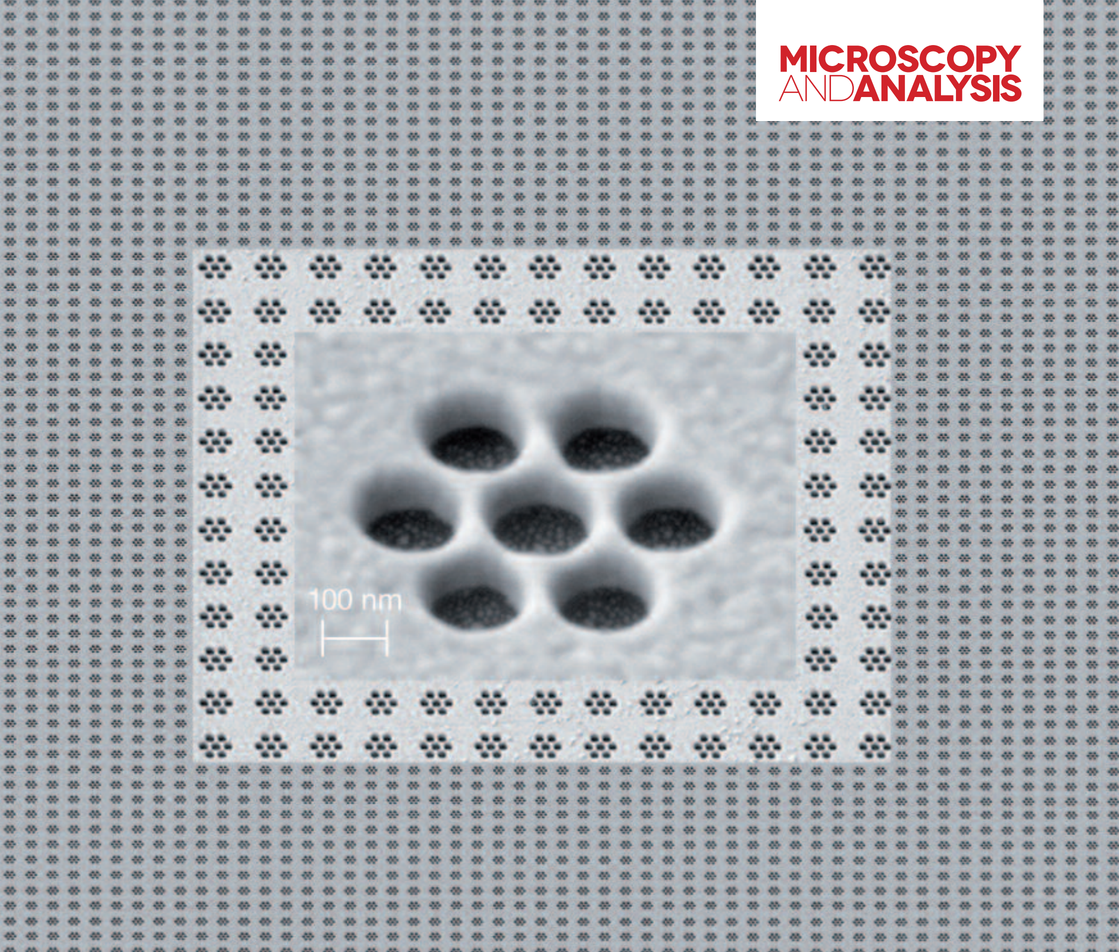
MICROSCOPY AND ANALYSIS



Focused ion beam nanofabrication

Essential Knowledge Briefings

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RAITH NANOFABRICATION

Front cover image: plasmonic device of oligomer array directly fabricated into Au film by focused ion beam with excellent homogeneity and placement accuracy over a large area. The remaining ribbons between adjacent circles scale down to 30nm in the 80nm-thick Au film. Sample courtesy of University of Stuttgart, 4th Physics Institute, and patterning done by Raith GmbH.

Microscopy EKB Series Editor: Dr Julian Heath
Spectroscopy and Separations EKB Series Editor: Nick Taylor

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INTRODUCTION

From the industrial processing of semiconductors to fabricating the latest microfluidic devices in academic laboratories, the focused ion beam (FIB) has proven itself to be an invaluable tool for manipulating and fabricating at the nanoscale. It can work on almost any material without needing a mask, directly fabricating 2D and 3D features ranging from a few nanometers to hundreds of micrometers in size.

FIB systems are similar to a scanning electron microscope (SEM), except they use a beam of ions, usually gallium ions, rather than electrons. Scanning this ion beam over a substrate can modify its surface in several different ways, allowing material to be added, removed or altered, depending on the properties of the beam and the substrate. This makes FIB a highly flexible and versatile fabrication technique, able to be used for nanoscale machining, device processing and sample preparation.

The initial development of FIB instruments was driven by their unique capabilities for computer chip repair and circuit modification in the semiconductor industry. But their ability to fabricate complex nanoscale patterns and structures has seen FIB adopted by a much broader range of scientific and technological disciplines. They are now being used to advance the latest microelectromechanical systems (MEMS), microfluidic chips, photonic devices and nanopore membranes for DNA sequencing.

This Essential Knowledge Briefing provides an introduction to the nanofabrication applications of FIB. It explains how the technique works, outlines the kind of structures it can produce and details how patterning can be controlled. The briefing goes on to outline various practical issues related to the technique, describes potential problems that may arise and how to solve them, and provides examples of how FIB is being used by scientists in their research. Finally, it reveals how the technique is poised to develop and advance in terms of both technology and applications over the next few years.

HISTORY AND BACKGROUND

FIB's popularity as a fabrication technique is down to its flexibility and versatility. This, in turn, stems from the fact that scanning a focused beam of ions across a substrate can modify its surface in several different ways, all of which can be useful for nanofabrication.

The most obvious way in which the ion beam can modify the surface is by removing and dislodging atoms. This allows FIB to etch patterns with a precision that extends from hundreds of micrometers down to a few nanometers without the need for a mask. In addition, the beam can also be used to deposit material onto the surface and even to implant ions into the surface, causing changes to the substrate's chemical composition and physical structure.

This versatility and flexibility has led FIB to find applications beyond fabrication, although they will not be discussed in detail in this Essential Knowledge Briefing. Reflecting its similarity to SEM, FIB can produce high-resolution images of a surface by collecting the secondary electrons generated when the ion beam hits the substrate. FIB is also commonly used for site-specific preparation of samples, including nearly any sample in biological and material sciences, for transmission electron microscope (TEM) investigation, by removing unwanted material and revealing interesting features.

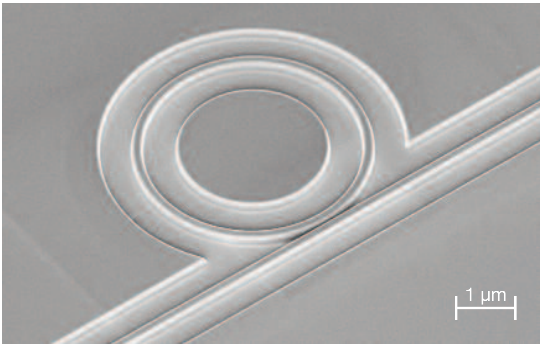
The beginnings of FIB can be traced back to 1975, when VE Krohn and G Roy Ringo at the Argonne National Laboratory in Illinois, USA, first produced an ion beam with high brightness (intensity) using liquid gallium as the ion source. Following on from this, Robert L Seliger and his team at the Hughes Research

Laboratories in Malibu, USA, used gallium as the ion source in the first scanning ion microscope successfully employed for direct patterning. They produced a beam with a resolution of 100nm and used it to mill 100nm-wide lines in a 40nm-thick gold film deposited over a silicon substrate.

By the mid-1980s, advances in ion beam optics had allowed researchers to improve the resolution to 10nm, leading to FIB's adoption by the semiconductor industry for machining devices, and analyzing and repairing circuits. For example, the ion beam can be used to cut electrical connections on a circuit or deposit conductive material in order to rewire it. More recently, FIB has been adopted by academic researchers to fabricate a wide variety of microscale and nanoscale structures, including microtools, nanorotors, nanopores and superconducting thin film devices.

The unique capabilities of FIB derive from the complex phenomena that occur as the ions, accelerated at high energy, hit the surface of a solid sample. One important physical effect is sputtering, whereby the ions dislodge substrate atoms from the surface. By scanning the beam over the substrate, the beam can remove material and thus etch 2D and 3D shapes very precisely (also called milling).

Not all the surface atoms will be dislodged when a sample is bombarded by a high-energy ion beam; some atoms will remain where they are on the surface but be kicked into an excited state. This effect gives mainly emission of secondary electrons that can be exploited to deposit materials onto a substrate using gas-assisted deposition, which involves exposing the sample to molecules in the gas phase while hitting the surface with the ion



Photonic device (wavelength and coupler) in a silicon substrate created by direct milling with a focused gallium ion beam. Image produced by Raith for Professor Peng, Peking University, China

beam. These materials can include metals such as platinum and tungsten, as well as insulator materials such as silicon dioxide.

First, a fine nozzle is used to spray a 'precursor' gas onto the surface: to deposit tungsten (W), for example, the precursor gas would be tungsten hexacarbonyl (W(CO)6). The gas adsorbs onto the substrate, where it can react with the ion beam, secondary electrons and excited surface atoms to produce various products. If volatile, these products will simply break free of the substrate and escape, but some of the products may chemically etch the surface of the substrate or build up on the surface as thin films.

These thin films can form part of the desired structure or they can be used as a 'sacrificial layer' to protect the underlying substrate from the destructive sputtering of the beam. The smallest features that can be deposited are usually in the order of 100nm (lateral dimension), although in special cases this can be reduced to 20nm, with a minimal thickness of around 10nm.

The FIB can also be used to modify a material's surface by implanting ions. When high-energy ions slam into the sample, as well as sputtering atoms from the surface, some of them will penetrate into the top few nanometers, becoming stuck. Implantation can be used to selectively dope a substrate with specific ions, such as doping compound semiconductor devices with gallium ions.

The dislodging of surface atoms by the ion beam, together with implantation, can also change a material's physical structure, making it more amorphous. In many cases, this is just unwanted damage and needs to be prevented, but it can also have useful applications. One of these is producing a mask with a defined shape. Because this mask has a more amorphous structure than the other areas of the substrate, it can be selectively removed with chemical etching. These techniques using well controlled low-dose ion beam damage include direct modification of the sample properties or directing a subsequent processing step, *eg* changing the etch selectivity, offering yet another fabrication option.

In today's FIB systems, the ion beam tends to be produced by liquid metal ion sources (LMIS), as they produce bright and highly focused beams (when connected to the appropriate optics). There are a number of different types of LMIS, but the most widely used is based on gallium.

Gallium has several advantages over other LMIS metals, including a low melting temperature (30°C) and low vapor pressure. The low melting temperature makes the source easy to design and operate, and the low vapor pressure ensures that evaporation is negligible.

Magnetic domains defined by specific low-dose ion beam damaging (intermixing of multi-layers) without creating any surface topography. Reproduced with permission from Gierak J, Mailly D, Hawkes P, *et al.* Exploration of the ultimate patterning potential achievable with high resolution focused ion beams. *Applied Physics A* 2005;80:187–94 © Springer

The ion beam is produced by a process similar to electrospray, in which the liquid gallium sprays from the tip of an electrically charged tungsten needle, inducing ionization. The resultant ions are accelerated to an energy of 5–50keV and focused onto the sample by electrostatic lenses. LMIS produces ion beams with high current density. A modern FIB can deliver tens of nanoamperes of current to a sample, or can image the sample with a spot size of just a few nanometers.

Although gallium is the most commonly used ion source for FIB, other options are available, which can be preferable for certain applications. These include metal alloys produced from combinations of the metals commonly used for LMIS, such as gold with silicon or germanium. Ion beams produced using metal alloys have reasonable stability and lifetime, but really benefit from the fact that they allow processing with distinct ions.

Another option is gas field ion sources (GFIS). These produce ion beams from hydrogen and noble elements such as helium in the gas phase by condensing the gases onto the electrically charged tungsten needle for field ionization. GFIS can produce narrower beams than are possible with LMIS, enhancing pattern resolution, but at the expense of limited stability and a relatively short lifetime.

There are also various other ways to convert the sources into ions, besides electrospraying, including electron bombardment and plasmas. For example, xenon plasma sources can produce beams that are better at milling than gallium beams because they have higher sputter yields.

IN PRACTICE

Of all the possible applications of FIB, nanofabrication requires the greatest level of ion beam stability and control, as the process of producing fine patterns and detailed structures over extended areas can take hours or even days. This means a stable beam current and ion emission, and fine control over beam size and positioning.

A focused gallium ion beam typically has an energy of around 30keV, but this can range from 5keV to 50keV. The diameter of the beam (spot size) and its current can be as small as under 5nm and 1pA, respectively; at the other end of the scale, the spot size can reach over 500nm and the current around 100nA. For fabrication applications, however, beam currents toward the lower end (1pA to 1nA) tend to be utilized, as they provide the required resolution.

In general, the smaller the diameter of the beam, the better the etching resolution. The beam diameter is mainly determined by the beam current, with larger currents producing wider beams, and by the lenses that focus the beam. The current generation of FIB instruments can etch features smaller than 10nm, depending on the beam size and specific interactions with the sample.

The other beam property that has an influence on resolution is beam profile, meaning how the intensity of the beam varies over its width. This profile comprises a central peak of maximum intensity surrounded by tails of declining intensity; for high-resolution fabrication, those tails should be as small as possible.

The ion dose, meaning the quantity of ions delivered by the beam, is usually divided into many portions and delivered in multiple rounds. The number of repeated scans (loops or passes) can range from 1 to 10 000 for milling and from 100 to 1 million for gas-assisted deposition. For the latter there is usually a set delay, up to around 1ms, between repeatedly scanned points; this is known as the refresh time. The length of time that an ion beam stops at a specific scanned point is called the dwell time, which can vary from 50ns to 1s, while the distance between dwell points (step size or pitch) is usually 50–200% of the spot size.

The substrate is placed on a stage that can move in three separate directions, with some stages also able to rotate and tilt, while the ion optics focus and shape the ion beam and scan it over the surface of the substrate. Most stages use standard position encoding systems. Stages equipped with laser interferometer position control provide the precision and stability for large area nanofabrication. Meanwhile, dedicated FIB nanofabrication systems are equipped with such ultraprecise stages , similar to electron beam lithography systems. The stage is usually oriented horizontally, with the ion beam placed vertically above it.

By moving both the stage and the ion beam, patterns defined with computer-aided design tools can be etched into the substrate. Software supplied with commercial FIB systems can automatically determine the scan path, dwell points and dwell times needed to implement the designs. This software can also take into account the composition of the substrate, as less robust substrates will require more rounds at shorter dwell times.

The great advantage of FIB for nanofabrication is its ability to conduct direct patterning, removing the need for complicated masks and pattern transfer processes. As a consequence, the fabrication process is simpler, requiring fewer steps.

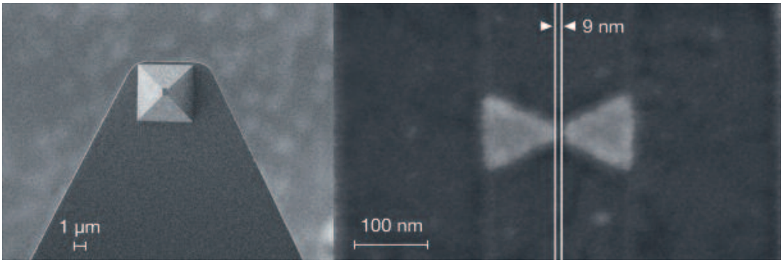
This helps to explain why the semiconductor industry was the earliest industrial user of FIB nanofabrication. Nowadays, the industry uses FIB to characterize and modify prototype-integrated circuit devices, as well as to analyze physical failures. Like a micro-soldering iron, the ion beam is capable of precisely milling a cut to disconnect a wire, but can also deposit conducting material in another area to connect two pieces of wire. Rewiring interconnects for circuit editing or selectively deprocessing a certain area on a chip to investigate a defective component are routine procedures today. This can all be done with minimal damage to the silicon substrate.

The hard disk drive manufacturing industry has also adopted FIB for trimming magnetic write heads. While read-write heads are fabricated mainly by optical lithography, an additional FIB milling step shapes the magnetic strip on the wafer used for writing.

FIB has also recently become a popular technique for fabricating high-precision microstructures on MEMS such as pressure sensors and actuators, as well as on photonic devices and scanning probe microscope tips. FIB techniques such as milling, etching and deposition are attractive for MEMS processing, because they allow direct high-resolution patterning on surfaces placed at various angles, such as vertical or curved surfaces. Apart from surface modifications, milling can also achieve the complete fabrication of structures such as cantilevers.

FIB is presently the only nanofabrication method capable of producing intricate and high-resolution 3D surface patterns on any solid material. This means FIB can fabricate layered structures like nanoscale-stacked ‘junction’ devices, which show promise as photovoltaic devices made up of two or more semiconductor materials stacked on top of a solar cell.

Another layered structure that lends itself to FIB nano-fabrication is graphite, which consists of atom-thick layers of pure carbon (graphene) stacked on top of each other. Thanks to its



Plasmonic nano-antenna on top of a gold-coated AFM tip. Direct milling, *ie* without the need for using a resist, enables high-resolution features even on topographic samples. Image produced by Raith for Professor Moerner, Stanford University, USA

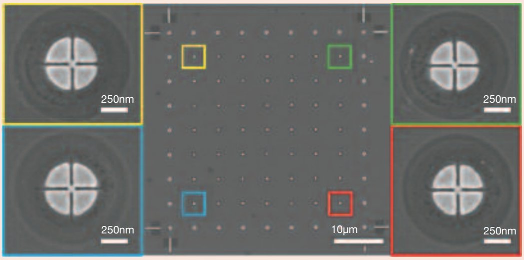
interesting electrical and electronic properties, graphene has recently attracted much research attention (see *Case study 2*). FIB 3D etching can fabricate sub-micrometer stacked junctions made from bulk graphite materials, as well as nanodevices such as graphene nanoribbons, graphene-based ultracapacitors and 3D single electron transistor (SET) devices.

Sometimes a fabrication task is uniquely suited for FIB processing. For example, 3D deposition and milling techniques can be used to fabricate microfluidic devices containing a network of tiny channels, with diameters ranging from 1µm to 100µm, for fluids to pass through (see *What's next?* section). These devices are used in a range of biological and chemical applications, such as cell culturing and DNA analysis.

At the small scales available on these devices, the mixing of fluids is particularly difficult to achieve. FIB can solve this problem by patterning sophisticated 3D channel shapes that produce mixing by exploiting geometrical patterns in a single fabrication step.

CASE STUDY 1

Saulius Juodkazis at Swinburne University of Technology, Hawthorn, Australia, is experimenting with using FIB techniques to create precisely patterned arrays of nanoparticles that show great potential as nano-tweezers.[1]



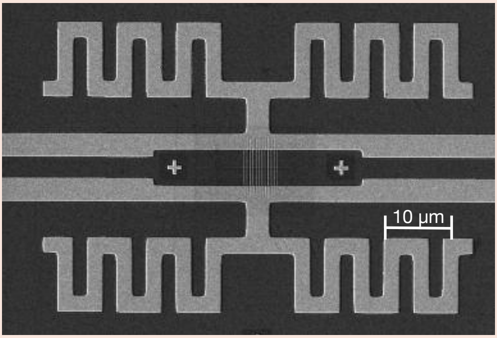
Array of precisely and reproducibly patterned gold nanoparticles for plasmonics applications. Reprinted with permission from Gervinskas G, Seniutinas G, Rosa L, Juodkazis S. Arrays of arbitrarily shaped nanoparticles: overlay-errorless direct ion write. *Advanced Optical Materials* 2013;1:456–9 ©Wiley-VCH Verlag GmbH & Co.[1]

These nano-tweezers exploit the phenomenon of plasmonics (the result of interactions between an electromagnetic field and free electrons in a metal). When light from a laser is focused onto an array of metal nanoparticles, often gold, it excites electrons at the surface of the metal. This creates rapid waves of electromagnetic charge that form 'hot spots' of enhanced fields around the edges of the particles. These 'hot spots' can act as tweezers, trapping specimens as small as a few tens of nanometers.

Fabricating the arrays of 3D tailored nanoparticles – arranged in patterns consisting of equal, narrow grooves separated by 10–20nm – has proved tricky using techniques such as electron beam lithography (EBL). One of the challenges is to produce these features over areas that stretch to hundreds of micrometers.

The solution adopted by Juodkazis and his colleagues is to combine EBL with FIB. First, they use EBL to deposit gold, fabricating the basic shape of the nanoparticles. Then they employ FIB to etch the pattern of grooves needed for the production of the hot spots.

According to Juodkazis, combining these two fabrication techniques provides large array fabrication with high precision, removing errors in patterning. This two-step approach could also be used for various other applications, including fabricating lab-on-a-chip devices, terahertz emitters and fractal antennas.



Terahertz emitter. Image courtesy of Dr Juodkazis, Swinburne University of Technology, Hawthorne, Australia

1. Gervinskas G, Seniutinas G, Rosa L, Juodkazis S. Arrays of arbitrarily shaped nanoparticles: overlay-errorless direct ion write. Applied Optical Materials *2013; 1:456–9. (http://onlinelibrary.wiley.com/doi/10.1002/adom.201300027.epdf)*

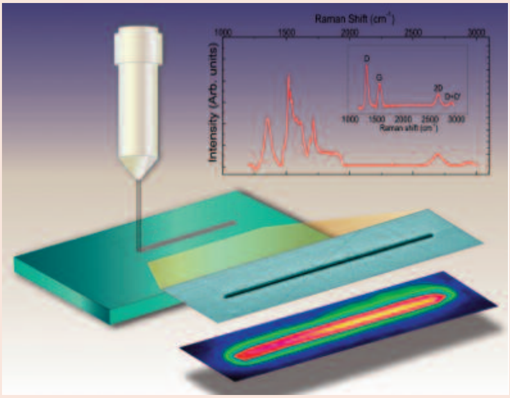
CASE STUDY 2

Brent Gila leads a team of researchers at the University of Florida, Gainesville, USA, who are using FIB to process the single-atom-thick sheets of carbon known as graphene. This is allowing them to produce patterns of several layers of graphene over large areas with nanoscale precision.[1]

Taking advantage of FIB's ability to insert ions into substrates, they are inserting a defined pattern of ions into crystals of silicon carbide (SiC) and then subjecting these crystals to controlled heating and cooling, known as thermal annealing. This annealing causes the silicon to sublime and results in several layers of graphene forming where the ions have been implanted, replicating the pattern.

Gila and his team employ a variety of different LMIS, including germanium, copper, gold and silicon, together with a filter, to produce nanometer-dimension ion beams accelerated through 15–40kV. These beams contain single or multiply charged ions or ion clusters. By multiple charging, ions can carry multiples of the primary acceleration voltage, eg *2 x 35kV = 70kV.*

When using thermal annealing combined with gold or silicon ion implantation, Gila and his team have been able to grow graphene nanoribbons (with dimensions ranging from 20 to 200nm in width) selectively on SiC crystals.[2] *Because graphene only forms on the implanted regions, the surrounding and underlying SiC recovers its crystalline form.*



Schematic representation of an example of an FIB-directed growth process. After patterned ion implantation and subsequent annealing of the SiC sample, graphene grows only in the implanted regions.[1] Image courtesy of Brent Gila

They have also shown that it is possible to grow graphene in implanted areas when using pulsed laser annealing (PLA). PLA is more attractive than thermal annealing because it is rapid, maintains the substrate surface near room temperature and requires only short processing times in many environments, including air.

1. *Lemaitre MG, Tongay S, Wang X,* et al. *Low-temperature site selective graphitization of SiC via ion implantation and pulsed laser annealing.* Appl Phys Lett *2012;100:193105. (http://dx.doi.org/10.1063/1.4707383)*

2. *Tongay S, Lemaitre M, Fridmann J,* et al. *Drawing graphene nanoribbons on SiC by ion implantation.* Appl Phys Lett *2012;100:073501. (http://dx.doi.org/10.1063/1.3682479)*

PROBLEMS AND SOLUTIONS

As an inherently destructive technique, a major drawback of FIB systems is the damage that can be caused by the ion beam. Bombardment with ions usually results in some gallium being inadvertently implanted in the surface layers of the sample. As the ion dose increases, so too does the ability of the ions to damage the substrate and penetrate further inside. However, this is not always a drawback as FIB's ability to alter the sample is actively desired for certain applications, such as doping semiconductors.

The ion beam can scramble the surface of crystalline materials (amorphization) and disrupt the crystalline order. By displacing atoms, the beam creates holes (vacancies) in the substrate, forming an amorphous layer at the surface. Because hundreds of vacancies may be generated by each ion impact, exposure to even relatively small ion doses can result in significant changes to a substrate's properties. Other effects of the ion beam include point defects, dislocations (where parts of the crystal lattice structure get displaced), and changes to crystal shape and size.

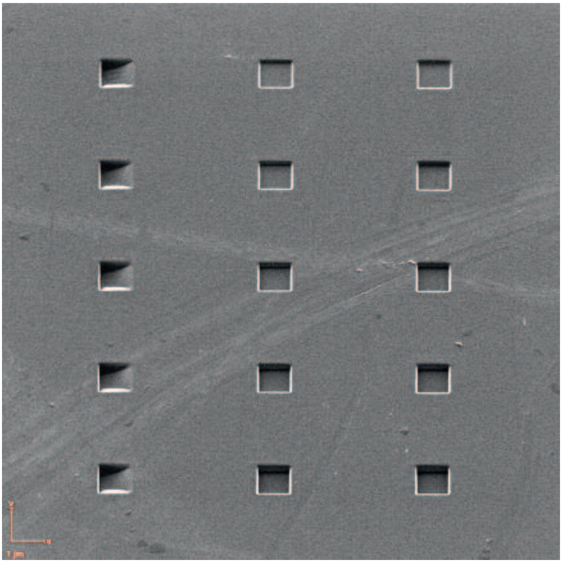
There are several approaches for reducing undesired sample damage, each with advantages and disadvantages. One approach for directly protecting against damage is to decrease the energy of the ion beam, thereby reducing the depth that ions can penetrate into the sample. This has to be balanced, however, against the loss of resolution that occurs at lower beam energies. Another strategy is to tilt the sample: researchers have found that the depth of damage can be decreased by tilting the specimen at 4–8 degrees.

In general, scanning only the outer parts of the sample and the alignment marks that guide pattern placement can also help to keep damage to a minimum, because this reduces the ion dose applied to crucial or sensitive parts of the substrate. A protective thin film coating can also help to block damage from the processing beam. This coating may be removed later if it's made from a suitable material.

Another way to reduce sample damage is by conducting gas-assisted etching (GAE), which can also improve pattern depth and definition. Similarly to deposition (see ***History and background***), a 'precursor' gas is injected onto the sample, where it adsorbs on the surface. The ion beam provides sufficient energy for the gas to react with the sample, producing volatile reaction products that subsequently break free and thus remove material from the substrate. Using a gas in this way allows the beam to remove layers faster with a smaller ion dose.

GAE can only be used with certain combinations of substrates and precursor gases, as it requires reactions that specifically form volatile products, rather than non-volatile products that deposit on the substrate surface. Furthermore, GAE sacrifices some of the precision of unassisted ion milling for higher speed, although features with sizes of just 20nm have been produced with GAE. Nevertheless, a two-stage process of initial GAE followed by ion milling is frequently used when an operation requires both the removal of bulk volumes of material and high precision.

Redeposition, where sputtered material resettles back on the surface resulting in unwanted residues and shapes, can also cause problems. Redeposited material can make it difficult for an FIB system to produce complicated, large-volume or 3D patterns with a uniform depth. One way to limit redeposition is to scan the beam in short, repeating loops over the surface. Sometimes it can take several thousand repeats to remove material precisely while limiting the chances of redeposition.



Milling results for 1µm squares in SiC with constant dose per line, while the total dose is applied in 1 (left) or 1000 loops (right). The scan strategy consists of lines in Y direction from right to left on all boxes and shows pronounced redeposition and edge effects in case of the single loop approach. Image produced by Raith

Finally, any work involving charged particle beams and materials in a vacuum is prone to the effects of charging. Electric charge can build up around the patterns on a substrate if the material is electrically insulating. This accumulated charge can cause the ion beam to drift off course or lose focus, and can sometimes produce melting and explosions when the sample does finally discharge.

Researchers have come up with various ways to alleviate this problem, such as by coating the surface with a conductive thin film or supplying negative charge with an electron flood gun, simultaneous electron and ion beam scanning, or electron beam irradiation. These approaches usually allow accurate milling of even the most insulating substrates, and can also be applied in an automated way.

One particular patterning issue faced by FIB users is ending up with rounded edges and 'side-walls' at an angle. This can happen because sputtering is more prolific on the side-walls due to the angle of incidence of the ion beam and also in some cases because material is redeposited. When it is important to create a pattern with sharp edges, one solution is to finish the edges last using a small beam current.

Additionally, a sacrificial thin film of material coated on the sample can take up much of the rounding and then be removed after patterning. In some applications, it may also be useful to consider tilting the sample a couple of degrees in order to produce edges that are vertical to the sample surface.

Another potential problem is that 'differential' milling can produce uneven surfaces and poor pattern definition. This occurs because milling rates depend on how crystals are oriented in the sample, as some orientations undergo less sputtering. One way to minimize this problem when milling thin films of polycrystalline material is to apply a small number of beam passes but long dwell times.

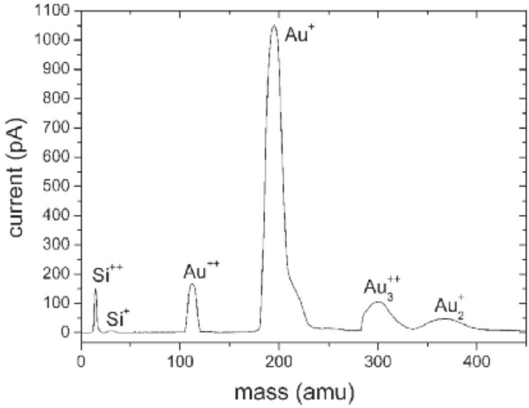
WHAT'S NEXT?

The performance of modern FIBs is principally determined by the properties of the ion source, and thus advances can be made by designing new ion sources and experimenting with new ion species. Although most FIB users still work with gallium LMIS, other ion sources and species are available (see ***History and background***). Novel techniques for producing ion beams are also being investigated all the time, potentially extending the range of applications of the instrument.

Alternatives to gallium as an ion source include gold and silicon. Advances in sources and column design have recently solved some of the issues associated with using these other ion sources, including stability, handling and lifetime.

When gallium is used as the ion source, the resultant beam comprises only Ga^{+}. In contrast, gold and silicon produce a beam made up of a range of different species of ion, including Au^{+}, Au^{++}, Au clusters, Si^{+} and Si^{++}. This gives operators more options, allowing them to select specific ions by introducing an ion separator or mass filter into the column, as different ion species interact with substrates in different ways. Light ions produce narrow beams, but tend to penetrate deep into the substrate rather than removing atoms via sputtering. In contrast, heavy ions don't implant as deeply and show a larger sputter yield, but can't be focused into narrow beams.

Gold and silicon beams could be particularly suitable for fabricating 'plasmonic' structures, which often use silicon or silicon oxide as a substrate with a layer of gold on top. These structures use finely patterned features to exploit optical phenomena caused by surface plasmons, which refer to the collective oscillation of electrons at the surface of certain metals, especially gold.



Mass spectrum of a AuSi alloy ion source providing a range of different ion species. Image produced by Raith

FIB milling can directly create the features required to manipulate surface plasmons. With gallium-based FIB, however, these features can be contaminated with implanted gallium ions, hampering their ability to manipulate the surface plasmons. As these features are already made from gold and silicon, however, any implantation with further gold and silicon ions doesn't result in any contamination.

Other applications that can benefit from the low contamination offered by gold and silicon ions include fabricating photonic structures (3D layered structures that can manipulate light, such as optical fiber sensors and waveguides), silicon-based devices, nanopores and semiconductors.

Gold ions implanted into samples by FIB can also acts as 'seeds' for subsequent processes such as binding biomolecules, growing graphene (see *Case study 2*), and creating nanoparticles and nanowires. Another promising application is the direct patterning of catalysts, where gold ion implantation is followed by thermal annealing (heating to a certain temperature and then cooling slowly) to form catalytic clusters on the substrate surface. For example, Carmen Marcus and her colleagues at the Institut de Ciència de Materials de Barcelona in Spain have used a gold FIB to assemble and pattern catalytic germanium nanowires on silicon substrates.

Completely novel ion source technologies are being developed that can work with lithium and caesium, offering the potential to produce beams with a narrow energy spread that can fabricate at high resolutions. For example, Jabez McClelland and his team at the Center for Nanoscale Science at the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, USA, are working on low energy spread FIB systems with a lithium ion source. This ion source works by cooling lithium atoms with a laser to just a few millionths of a degree above absolute zero and then photo-ionizing the atoms in an electric field to create an extremely cold ion beam.

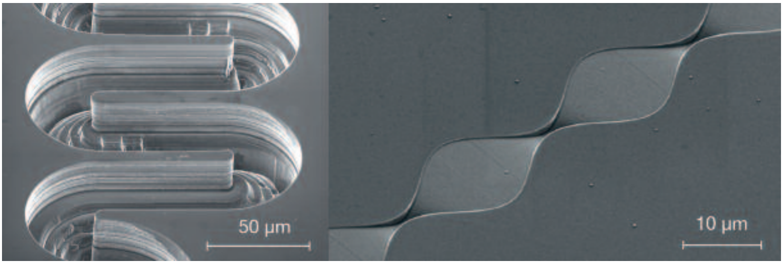
Removing the heat and associated 'randomness' from the ions can produce ion beams that are brighter and more sharply focused. McClelland's team has developed an instrument with probe sizes of a few tens of nanometers and beam energies of 500–5000eV. These beam energies are much lower than the typical operating energies of gallium-based systems.

FIB milling is also being put to new and exciting uses, such as helping to produce microfluidic devices with finely designed microchannel walls and precisely shaped submicrometer channels. For example, Leo Ocola's team at the Argonne National Laboratory in Illinois, USA, has been working on microfluidic mixers containing 3D patterns with complex geometries that are several tens of micrometers deep. They use techniques such as photolithography and dry and wet etching to remove large volumes before fine patterning with a FIB. This allows them to produce microfluidic channels that are tens of micrometers in width and depth, but hundreds of micrometers in length. So far, their 3D designs appear to be significantly faster mixers than standard mixers with no 3D texturing.

These kinds of devices and structures typically extend single scan fields of the ion column and can easily cover millimeters or even centimeters. Therefore new fabrication methods are required that include precise stage movements and need to be combined with beam scanning in a synchronized way. Methods like write field stitching or truly continuous patterning by using precise stage movements are well known from other areas of nanofabrication, *eg* electron beam lithography, and enable a complete new area of possibilities. Recently there has been significant progress in adapting it to FIB in order to overcome some specific challenges like edge effects at the write field border.

Another example of a large area, *ie* wafer scale, application for which FIB is particularly suited is making ultra-thin membranes containing nanopores with diameters less than 10nm. Membrane-based solid-state nanopore devices are used for DNA sequencing, and other biological sensing and filtering applications.

Since these applications require either a large area covered with nanopores or a large number of devices with a few or single nanopore, challenges like process time, automation, large area precision and reproducibility play a major role besides the ultimately small pore diameter. The instrument capabilities and achievable devices depend on many aspects like the selected ion species, *eg* light for small pore diameters versus heavy for fast process



Microfluidic mixer structures can be fabricated by 3D FIB machining both on a large micrometer scale (left, compare Ocola *et al*, 2013) as well on a micrometer/submicrometer scale (right, compare Palacios *et al* 2010) by complex and precise patterning control in combination with sophisticated sample positioning. Reprinted with permission from Ocola LE, Palacios E. Advances in ion beam micromachining for complex 3D microfluidics. *J Vac Sci Technol* 2013;31:06F401

times, the source technology defining the long-term stability and spot size, and the overall system architecture, *ie* large area, unattended and automated fabrication capabilities. Finally, also the membrane quality and thickness determine the shape and size of the nanopores, which is an active research area looking at ultimately thin membranes like self-assembled monolayers or graphene.

FIB systems are likely to continue to play a major role in nanofabrication because of their superior flexibility, resolution and precision, and their ability to conduct material addition, removal and modification. The number of different FIB techniques currently available for nanofabrication in a single instrument is impressive, and so is the range of applications where they can be used. Both are set to expand as exciting new instruments, employing various ion species, continue to appear.

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