Various Ion Species from a Universal Ion Source for FIB Nanofabrication

Nanofabrication requirements for FIB technologies impose specific demands in terms of patterning resolution, stability, and the support of new processing techniques[1]. Additionally, the type of ion defines the nature of the interaction mechanism with the sample. Therefore, Raith has extended the technology to include stable delivery of multiple selectable ion species for a nanometer scale focused ion beam by employing a liquid metal alloy ion source (LMAIS)[2] (Figure 1). This LMAIS provides single and multiple charged ion species of different masses, resulting in significantly different interaction mechanisms. Nearly half of the elements of the periodic table are thus made available in FIB technology thanks to continuous research in this area[3]. This range of ion species with different mass or charge can be beneficial for various nanofabrication applications. Recent developments make these sourcies available for challenging nanopatterning. This white paper presents the operating principle, LMAIS capabilities for FIB nanofabrication, and results of modern FIB direct nanopatterning applications[5].

Impact of ions, triggered processes and influence of ion species

The interaction between ions and a substrate has consequences for the resulting nanostructures as well as for fabrication strategies. When ion impacts into a solid, it loses kinetic energy by interacting with the sample atoms. This transfer of energy from the ion to the solid results in several different processes (Figure 2), with processes such as electron emission, atomic sputtering, ion emission, and sample damage being relevant for FIB nanofabrication.

The ion beam can be scanned over a sample surface in the same manner as an electron beam and produces FIB images by detecting emitted electrons. Images generated in this way can either be used as reference for drift compensation by automated mark recognition, or for endpoint detection of layered samples. However, the main purpose of focused ion beam sample processing is material removal and modification of the targeted substrate. Sputter yield and crystal lattice damage are determined by ion energy as well as ion mass. Along with these processes, the ion typically comes to rest in the solid, resulting in implantation of the ion[6]. While the effect of ion implantation can be desired in case of sample functionalization and doping, it can also be considered as sample contamination and may harm the nanostructure in question.

The choice of ion species for FIB nanofabrication processes has a huge impact on the resulting nanostructures

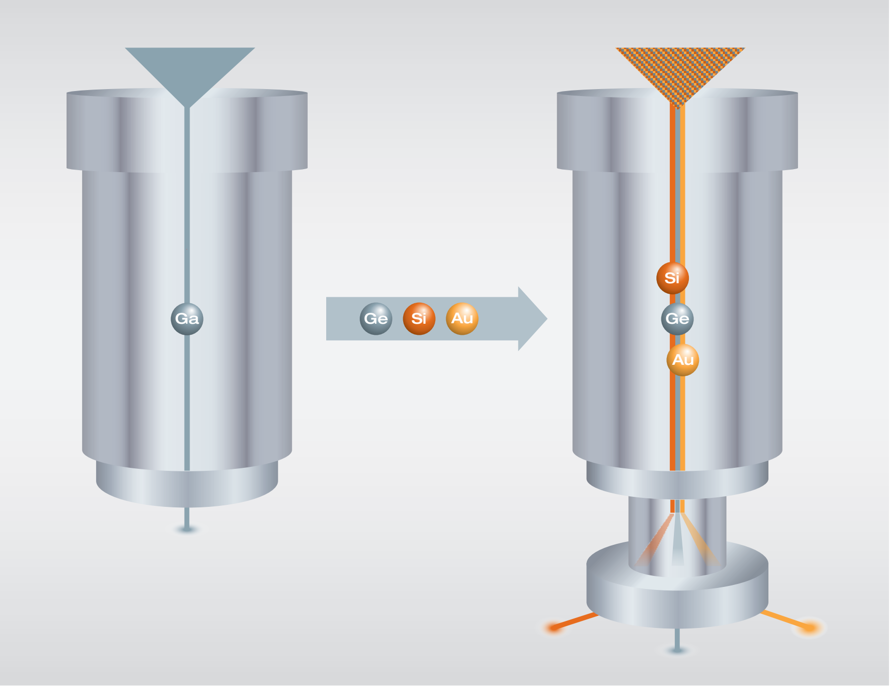

Figure 1: Schematics of nanoFIB column for Ga FIB (left) and nanoFIB with IONselect extension including LMAIS and Wien filter (right)

and reveals the high demand for providing various ion species from a universal ion source for the purpose of tailoring the nanostructures by means of FIB nanofabrication.

IONselect technology: From Gallium LMIS to universal LMAIS for FIB nanofabrication

Raith's proprietary FIB technology has evolved to enable advanced nanofabrication with different ion species. With unique universal ion sources, it truly defines a new state of the art in FIB technologies. The IONselect setup comes with a multi-species ion source consisting of a eutectic alloy containing various ion species. This liquid metal alloy ion source (LMAIS) replaces the Gallium (Ga) liquid metal ion source (LMIS) to make universal combinations of ions such as AuGeSi or GaBiLi with nanometer beam diameters accessible (Figure 1). The low-aberration ion selection optics is based on a special flow-optimized tip geometry for the eutectic alloy material, ensuring long-term stability and improved lifetime. With automated ramp-up and emission control, the source is an integral part of the nanoFIB® column for operation in Raith's VELION system, which defines a unique tool for nanofabrication.

Source technology

Unlike Gallium, eutectic alloys that are used for LMAIS are not liquid at room temperature and consequently require heating. Eutectics typically require a few 100°C for liquefaction. This temperature is much lower than the melting temperature of the pure components of the alloy. Thus, LMAIS are an excellent way to make various ions available by moderate source heating.

Ga sources and LMAIS have a common mechanical setup and working principle. A reservoir is filled with either Ga or eutectic alloy and connected to a metallic needle. To allow ion emission from sources, an extractor voltage is applied to create a strong electric field near the tip to start ion emission[3].

Schematics of the operation principle of Ga-LMIS and LMAIS is shown in Figure 3.

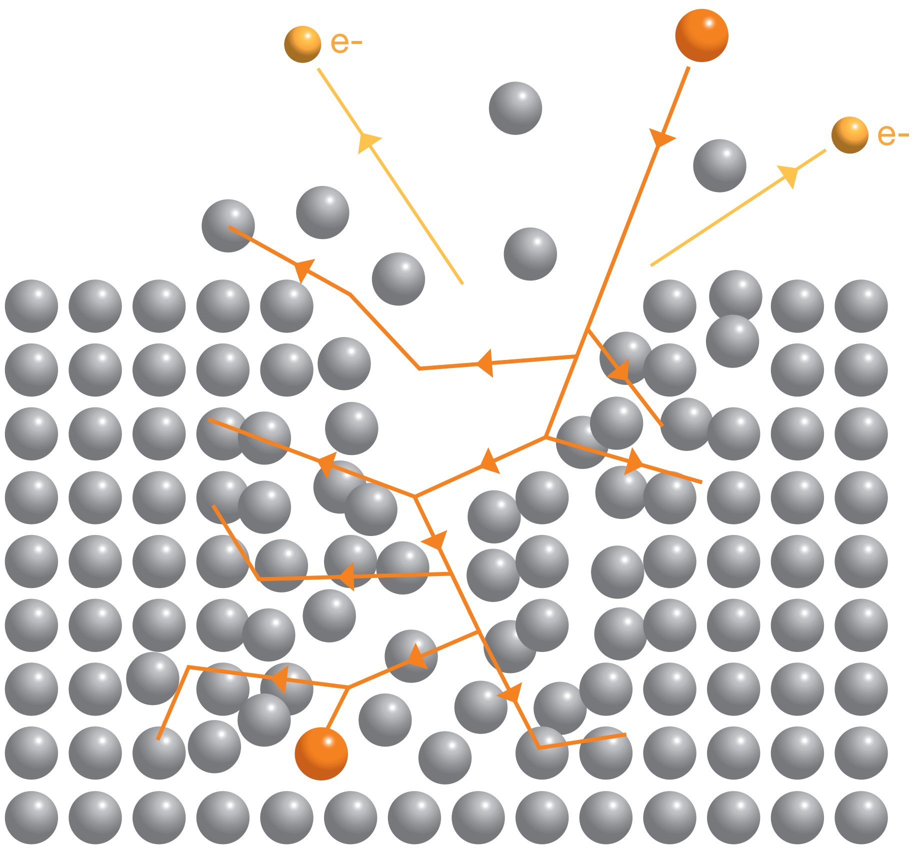

Figure 2: Ion beam and substrate interaction. Impacting ions generate electron and ion emission and are finally implanted into the substrate

ExB Filter

Since the liquid metal alloy ion source provides simultaneous emission of various ions or clusters of different charge states, switching between multiple ion species (m/q) is required. By employing an ExB filter system, this is easily achieved without forfeiting the high resolution of the nanoFIB performance. A permanent magnet is mounted outside the vacuum environment of the column and provides a constant B field to eliminate deleterious hysteresis effects. The required electrical field can easily be changed by changing voltages to

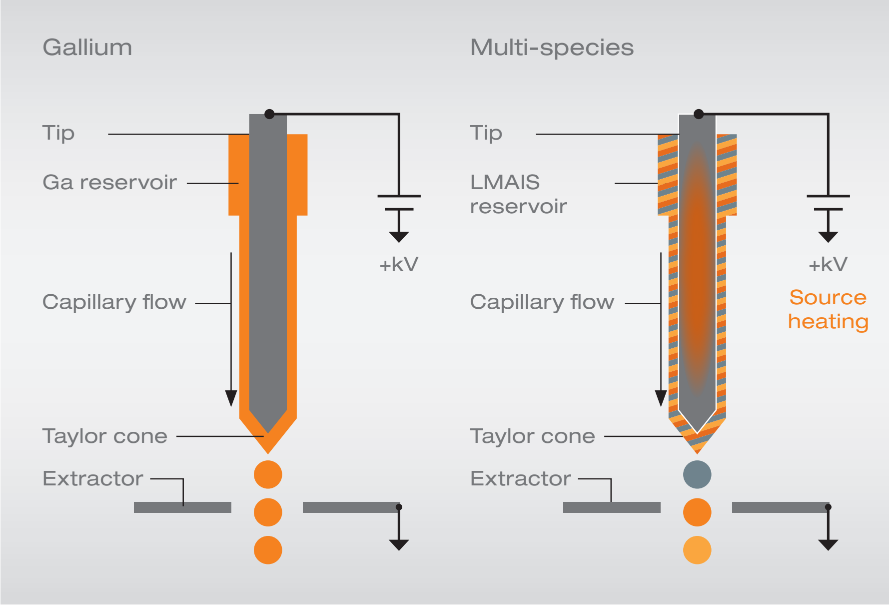

Figure 3: Schematics of Ga FIB ion emission (left) and schematics of LMAIS FIB including source heating (right)

separate different (m/q) ions. Quick, reliable, and dependable toggling between different ion species, clusters or ion charge states is possible. No mechanical realignment is required as only the electrical field of the ExB filter is changed to switch between different ions (Figure 4).

Available ion sources

At present many elements of the periodic table are suitable for FIB applications. Elements highlighted in orange are basically available as single or double charged ions and clusters in LMAIS, but certain constraints and requirements must be met to use these ions for FIB nanofabrication (Figure 5).

Requirements for LMAIS usage

Both the mechanical manufacturing processes and the material combinations for tip, reservoir, and LMAIS must meet high technical demands in order to maintain long-term beam current stability and superior source performance capable of focusing ion beams into smallest spot sizes.

One of the best alloys for use in LMAIS is AuSi eutectic. This eutectic metallic glass represents a stable source and is very well suited for FIB due to its low melting temperature and low vapor pressure. Thermal evaporation of the source is therefore minimized, extending the lifetime of the source. Another important characteristic of AuSi-based eutectics is the good wettability of the reservoir and tip, which ensures a continuous flow of liquid towards the tip to guarantee stable ion emission even at low currents.

AuSi is also well suited to accommodate additional elements such as Germanium (Ge) or Lithium (Li)[7], and to act as basis of a ternary compound. The mass spectrum of a AuGeSi shows that this source matches the requirements of the ExB mass separation filter. Peaks do not overlap, so that ions of different m/q can easily be separated (Figure 8). These characteristics make AuGeSi a good choice from an engineering perspective, as this eutectic meets all criteria for LMAIS FIB technology. In addition, AuGeSi as an ion source is highly sought-after from an application point of view. Extensive research is conducted into semiconductor-based materials and silicon-containing substrates. Plasmonic structures are often fabricated of Gold (Au) on Silicon (Si) substrate[8], a scenario in which contamination can be minimized or even eliminated. In particular, Germanium is highly sought-after for the creation of vacancy defects.

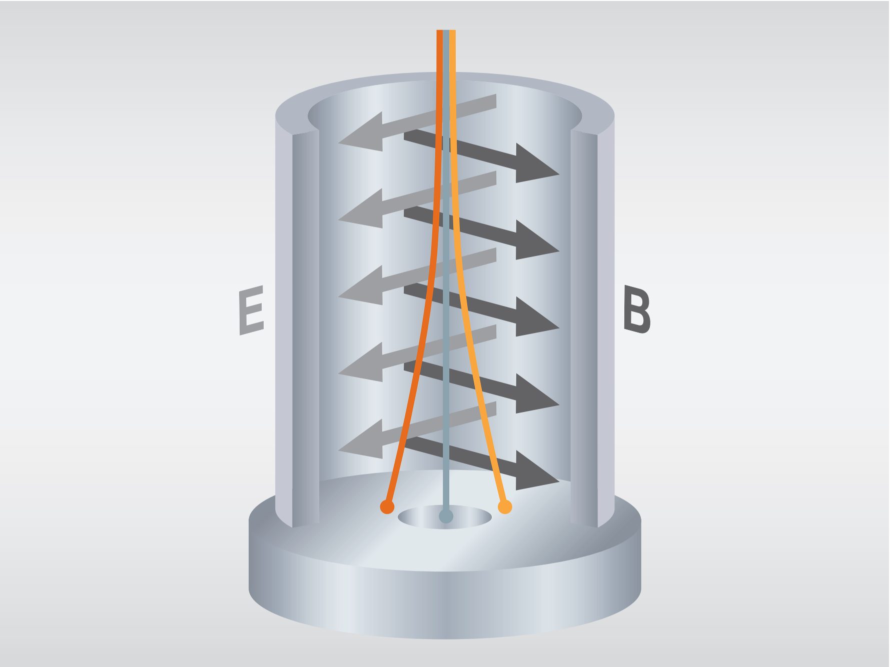

Figure 4: ExB filter setup to separate ions of different mass and charge

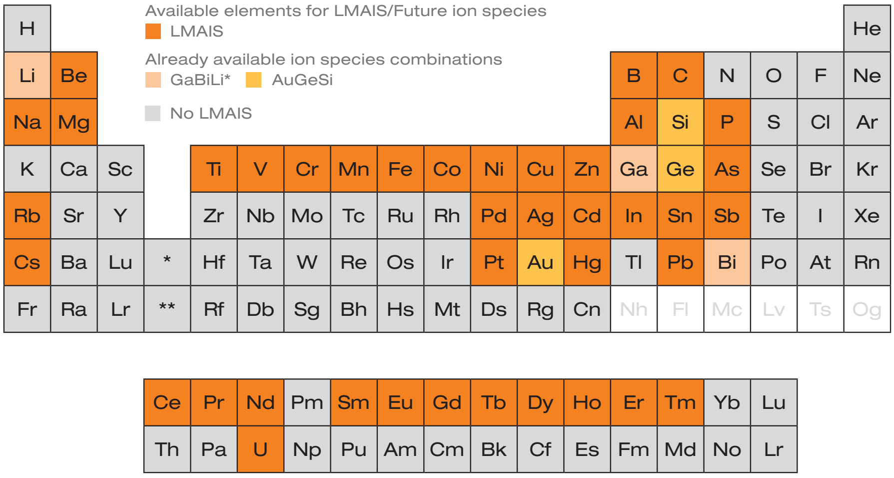

Figure 5: Available ions from LMAIS (orange) and Universal Ion Sources AuGeSi/GaBiLi (yellow/gray)
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Another very interesting LMAIS contains Lithium as the lightest element and Bismuth (Bi) as the heaviest element available from the periodic table. Ga can be added to this eutectic, which is well known and frequently used for FIB applications. Light lithium-ion beams have the smallest beam diameter, which results in the highest lateral resolution of patterned nanostructures, whereas heavy Bi ions and Bi clusters have the best vertical resolution at higher sputter yield (Figure 9).

Both combinations – AuGeSi and GaBiLi – fulfill important engineering-related demands with respect to materials.

In addition, crucial parameters for ion sources are virtual source size d_v and energy spread ΔE, due to their influence on the final spatial resolution of a FIB system.

The virtual source size of a LMAIS reflects the disk from which the ions seem to be emitted. The value is about 5 to 10 times higher than the physical size of the emission point because of

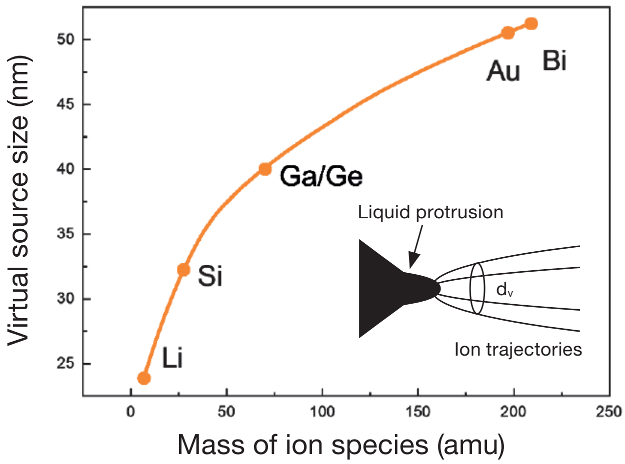


Figure 6: Virtual source size depends on ion mass, therefore light ions have a small virtual source size (d_v)

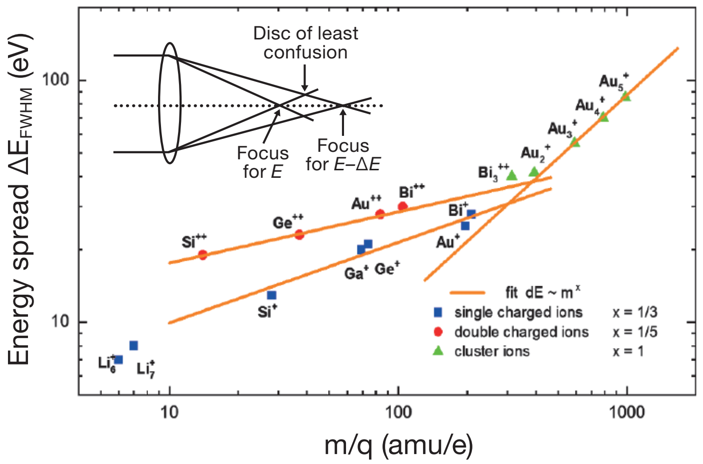


Figure 7: Energy spread and chromatic aberration of AuGeSi and GaBiLi LMAIS

lateral motion of the liquid jet, space charge effects, or Coulomb interaction of the ions. This parameter depends on the mass of the source material and emission current: $d_v \sim m^{1/4}$ (Figure 6).

The second source parameter which impacts highly on the ion beam diameter at the focal spot is the energy spread. The energy spread influences chromatic aberrations and determines the final spatial resolution of a FIB system according to $\Delta E \sim m^{1/3}$.

Experimental data on the energy spread of single and double charged ions and clusters as function of m/q ions can be found in Figure 7.

Lithium-containing LMAIS such as GaBiLi has the smallest energy spread and virtual source, and therefore the smallest ion beam diameter[2].

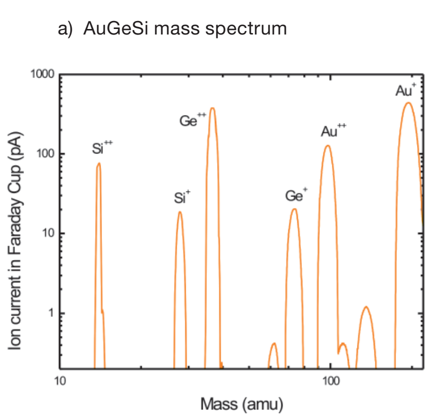
Universal ion sources

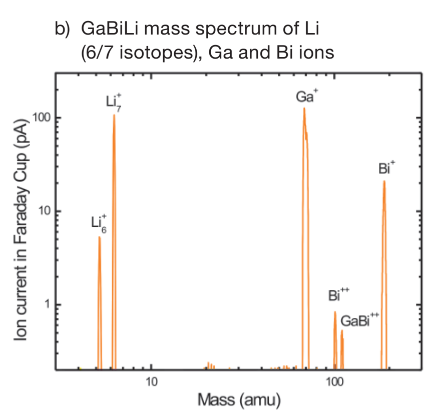
Both sources, AuGeSi and GaBiLi, enable sample processing to be performed with light ions or sample modification using heavy ions from a single ion source. Their specific characteristics are well suited to tailor nanofabrication processes for resulting nanostructures (Table 1).

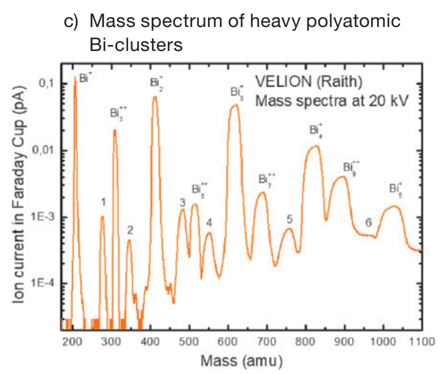
The choice of ion species is always driven by applications. As different ion species have different properties, they also offer different advantages and possibilities or disadvantages for specific applications. For instance, many applications are very sensitive to ion implantation. In some cases impurities are considered to be contamination and must be avoided. For material functionalization, on the other hand, impurities are desired and need to be implanted in a well-controlled manner. Sputter yield is a further parameter that is important for FIB nanofabrication; it depends on the mass of the impacting ion. Ion energy and ion species determine the number of ions that are emitted from the targeted sample per incoming ion and define the speed of material removal. Available ion beam current for the ion species, sputter yield, ion implantation, and ion beam diameter define the choice of ions which can be employed to the nanofabrication processes.

LMAIS	Available ions	Mass range
AuGeSi	$Si^{+,++}$; $Au^{+,++}$; Au_{2-5}^{+}; $Ge^{+,++}$	28 ... 985
GaBiLi	$^{6}Li^{+}$; $^{7}Li^{+}$; $Bi^{+,++}$; $Bi_{3,5,7,9}^{++}$; Bi_{2-7}^{+}; Ga^{+} (GaBiLi contains ^{6}Li and ^{7}Li isotopes)	6 ... 1.463

Table 1: Mass range of LMAIS ions







1. $BiGa^+$ 2. Bi_3Ga^+ 3. Bi_2Ga^+ 4. Bi_5Ga^+ 5. Bi_7Ga^+ 6. Bi_9Ga^+

Figure 8: Mass spectra
a) Au and Si ion peaks are well separated and Ge ions can be added between AuSi eutectic ions without overlap
b) Even different Li isotopes can be resolved by ExB filter
c) Many heavy Bi clusters are available from GaBiLi LMAIS

GaBiLi source

The expected and demonstrated beam diameter for Li from LMAIS is below 4 nm[4]. This ion species is the best choice with respect to high-resolution patterning and ion beam imaging from LMAIS. However, nanofabrication also requires consideration of the interaction between ion beam and substrate.

TRIM calculations shown in Figure 9 reveal the diverse interaction properties of light and heavy ions with the substrate. As the lightest ion available from LMAIS, lithium has the best lateral resolution. They penetrate much deeper into the substrate compared to heavy ion species like Bi or even Bi clusters.

Heptamer-arranged nanohole (HNH) array is a challenging structure and regarded as a benchmarking nanostructure for resist-free, maskless direct write nanofabrication[9]. Li FIB can fabricate adjacent holes as close as 15 nm without sacrificing ribbons between adjacent holes (Figure 10). Sidewalls are vertical without material redeposition.

Beyond single ion species FIB nanofabrication, ions from LMAIS allow for a wider application spectrum due to the vast number of different ion species, various charge states of ions, and rapid switching between ion species. Based on the varying interaction properties of the ions with the substrate, the ion species can be selected and adapted to the respective strengths of the particular task (Table 1).

Figure 11 shows the stepwise fabrication of bowtie structures. In the first step, a large volume of material was removed by Bi patterning to excavate rectangular structures (a). This process step takes advantage of the high Bi sputter yield. In a second step, the bowtie structures were refined (b) and adjusted (c) using a Li ion beam for high resolution direct patterning capabilities. The entire process can be run automatically with quick and reliable switching between ion species. This approach results in a faster and more accurate fabrication process overall compared to using just one ion species.

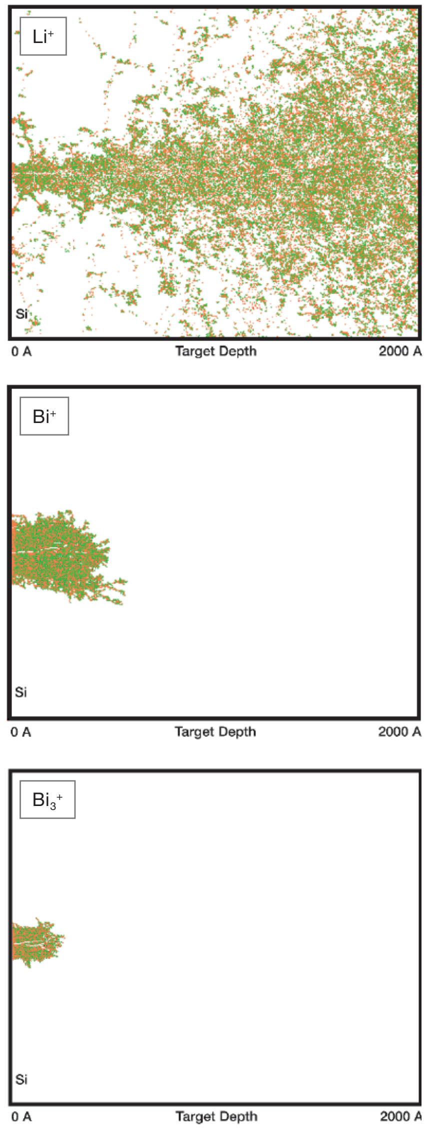


Figure 9: TRIM acceleration voltage 35 kV, target Si substrate

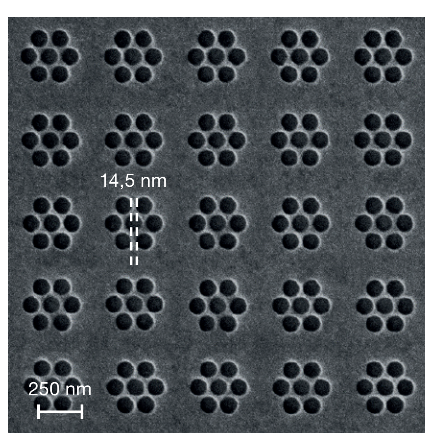
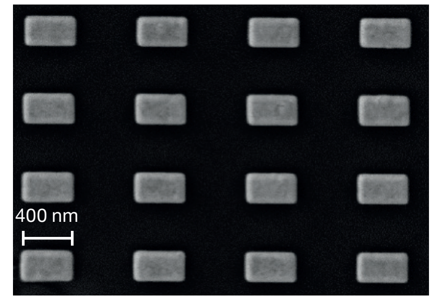
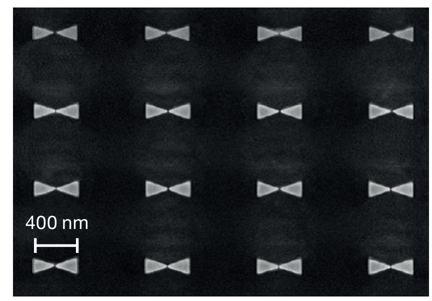


Figure 10: Li FIB patterning of HNH in 50 nm Au substrate and subsequent Li ion beam imaging

a) Step 1: Bi FIB for large-volume milling at high sputter yield to excavate rectangular boxes



b) Step 2: Li FIB for fine shaping of rectangular boxes to fabricate bowtie structures at highest resolution



c) Li FIB adjustment of the gap size of the bowtie nanoantenna

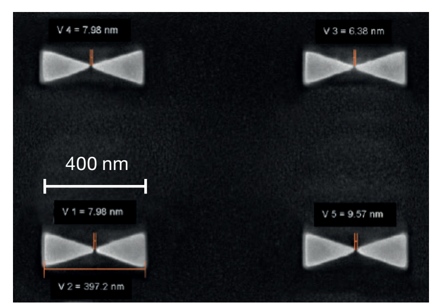


Figure 11: Stepwise fabrication of bowties/50 nm Au on Si substrate

AuGeSi applications

AuGeSi ion beams are broadly used for nanofabrication applications to avoid sample contamination. AuGeSi ion nanopatterning is usually an optimization process in selecting the ideal ion for achieving the targeted results to tailor resulting nanostructures.

Although Si^{++} has the smallest beam diameter compared to Ge^{++} or Au^{++}, it has the lowest sputter yield of ions from this source. Sputter yield is defined as the number of atoms ejected from the target per incoming ion. At maximum acceleration voltage of 35 kV, the kinetic energy of single charged ions is 35 keV, and 70 keV of doubly charged ions. Figure 12 shows the sputter yield of Si on a Silver target, which is at a kinetic energy of 35keV 2 to 3 times lower as of Ge and about 5 to 6 times lower as of Au respectively. Thus, Si will take 3 times longer to achieve the same milling effect as Ge^{++} at similar ion beam currents. As the different ion species can be switched within a very short timeframe, many different ion species and m/q from AuGeSi are available to adjust the chemical or physical properties of the resulting nanostructures. A typical workflow is illustrated in Figure 13 and represents the direct patterning of this process for a plasmonic device from a layered sample.

The goal of this application is to create a plasmonic device by excavating squares from functional layer on a layered sample. The top Ag layer is placed on a Si_3N_4 interface on Si substrate. It is very important for the functionality of the device not to expose the interfacing layer with ions other than silicon to avoid contaminations. The fabrication process of this plasmonic device was designed to utilize different ion species with the best properties for the respective layer. The top 80 % of the silver layer was milled using the Au ion beams to speed up the process, as the sputter yield is 5 times higher compared to Si. The remaining 20 % of the layer was cleaned up using Si^{++} to avoid contamination of the Si_3N_4 layer.

This process is possible because Au and Si ions are available from a single ion source and because of VELION's capabilities to accurately overlay the Si milling pattern on top of the previously Au ion patterned areas, enabled by a laser interferometer stage and automated mark recognition functionalities[13].

In addition, sample functionalization such as doping or ion implantation requires a wide range of different ion species.

For instance, Au ion implantation into SiC substrate is a straightforward technique for selective graphene growth. For this application it offers alternative avenues that can be applied to graphene based devices and nanoribbon synthesis. The desired regions of SiC for growing graphene layers are functionalized with Au ions, followed by an annealing step to a temperature of 100° C below the graphitization temperature of SiC. Implanted Au ions lower the graphitization temperature of SiC and allow selective graphene growth at temperatures below the graphitization temperature of pristine SiC, but above graphitization temperature of implanted SiC. In this way a graphene layer can easily be drawn onto the SiC surface[10].

Germanium ions are of high interest for creating color centers in diamond, as Ge vacancy emitters represent promising solid-state qubits for scalable quantum photonics applications. The preferred nanofabrication method of Ge vacancy emitters as a leading platform for spin-spin interactions within integrated photonic structures is the usage of Ge focused ion beams for mask less direct ion implantation at spatial accuracy of tens of nanometers[11].

The same nanofabrication approach can be employed to create vacancy defect arrays in silicon carbide by means of Si ion implantation. Reliable production of Si vacancy defects in silicon carbide could pave the way for its application in quantum photonics and quantum information processing. Essential requirements for spin-based quantum information processing, such as optical initialization, readout, and microwave control of the spin state, are met for Si vacancy defects in silicon carbide[12].

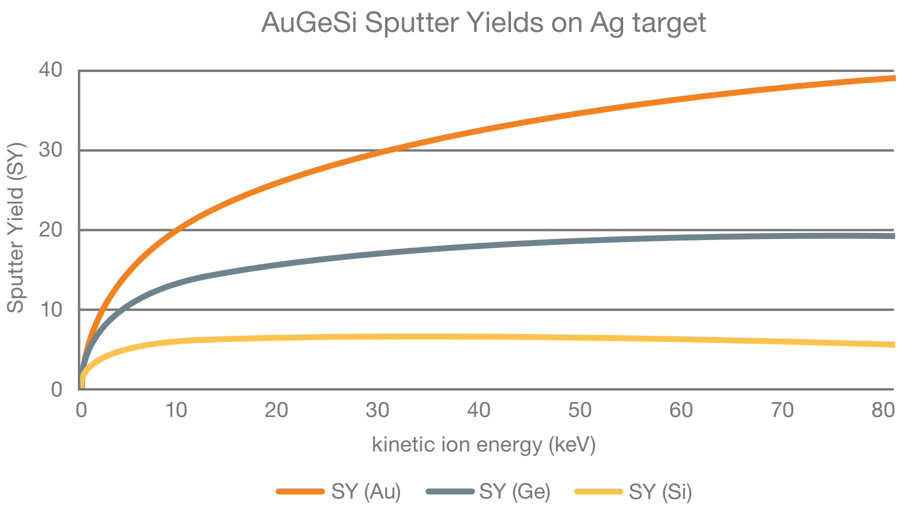
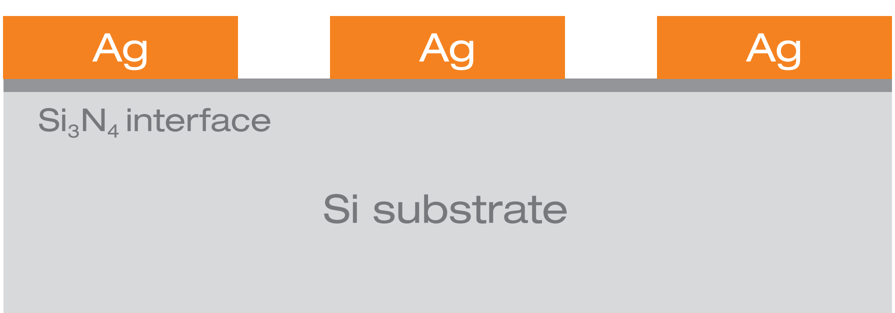


Figure 12: AuGeSi sputter yield as a function of ion energy



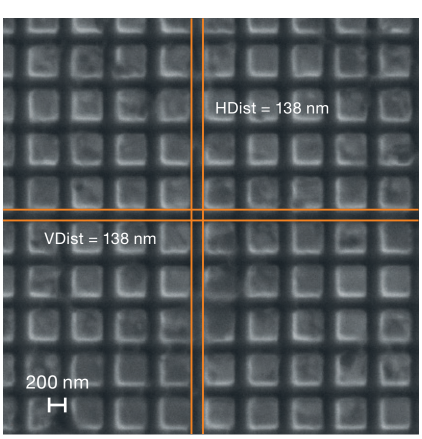


Figure 13: Illustration of the milling strategy on a functional film of a layered sample to avoid contamination

The VELION system

IONselect technology with universal ion sources is used in the VELION system.

VELION features a vertically mounted FIB-column and side-mounted SEM. In combination with Raith's Laser Interferometer Stage and matured lithography technology, VELION represents a unique FIB-SEM nanofabrication setup. The dedicated FIB-centric system architecture ensures unrivaled stability, accuracy, and automation. Even most complex structures in both 2D and 3D can be fabricated completely unattended and to the highest precision standards over extensive areas and long periods.

The combination of laser interferometer stage and multi-ion species from LMAIS has demonstrated its strength of achieving results faster than with any other nanofabrication workflow. Nanofabrication processes can be simplified and deliver results in one go without a complex and costly process chain [14]. Direct FIB nanopatterning with universal ion sources in combination with FIB-SEM on a lithography platform expands the possibilities of FIB nanofabrication and delivers solutions for today's nanofabrication challenges.

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Final Note

Raith's proprietary nanoFIB technology with IONselect technology has been specifically developed to meet requirements of advanced ion beam nanofabrication. The nanoFIB column employs universal ion sources providing various ion species to tailor the properties of resulting nanostructures.

- IONselect provides a user-friendly, easy-to-use ion access to FIB sources with multiple ion species
- IONselect enables fast switching between light or heavy and fast or slow ion species from universal ion sources
- nanoFIB column exhibits superior capabilities and delivers cutting-edge application results
- nanoFIB column incl. multipurpose ion source options is the central component of the VELION system. Combined with a laser interferometer stage and an SEM column, the VELION forms a versatile and outstanding nanofabrication system and sets a new benchmark for nanoengineering applications

Explore the huge potential of next-generation research instrumentation in nanoscale science, and experience the advantages of universal ion species for FIB applications!
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