Chapter 1 Focused Ion Beam (FIB) Technology for Micro- and Nanoscale Fabrications

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 Abstract The focused ion beam (FIB) technology has become increasingly popular fabrication and characterization tool across many research areas from nanotechnology, material science, microelectronic industry, life science, biology, and medicine. FIB was specially recognized as an attractive tool or the fabrication of micro- and nanostructures with complex geometries and shapes. This chapter presents the basic introduction of FIB dual-beam system and its operation modes, followed by description of instrument in more details. The review has emphasis on FIB fabrication of nanostructures by milling and deposition methods with particular focus on fabrication of nanopatterns and nanopore arrays. Various efforts to fabricate micro- and nanoscale structure and geometrically complex structure are described. Finally, recent developments of applications of FIB in different areas of material science and life science are briefly reviewed.

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1.1 Introduction

 Fabricating materials with micro- and nano-dimensions is one of the biggest challenges in the material science and nanotechnology, and focused ion beam (FIB) is an important technology to address these challenges. The FIB technique not only offers the fabrication of nanostructures by the removal of material from the solid surface (milling) or their deposition or surface modifications but also at the same time has the capability to provide their topographical analysis. The dual-beam FIB or more advanced FIB instruments combined with the scanning electron microscope (SEM) and other imaging technologies are recognized currently as the most powerful tools for fabrication and characterization of nanostructured materials.

 The FIB technique was introduced in the 1970 and the early 1980s by the invention of liquid metal ion source (LMIS), but the first commercial instruments were introduced more than two decades ago $[1]$. The FIBs initially have been mostly used in the semiconductor industry for repairing lithographic masks, correction and failure control of electronic circuits, and debugging integrated circuits (ICs) with increasing circuit density and decreasing feature dimension [2]. Since the 1990s an enormous growth in nanoscience and nanotechnology took place and the FIB started to be a very demanding technology for rapid prototyping of micro- and nanoscale structures and development of new areas of applications including microelectromechanical and bio-microelectromechanical systems (MEMS and bio-MEMS) for fabrication of micro-chips, microfluidic devices, miniaturized sensors, biosensors, photonic devices, scanning probe microscope (SPM) tips, magnetic head trimming and other micro-tools $[3, 4]$. The FIB showed to have many advantages for these applications over electron beam lithography, as it is a mask-free process, has the capability of very fine focusing with choice of a wide variety of ion masses and various ion species, and provides shorter penetration depth in solid. By controlling FIBs, it is possible to achieve an improved reproducibility and precision in fabrication of nanostructures with capability to create very complicated 3-D structures [5, [6 \]](#page-18-0). Ion beam is produced by the high-energy ionized atoms with a relatively high mass of atoms and directed to the sample surface with the help of lenses and apertures provide high resolution in nanofabrication. In general FIB has four types of functionality including milling, deposition imaging, and implantation with highenergy ions $[7, 8]$. The collision of ion beam on sample surface produces the elastic and inelastic collision, and dual beam uses both of them for milling and imaging. Apart from the fabrication by milling FIB is used for the formation of nanostructures by the metal deposition with the help of gases from metal sources (LMIS) including the ion $(Ga⁺)$ directed as a beam with high electric field $[9-11]$.

 The development of FIB technology is a very active research area over the past years to improve the performance of FIB instruments and extend their applications into new research areas and industry. Even though the FIB is an expensive instrument it is today widely used in laboratories for material research and development, physical and chemical analysis, biological research sectors, and manufacturing companies. While the mainstream of FIB usage still remains within the

 Fig. 1.1 Schematic illustration of a dual-beam FIB–SEM instrument. Expanded view shows the electron and ion beam sample interaction [13]

semiconductor industry and nanofabrication, the FIB usage has significantly expanded for applications in metallurgy, earth science, ceramics, polymers, geology, art, biology, pharmaceuticals, and forensics $[12]$. The FIB provides huge advantages to the preparation of specimens for microscopy analysis including SEM, transmission electron spectroscopy (TEM), and other characterization techniques.

In this chapter we firstly show the basic introduction of dual-beam system to understand the basic principle of FIB and difference and advantages compared with SEM. Then, basic operation modes of FIB followed with description of instrument with more details are discussed. The review has emphasis on FIB fabrication of nanostructures by milling and deposition methods with particular focus on fabrication of nanopores and nanopore arrays. Finally, recent developments of applications of FIB in different areas of material science and biological applications are briefly reviewed.

1.2 Principle of FIB System

 The basic components of an FIB system consist of an ion source, ion optics, a substrate stage, and a vacuum chamber with auxiliary equipment (Fig. 1.1) [13]. An FIB instrument looks and operates as an SEM as both instruments are based on a focused electron beam for image and an ion beam for the FIB fabrications.

In the FIB secondary ions can be detected and used to construct image of the sample with magnifications up to 100,000 times. The operating ion beam in FIB is based on LMIS of gallium $(Ga⁺)$ positioned in contact with a sharp tungsten (W) needle. The Ga^+ wets up the needle and flows to the W tip. A high extraction field (>10⁸ V/cm) is used to pull the liquid Ga^+ into a sharp cone of up to 5–10 nm radius as it is shown in Fig. 1.1. The use of $Ga⁺$ has its advantages: (1) $Ga⁺$ has a low melting point, so it almost exists in the liquid state near room temperature, and (2) $Ga⁺$ can be focused to a very fine probe size <10 nm in diameter. The FIB generally operates with an accelerating voltage between 5 and 50 keV. By a fine controlling of the strength of the electrostatic lenses and adjusting of the effective aperture sizes, the ion probe current density may be altered from tens of pA up to several nA. The ions of beam collide with the surface atoms of a solid sample, and they lose energy to the surface electrons. The important physical effects of collision of ions on the substrate are as follows: sputtering of neutral and ionized substrate atoms which lead to the substrate milling, and the electron which is emitted out as secondary electron provides the imaging of surface and also makes sample charged. In the presence of gases chemical interactions between the surface and breaking of their chemical bonds help in the deposition process of ion milling $[13]$.

1.2.1 Topographical Imaging by FIB

The finely FIB is used to scan over a substrate, and secondary electrons are generated from the surface of the sample and used for imaging purpose (Fig. $1.2a$) [14]. The electrons or the ions are collected on a biased detector for secondary electrons. The detector bias is a positive or a negative voltage with respect to the sample for collecting secondary electrons or ions. These secondary ions can be used for secondary ion mass spectroscopy (SIMS) of the target material in a mass spectrometer attached to the system. During the FIB operations, a small amount of $Ga⁺$ ions collide on the sample surface and large numbers of secondary electrons leave the sample. The system also prevents the excess of electron collecting when it is used only for imaging due to electrostatic discharge, and it enables the reliable imaging of nonconducting materials such as glass which is largely used in microsystems.

 The resolution of FIB images depends on the ion beam spot size, i.e., below 10 nm. In crystalline materials such as aluminum and copper, the ion penetration depth varies due to channeling along open columns in the lattice structure of the metals and its oxides. The rate of secondary electron emission depends on the penetration depth of ions. It is worth noting that imaging with FIB inevitably induces some damage to the sample. Most of the $Ga⁺$ ions that arrive at the sample surface enter the sample; thus, the ion implantation takes place; besides implantation some milling also occurs when the ion beam scans over the sample surface. This type of milling effect can be reduced or avoided by using a fine ion beam with a fine spot and low ion current.

1.2.2 Milling of Solid Structures by FIB

 The removal of sample material and making of patterns and structures can be achieved by using a high-current ion beam. The result of high-current FIB is that the high-mass atom of material comes out and leaves empty space at the site as shown in schematic in Fig. [1.2b](#page-4-0). Scanning the beam over the substrate in a particular manner can achieve any type of shape or structure in surface. The sputtering yield is generally dependent on the angle of ion beam incidence; it increases with $1/cos(\theta)$, where *θ* is the angle between the sample surface and the ion beam direction. At the end of the milling process an insulator will appear dark in the SEM image than a conductor because conductor loses secondary electron. The resolution of the milling process is a few tens of nanometers, and the maximum aspect ratio of the milled holes is up to 10–20 nm. To speed up the milling process or increase the selectivity of beam towards different materials, an etching gas can be introduced into the work chamber during milling. It will increase the etching rate and the selectivity towards different materials by chemical removal of reaction products. This technique is called gasassisted etching (GAE).

1.2.3 Material Deposition by FIB

 FIB has the capability of localized mask-free deposition of both metal and insulator materials. It works on the principle of chemical vapor deposition (CVD), and it is similar as laser-induced CVD $[15, 16]$. The only difference is the better resolution with FIB but with lower deposition rate. The metals, which generally used for deposition on sample surface is platinum (Pt), but in the case of tungsten (W) deposition organometallic precursor gas $(W(CO)₆)$ can also be used. The deposition of insulating material requires specific precursors; for example, silica $(SiO₂)$ deposition requires gases such as 1,3,5,7-tetramethyl-cyclotetrasiloxane (TMCTS), oxygen $(O₂)$ or water vapour are required. The deposition process is shown in schematic in Fig. $1.2c$. The precursor gases flow on the surface of sample with the help of gas jet, and it gets adsorbed on surface molecules. When the ion beam hits the sample surface it decomposes the adsorbed precursor gases. The volatile reaction products come out from the surface and are removed through the high-vacuum system, and the desired reaction products (W or $SiO₂$) remain on the sample surface as a thin film. In general the deposited material is not very pure because some organic contaminants as well as $Ga⁺$ ions of ion beam are also present. The deposition has a bitmapped shape instead of a simple rectangle shape. The smallest features that can be deposited are of the order of 100 nm (lateral dimension) and up to 10 nm thicknesses [14].

1.3 The FIB Instrument Description

 A schematic diagram of a FIB/SEM dual-column instrument is shown in Fig. [1.3](#page-6-0) [[17 \]](#page-18-0). The instrument consists of a high-vacuum system, a chamber with sample stage, LMIS, ion column, gas jet, and a computer to run the complete instrument. The configuration is almost similar to the SEM, and the only difference is the use of a

gallium ion $(Ga⁺)$ beam instead of electron beam. The ion beam is generated from LMIS by the application of a strong electric field. This electric field causes the emission of positively charged ions from a liquid gallium cone. The operating extraction current is 2 μA, which is formed on the tip of a tungsten needle and has an extraction voltage of 7,000 V. The ion beam energy is typically between 10 and 50 keV, with beam currents varying between 1 pA and 10 nA. Using the variable aperture mechanism, the beam current can be varied, allowing fine beam for high-resolution imaging on sensitive samples and a heavy beam for fast and rough milling.

 The sample is mounted on a motorized stage, inside the chamber. The chamber is divided into two parts with the valve: one is beam line chamber, and the other one is working chamber for sample loading and unloading. Under normal operating conditions, inside this chamber a high vacuum up to 10^{-7} m bar range is maintained. In order to preserve this vacuum the valve gets closed while loading or unloading the sample. A system of high vacuum is needed to maintain the vacuum inside the column and the work chamber where a rotary pump is used in combination with a turbo pump. The ion column is separated with one pump. Most of the FIB systems are available for delivering a variety of gases in working stage to help milling to the sample surface. To provide gas for working chamber a gas cabinet containing all applicable gases is present outside the vacuum chamber. The gas containers are connected to a gas jet nozzle assembled inside the working chamber through an

Scheme 1.1 Classification of FIB parameters important for performances of FIB system

appropriate piping system. The gases are used for faster and more selective etching as well as for the deposition of materials [\[17](#page-18-0)]. All operations in FIB instrument such as loading of samples (partly), adjustment of sample stage, manipulation of the ion beam and beam energy, and control of the vacuum system and gas jets for gas delivery are controlled with the computer and software. Indeed, the complete user interface is run by means of a computer workstation. There are a number of FIB parameters which influence performance and efficiency of FIB system which are summarized in Scheme 1.1. The influence of these parameters using different FIB modes and applications was investigated by many authors and presented in a recent review [18].

1.4 Fabrication of Nanostructures by FIB and Their Application

1.4.1 Nanopatterning and Nanolithography

 The FIB technology is widely used in the nanopatterning or mask-free lithography providing many advantages compared with conventional and electron beam

 Fig. 1.4 Examples of patterned nanostructures fabricated by FIB milling and deposition: (**a**) Aligned 2-D structures used as a diffractive optical element of a fast parallel processing spectrometer were milled into Si with a 21 nA FIB beam which was [19]. (**b**) 2-D periodic pattern of closed nanoholes directly written on wave guide slab by the FIB milling. The cross section of the small pattern shows the 600 nm deepness [20]. (c) SEM images of square patterns fabricated by milling into a 30 nm permalloy thin film by the 30 keV Ga⁺ ion beam perpendicularly on substrate. (**b**) Spin-valve nanowire of 200 nm width and 16 μm length with FIB-deposited platinum electric contacts used for the study of domain wall motion [19]

lithography. A mask-less lithography is a one-step process based on direct ion beam writing, i.e., transferring a pattern by direct printing of the FIB onto the sample. The technique can be used for any type of materials including silicon, metal oxides, metals, polymers, and soft and hard biological samples. Most of the FIB applications have aimed to achieve the smallest possible sizes for pattern elemental structures. FIBs with high beam current, small spot size and high accuracy in patterning open the opportunities to expand FIB prototyping to larger area and complex structure. The dual-beam FIB instrument has control not only on the lateral dimensions but also on the depth of each individual pattern element independently. These 2-D patterns on the surface can be fabricated using both milling and deposition mode depending on the required application.

 Figure [1.4a](#page-8-0) shows an example of FIB machining for fabrication of a diffractive optical element for UV–Vis spectrometer. The 2-dimensional trenches with length of 125 mm and width of 2.5 mm are patterned in area of 1×0.5 mm² and arranged in groups that differ in pattern depth of 35–3.5 mm in step of 35 nm. These trenches were patterned with a 21 nA current beam, which usually takes about 7 h to write [19]. In another example a pattern of nanohole arrays was made with the 10 pA beam current, and cross section is milled at a particular region to check the correct depth of the holes (Fig. 1.4_b). These structures were used on wave guide slab for photonic applications $[20]$. The FIB is also used as a useful tool for the fabrication of 2-D patterns with the implantation of the ions in silicon nitride for various types of semiconductor device fabrication which is shown in Fig. [1.4c](#page-8-0) . Another example is fabrication of nanowires of 200 nm width and 16 μm length with FIB-deposited platinum electric contacts used for the study of domain wall motion [19].

1.4.2 FIB Nanopore Fabrications

Nanopores, defined broadly as holes or channels that range from less than 1 to 100 nm in diameter, have attracted considerable interest in recent years for molecular separations, biosensing, molecular electronics, optical devices, and nanofluidics. Solid-state in comparison with soft materials were seen more advantageous for fabrication of nanopores due to its high stability and rigidity, controllable diameter and channel length, and adjustable surface properties [\[20](#page-18-0) , [21](#page-18-0)]. Several approaches for fabrication of solid-state nanopores such as FIB, electron beam drilling, electron sculpting, reactive ion etching, and nuclear track etching have been explored in previous years $[22-25]$. However, the FIB milling has been particularly accepted as the most popular method for preparing nanopores in thin film because it provides superior resolution and the ability to perform direct patterning of pore arrays $[26, 27]$. In this case if we look towards the formation of nanopores into silicon wafer it needs to have very high control in accuracy and beam focus.

 A typical example of nanopore array electrode fabricated on oxidized silicon wafer substrates with the FIB method is shown in Fig. 1.5 [27]. To fabricate the single nanopore or nanopore array with controlled-pore diameters and inter-pore distance the sequential FIB milling is the best choice. The fabrication of single or array nanopores was particularly attractive for fabrication of DNA sensing devices and biosensors $[26]$. In Fig. [1.5](#page-10-0) shows the disk-shaped pores produced with different size by sequential milling in the silicon nitride layer. The fabrication process requires two steps including making of the pattern by the software which will work as the guide mark for the ion beam to do the milling over the surface. The process provides the flexibility to make the number of patterns and run the instrument in auto mode for long-time milling on sample. The ion beam current is the key factor to know the milling time of the pattern; if the ion beam current is high the milling time will be lower. The lower ion current is useful but has some disadvantages: long time of milling and beam displacement if it is not very stable with sample.

Fig. 1.5 Schematic of the fabrication strategy (*left*). The Pt-coated surface covered with silicon nitride is milled by focused ion beam to open up nanopores through to the underlying Pt. SEM images from a selection of arrays and single electrodes with varying diameters. Schematic (*right*) illustration of the concept of threading of the DNA/beacon complex through a nanochannel that allows optical detection of the target DNA sequence [27]

 The milling rate varies with the ion beam current, and a small beam current is always preferred to use for milling process of structures with nanoscale dimensions. It is reported that the limitation of 150 nm is the lowest pore diameter achieved for longer pores due to the high aspect ratios of the pores and the beam diameter [27]. Close examination of these nanopores fabricated by FIB method shows to have a conical rather than cylindrical shape. The formation of conical pores by FIB milling has been explained to be caused by redeposition of milled material [28, 29]. To address this problem, the use of XeF2-enhanced FIB milling is demonstrated to reduce the effects of material redeposition in the fabrication of inter-digitated nanogap nanoelectrodes $[30]$. These nanopore arrays produced by FIB were explored by many groups to mimic cell membrane nanopores and for DNA biosensing applications $[31]$.

1.4.3 FIB Combined with Anodization Process for Nanopore Array Fabrication

 In recent years, porous anodic alumina (PAA) fabricated by electrochemical anodization process with self-organized nanopores in hexagonal arrangement has created great interest due to its potential to be used for many applications including molecular separations, biosensing, energy storage, photonics, drug delivery, and template synthesis. However this fabrication process has limitations regarding pore diameters, pore inter-distance, and pore patterns and shapes. To address this problem, the FIB and FIB-based lithography is combined with anodization process. The FIB lithography on silicon has been previously demonstrated to produce

Fig. 1.6 (a-d) Schematic diagram showing the process for fabricating anodic alumina film with ordered nanochannels [\[35 \]](#page-19-0). (**e**) Alternating FIB pattern after the anodization, and the inset is the schematic of the pore shape and oxide wall shape development [37]

hexagonal patterns to guide the growth of highly ordered PAA with controlled inter-pore distances and patterns. The depths of the FIB patterns range from 5 to 20 nm for effective pore growth during anodization. With the FIB gradation in the size and shape (rectangle, square, and circle) can be easily achieved with the array of the nanopatterns [32–34]. Another approach is used by FIB to create arrays of hexagonally close-packed concaves with variable depths on polished Al surfaces and study their effects on guiding the growth of the nanopores. Figure 1.6 [35] shows the schematic of the fabrication of PAA membranes by FIB patterning process. It was found that concaves as shallow as few nanometers can effectively guide the growth of the PAA nanopores during anodization process. Therefore the fabrication of pores with different pore sizes, inter-pore distance, shapes, and patterns is possible by combining FIB and anodization method [36]. It was found that the Ga⁺ implantation in Al, during the FIB patterning, has important role in growth of nanopore in the anodization process; the pores start growing from the bottom side of the FIB-patterned concaves $[37]$. At the same time, the pores have a tendency to expand, and the neighboring walls of these pores approach each other until two alumina layers merge. Since all the nanopores develop at the same rate, the neighboring pores restrain the change of the inter-pore distance. The nanopores developed from the FIB-patterned Al grow faster and have thicker oxide walls. Despite lot of advantages in patterning with FIB for shape and size, the *z* -direction pore growth is not well controlled and it depends on the anodization conditions. If we monitor the effect of FIB patterning on the anodization process we can find that the shallow pattern created by the FIB can effectively guide the growth of the anopores during the anodization. Based on this concept the FIB is used combined with other synthetic approaches including chemical vapor, atomic layer, and electrochemical deposition to grow highly ordered nanorods, nanowires, and nanotubes in a hexagonal pattern on a substrate [38].

 Fig. 1.7 Schematic diagram of the concept of using focused ion beam (FIB) technique for fabrication of single nanopore and nanopore arrays using porous anodic alumina (PAA) as substrate. (**a** , **b**) Fabrication of PAA by anodization of aluminum. (**c**) PAA substrate with exposed bottom surface for FIB milling. (**d**) The pore opening of PAA barrier film by FIB milling. (**e**) PAA with opened pore and (**f**) PAA with single pore or nanopore arrays. SEM images of nanopore fabricated on PAA substrate by removing of barrier film using FIB milling. (**g**) Single pore 50 nm fabricated by FIB technique with (**h**) corresponding model of the barrier film removal [38]

 In addition to FIB-guided anodization method there is another approach to use FIB for fabrication of PAA with single or nanopore arrays. The method is based on the pore opening from the bottom side of PAA by FIB milling and presented in Fig. 1.7 [\[38 \]](#page-19-0). To obtain a single nanopore opening on PAA the ion beam milling is done at the central part of a single hexagonal PAA cell using a 30 keV Ga⁺ focused beam and very low beam current of 1.5 pA for a period of 5 s. A single pore with a diameter that corresponds to the diameter of an internal pore structure is obtained and presented in an SEM image in Fig. 1.7g . The pore was opened by removing the barrier oxide layer from a single PAA cell. The milling of this smallest pore opening is more delicate because very fine adjustments of position and ion beam are required to accomplish milling in exactly the middle of the PAA cell. It was demonstrated that the FIB milling can be successfully used for controlled removal of the oxide barrier film and pore opening of PAA to form a single nanopore or nanopore arrays.

1.4.4 Fabrication of Specific Micro- and Nanostructures

 FIB-based fabrication methods were successfully used in recent years for preparation of numerous morphologies and forms from different materials including metal sheets, fibers, powders, and films. The FIB is largely used for sample and tip preparation for TEM and AFM where two types of milling processes were applied: the annular milling process and the site-specific milling. One specific example is the fabrication of atom probe which is a smoothly tapered needle with a circular cross section and an end radius of 50–150 nm, Fig. [1.8](#page-13-0) [39]. In this type of milling method, the sample is mounted in a copper tube and oriented towards end on to the ion beam. An annular mask is used to mill away the edge; the outer diameter of the mask is

 Fig. 1.8 Annular milling stage of atom probe specimen preparation. The size of the annular mask and the ion current are decreased as shown during the annular milling procedure [39]

chosen to be slightly larger than the maximum diameter of the sample to avoid the generation of multiple sharp edges with the sample. It is a multiple-step process with decreasing inner and outer diameters and ion currents. To maximize the milling rate, milling should be performed concentrically from the outer diameter to the inner diameter of the mask as the milling rate is greater on an exposed edge. This procedure can be completed in about 30 min. Annular milling method is useful in correcting wedge-shaped electropolished samples or simply re-sharpening blunt or previously analyzed samples $[40, 41]$. At lower ion beam energy $2-5$ keV, it may also be used to remove contamination or surface oxide films from sample surface and to prepare tips for scanning tunneling microscope [42].

1.4.5 Fabrication of 3-D and Complex Micro- and Nanostructures

 The most attractive application of FIB technique is to fabricate complex 3-D microand nanostructures which are not possible to make by any other existing technologies [\[43](#page-19-0)]. The high density and uniform supply of reactive gases are commonly used along with FIB milling for these applications. When the sample is exposed to the ion beam and surface of the sample started etching, the deposition of material takes place with higher deposition rate than etching rate. It is also important that ion beam current should be lower for deposition and etching. The ion beam-induced CVD is introduced with the help of excited secondary electrons as well as the primary ions used in reaction. The principle is presented in Fig. [1.9](#page-14-0) [43]. The ion beams itself produce secondary electrons at the beam point, and the ion beam CVD reactions take place at lower source-gas pressure levels than the pressure which is required in electron beam CVD processes. It is also observed that $Ga⁺$ beam ions are available on the surface of the structures up to 40 nm depth. A lower current beam is caused as low lateral growth rather than a perpendicular growth, so it helps to form an

upstairs. By combining the lateral growth with the rotating beam scanning, complex 3-D structures can be fabricated.

 In addition to the low scan, the faster FIB scanning enhances both the lateral and bottom growth to produce downstairs structures. It is reported that with change in gas pressure these two growth modes share their boundary in the same space, where the upstairs growth at the bottom and the downstairs growth at the higher region take place. The secondary electrons which are produced by the ion beam by sample surface decomposition enhance the lateral growth on sample. A slight shift in beam position causes shifts in secondary electrons and its distribution, so the growth moves from its place and starts from the side of the fabricated structure. The combination of this lateral growth mode with shifting beam scanning provides the growth of 3-D structures $[45]$. At the same time with the continuous rotation in the lateral growth it is possible to achieve a rotational symmetry like a wineglass or a boll. Based on the growth mechanisms very complex 3-D nanostructures such as wineglass and coils were produced (Fig. 1.10) [44].

1.5 Emerging Applications of FIB Technology

 The main applications of FIB methods based on milling, deposition, patterning, implementation, doping, and imaging are summarized in Scheme 1.2 [18] and Table [1.1](#page-16-0). These applications are broad and include a number of areas from

Fig. 1.10 Examples of FIB-prepared three-dimensional (3-D) structures: (a) wineglass and (**b**) coil [44]

Scheme 1.2 Classification of FIB methods and their applications

| FIB methods | Applications | Reference |
|-------------------------------------|---------------------------------|--------------|
| Nanopatterning | Optoelectronics | $[46 - 48]$ |
| Preparation of TEM samples | Geosciences/mineralogy | $[49 - 52]$ |
| Metal grain boundary examination | Metal research | $[53 - 57]$ |
| Device fabrication | Semiconductors | $[58 - 61]$ |
| Imaging and sample preparation | Life science | $[62 - 66]$ |
| Sample analysis | Secondary ion mass spectrometry | [12, 67, 68] |
| Fabrication of nanopores | Nanopore biosensing | $[69 - 71]$ |
| Sample preparations | AFM, TEM sample | $[72 - 74]$ |
| Patterning and nanostructure growth | Nanowire fabrication | $[75 - 78]$ |

Table 1.1 FIB technology and its application in various fields

 Fig. 1.11 (**a**) Pollen grains shown by SE imaging with the FIB. (**b**) Cross section by the ion beam (FEI Company)

microelectronic industry, materials, life and earth science, biology, and medicine. A number of researchers working in the field of nanoscience and nanotechnology are using the FIB in their daily work as a basic fabrication or characterization tool. Some of the selected application examples in the life science are presented.

1.5.1 Application of FIB in Life Science

 The FIB for imaging, cross-sectioning, and 3-D machining is a rapidly growing application in life science and biological research. Many of the conventional methods of sample preparation used within life sciences are directly compatible with investigation by the FIB. It is difficult to examine biological material in its natural state with scanning or transmission electron microscopy, because biological samples are not compatible with high vacuum. To overcome this problem there are few techniques which can maintain the original state of the sample in such conditions. The chemical fixing, drying, and cryogenic preparation such as high-pressure freezing are generally being used for biological sample preparation. Some of these samples are robust enough to be used directly in the FIB for site-specific investigation. Specimens with hard cell walls such as plants or with an outer skeleton of insects can be applied in the FIB without any sample preparation. The imaging of pollen structure which includes external and cross-sectional imaging after FIB cross-sectioning is shown in Fig. 1.11 . A stamen was removed from flower and pollen tapped onto double-sided carbon tape loaded in the chamber, cross-sectioned, and imaged by FIB. The same method is used for many other biological samples (algae, leaves, tissues, etc.) showing the capability of this method for direct and in situ examination of internal structures of biological samples.

1.6 Conclusion

 The FIB technology has shown tremendous development in last decade with extensive spread of applications in many areas from microelectronic industry, materials, life and earth science, biology, and medicine. This was possible because the FIB technology adopted the developments from all the previous advances in related fields like field emission, charged particle optics, and applied material science. The optimization of specific gallium-based FIB instrument, together with innovative patterning and milling process, was a key progress in these developments. All these advancements make FIB currently the most powerful tool for characterization and fabrication in modern laboratories and for small-scale post-processing or prototype fabrications. The nanofabrication by FIB has been mainly based on the functions of milling, depositing, implanting, and imaging. Specially combination of FIB and CVD has recently become to be attractive for fabrication of complex 3-D microand nanoscale structures. The FIB technique is now well established for the diverse nanotechnological applications especially with the aim to explore "bottom-up" approach and use for fabrication of advanced materials and devices. The technique is of major interest for future applications to nano-electronics, nanomagnetism, spin-electronics, nano-optics, and biosensing. However, still there are some obstacles to address the limitations of physical and chemical phenomena in having structural stability, especially in milling mode due to its inherent error and material redeposition. The FIB technique, as one of the most powerful nanofabrication technologies, is expected to lead to numerous new breakthroughs and applications in nanotechnology research in the coming decade.

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