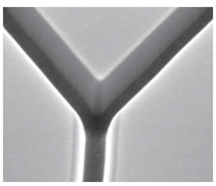
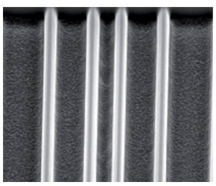
APPLICATION NOTE

Nanofabrication and rapid prototyping with DualBeam instruments

Nanopioneering the next generation


Focused ion beam (FIB) milling of patterns in any kind of material and the precise beam induced deposition of various materials in one single instrument are recognized as novel ways to achieve true rapid prototyping.

The capabilities to observe the patterning process live and to immediately image the resulting structures with high resolution offer unique control over the patterning process and provide an immediate feedback loop for the operator. Successful nanoprototyping requires dedicated strategies for the execution of pattern designs owing to the characteristics of the FIB-substrate interaction. The impact of different patterning strategies is illustrated in this application note and may serve as a guideline for successful nanofabrication.

Introduction

Conventional nanofabrication batch processes require for each pattern layer the combined use of different machines, typically: a spin coater for resist application, a lithography tool, wet chemistry for resist development, a plasma cleaner for descum, deposition or etching equipment for pattern transfer onto or into the substrate, facilities for clean resist removal and along with all of the above—suitable inspection capabilities.

On top of that, nanodevices that are built in several pattern layers have to ensure accurate alignment of all individual layers. As researchers push nanotechnology towards smaller dimensions, new geometries and new materials, the established procedures and recipes might no longer be applicable and the entire nanofabrication process may need refinement in a series of iterations. In practice, this way of achieving advances in nanotechnology takes up a considerable amount of total project times, especially in facilities where the equipment is shared between many projects and machine availabilities need to be taken into account.

The patterning capabilities of Thermo Scientific™DualBeam™ instruments hold the potential to largely cut short development times in Research. Rapid prototyping with the FIB enables tests of prototype functionality before the final layout of a device is established for batch fabrication. Beam-induced deposition of different materials can be combined with FIB milling without the need of several aligned lithography steps; stacks of dissimilar materials can be structured in one single milling process; patterns can be added to existing structures on a substrate or existing patterns can be modified. The patterned substrates are immediately available for further processing or characterization.

Once a prototype has been tested successfully, a batch nano-fabrication process can be qualified by integrating electron beam lithography with the SEM column of the DualBeam instrument as patterning step. The ability to rapid prototype and do resist patterning for nanofabrication in one instrument facilitates the delivery of a proof of concept for a device much faster and allows pointing out a way towards volume manufacturing at the same time [1][2]. All patterning is hereby accompanied by the inspection, analysis and characterisation solutions of DualBeam instruments: ultra-high resolution electron microscopy, FIB imaging, cross-sectional analysis, 3D reconstruction, EDX and EBSD analysis and *in situ* electrical testing.

Examples

A detail view of a Fresnel zone plate is shown in Figure 1. The pattern was milled into a silicon substrate with a 30 kV FIB at 100 pA beam current. The total milling time to a pattern depth of 500 nm was 4:23 minutes. Excellent sidewall quality and good control of the groove profiles were achieved. This pattern could for instance be added to an existing nanoimprint mould for qualification of a nanoimprint process on the same day—without the need to fabricate an entirely new mould. The short process times even allow creating a series of repetitions of the pattern with different pattern depth, for evaluation of the maximum feasible aspect ratio.

DualBeams enable the inspection of the fabricated patterns in great detail by site specific cross sectioning. The cross-sectional image in Figure 2 includes the measurements of one of the zones of the Fresnel zone plate in Figure 1.

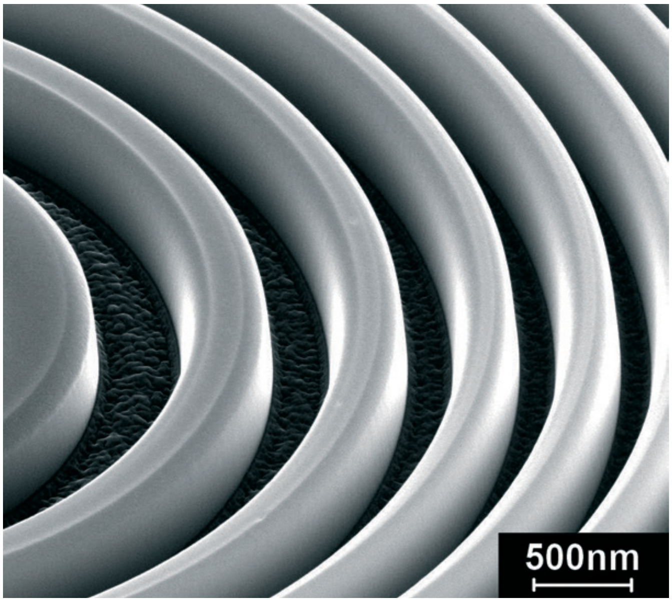


Figure 1. A Fresnel zone plate pattern fabricated by direct FIB milling into Si.

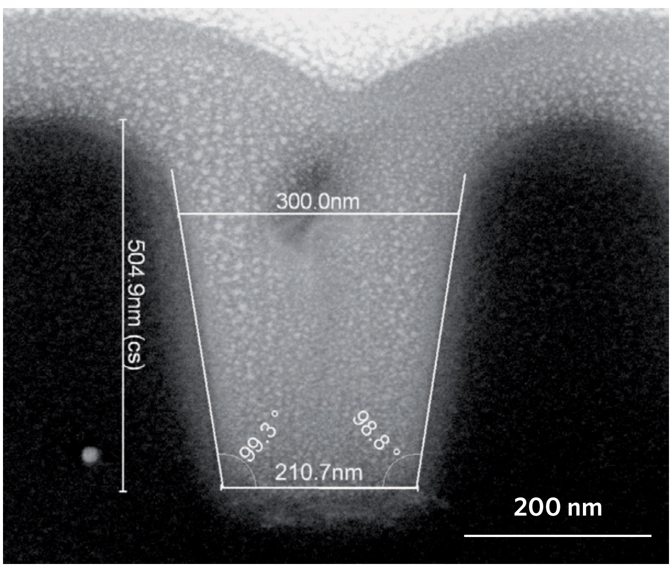


Figure 2. A strap of Pt was deposited with the electron beam across the zone plate structure to protect the Si surface during cross sectioning. The Pt can be recognized inside the groove by its grain texture.

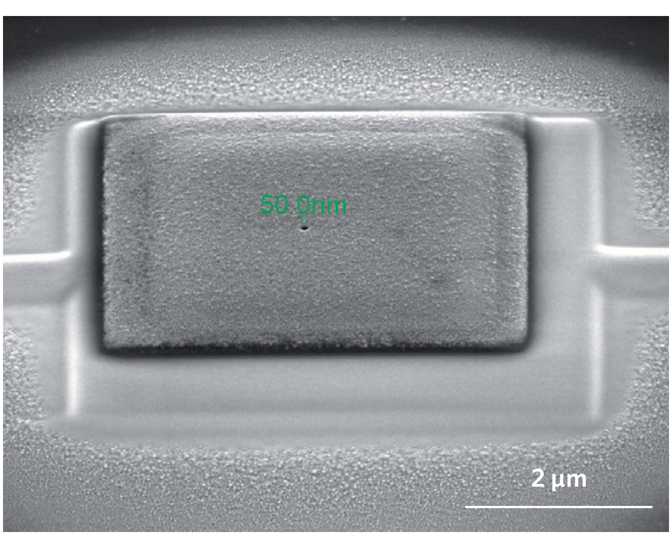


Figure 3. Intermediate step of a crossbar architecture intended for self-assembly of alkanethiol based functional molecules. Bright 5 µm square with leads to either side: W deposition; concealed square: Au deposition; covering layer: insulating SiO_x. 50 nm FIB-milled hole to the Au in the center.

A second example is the combination of deposition steps with different gas chemistries and a final milling step for the fabrication of a crossbar architecture (Figure 3) for the test of the electrochemical properties of alkanethiol based functional molecules, which self-assemble on Au electrodes [3][4].

A bottom electrode pattern was generated by ion beam induced W deposition on a SiO_2 substrate. In a second step, an Au deposition pattern was placed onto the W pads of the bottom electrode layer. An insulating SiO_x layer was deposited across the Au squares and the FIB was used to mill 50 nm pores through the insulating layer with the intention to open a small, defined area for self-assembly of molecules inside the pores on the Au. A 4×4 array of such electrode pads was produced in half a day, leaving a substrate ready for self-assembly experiments.

Figure 3 shows one element of the array pad of the bottom electrode structure. The top electrode can be deposited by e-beam induced deposition in order to cause no FIB damage to the molecules inside the pores.

Figure 4 shows the layer sequence of the crossbar architecture including measurements of the individual layer thicknesses in a FIB cross section (after deposition of the W top-electrode).

The Thermo Scientific pattern engine

The design and execution of patterns on DualBeam instruments makes use of dedicated and fully integrated building blocks: tools for pattern design, automated or manual parameter set-up and patterning hardware that steers the respective beams and controls the gas injector systems.

The choice of the most suitable method to define patterns for DualBeam prototyping depends mostly on the complexity of the structures. The presently available design tools are listed in Table 1. The Fresnel zone plate in Figure 1 for instance was designed using Thermo Scientific scripting. The script is using the text book formula for the radii of zone plates. Input parameters are the wavelength of the light in the intended application, the focal length and the number of zones. This way the design is fully parameterized and the geometry can be altered by simply keying in new input parameters rather than getting back to the drawing board. Once the geometries are defined, the strategy for pattern execution is set by selecting the matching application file in the DualBeam user interface. The instrument comes with a library of application files for a number of common materials.

This library can be expanded with application files for new materials as they become available or users can easily add and modify application files themselves. The application files use the volume sputter rate of the respective material and an empirically optimized beam dwell time together with the actual beam parameters to calculate the best pitch and number of passes. All Thermo Scientific DualBeam instruments are equipped with digital patterning boards to steer the beams according to a given design and application file. The key specifications of the latest generation that are relevant to patterning applications are:

- 16-bit DAC resolution
- 25 ns minimum pixel dwell time

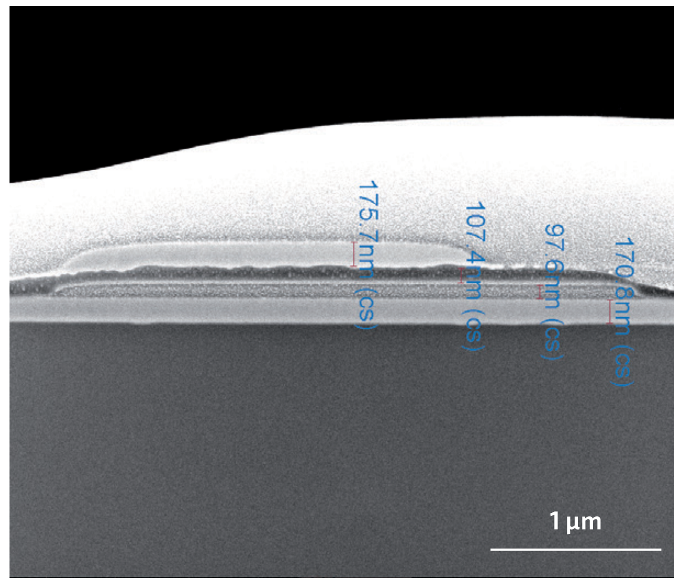


Figure 4. The FIB cross section of one electrode pad including the deposition of a W top electrode shows the thickness of all individual layers. Layer order from bottom to top: substrate, W bottom electrode, active Au layer for the SAM (self-assembled mono-layer), insulating SiO_x layer, W top electrode, protective Pt for cross sectioning.

	Advantages	Practical limitations
User interface	Direct drawing on the UI, quick & easy, intuitive to first timers, overlay on image guarantees alignment with existing structures.	Number of pattern elements.
Scripting	Complex pattern can be fully parameterized. Patterning jobs can be automated. Off-site design does not take up machine time.	Limited to combinations of the basic shapes available in the user interface design.
Bitmap	Grey-level exposure dose for 3D structures. Accepts any graphical template.	Raster scan only. Number of pixels defined in bitmap, no variation of pitch.
Stream file	Direct control of the DAC output. Greatest flexibility. Accepts xy data from any source. Dwell time control for each individual pixel.	Instrument settings such as beam current, magnification are incorporated at design stage.

Table 1. Thermo Scientific design tools for pattern definition.

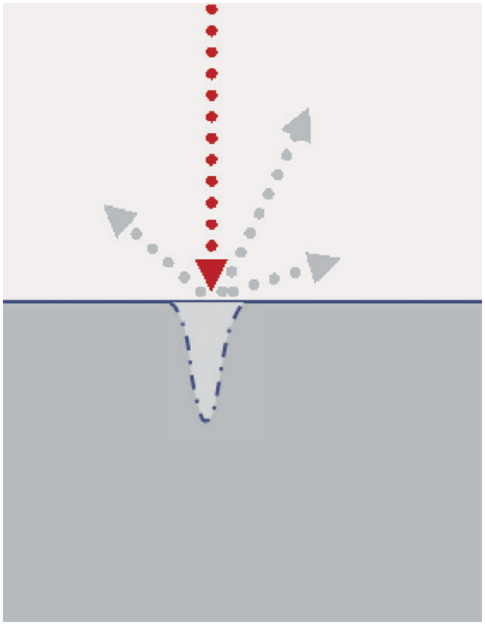


Figure 5. Illustration of FIB milling on one position for a prolonged dwell time.

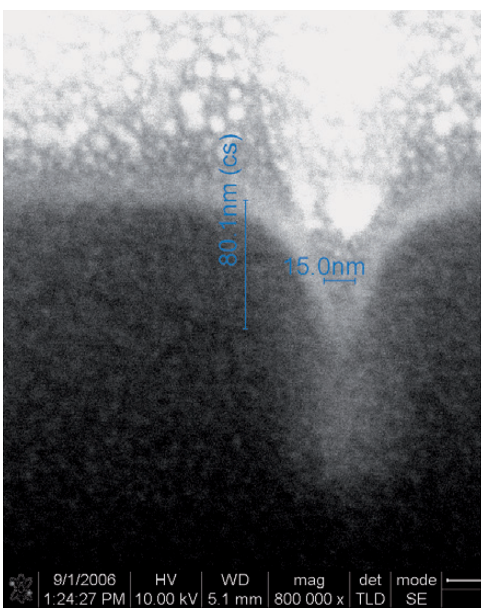


Figure 6. Cross section of a single pixel line milled into Si with a 30 kV, 10 pA FIB in a single pass at a 1 ms pixel dwell time.

Dedicated FIB patterning strategies

The patterning functions for the FIB have at first glance apparent similarities to e-beam lithography, a technique a lot of users are more familiar with. This has often lead researchers to carry forward electron beam lithography exposure strategies. This however is neglecting the fundamental differences of accumulating a certain exposure dose before resist development in electron beam lithography, in contrast to the instantaneous removal or deposition of material with a FIB [5]. The following examples illustrate the significance of adequate pattern execution for successful FIB patterning by practical examples.

1. Parallel multi-pass vs single pass milling

The profile of a single point milled into any substrate will partly be determined by the profile of the FIB itself. On the other hand, since the FIB immediately sputters away substrate material, a hole forms while the FIB continues milling at the same position. Consequently, ions hit the sloped sidewalls of the forming hole, and as sputter yields depend on the angle of the incident ions, milling rates depend on pixel dwell time. Another aspect that needs to be taken into consideration is the redeposition of sputtered material that will occur inside the milled structures.

Single pass milling (lithography approach)

Figure 5 illustrates the FIB milling of a single pixel with the FIB being represented by the red arrow and the sputtered material by the grey arrows. The profile in the substrate material is a first order approximation, basically representing the FIB profile. Figure 6 shows the resulting trench when milling a single pixel line in a single pass at long dwell time. This of course does include the effects of the interaction of beam and substrate. The intrinsic V-profile of a single pass trench is limiting the use of patterns in nanotechnology applications.

When the line is designed with a target width of 100 nm, the pattern is represented by a 100 nm wide rectangle. This rectangle will be patterned with the beam sweeping across its area in a serpentine fashion with the direction defined in the design. In order to fill the pattern continuously the Thermo Scientific pattern engine calculates a pitch in between pixels that ensures a 50% overlap of adjacent milling dots. With a single pass strategy, the second line of the serpentine sweep would be hitting the sloped sidewall of the first line as indicated in Figure 7. The higher milling rate of a FIB hitting a surface under an angle makes the second line mill deeper than the first one. In addition, substrate material that is removed when milling the second line will partly redeposit into the first line.

Figure 8 shows how both effects will add up at continued milling, clearly not delivering a desired profile and not allowing appropriate control of the pattern depth. This is well visible in the actual milled 100 nm line in Figure 9, showing the redeposited material as a bright zone at the left hand side of the trench and too deep milling on the right hand side.

The above sketches and micrographs explain why patterning strategies adopted from electron beam lithography are not suitable for prototyping or nanofabrication with a FIB. For illustration purposes a nanofluidic structure was first milled in a single pass into silicon with a 1 ms dwell time. The Y-junction of the three trenches in Figure 10 shows how material is redeposited into the previously milled trenches when milling them one after the other. Redeposition of milled materials on the sidewalls is obvious from the difference in texture of the sidewall as compared to the silicon surface.

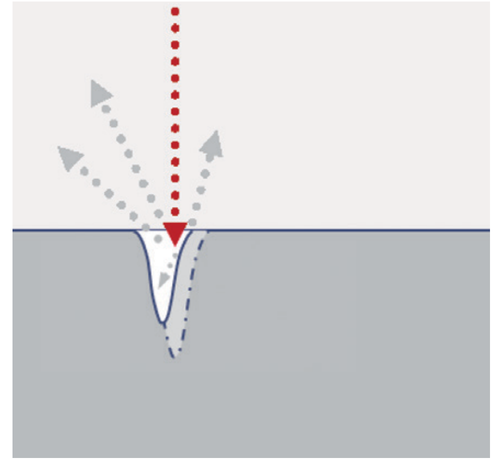


Figure 7. When milling a pattern in a single pass strategy, the FIB will hit the sidewall of the forming pattern. This results in an uneven pattern depth and difficult control of the pattern depth.

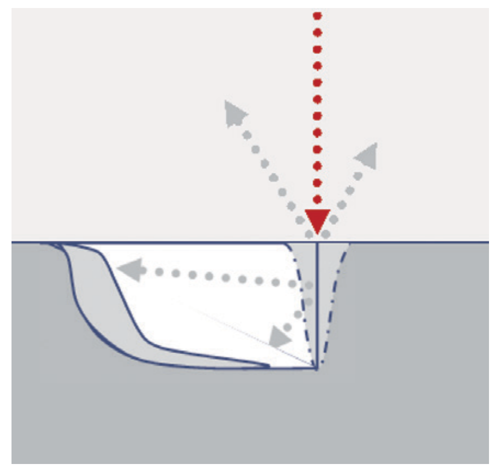


Figure 8. Accumulated effect of redeposition and a progressing milling rate in single pass strategy.

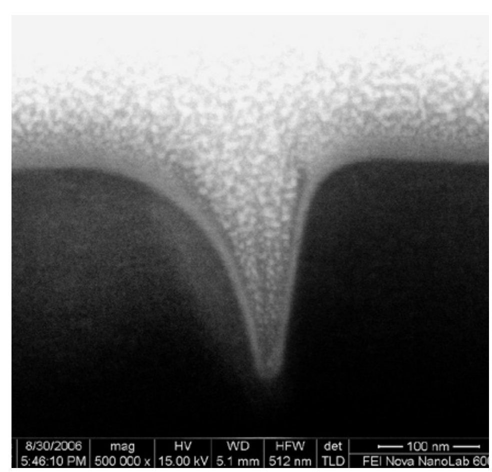


Figure 9. Cross section of a trench designed 100 nm wide and 200 nm deep after single pass milling from left to right. The pattern is getting deeper to the right; the brighter zone underneath the left flank is the redeposited material.

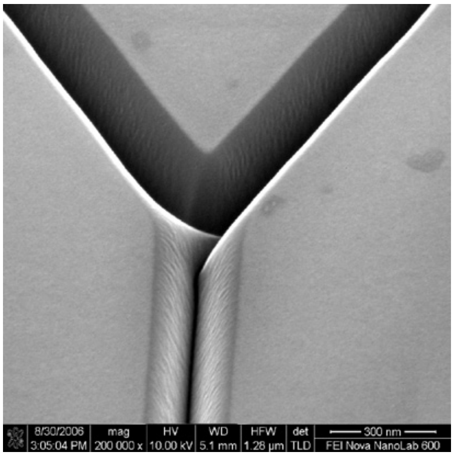


Figure 10. Junction of three 100 nm trenches in a nanofluidic structure done by single pass milling. The trench at the bottom was milled first, followed by top left, then top right.

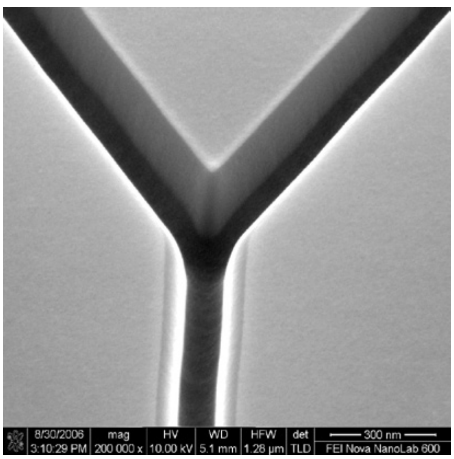


Figure 11. Junction of three 100 nm trenches when a dedicated FIB patterning strategy is applied. The junction comes out as designed and the individual trenches show a well defined profile without redeposition artifacts.

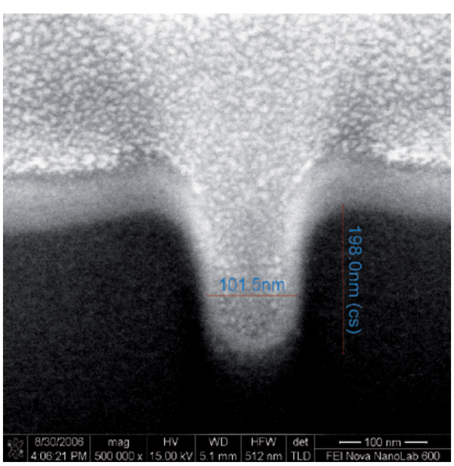


Figure 12. Cross section of a trench designed 100 nm wide and 200 nm deep when a FIB milling strategy is applied.

Parallel multi-pass milling (Thermo Scientific approach)
DualBeam instruments are fitted with application files that provide the user with the best known strategies for FIB milling, FIB deposition and electron beam deposition for a certain material. The specifics of the interaction of the FIB and the substrate material are implemented.

Figure 11 shows the same pattern as Figure 10, just that this time the Thermo Scientific application file for silicon and parallel milling was applied. The pixel dwell time is kept at 1 µs and the target depth of 200 nm was calculated to require 884 passes at a FIB current of 93 pA at 30 kV. The difference of the outcome is striking: using a dedicated FIB patterning strategy, the junction is now matching the design and the nanofluidic channels show a well defined profile. The target width and depth are met as shown in the cross-sectional image in Figure 12.

The difference to the structure in Figure 10 can intuitively be understood when visualizing the parallel multi-pass milling. The short pixel dwell times in multi-pass milling avoid the formation of strong topography in one pass, hence yielding homogeneous pattern depth (keeping the dwell time short and merging all patterns into one, minimizes the depth of the profile in Figures 5, 7 & 8). Passes are repeated until the target depth is reached. The milling of the entire pattern until completion prevents the build-up of redeposition. Any redeposition that does occur during the milling will be removed in the subsequent pass.

A structure as in Figure 11 could directly be used as a nanofluidic prototype. While it remains of course to the judgment of the scientist whether a FIB milled pattern is suitable for the final application, the FIB prototype is well suited to tackle some of the initial project challenges.

A technique for bonding a tight top seal to the fluidic channels can be developed while the development of a batch nanofabrication process is still ongoing. The connection with external pumps, reservoirs or filters can be tested, experimental flow rates can be determined and compared with design specifications, particle filtration can be worked out. Rapid prototyping with a DualBeam allows generating experimental data for quicker design optimization and the development of a batch nanofabrication process in parallel on one instrument, which is possibly decisive for fulfilling deliverables in a research project.

Figure 13 shows a crossing of two 40 nm trenches as an example of how nanoengineering with good control of dimensions and proper structure profiles can be applied to smallest dimensions. The micrograph is a detailed view of a pattern that is covering a total area of 50×50 μm^2. The 16-bit patterning board provides sufficient resolution to position the beam with a pitch small enough to put several pixels into a 40 nm width even at larger pattern areas. This is the key to a reasonable process latitude with lateral dimensions less dependent on focus and shot noise and a control of the pattern profile less dependent on the profile of the FIB itself.

The relevance of having the right milling strategy does not only apply to fine features. It becomes equally obvious at larger structures. As an example, two rectangular boxes of 2×10 μm^2 were milled into Silicon (Figure 14). The left hand side box was milled with a serpentine sweep parallel to the longer 10 μm side, the right hand side box with a serpentine sweep along the shorter 2 μm side. The Thermo Scientific application file was used for the boxes in Figure 14 showing no difference for the two different milling directions, yielding in both cases a usable pattern. Enforcing a single pass strategy by fixing the number of passes to 1 and increasing the dwell time resulted in the pattern shown in Figure 15; neither sweep direction gave the desired pattern.

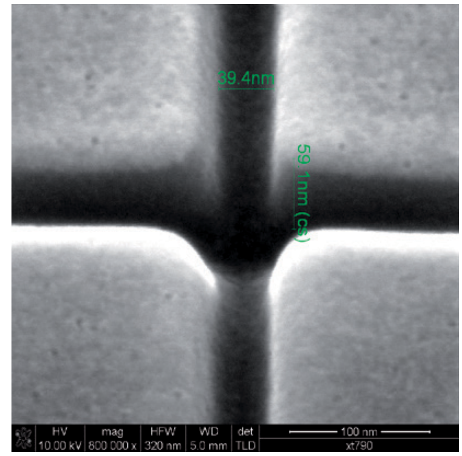


Figure 13. Line crossing of two 40 nm trenches. The application files permit accurate patterning down to the lower nanometer range.

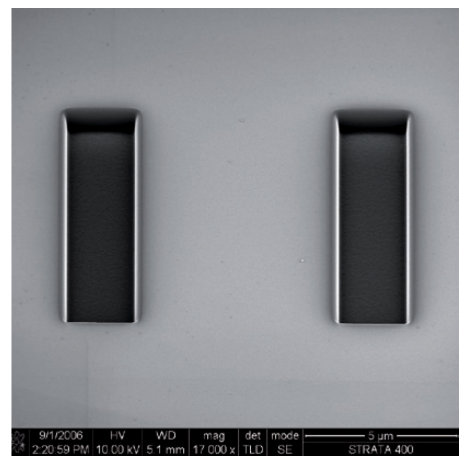


Figure 14. Two rectangular boxes milled with the Thermo Scientific application file for Si. The left box is milled with a serpentine sweep along the long, vertical side, the right box with a serpentine sweep along the short, horizontal side.

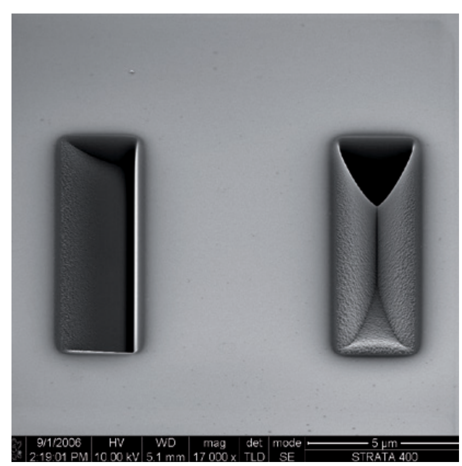


Figure 15. When the patterning is done in a single pass (e-beam lithography style) pronounced artifacts are present for both sweep directions.

Figure 16. The Thermo Scientific parallel multi-pass milling strategy delivers a smooth bottom and clean sidewalls for all pattern elements.

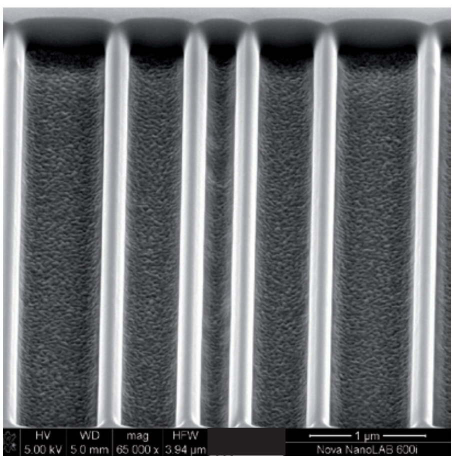


Figure 17. Milling the center part first results in strong redeposition into the central features and an overall unsatisfactory pattern quality.

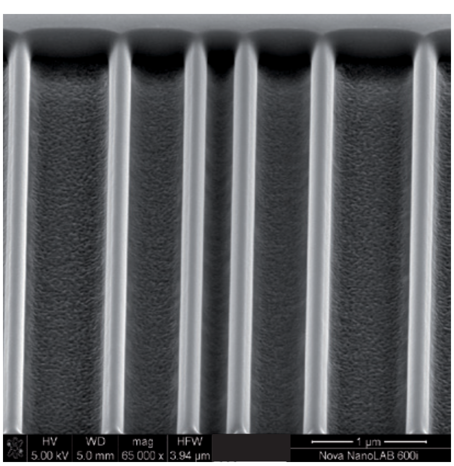


Figure 18. Milling the center part last results in less significant redeposition. However, the overall pattern quality is still compromised by redeposition.

2. Milling order and redeposition

An interesting observation in Figure 15 is that the redeposition of sputtered material is generating strong artifacts only within the pattern elements. The substrate surface to the contrary is not showing any redeposition. This is due to the fact that inside a milled pattern exist straight lines from the origin of the sputtered material (the actual dwell point of the FIB) to the pattern sidewalls, which represent a direct path for redeposition. As there is no straight line onto the substrate surface or into adjacent pattern elements, redeposition could occur only in the case that charging effects are present.

FIB milling will charge a pattern area electrically as a combined effect of the charge being introduced by the FIB itself and charge being carried away in the form of ionized sputter fragments and secondary electrons. Until all charge is dissipated a pattern area will attract the redeposition of ionized sputter fragments and thus confine the redeposition. The Thermo Scientific parallel multi-pass milling strategy prevents redeposition as any redeposited material in a pattern will be removed again in the consecutive pass. This is demonstrated in Figure 16 by the example of a series of boxes that were milled 400 nm deep into Si. The entire pattern shows a smooth bottom and clean and vertical sidewalls.

When the use of single pass milling can not be avoided, the choice of an appropriate milling order can still minimize redeposition artifacts. The impact of the milling order is illustrated in Figures 17 & 18. In Figure 17 the milling started with the central 200 nm box then continuing to the outside. The larger volume of sputtered material from the outer boxes redeposits into the small area of the inner boxes, covering the bottom of the pattern and being visible along the sidewalls.

For Figure 18 the milling order was reversed, milling the central 200 nm box last, and thus not exposing it to redeposition. In this order, the milling is removing a smaller volume of material towards the end, which partly redeposits across the relatively larger area of the outside boxes. As a result, redeposition is still visible at the bottom of the patterns but clearly less significant when compared to Figure 17. In general, when a small pattern is placed close to a large pattern and serial milling can not be avoided, the large pattern should be milled first and the fine pattern last.

In practice, patterns that comprise features across a larger range of critical dimensions are most efficiently milled with different ion beam currents for optimization of the overall milling time. The necessary changes of the FIB current make serial milling inevitable. Changing the FIB current involves a short interruption of the milling process for the change of an aperture in the FIB column. This interruption allows for charge dissipation, which reduces the effect of redeposition caused by charging effects. The pattern in Figure 19 shows the impact of redeposition in a more application related example. The two large patterns were milled with a FIB current of 6.5 nA. The 250 nm trench passing through the gap in between the large patterns was milled with a FIB current of 93 pA.

For the purpose of comparing the redeposition the pattern was milled twice: first milling the large patterns followed by milling the trench (Figure 20), then milling the trench first followed by the two large patterns (Figure 21). Although the definition of the trench is slightly deteriorated by redeposition in the latter case (Figure 21), the charge dissipation during changing the FIB current resulted in usable structures in both cases. The images also show nicely that accurate placement of patterns relative to each other is achievable irrespective of the milling order.

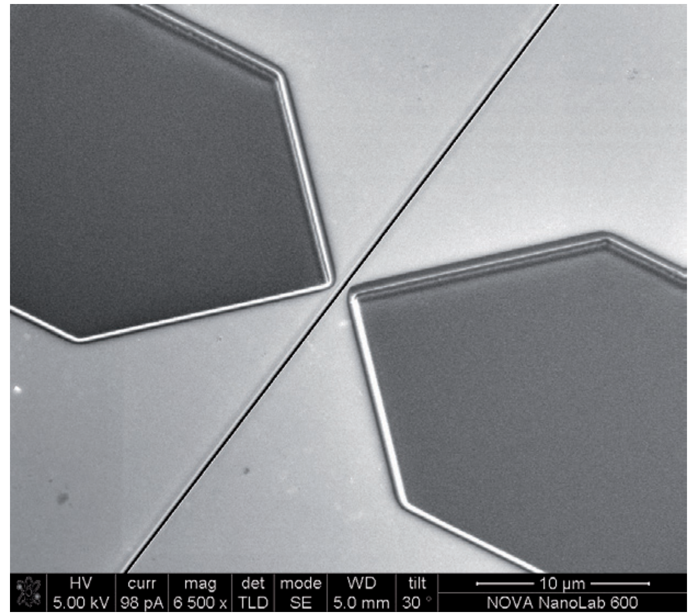


Figure 19. The large patterns were milled with 6.5 nA, the trench through the center with 93 pA.

Practical considerations

The prototyping of individual devices with the FIB often takes only a few minutes per layer. Larger arrays of devices however will need considerable patterning times, up to several hours. In order to avoid possible drifts, which could degrade the definition of individual pattern elements when milling multi-pass for prolonged times, dividing the pattern elements into “parallel mill” groups is considered the best solution. Doing so, typical drifts will lead to only negligible placement errors of the groups relative to each other.

When mix and match strategies require overlaying a FIB pattern with an existing pattern, the FIB pattern can very practically be placed based on quick image recognition. If the substrate doesn’t allow any exposure to the FIB in areas that are not part of the pattern itself, a reduced area can be defined for overlay positioning. When using Thermo Scientific scripting or in cases where an e-beam lithography pattern generator is used for FIB patterning, the overlay can be achieved by registration of alignment marks. For registration of alignment marks however it has to be taken into account that the FIB is milling the alignment marks during registration and appropriate methods for mark recognition need to be implemented.

The properties of materials deposited by beam induced deposition can be substantially different to materials deposited by conventional nanofabrication techniques. The inclusion of fragments of the gaseous precursor molecules will generate high contents of carbon in the deposits (ion beam induced deposits will also include gallium). Although material properties will be different from the final device made by batch fabrication, FIB prototypes are well suited for electrical testing or catalytic functionality for instance. For FIB milled patterns, gallium implantation and amorphisation of a surface layer on sidewalls and structure bottom need to be considered. Advanced methods from DualBeam TEM sample preparation techniques can—within limitations—be applied for reducing the Ga implantation and amorphisation to a minimum layer thickness [6].

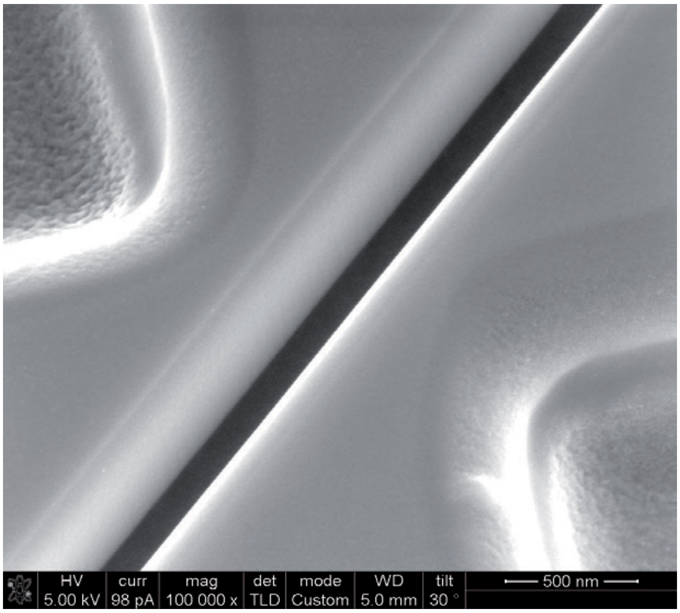


Figure 20. The trench was milled last. The sidewalls and bottom are clean and well defined.

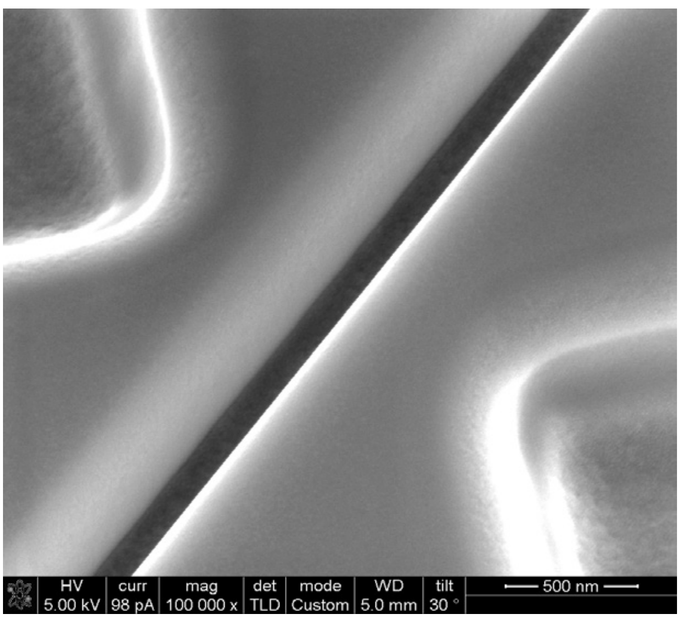


Figure 21. The trench was milled first. Redeposition is covering sidewall and trench bottom.

Conclusions

The intention of the material presented in this application note is to bring out the capabilities and benefits of DualBeam instruments in top down nanotechnology processes. The emphasis is put on robust patterning functions that deliver reproducible results according to engineering criteria, which are mainly seen as controllable lateral dimensions, pattern depth, sidewall roughness and pattern profiles. Although the material characteristics might impose limitations on the use of the prototype in the eventual application, FIB prototypes need to be seen as means to shorten development times in the initial and intermediate phase of a research project.

The patterning concepts introduced here generate patterns that are transferable to batch nanofabrication processes, providing a viable route for nanofabrication in a pilot series. Batch nanofabrication will depend on e-beam lithography as a patterning step due to the small dimensions of the patterns. The e-beam columns of DualBeam instruments have a successful track record in many e-beam lithography applications. Details about this aspect of prototyping and nanofabrication however are beyond the scope of this application note.

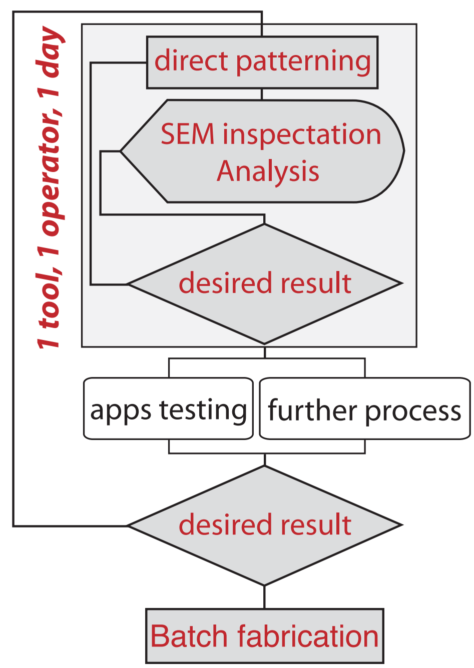


Figure 22. Flow chart of a typical rapid prototyping process. The transfer to batch fabrication as the outcome can refer back to the same instrument, using the e-beam column for lithography.

All micrographs in this application note are evidence of the excellent imaging capabilities for SEM tasks as well as for site selective FIB cross sections. How the inspection and imaging capabilities in combination with the patterning functions add to the total solution that a DualBeam instrument represents for rapid prototyping is visualized in the flow chart in Figure 22.

Key to successful patterning in any kind of nanotechnology application are the tools in the hands of the researcher. These are, apart from high quality FIB and e-beam columns: an integrated digital patterning board, the provision of application files with the implemented best known patterning methods and the expertise of dedicated patterning strategies.

The enormous versatility of DualBeam instruments as patterning and inspection tools represents a high value to any nanotechnology lab. They contribute to a streamlined development process, cutting short development cycle times and reaching deliverables faster, they free up machine time on other equipment and will captivate researchers. Seeing nanotechnology unfolding live in front of your eyes will surely conquer the heart of anyone working in this fascinating discipline.

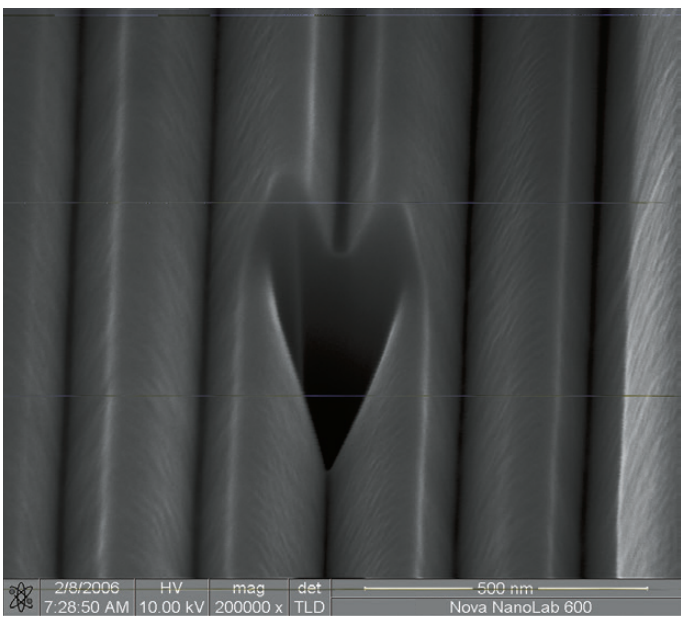


Figure 23. Capturing the heart of nanofabrication. This nanoheart was formed by cross sectioning very narrow FIB-milled lines in a silicon substrate. The horizontal field of view is just 1.2 µm.

References

1. *P.M. Nellen et al, MNE'06 Micro- and Nano-Engineering, 17-20 September 2006, Barcelona*
2. *Y.Q. Fu, N. Kok, A. Bryan, Microelectron. Eng. 54 (2000) 211*
3. *M.A. Reed and J.M. Tours, Computing with Molecules, Scientific American, June 2000, p. 86*
4. *Deng J et al, Thin Films & Nanotech 2004, 13-17 July 2004, Singapore*
5. *L.A. Giannuzzi, B.I. Prenitzer, B.W. Kempshall, in: L.A. Giannuzzi, F.A. Stevie, eds., Introduction to Focused Ion Beams, Springer, New York, 2005, 13*
6. *B.I. Prenitzer et al., Microsc. Microanal. 9 (2003) 216*
7. *L.A. Giannuzzi, R. Geurts, J. Ringnalda. Microsc. Microanal. 11 (Suppl 2) (2005) 828-829*

This application note does not cover any Thermo Fisher entries into the leaderboard of the FIBlympics in most narrow line, tiniest hole or highest aspect ratio. It also does not go into the discipline of the creation of elaborate 3D nanoconstructions. Also for these applications DualBeam instruments have proven the high degree of sophistication of their patterning functions. Though, as those types of structures are not transferable to batch processing, they are considered a different category of applications than nanofabrication. Nevertheless the electrical contacting of individual nanowires or the study of material and device properties in tailor-made single nanostructures have provided great scientific insight and paved the way for nanodevices that eventually could go into batch fabrication.

thermo scientific

Notes

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

Thermo Fisher SCIENTIFIC