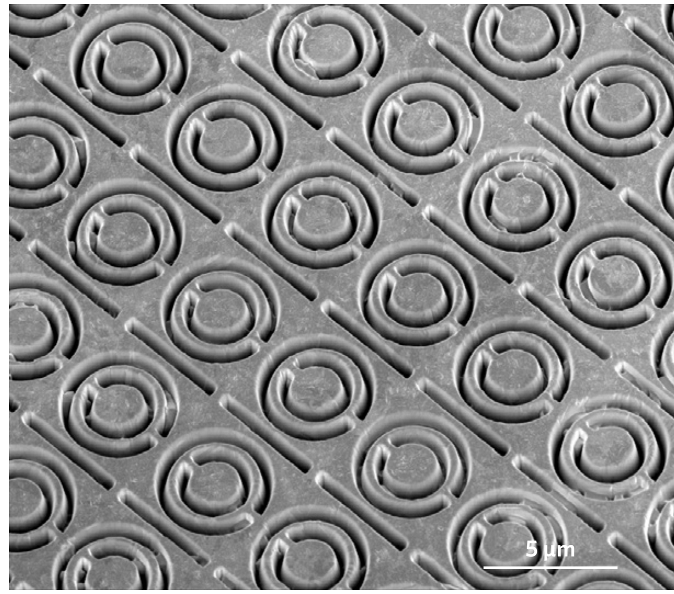


Figure 7a. A split-ring oscillator pattern directly milled using FIB. The FIB used a "vector scan" that allows the ion beam to be moved in the optimum direction for each portion of the pattern and allows multiple passes of the pattern to avoid any redeposition artifacts.

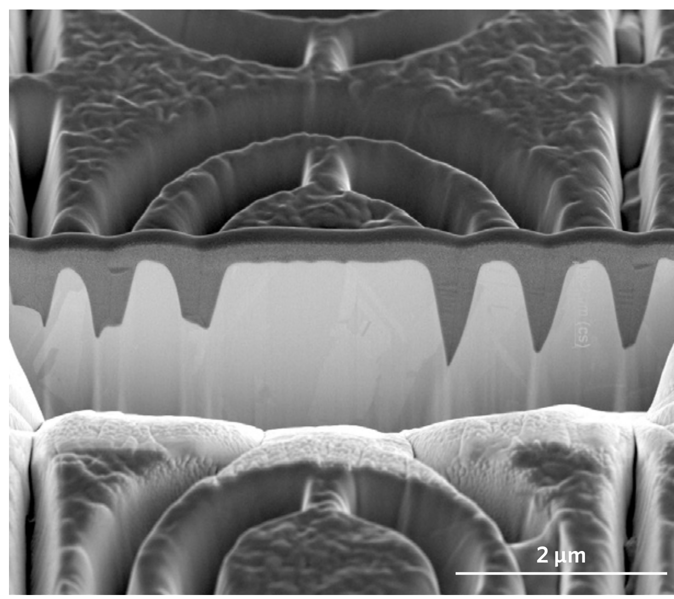


Figure 7b. A FIB-cut cross section through the split-ring oscillator.

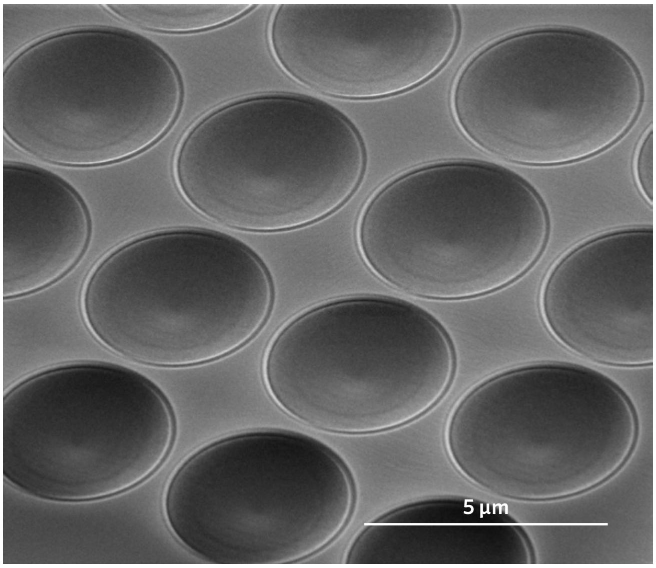


Figure 8. Secondary electron image of a micro-lens array milled into a silicon substrate. The lens shapes are accurately formed using our integrated patterning engine designed for advanced patterning.

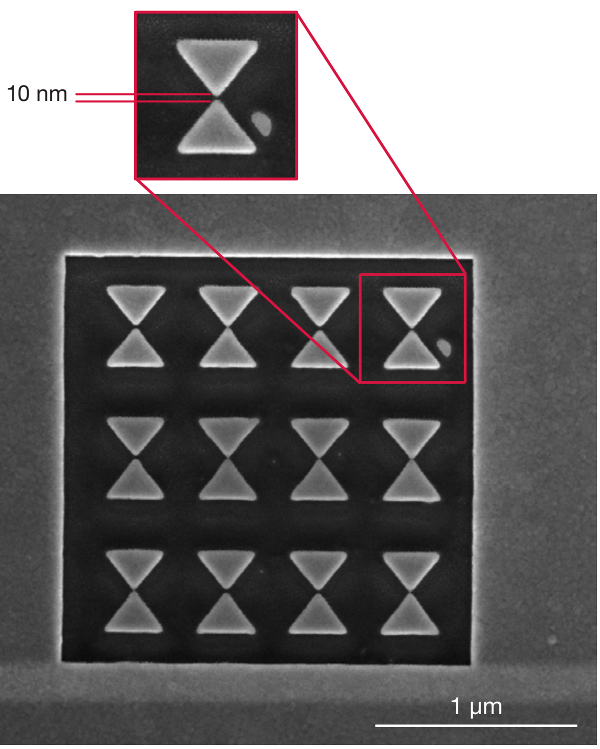


Figure 9. Nanoantennas in IR and VL – bowtie structures patterned on 40 nm Au/Si. *Image courtesy of CIC Nanogune - Spain.*

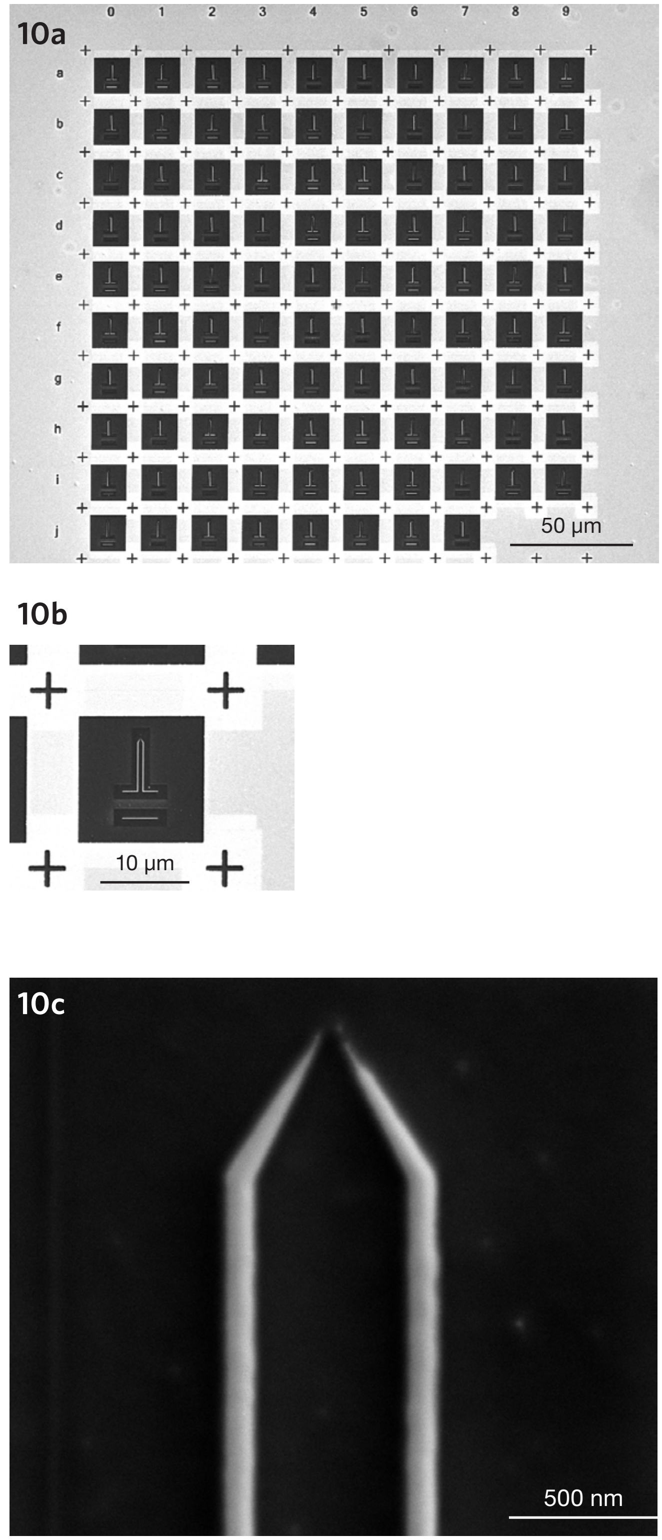


Figure 10. 10x10 array of IF plasmonic waveguides, min. feature size < 50 nm, written on a 40 nm Au/Si sample. (a) full array (b) one waveguide (c) waveguide detail. *Images courtesy of CIC Nanogune – Spain.*

Gas injection system

Enhanced FIB milling and direct deposition of materials using beam chemistries

Beam chemistry describes the injection of gases into the vicinity of the ion/electron beam impact zone. This gives some interesting additional abilities to FIB and SEM. For example, structures can actually be grown rather than milled when gas is introduced under an ion beam (Figure 11). Similarly, the electron beam is not only useful for imaging and analysis; it can also be used to deposit material, generating some of the smallest individual features currently possible.

Gas injection can either generally enhance milling by the ion beam or, more importantly, preferentially remove one material over another. These etching gases improve job processing speed and are often used on a micron or sub-micron level. Although depositions can be applied in the micron range, they are mostly performed at a nanometer scale where they are more relevant for prototyping. Depositions can be used to make mechanical, electrical (conducting or non-conducting) and optically interfering features.

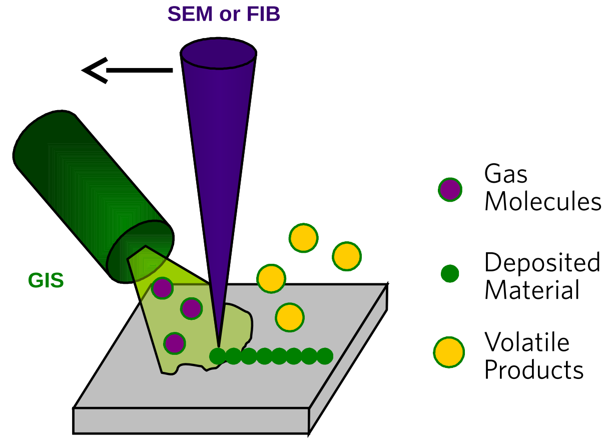


Figure 11. Illustration of FIB/SEM beam chemistry used to deposit material onto the surface of a sample.

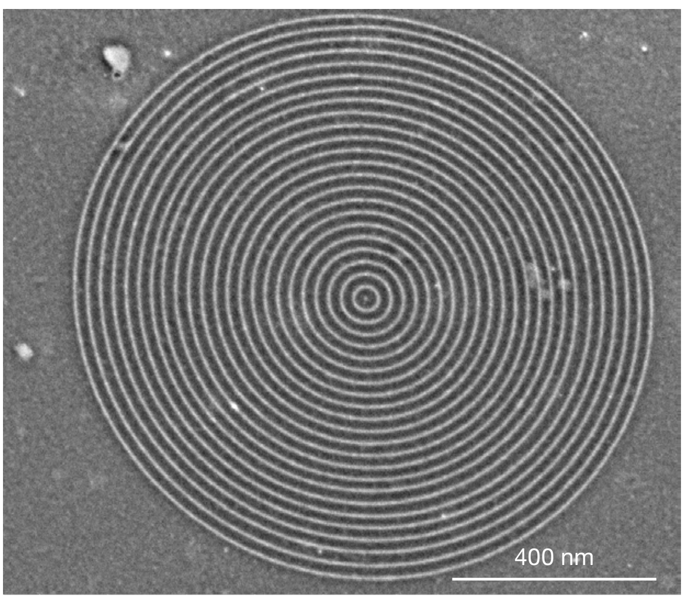


Figure 12. Electron beam induced direct deposition of Pt lines, arranged in concentric circles. Each line has a width of 7 nm, the pitch between two lines is 22 nm.

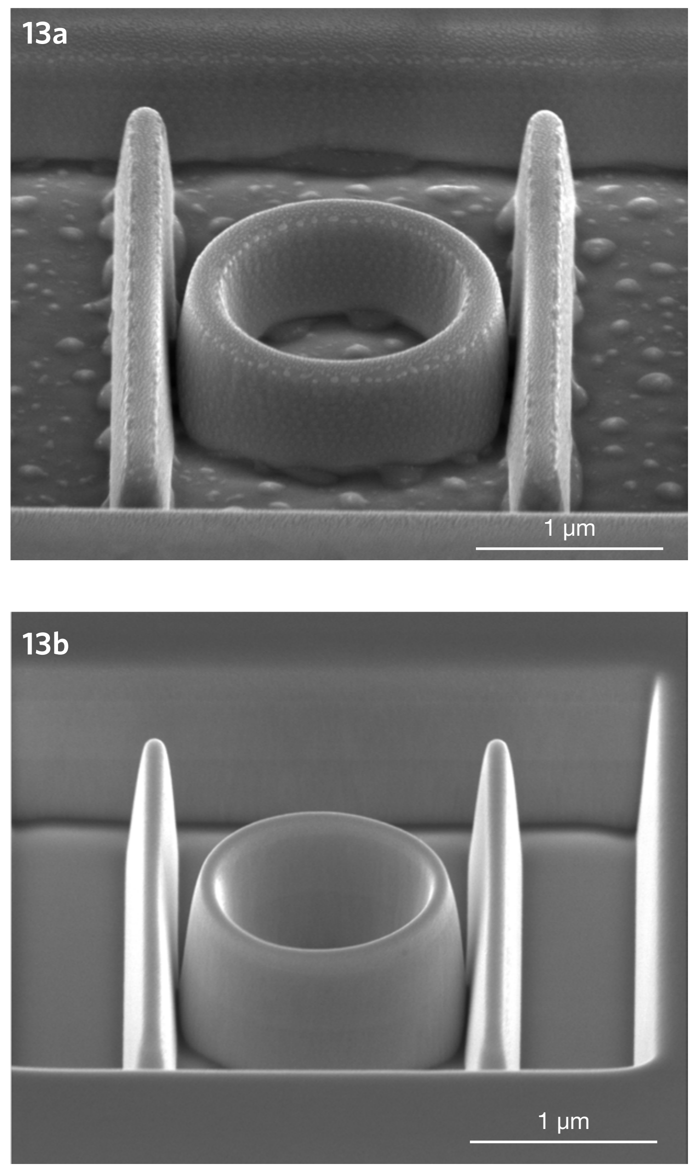


Figure 13. A ring oscillator milled by FIB into GaAs, without (13a) and with (13b) an enhanced etch beam chemistry. This specific beam chemistry passivates the milled surface and prevents the formation of gallium droplets.

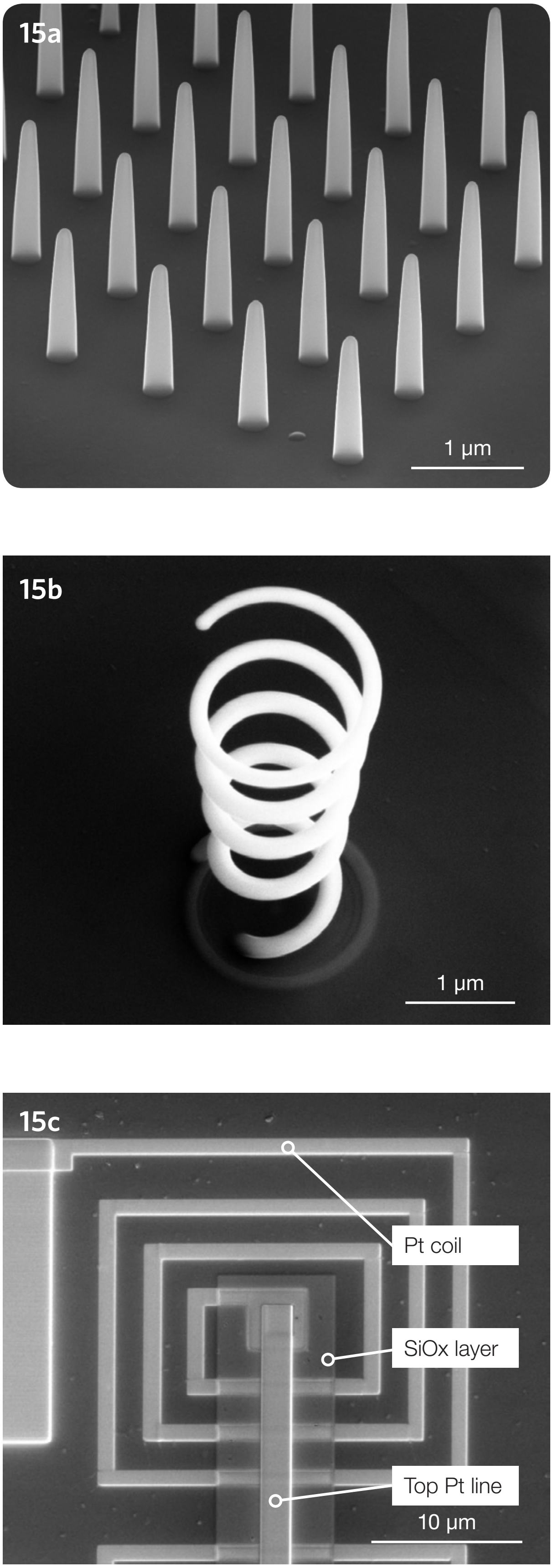


Figure 15. Examples of structures created by FIB induced deposition: (a) array of platinum pillars, (b) direct deposition of a nanospring, demonstrating the ability to produce intricate 3D structures and (c) detail of a dual-coil inductor device, composed of a platinum coil, an intermediate insulating layer (SiOx), and a top connecting platinum line. All are directly deposited in the same instrument, without any masking.

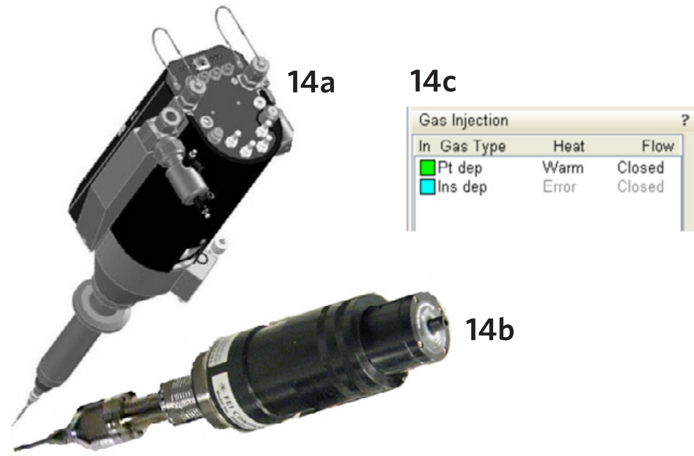


Figure 14. In-house Thermo Scientific technologies for optimized beam chemistries: (a) versatile single gas injector, (b) MultiChem advanced multi-channel injection system and (c) integrated user interface.

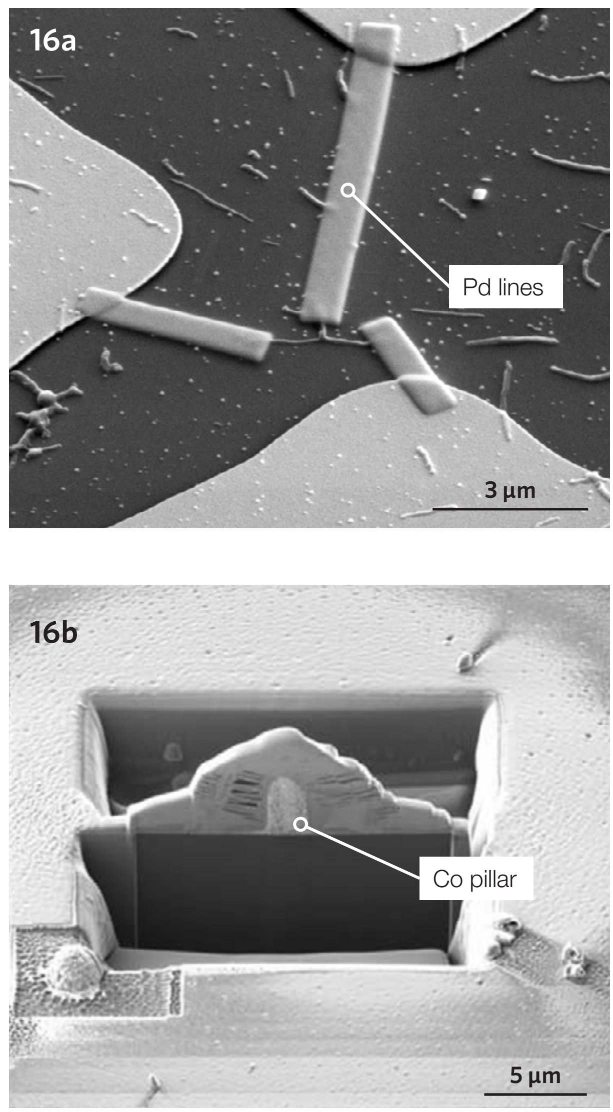


Figure 16. Examples of innovative Thermo Scientific beam chemistries: (a) beam induced deposition of palladium lines, for improved contacting of nanotubes and (b) cross-section through a cobalt pillar deposited by FIB. Cobalt deposition can be useful for generating nanowire and nanoparticle growth sites. Beam-induced deposition can also make regular patterns of magnetic materials.

Electron beam lithography

Electron beam lithography (EBL) has been used to prototype micro and nano features over a medium scale (square mm to cm) for some time. It is reasonably cost effective, and allows small features to be written consistently. It does, however, need a method for laying down and removing a resist layer, etching the substrate, and depositing specific materials for permanent features. Dedicated EBL systems require very accurate stages, very fast and accurate electron beam steering and switching, and are very expensive. However, new commercially available SEM systems are approaching some of the capabilities of EBL systems; this has made prototyping available to a whole new set of researchers. When these EBL capabilities are combined with a FIB system, it provides a wide range of possibilities. The ion beam can be used to make very small, site-specific features in any material; meanwhile the EBL can be used to make the more uniform structure over a much wider area. This would take a disproportionately long time if the FIB alone were used.

Figure 17a

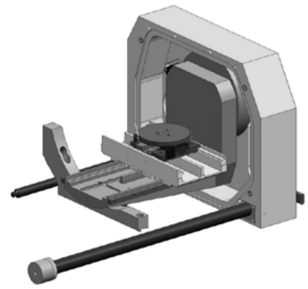


Figure 17b

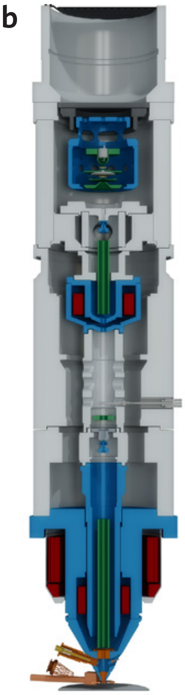


Figure 17. The Thermo Scientific™ Verios SEM and the Helios NanoLab DualBeam, formerly produced by FEI, feature unique integrated technologies that allow state-of-the-art electron beam lithography: a stable platform, highly precise and reproducible movements thanks to Piezo stages (a), and a high-resolution FEG column with conjugated fast beam blanker (b).

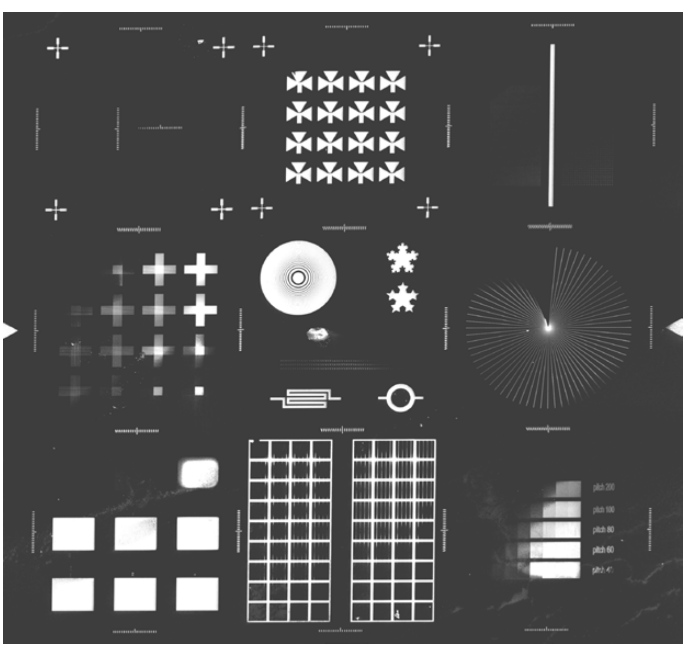


Figure 18. An electron beam lithography test pattern made over a 300 µm x 300 µm area. The high accuracy of the Piezo stage is used to place patterns in a serpentine fashion, starting horizontally at the bottom left. An array of 3 x 3 fields (100 x 100 µm each) were stitched with an accuracy of >300 nm. The Vernier scales indicate stitching positions (displacement of 20 nm per tick in opposing scales. Tick width 200 nm).

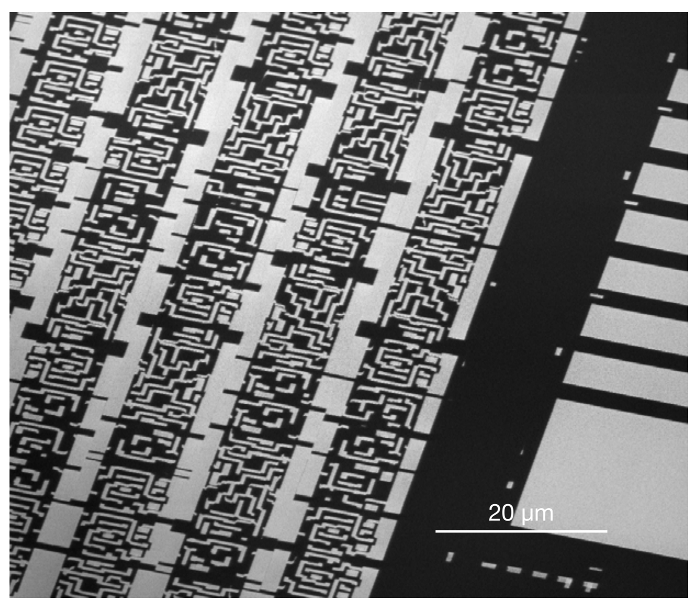


Figure 19. Example of electron beam lithography of an electronics device, accurately written at high speed with a high current (1 MHz writing speed with 1.4 nA beam current).

In addition to being able to make complex patterns over a reasonable area, electron beam lithography can also be used to make features with a critical dimension as low as 20 nm, in some cases. As it is a resist technique, different etching and deposition parameters can produce special effects. For example, reactive ion etching can be used to create very high aspect ratio holes. Etching times can be used to undercut or etch preferentially on a crystal orientation to give an angled hole. Thermo Scientific DualBeam instruments offer a unique integrated environment that takes advantage of the complementary benefits of electron beam lithography and ion beam patterning. They can perform rapid writing over multi-milimeter areas as well as direct, maskless fine writing. Both are accessible from the same instrument and even same interface (NanoBuilder Software, see pages 10-12) using the same CAD design. Precise and high-stability large-stroke Piezo stages are standard; they are built in to support accurate, multi-field writing over larger areas.

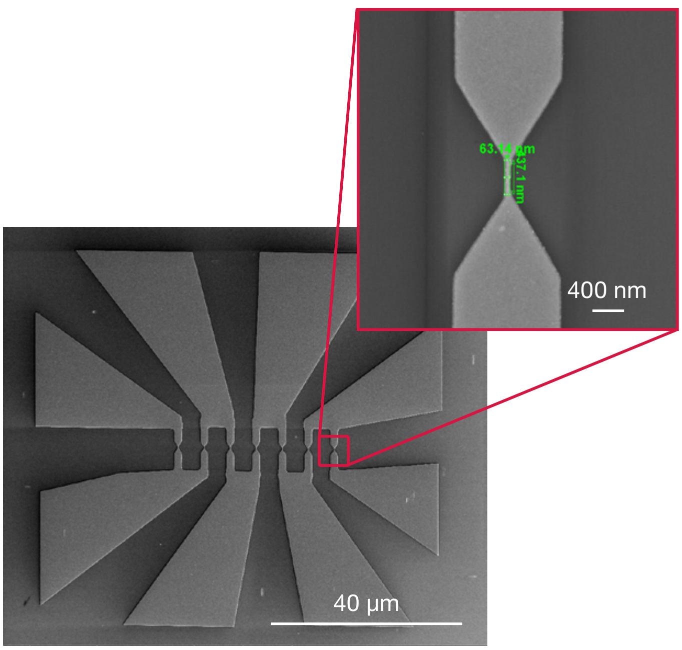


Figure 20. Electromigration structure created by electron beam lithography. NanoBuilder Software was used to drive the electron beam to transfer the structures on the resist.

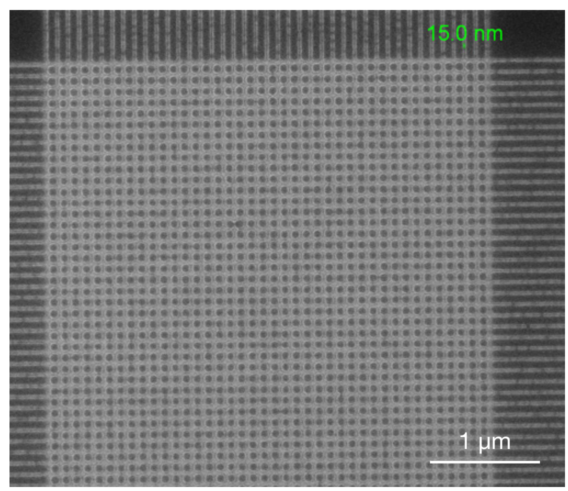


Figure 21. Series of crossing horizontal and vertical lines, transferred in an electron beam lithographic resist using a Helios NanoLab DualBeam. The line width is 15 nm.

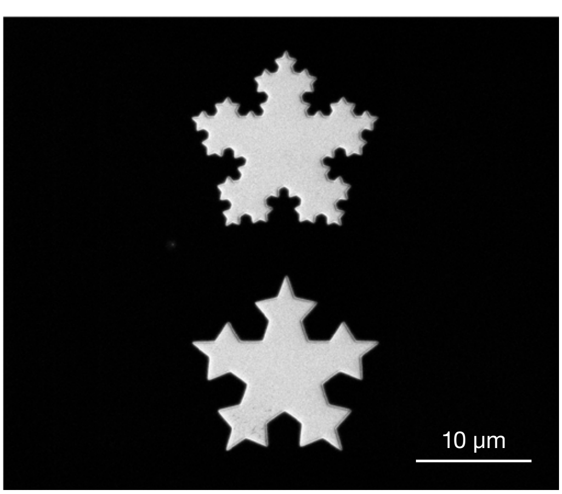


Figure 22. Electron beam lithography pattern in a resist, showing the precision build-up of a fractal pattern.

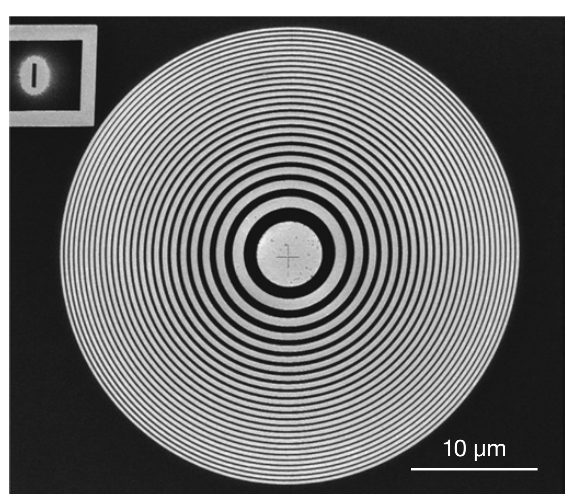
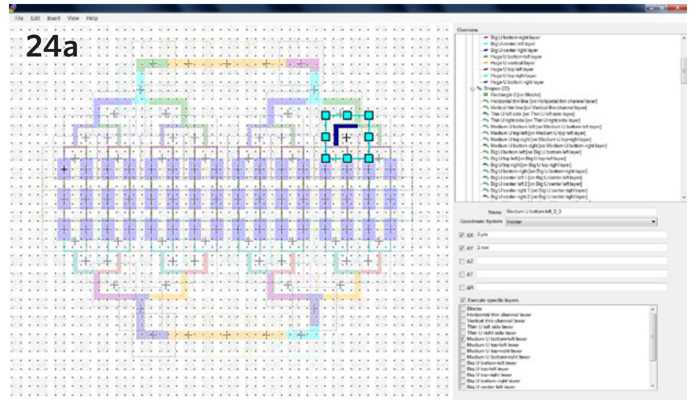


Figure 23. A zone plate diffraction grating written and imaged with electron beam lithography using low vacuum conditions to eliminate any charging artifacts in the patterning or the imaging.

CAD-based prototyping

The 16-bit digital patterning engine built in to every Thermo Scientific DualBeam instrument is extremely powerful, capable of making complex patterns by steering both the ion and electron beam. It can switch between any of the points and dwell at them as necessary. However, to achieve the complex patterns that researchers require, the instrument needs not only the power of the patterning engine but also a way to program the points into it. Doing this per point by trial and error would obviously take far too long. Thermo Fisher has enabled users to employ several different methods of programming the patterning engine. In its most basic form the user can just use the simple patterns built into the DualBeam software to achieve their desired shapes. In its most powerful form the user can individually program each point on the patterning engine. To facilitate this the DualBeam allows structures to be designed in a standardized CAD environment (both offline or directly on the instrument); these can be written immediately afterwards. Very complex 3D patterns incorporating several different milling and deposition runs, and varied operating conditions such as beam current or dwell time, can be created. This means researchers can quickly go from a design on paper to a real device. Since it is such a fast process several trials can be made without incurring any undue cost or spending large amounts of time — this really does bring nanoscale prototyping into the real world!



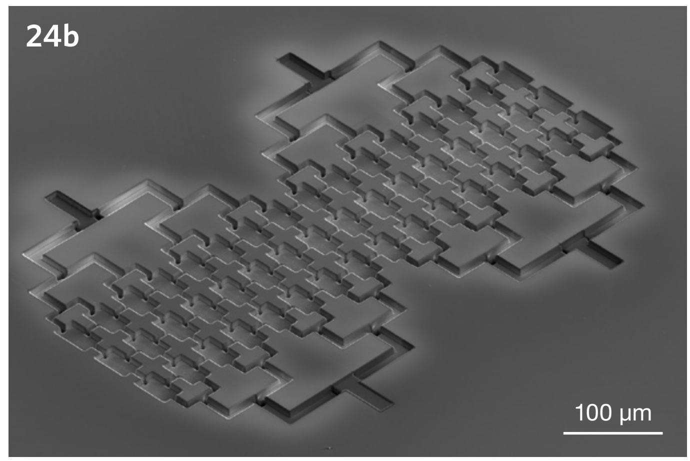


Figure 24. One millimeter long microfluidic device viewed from the NanoBuilder Software interface (a) and at an angle with the SEM, after completion of the automated FIB writing (b). As the FIB cannot write such a large device with high accuracy, NanoBuilder Software subdivides the design into smaller fields and precisely repositions and writes each of them. Here about 100 writing fields were used.

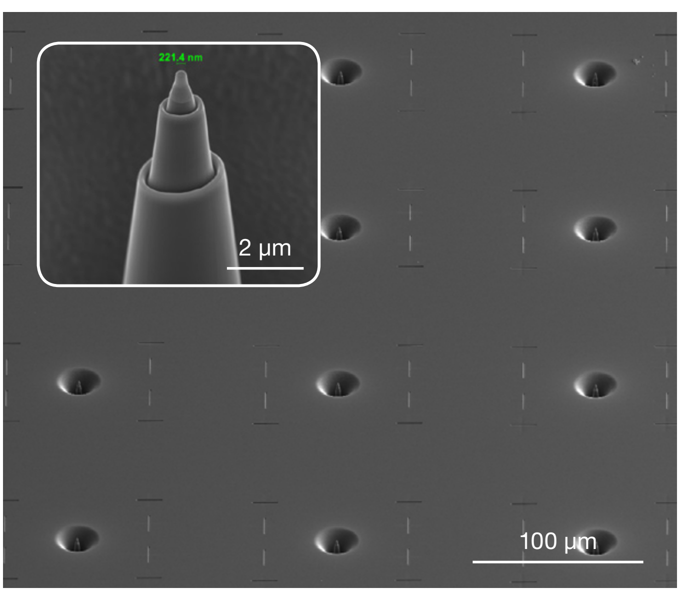


Figure 25. Automated preparation of an array of 10 x 3 AFM tips, with a pitch of 150 µm, using NanoBuilder Software. For each tip, FIB currents from 20 nA down to 10 pA were used, including a beam re-alignment after each current change using the fiducials. The inset shows the detail of one tip.

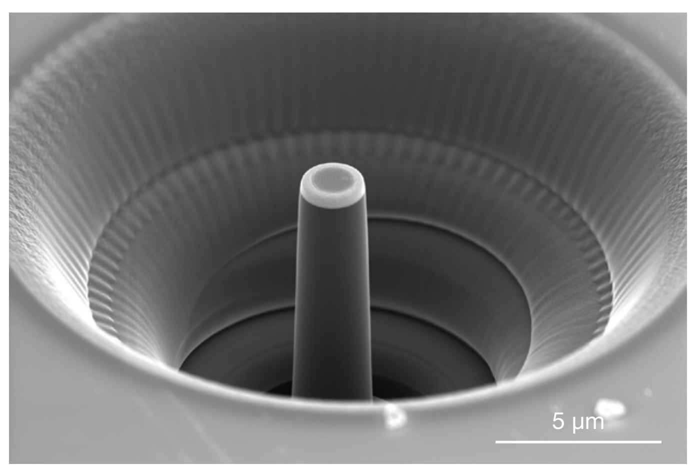


Figure 26. Non-tapered pillar for micro-mechanical testing. An automated multi-step FIB milling process, driven by NanoBuilder Software and consisting of a sequence of decreasing beam currents and alignments, was used for optimum shape control.

To achieve the most detailed and complex structures, Thermo Scientific has designed a software package — NanoBuilder— which utilizes dedicated patterning strategies for FIB milling and beam induced deposition. It combines pattern generation with routines for alignment, mark recognition and write-field calibration that have specifically been developed for DualBeam instruments. Pattern placement means that all individual pattern layers within a single writing field need to be positioned accurately by means of alignment mark recognition. When this alignment is performed with the FIB it causes minimum damage to the alignment marks, such that they can be re-used for several layers. Field-to-field stitching writes alignment marks as part of the pattern in each individual field. It then reads back these marks as the stage moves from field to field. This passes the exact pattern position of the previous field to the actual writing field.

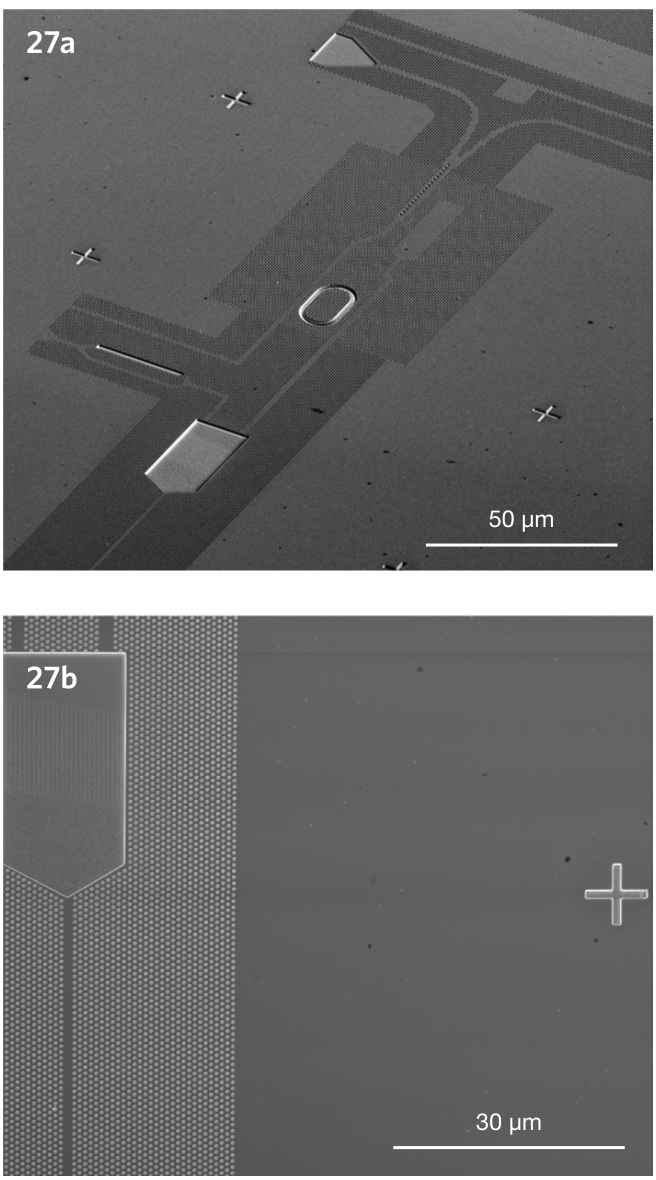


Figure 27. A photonic crystal pattern in InP, covering a total area of 500 × 100 µm^2, was split into 5 writing fields. The alignment crosses were FIB milled as part of the photonic crystal design and serve as a reference for the exact position of each field. The position of the crosses is registered after each stage move to align the pattern in the actual writing field accurately to the previous one.

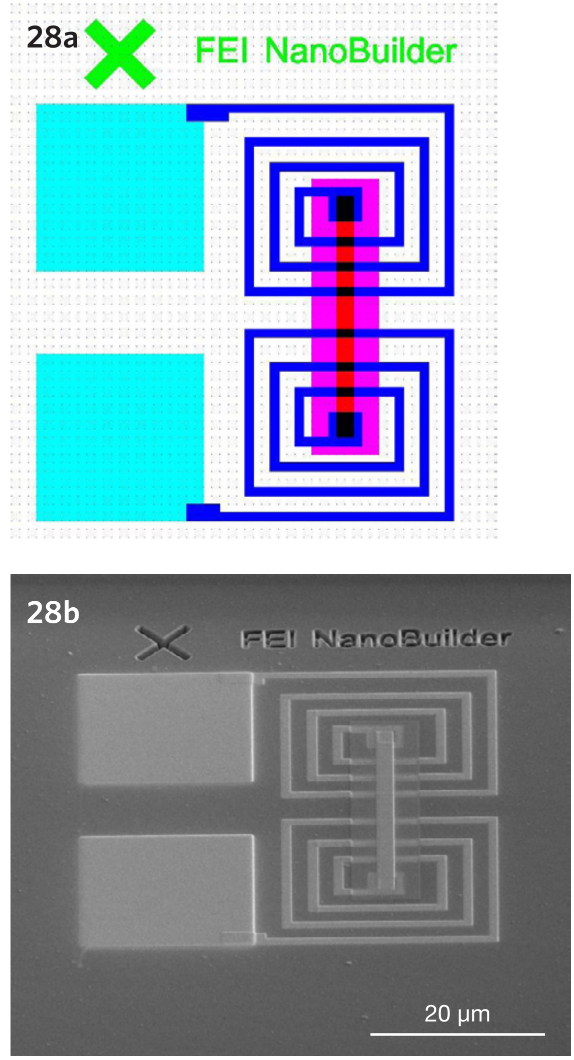


Figure 28. Same dual coil inductor device as the one presented in fig. 15. Its design (28a) consists of different layers, one for each of the building blocks of the device: deposition of a specific material, milling with a specific ion current, etc. Once the design is complete, NanoBuilder Software automatically executes the writing of all layers (28b).

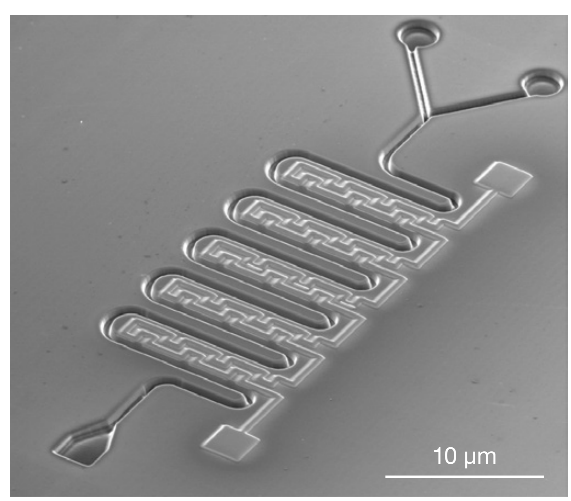


Figure 29. A nano-fluidic system made with a multi-layered pattern. A layer may consist of a deposition sequence, like for the Pt resistive strip of this device, or a milling sequence, as was used to create the fluidic channel. The device is written in one single automated step using NanoBuilder Software .

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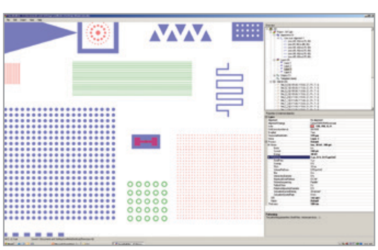
Scanning Electron Microscopes

SEMs are used for inspecting the surface of materials with a magnification range from the micro- to the nano-scale. Thermo Fisher offers a variety of advanced SEMs to meet customer requirements and application needs. A Thermo Scientific SEM can scan the surface of a sample with a finely focused electron beam, producing an image from the beam-specimen interactions. A variety of detectors are available, from secondary electron detectors that provide surface information, to backscattered detectors for compositional information in both high or low vacuum modes.

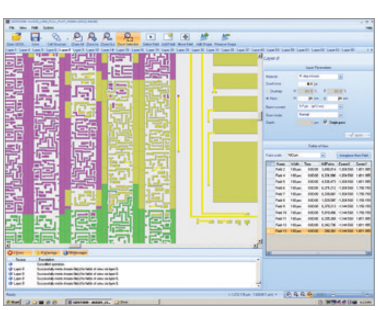
Small DualBeam Systems

A Thermo Scientific DualBeam (FIB/SEM) system is the preferred solution for 3D microscopy and analysis, serving material characterization, industrial failure analysis and process control applications. DualBeam instruments deliver fast, precise and integrated sample preparation, all the way up to thin samples for high-resolution S/TEM. Using FIB nanomachining, they provide access to in-depth information and can automatically serial-section a object to analyze its morphology, chemical or crystallographic composition in 3D.



NanoBuilder Software

With Thermo Scientific NanoBuilder Software, you easily plan construction of multi-layer structures by using its integrated CAD environment to design, parametrize and optimize the writing sequence. NanoBuilder Software allows patterning of large and complex nanostructures accurately on multiple sites, supporting all DualBeam patterning processes: focused ion beam (FIB) milling, gas assisted FIB milling, ion or electron beam induced deposition, and electron beam lithography. Different pattern processes can be assigned to each individual layer in the GDSII layout, and the user can define the sequence in which the NanoBuilder Software executes these. Advanced alignment algorithms ensure that the individual layers are accurately aligned without unwanted exposure of the pattern area to ions or electrons. Finally, the patterning of all layers is executed fully automatically. Site lists can be created to repeat the nanostructure on multiple sites on a substrate.



GDStoDB Software

Thermo Scientific™ GDStoDB Software allows CAD to be used as a starting point for designing complex patterns, which can then be fed directly into the instrument. Unlike other packages, such as external electron beam lithography kits, GDStoDB Software takes direct advantage of the SEM or DualBeam instrument's on-board prototyping capabilities, such as vector scanning and individual pixel dwell time, available on the digital patterning engine. It integrates our extensive knowledge related to FIB and GIS optimized writing strategies, such as material sputter/deposition rates, redeposition control and more.

Find out more at **thermofisher.com/EM-Sales**

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