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Focused Ion Beams (FIB) – Novel Methodologies and Recent Applications for Multidisciplinary Sciences

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Additional information is available at the end of the chapter

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Abstract

Considered as the newest field of electron microscopy, focused ion beam (FIB) technologies are used in many fields of science for site-specific analysis, imaging, milling, deposition, micromachining, and manipulation. Dual-beam platforms, combining a high-resolution scanning electron microscope (HR-SEM) and an FIB column, additionally equipped with precursor-based gas injection systems (GIS), micromanipulators, and chemical analysis tools (such as energy-dispersive spectra (EDS) or wavelength-dispersive spectra (WDS)), serve as multifunctional tools for direct lithography in terms of nano-machining and nano-prototyping, while advanced specimen preparation for transmission electron microscopy (TEM) can practically be carried out with ultrahigh precision. Especially, when hard materials and material systems with hard substrates are concerned, FIB is the only technique for site-specific micro- and nanostructuring. Moreover, FIB sectioning and sampling techniques are frequently used for revealing the structural and morphological distribution of material systems with three-dimensional (3D) network at micro-/nanoscale. This book chapter includes many examples on conventional and novel processes of FIB technologies, ranging from analysis of semiconductors to electron tomography-based imaging of hard materials such as nanoporous ceramics and composites. In addition, recent studies concerning the active use of dual-beam platforms are mentioned.

Keywords: Focused Ion Beams, Electron Microscopy, Dual-Beam Platforms, Nanostructuring, Nanoanalysis

1. Introduction

The miniaturization of novel materials, structures, and systems down to the atomic scale has assigned *electron microscopy*, a complementary branch of *nanotechnology*, for multidisciplinary sciences. In particular, transmission electron microscopy (TEM), scanning electron microscopy

(SEM), focused ion beams (FIB), and atomic force microscopy (AFM) can be considered as the most comprehensive techniques for advanced and precise analysis on different material species. Among all, *focused ion beam* microscopes are becoming more popular due to their versatility and configurational flexibility, as numerous tasks can practically be carried out with a single tool. Currently, many cutting-edge integrated microscope systems allow for nanosurgery applications inside a pressurized microscope chamber using in situ applications by means of using the capabilities of related tools and attachments that are coupled to related equipment. The best candidate is the dual-beam (also called two-beam or multi-beam) platforms.

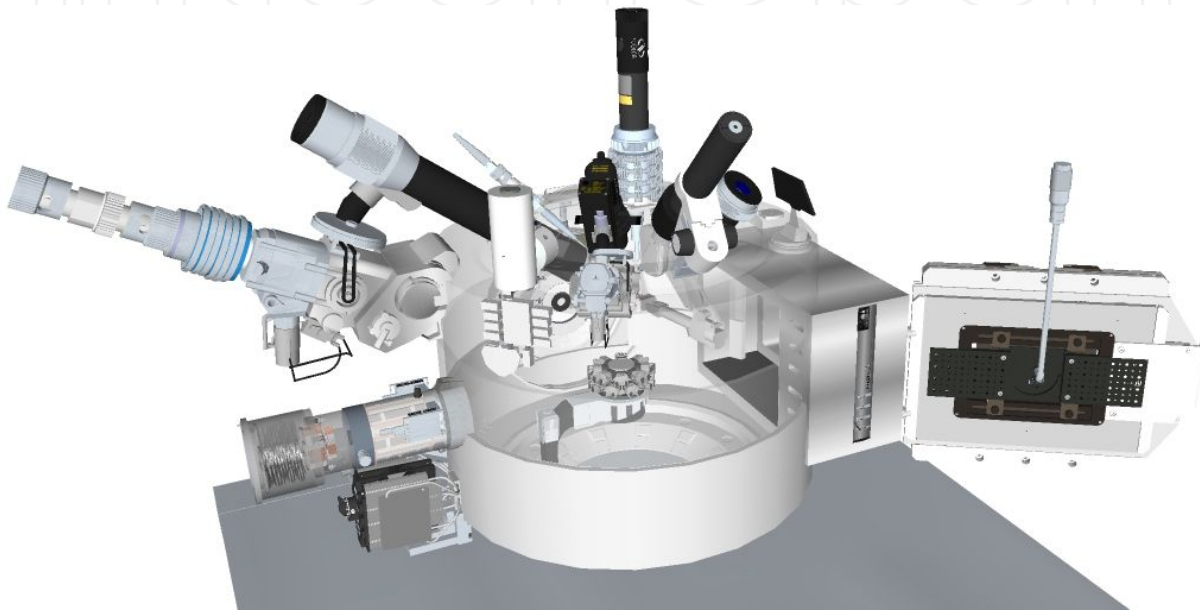


Figure 1. Illustration of a dual-beam platform with several attachments

Dual-beam platforms, consisting of an HR-SEM and an FIB column and additionally equipped with gas injection systems (GIS), micromanipulators, and detectors, serve as multifunctional tools for direct lithography in terms of nano-machining and nano-prototyping. A commercial dual-beam platform is shown in Figure 1. In the dual-beam platforms, in addition to imaging and chemical analysis of matter at ultrahigh resolution, many more processes can successfully be carried out from the micro- down to nanoscale, such as fabrication, structuring, deposition, prototyping, machining, 3D sculpturing, and manipulation. Using electron and ion beams in an integrated dual-beam platform, a wide variety of applications can be performed for multidisciplinary fields of nanotechnology: from material sciences and semiconductor technologies to biosciences. Whereas the initial development of focused ion beam (FIB) instruments was driven by their unique capabilities for computer chip repair and circuit modification in semiconductor technology, present FIB applications support a much broader range of scientific and technological disciplines [1,2]. The versatility and multifunctionality of these platforms give users inspiration and *enthusiasm* for developing new methodologies of nanostructuring and nanoanalysis upon the needs and the properties of diverse materials and systems.

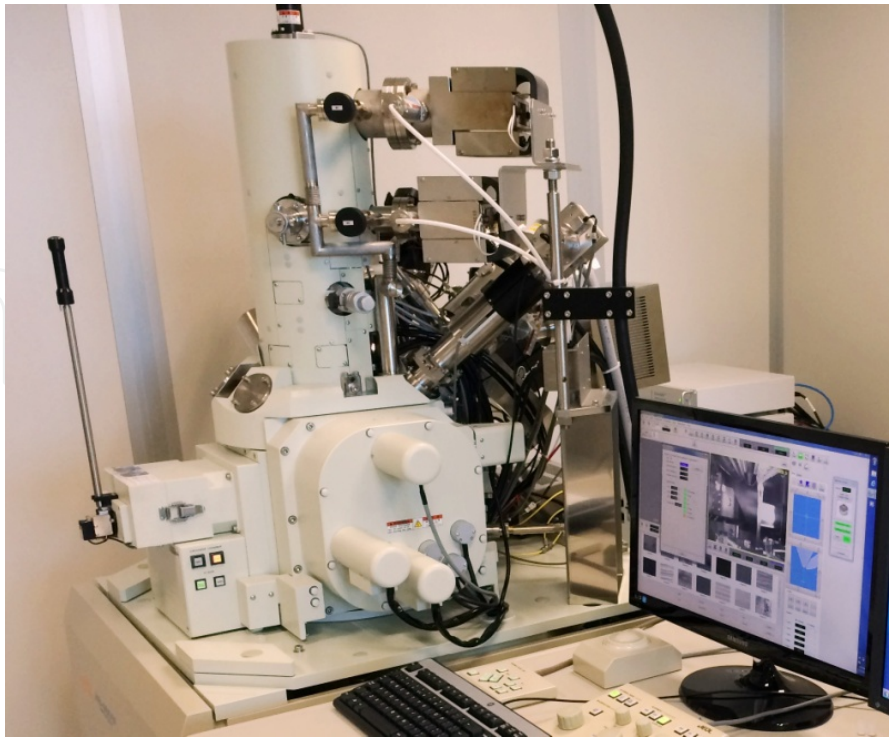


Figure 2. A commercial FIB/SEM workstation equipped with GIS, micromanipulator, and EDS detector (JEOL JIB 4601F MultiBeam System at Sabanci University Nanotechnology Research and Application Center-SUNUM)

Fundamentally, an FIB column produces and directs a stream of high-energy ionized atoms of a relatively massive element, focusing them onto the sample both for the purpose of etching or milling the surface and as a method of imaging. The ions' greater mass allows them to easily expel surface atoms from their positions and produces secondary electrons (SE) from the surface, allowing the ion beam to image the sample before, during, and after the lithography process. The ion beam has a number of other uses as well, including the deposition of material from a gaseous layer above the sample [3]. Most of the recent commercial FIB systems and dual-beam platforms use gallium (Ga^+) as the ion source. The reason for preferring gallium element as the ion source is explained in the subsequent sections.

Concerning micro- and nano-sectioning processes, rapid FIB slicing techniques are frequently preferred for especially hard materials (e.g., metals, glass, ceramics) or layered structures (e.g., semiconductors) with hard substrates (silicon, glass, ceramics, etc.) because corresponding materials cannot be mechanically sliced by cutting tools. It has been always reported that ion milling is not a proper method for soft materials due to their sensitivity to beam damage and heat-dependent shape distortion. Therefore, for soft materials, ultramicrotomy has so far been accepted as the most convenient preparation technique, which is a mechanical serial sectioning process that uses diamond knives or blades for cutting cross sections from the materials that were embedded in epoxy resin. However, currently, due to the progress in the FIB technologies and due to advanced procedures and novel recipes developed by experienced users, some processes can also be allowed for soft materials as FIB applications still have many advantages over mechanical sectioning methods. Most of the newest procedures include the application

of low voltage, low current, and gentle instrumental parameters, and bringing the microscope chamber to low temperatures and to even cryogenic conditions might also be necessary for specific experimental studies particularly when soft cells and organic materials are to be examined and structured.

FIB is recently considered to be the best technique for site-specific TEM specimen preparation. For an efficient TEM tomography analysis, the specimen has to be precisely structured so that it represents all its properties originally in 3D at the nanometer scale. Such samples can be successfully structured using the capabilities of FIB-SEM systems, e.g., ion milling, micro-/nanostructuring, deposition, manipulation, and polishing. Another application of dual-beam instruments is the 3D FIB tomography, where the material is cross-sectioned by ion milling sequentially, and from each sliced surface, an electron image (e.g., SE) or elemental information by energy-dispersive spectra (EDS) is acquired. Consequently, the collected serial two-dimensional (2D) data can be stacked and reconstructed to form ultimate comprehensive 3D data. Using this procedure, different material systems can be characterized for their morphological and elemental distributions in 3D.

Three-dimensional electron tomography is extensively used for revealing the structural and morphological distribution of material systems with 3D network at micro-/nanoscale. Accordingly, specific sample preparation techniques that keep the original structures to be investigated at the TEM are often required. FIB technologies, which are gaining high impact for their applications on diverse fields of science, provide material-dependent and TEM analysis spectrum-based solutions that are highly rapid, practical, creative, and reliable. The main approach is to overcome the limitations and difficulties (e.g., to be able to acquire only 2D data instead of 3D, projection problem that occurs at TEMs), which can be faced during electron microscopy-based characterization. In order to investigate the structural properties of the related materials in detail, practical, rapid, and reliable electron microscopy techniques and characterization methodologies have to be determined. This is very supportive in the direction of improving the quality of the production and the final product. TEM tomography enables to image the samples for different angles of tilting, which can be performed by 2D tilt series, and consequently, 3D reconstruction and segmentation can be carried out. Eventually, the resolving power of electron tomography (3D atomic resolution achieved) can be accompanied with analytical possibilities such as electron energy loss spectroscopy (EELS) and EDS. For performing TEM tomography work, special FIB samples (e.g., pin-like) that enable tilt series without projection problems are also needed to be prepared by experts.

Nevertheless, *irradiation damage*, caused by the use of beams in the electron microscopes, which leads to undesired physical/chemical material property changes or uncontrollable modification of structures that are being processed, should not be underestimated. Especially, soft matter such as polymers or biological materials is highly susceptible and very much prone to react on electron/ion beam irradiation. The effect is even higher when the ions are used as incident particles, and the end effect might even be the total loss of the material properties. The reason for that is, focused ions (in case of FIB, this is usually Ga^+) are energetic species with a high momentum and relatively low mean path, due to their mass. Therefore, they strongly affect surface composition, leading to extensive chemical modification and sometimes

resulting in graphitization. Nonetheless, it is well possible to turn degradation-dependent physical/chemical changes from negative to positive use when materials are intentionally exposed to beams. Especially, controllable surface modification allows tuning of surface properties in intended directions and thus provides the use of the ultimate materials and their systems toward desired and predefined concepts. Moreover, FIB is capable of performing maskless site-specific structuring, which are considered to be the major advantages of FIB over e-beam lithography. In particular, surface modification processes can be carried out through gas-assisted etching (GAE) in an FIB-SEM dual-beam instrument equipped with gas injections systems (GIS).

In the following sections, fundamentals of dual-beam technologies are revealed and examples of conventional and novel dual-beam applications and processes as well as structuring and modification of structures and surfaces are explained in detail.

2. Fundamentals of FIB technologies

2.1. Focused ion beams and FIB/SEM platforms

FIB systems are very similar to SEM, while the only difference is the use of an ion beam for scanning the sample surfaces, instead of an electron beam. In the FIB systems, a focused beam of metal ions is generated by a liquid metal ion source (LMIS). The LMIS is able to provide a source of ions of ≈ 5 nm in diameter, and a typical LMIS contains a tungsten (W) needle attached to a reservoir that holds the metal source material. There are several metallic elements or alloy sources that can be used in LMIS [4]. Among all, gallium (Ga^+) is commonly preferred in commercial FIB instruments owing to its advantages that are summarized as follows [2]:

- i. Gallium has low melting point ($T_m = 29.8$ °C), minimizing reaction or inter-diffusion probabilities between the liquid and the tungsten needle, from where the ions are emitted.
- ii. The low volatility of gallium at the melting point protects the supply of metal, and this yields a long source life.
- iii. The low surface free energy leads to viscous behavior of the liquid on the substrate.
- iv. The low vapor pressure allows gallium to be used in its pure form rather than in the form of an alloy source. This yields a long lifetime of the source as the liquid will not evaporate.
- v. Gallium has excellent mechanical, electrical, and vacuum properties.
- vi. Gallium's emission characteristics provide high angular intensity with a small energy spread.

Once the Ga^+ ions are extracted from the LMIS, they are accelerated down to the ion column up to 30 keV and subsequently focused onto the sample using electrostatic lenses. The ion column typically has two lenses, a *condenser lens* and an *objective lens*. The condenser lens is the

probe-forming lens and the objective lens is used to focus the beam of ions at the sample surface. Beam currents from a few picoamperes up to 60 nA can be obtained.

An optimum ion probe can be achieved by adjusting apertures, tuning all lenses, and doing final settings of the beam, such as stigmator and focus corrections. Cylindrical octopole lenses placed in an FIB system have multiple uses, which include beam deflection, alignment, and stigmation correction [2]. It should be reminded that, in both SEM and TEM systems, *magnetic lenses* are used for focusing the beam. Because ions are massive and they travel at much lower velocities, their *Lorentz force* is lower and magnetic lenses are less effective on ions than they would be on electrons at the same accelerating voltages. As a result, FIB columns are equipped with electrostatic lenses rather than magnetic lenses.

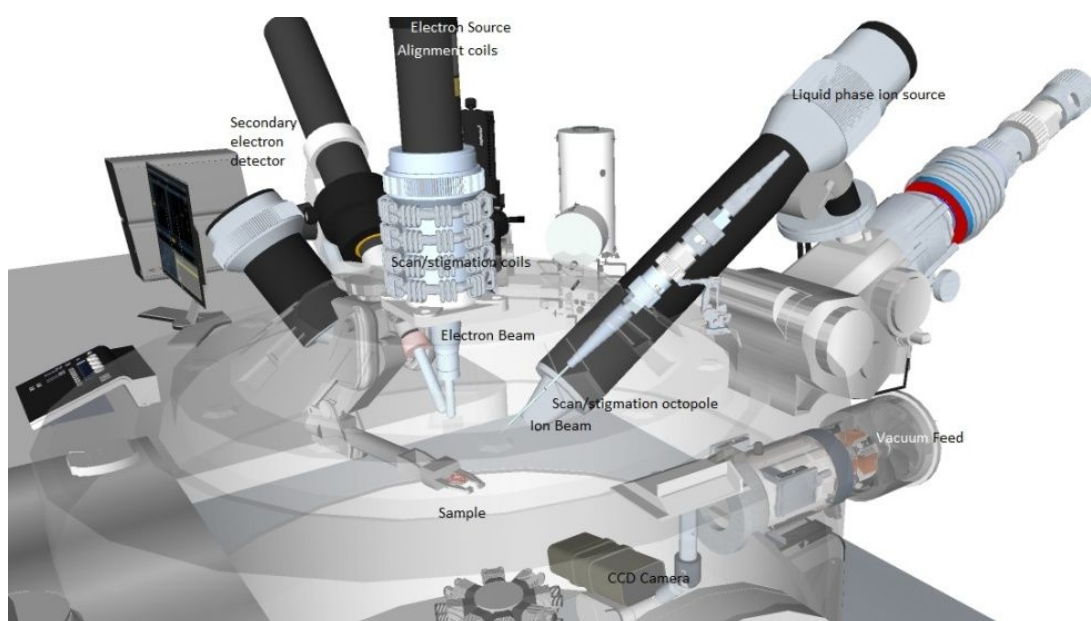


Figure 3. Construction of a dual-beam chamber

Commercial dual-beam platforms incorporate both a FIB column and a SEM column in a single system. This combination is especially useful for sample preparation of cross-sections using the electron beam to view this cross-sectional face as the ion beam mills normal to the sample surface. This monitoring allows the milling to be stopped precisely when the task is completed. The typical dual-beam column configuration is a vertical electron column with a tilted ion column. In dual-beam platforms, the tilt angle of the FIB column can vary from manufacturer to the model, but usually it is between 52° and 55° tilt to the vertical.

Both SEM and FIB can be used to acquire high-resolution images by collecting the secondary electrons (SE) that are emitted from the interactions between the beam and the surface atoms, although backscattered electrons (BSE) and/or secondary ions (SI) can contribute to form images. For secondary electron detection, Everhart–Thornley electron multiplier detector is the most common design used recently [3]. The main difference between scanning and/or transmission electron microscopy and focused ion beams is the use of ions as the beam that is

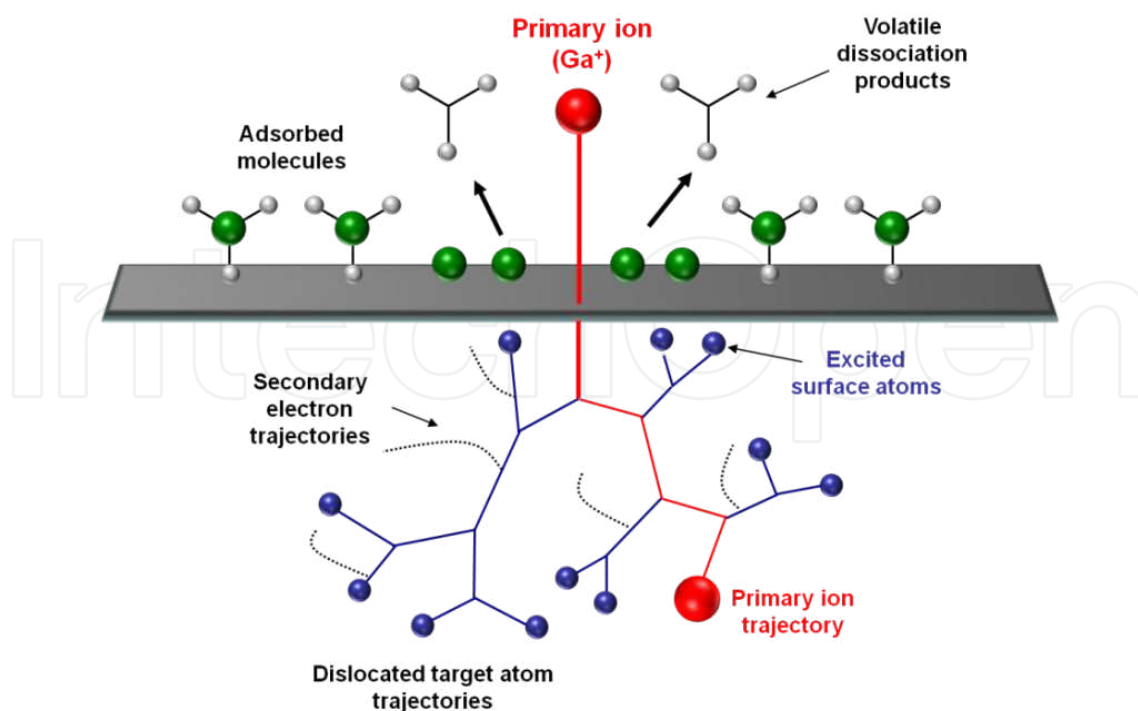


Figure 4. The interactions of primary ions generating secondary electrons and the collision cascade. The non-sputtered target atoms remain as excited surface atoms contributing to the molecule dissociation (modified from [5])

also responsible for many interactions occurring at the sample surface. Because ions are much larger in size than electrons, they are not able to penetrate within specimens' individual atoms, and outer shell interaction results in atomic ionization and breaking of chemical bonds of the target material. Secondary electrons are formed and alterations in the chemical stability are generated by this means. The interactions of primary ions and the target surface are responsible for the generation of secondary electrons, while the non-sputtered target atoms remain as excited surface atoms and contribute to dissociation of molecules [5].

2.2. Beam–specimen interactions

Dual-beam platforms allow the use of electron and ion beams for several applications through particle–sample interactions and/or reactions. As already very well known by electron microscope users, electrons may adversely affect organic or inorganic samples when they are placed in an electron microscope (e.g., SEM or TEM) for different purposes and may cause temporary or permanent changes within the specimen. The main effects might be in the form of electrostatic charging, ionization damage (radiolysis), displacement damage, sputtering heating, and hydrocarbon contamination. Typically, the amount of radiation damage is proportional to the electron dose, and the extent of damage depends on the amount of energy deposited in the specimen [6].

When ions are accelerated from a source and hit on the specimen surface in an ion column, they enter the target material as they might interact with specimen in various ways within a penetration volume. This interaction, depending on the ion energy, can be in the form of

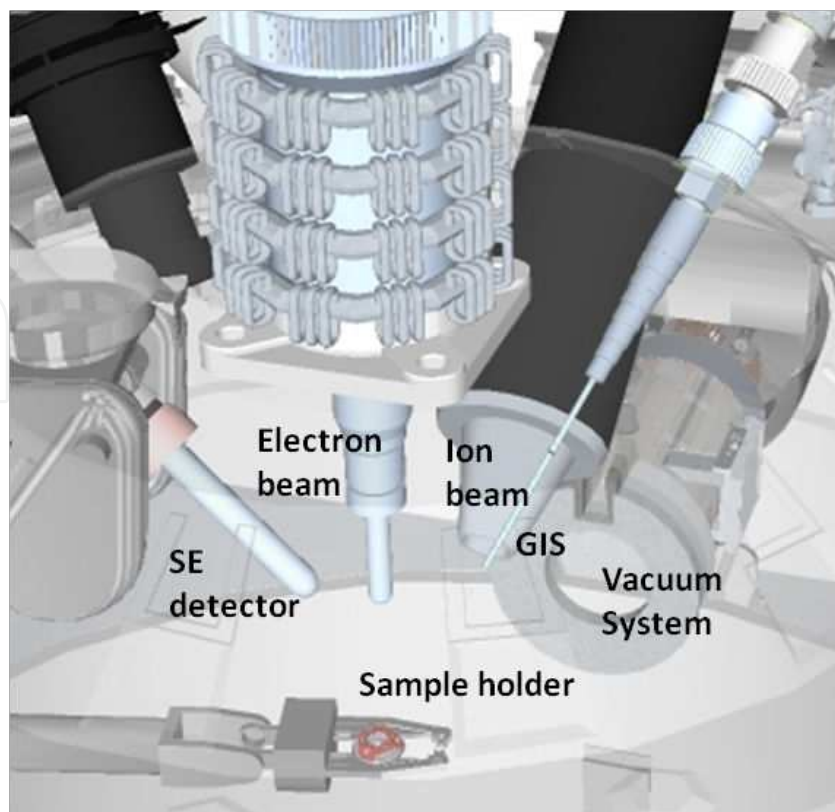


Figure 5. Illustration of the specimen chamber of a dual-beam platform

sputtering, amorphization, swelling, deposition, redeposition, implantation, backscattering, or nuclear reaction. However, many of the interactions are not completely separable and occur simultaneously, and it is often not very well understood which mechanism is dominating in the degradation process of specimens being irradiated by ions. Radiation damage not only leads to morphological changes but also alters intrinsic physical properties (crystallinity, elasticity, conductivity, electrostatic charge), as well as chemical characteristics (hydrophilicity, surface composition) of the surface.

The most dominating mechanisms for ion irradiation are discussed below:

- i. **Sputtering:** Sputtering is actually the same as ion milling process and is the major mechanism for material removal. Usually, sputtering range increases with increased ion energies, while heavier ion sources or lower surface binding energies of target materials can contribute to higher sputtering regimes.

A software package called SRIM (Stopping and Range of Ions in Matter)/TRIM (TRansport of Ions in Matter) has been widely used for predicting the range of sputtering for many different ions at a wide energy range. SRIM/TRIM uses a Monte Carlo treatment of ion–atom collisions to calculate the stopping range of ions into the matter. SRIM/TRIM calculations agree very well with the experimental data for the cases considered, and the sputter yield is dependent not only on the material but also on many processing parameters, including the ion energy, angle of incidence, and scanning procedures.

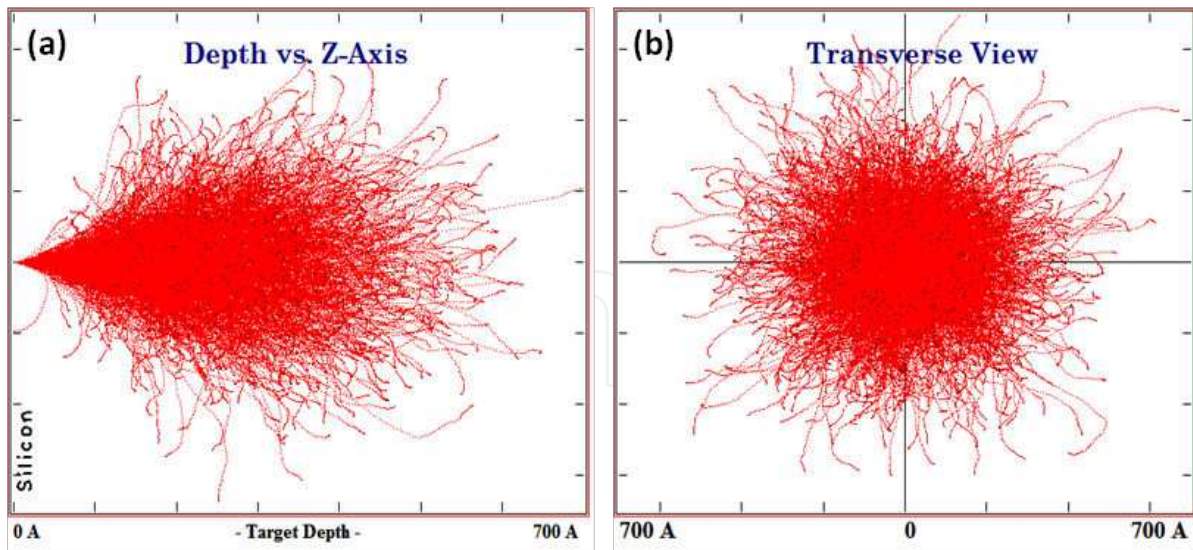


Figure 6. TRIM calculations for gallium penetration on silicon (for 30 keV ion energy, 0° angle and 5000 ions): (a) through z-axis, (b) transverse projection

- ii. **Gallium Implantation:** Sputtering mechanism may cause gallium ions to be implanted and mixed into the specimen. This might be responsible for the alteration of the specimen's local composition within the interaction volume. Gallium implantation may lead to structural changes, as well as alteration in, e.g., thermal, electrical, optical, and mechanical properties.
- iii. **Redeposition:** Sputtered particles leaving material's surface in gas phase are very prone to condense back into the solid phase upon collision with solid surfaces, as they are not thermodynamically in equilibrium. As a result, a portion of ejected atoms tend to stick back into the sputtered surfaces and redeposit. Redeposition can be minimized via using low ion doses (e.g., currents), deposition of protective layers using low ion energies, and optimizing ion milling geometries.
- iv. **Amorphization:** Amorphization of the materials that are processed with focused ions may occur in the bombarded area of a crystalline substrate and may induce the substrate to swell. This mechanism can be attributed to sufficient atom displacement within the collision cascade, resulting in the loss of crystalline orientation. Amorphization is usually faced during TEM specimen preparation and might be a serious problem for the crystalline structures to be investigated in TEM. Hence, the use of low ion energies during the polishing step of the preparation process can drastically help in minimizing amorphization effects.
- v. **Swelling:** Swelling of the target material due to ion bombardment during FIB processing is dependent on two major mechanisms: amorphization and ion implantation. Swelling is mostly attributed to material amorphization, while ion implantation does not seem to remarkably contribute to volume expansion. Distortion of the crystalline orientation to amorphous structures leads to volumetric alterations and

hence swelling of the material. Also implanting of gallium ions into the target material is found to be another reason for swelling mechanism during FIB processing.

2.3. Basic applications of FIB

2.3.1. Ion milling

Ion milling, as the fundamental application of FIB systems, is a continuous sputtering process that occurs during ion beam exposure on the sample. Milling is actually an atomic collision process that ends up in the removal of the material from the ion-sample interaction volume, mainly depending on the beam current and voltage used in the process. Ion milling can be used to create both simple structures, such as lines, rectangles, or circles in the material, and complex patterns, bitmaps, and streamline files. In addition, specific singular or serial patterns can be created by internal patterning engines or external lithography software and can be easily imported to FIB user interfaces.

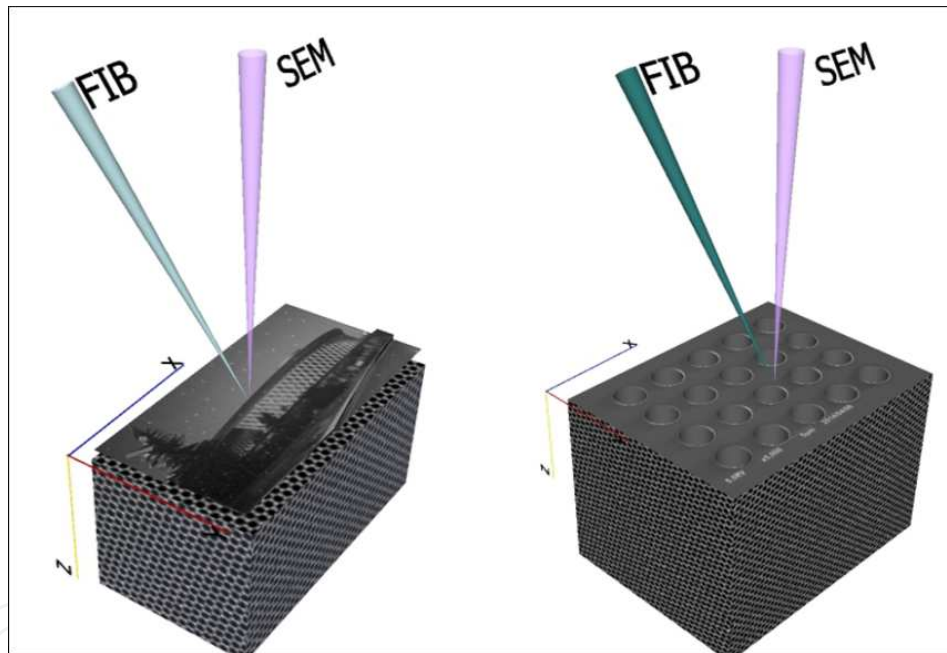


Figure 7. Representation of ion milling: maskless lithography and nanostructuring

Milling allows for creating cross sections or developing structures with desired geometries to control not only the lateral position but also local depth. Ion milling can also be described as a “direct writing technique” via etching the material surfaces with the exposure of ion beams. This process is similar to lithography; however, the advantage here is that it does not require the use of masks. Local assisting gases exposed by gas injection systems (GIS) that are integrated into dual-beam platforms can help in enhancing the removal of atoms from the material surfaces. Local gas delivery systems can change the oxidation state of released particles and speed up the milling process, while exposure of gases into the region of interest may also reduce the local redeposition of atoms released from the surface. XeF_2 and I_2 are the

most common gases that are used in FIB to enhance ion milling processes, and their supply is usually dependent on the manufacturer.

2.3.2. Deposition

Deposition is the second most powerful feature of FIB technologies, as the ion or electron beams can be used in a deposition system, allowing the addition of material instead of removing the material. Deposit materials are often supplied by an internal gas delivery system that locally exposes a chemical compound close to the surface impact point via gas injection systems (GIS) that can be incorporated into dual-beam platforms. The chemical gas compound is usually in the precursor form and consists of organometallic molecules. When this compound is exposed to the region of interest, beams decompose the molecules locally and deposit almost-pure material onto the surface. In other words, gas compound is exposed onto the target specimen and adsorbed on its surface, and this is followed by bombardment of focused beams on the adsorbed molecules within predefined patterns. Finally, as a result of complex beam-induced reactions, gas molecules dissociate into deposits and volatile fragments, while dissociated molecules are adsorbed and deposited into desired structures. This procedure is demonstrated in a scheme in Figure 8.

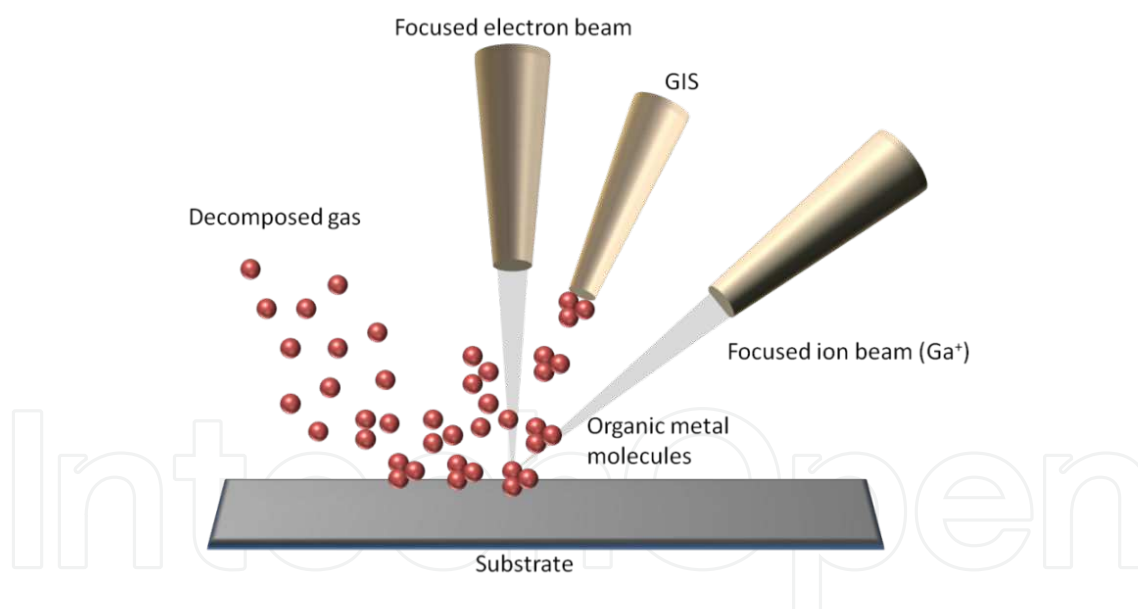


Figure 8. Illustration of deposition process in dual-beam instruments via electron beam-induced deposition (EBID) and ion beam-assisted deposition (IBAD)

Usually the decomposition of the precursor molecules is not hundred percent, and therefore some additional matrix molecules (e.g., organic residues) are also deposited together with the diverted material. For this reason, the purity of the deposits is lower when compared to other deposition techniques such as chemical vapor deposition (CVD) or physical vapor deposition (PVD). The materials used for beam-induced deposition in the dual-beam platforms are determined by their different gas chemistries, and several precursor gases are commercially

available for the deposition of Pt, W, SiO₂, and C provided by different manufacturers and suppliers. There has been already a COST Action (CM 1301 – CELINA) ongoing that aims to develop novel gas chemistries for beam-induced deposition techniques and to perform applications for multidisciplinary sciences using deposition capabilities of precursor materials.

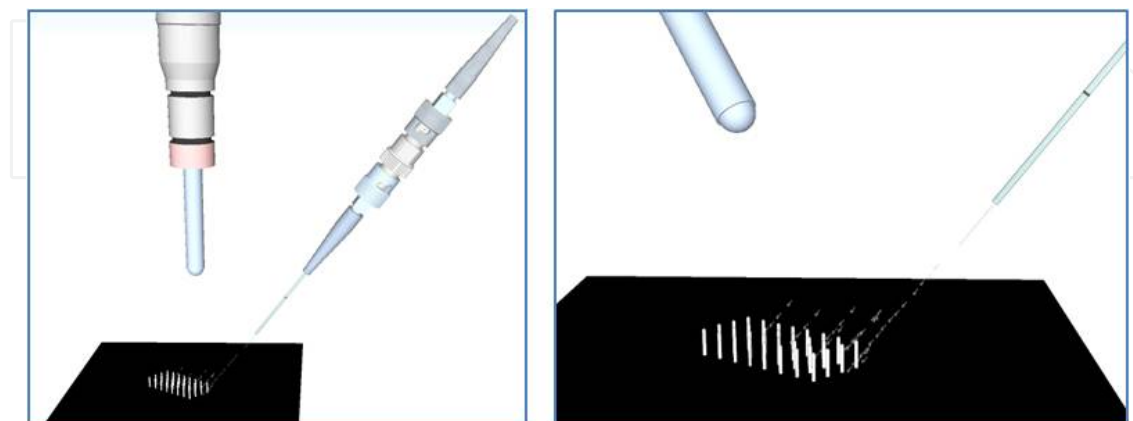


Figure 9. Illustration of gas-assisted deposition process

3. FIB-based conventional and novel processes

3.1. TEM specimen preparation

One of the most important applications of dual-beam instruments is preparing samples for transmission electron microscopy, as an important capability owing to controlled ion milling abilities is the production of ultrathin and uniform lamellae that are electron-transparent and hence can serve as TEM samples. Advantages of using an FIB for TEM specimen preparation are listed below:

- TEM lamellae can be prepared site-specifically with a spatial accuracy as fine as ≈ 30 nm from any region of interest.
- When compared to other techniques (microtomy, low-energy ion milling, dimpling, etc.), the duration for site-specific and ultrathin specimens' preparation process is considerably short, varying from less than 1 hour for noncomplex structures to 4–5 hours for challenging specimens.
- FIB allows for the preparation of TEM samples from both hard and soft materials, regardless of how brittle, ductile, or mechanically sensitive the material is. Even FIB serves for the TEM investigation of samples of life sciences studies when proper conditions of dual-beam systems are maintained (e.g., using cooling stage and cryo-conditions; low ion energy and ion current options), and the experience of the FIB user is sufficient.
- It is possible to develop new methodologies and specific geometries according to the nature and properties of the materials from which TEM specimens are to be prepared. The main

approach in special TEM sample designs is to overcome the limitations and difficulties which can be faced during TEM investigations.

The most common route for TEM specimen preparation is the “in situ lift-out technique,” for which the dual-beam instrument has to be equipped with a micromanipulator unit for allowing the transfer of the lamella to the TEM grid, when both are placed in the microscope chamber at the same time. In this procedure, initially a metal protection layer is coated on the region of interest via beam-induced deposition and two opposing trenches are milled away, leaving behind a 1–2- μm -thin section on the block sample. This section can be named as “pre-lamella.” The next step involves cutting the bottom part and the side trenches away, until the section is held by the bulk sample from its shoulders. Then, this pre-lamella can be welded to the micromanipulator using ion beam-assisted platinum deposition (IBAD) by simultaneously cutting away the shoulders, and can be lifted out from the sample, transferred, and welded to a TEM grid. Afterwards, final thinning and polishing down to a thickness of < 100 nm is achieved using low incident angles and low ion currents. Finally, the sample is ready for TEM analysis. This type of TEM specimen preparation method is demonstrated in Figure 10, giving an example of preparing a cross-sectional lamella from an organic thin film transistor.

In addition to in situ lift-out route, there are many more procedures in the literature that have been developed for more than a decade. Specimen preparation by FIB is often preferred for hard materials (e.g., metals, glass, ceramics) or layered structures (e.g., semiconductors) with hard substrates (silicon, glass, ceramics, etc.) since ion milling is not a proper method for soft materials due to their sensitivity to beam damage and heat-dependent shape distortion. For soft materials, ultramicrotomy is considered to be the most convenient preparation technique, which is a mechanical sectioning process using a diamond knife. As far as TEM investigations for organic and inorganic electronics are concerned, dual-beam instruments are effective for preparation of cross-sections from multilayer integrated devices with hard substrates as the active layer is often very thin (in nanometers) and sandwiched between supporting materials. However, it should be reminded that, for all conditions ion milling and deposition parameters have to be optimized in order to avoid potential ion irradiation damage. Hence, special care should be taken during the entire FIB-based TEM specimen preparation process for minimizing the radiation effects triggered by damage mechanisms mentioned in the previous section (2.2), such as amorphization, gallium implantation, and swelling.

Moreover, when three-dimensional investigations of materials are of interest, TEM tomography-based analysis can be performed. For this type of characterization, the specimen has to be precisely structured so that it has to be representing all its properties of the original material in 3D at the nanometer scale. Such samples can be successfully structured using the capabilities of dual-beam systems, as the details of the corresponding technique are given in the following parts of this section.

3.2. Serial slicing and imaging

Dual-beam platforms provide the use of electron and ion beams simultaneously, which opens a way to perform cross sectioning by means of sequential ion milling and monitoring and/or

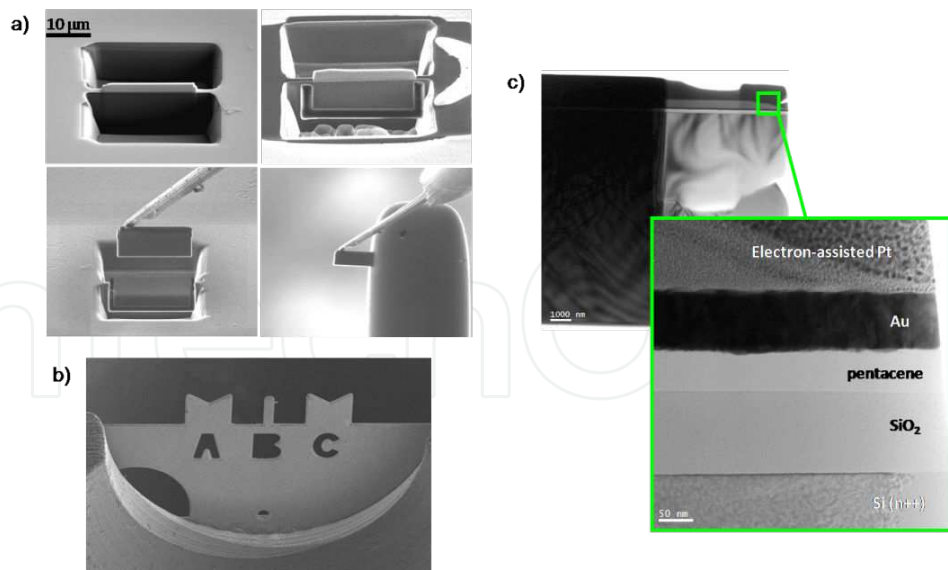


Figure 10. TEM specimen preparation of an organic thin film transistor (O-TFT) structure using a dual-beam tool: (a) coarse milling, lift-out, and mounting steps, (b) lamella on a TEM semi-grid, (c) thinned and polished cross-sections investigated by TEM in bright field (BF) mode (images taken at Felmi-Zfe, TU Graz, Austria)

acquiring images of the corresponding cross section of the specimen at the same time using the electron beam. There are mainly two different ways to collect a stack of SEM images of sectioned surfaces: static mode and dynamic mode. In dynamic image acquisition mode, SEM images are acquired in real time during the FIB milling process. In static image acquisition mode, after each slicing, the process is either paused or stopped and therefore slow-scan high-resolution SEM images are acquired.

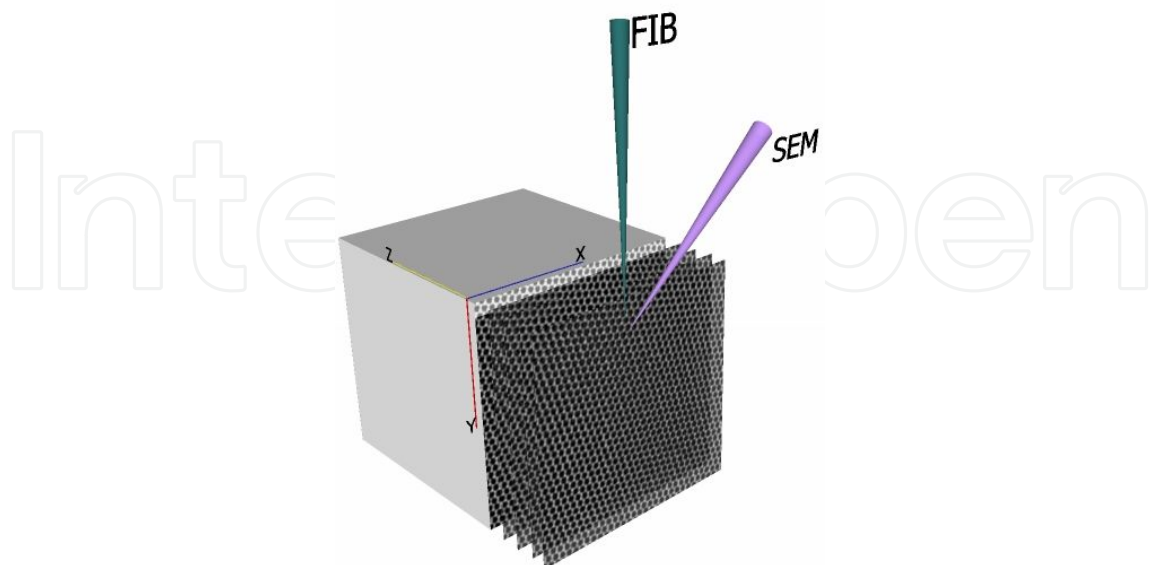


Figure 11. Illustration of serial slicing and imaging application of dual-beam platforms. Ion beam is used for creating cross sections, while electron beam allows for monitoring and imaging of the sliced regions.

In particular, the application of this process is very useful for failure analysis of semiconductor devices because it is much faster than TEM specimen preparation. Also rapid monitoring of inner structures of several materials and gaining information of the features down to a few nanometers possible with serial slicing and imaging techniques.

3.3. 3D microstructural characterization and FIB tomography

Dual-beam platform also enables three-dimensional information methodologies, especially for quantitative characterization of materials, while the measurement of a number of important geometric properties that cannot be obtained using a 2D analysis can be performed using FIB tomography methods. These are, for instance, the number of features per unit volume, feature connectivity, real feature shapes and sizes, and spatial distribution information.

One step further of serial slicing and imaging application is the 3D FIB tomography, which is based on the principle that continuous 2D data are collected from the surface of the bulk material by serial-sectioning and are stacked together to form reconstructed data, giving information in 3D. By the removal of each section, SE images, BSE images, EBSD maps, and/or EDS data can be acquired from the specimen surface, collected for 3D reconstruction and processed for 3D material characterization. Consequently, dual-beam microscopes are capable of high-fidelity characterization of the morphology, crystallography, and chemistry of micron- and submicron-sized features in 3D. The FIB slicing and HR-SEM imaging-based reconstruction showing the distribution of dentinal tubules in human tooth is shown in Figure 12.

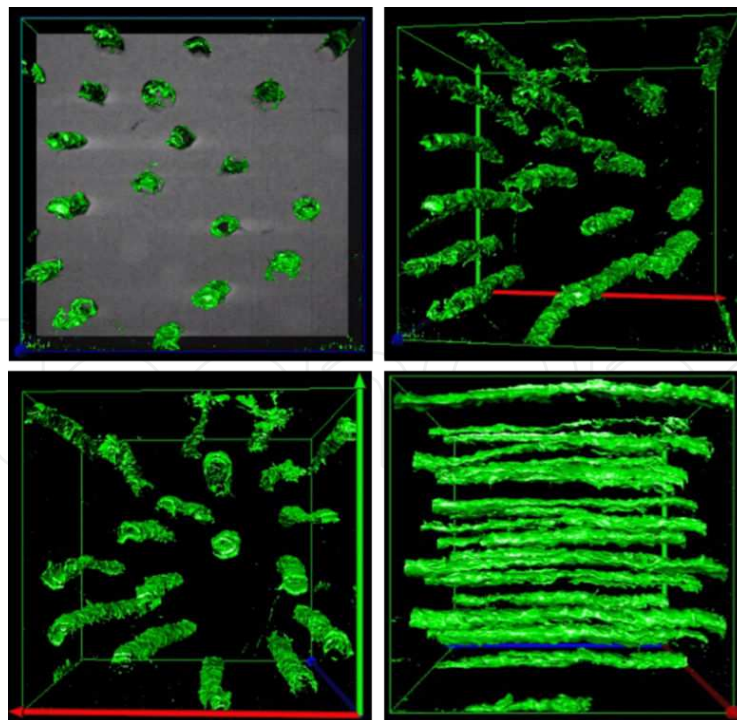


Figure 12. The 3D reconstruction of dentin showing the tubule distribution: red arrow corresponds to x -axis, while blue arrow represents the y -axis and green arrow the z -axis. For the reconstruction, Stack N-Viz software was used (reprinted by permission from [7])

3.4. Micro-/nano-fabrication, micro-/nano-modification, and other applications

In addition to applications mentioned above, structuring capabilities of dual-beam platforms in small scales fall into two major categories: one is *fabrication* and *machining*, while the other is *rapid prototyping* or *modification* abilities of structures and devices using both ion and electron beam-based processing. For the former, FIB is used for preparing structures that are difficult to form using conventional processes due to material or geometry constraints. The latter, when FIB is utilized, processes can be carried out in more practical and less time-consuming steps than conventional routes. Within user-defined patterns, it is possible to mill away lines down to 10 nm in width, and deposit materials as small as 30 nm by the breakdown of organometallic precursors. The finest ion beam spot size is approximately 5–10 nm, enabling small features to be patterned, while the shape of an FIB cut is dependent on many factors, such as its geometry, milled depth, ion beam profile, and the redeposition of sputtered material.

As already mentioned, FIB provides a very convenient technique for material removal using gallium ions. The advantage of the instrument is that the structure that is to be milled can be predefined as patterns and the process can be performed in an automated way. Milling patterns may be defined in different forms such as scripts, stream files, or image files. However, as already given in the previous section concerning platinum deposition, the quality and the efficiency of the ion milling process are dependent on the instrumental and process parameters, and those have to be optimized for the achievement of the desired structures.

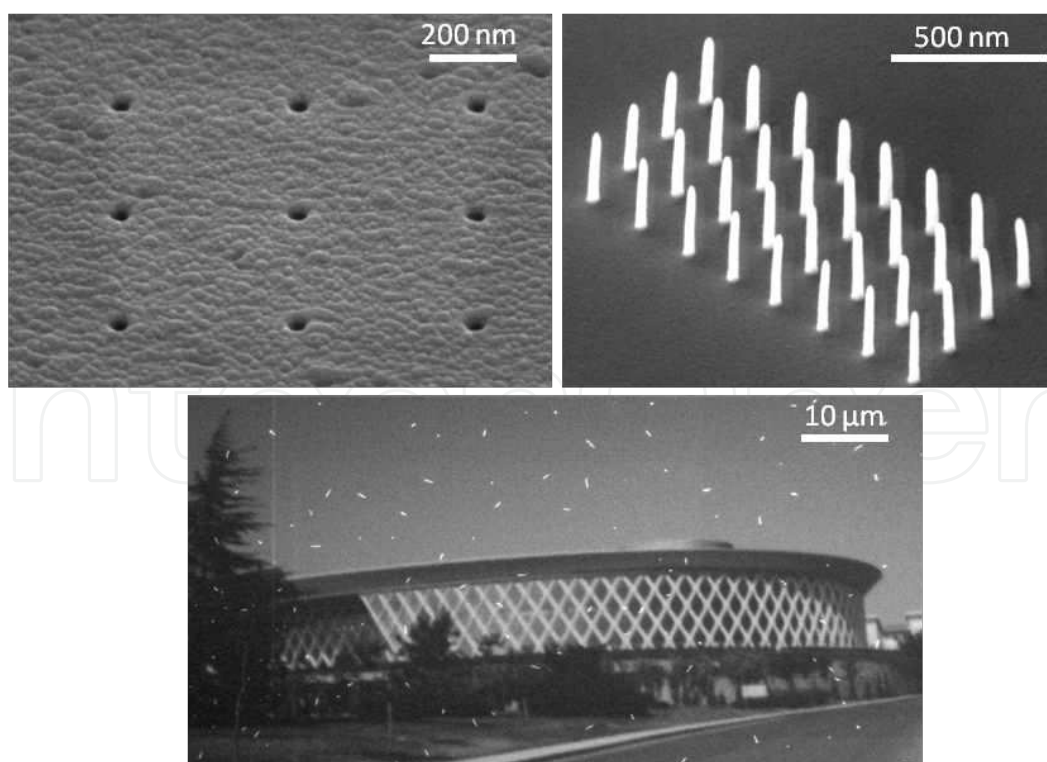


Figure 13. Nanostructuring, nano-fabrication and maskless ion lithography examples performed by dual-beam instruments

Recently, FIB technologies are becoming more popular for machining miniaturized samples to investigate the influence of sample dimensions on mechanical properties, in terms of determining size-dependent effects, particularly in metals, alloys, and ceramic composites. Sometimes dual-beam platforms enable not only the fabrication of the test structures but also the application of in situ examination in micro-/nano-size when they are coupled to proper mechanical test stages and nano-indentation devices. The most popular mechanical behavior tests that are recently in application and development include tensile strength and yield strength measurements, giving out the data for deformation in the form of stress–strain curves. The acquired data are very important to get compared to the data in macroscale, for the evaluation of size-dependent effects. Also in situ hardness tests can be performed on the coatings that include applications on several material systems used for multidisciplinary sciences and many fields of industry.

3.5. Special sample designs for TEM tomography

Electron tomography applications are recently becoming more popular for imaging of three-dimensional material systems such as alloys, composites, or samples having spatial features, such as porous network or multicomponents. Especially, in order to reveal the geometric and elemental distribution of material systems that are based on nanostructures, electron tomography applications are being widely used. For this reason, specific sample preparation techniques that keep the original structures of the sections to be investigated at the TEM are often required. Dual-beam technologies provide material-dependent and TEM analysis spectrum-based solutions that are highly rapid, practical, creative, and reliable.

The main approach for novel specimen designs is to overcome the limitations and difficulties (e.g., to be able to acquire only 2D data instead of 3D, projection problem that occurs at TEMs, etc.) that can be faced during electron microscopy-based characterization. In order to investigate the structural properties of these materials in detail, unique electron microscopy techniques and characterization methodologies have to be developed according to the needs and what is expected to be analyzed.

When TEM tomography investigation of a sample is coupled to its FIB slicing and imaging and FIB tomography work, while TEM will be able to provide 3D information at the nanometer scale and below, via FIB tomography sectioning, the information in the scale ranging from micrometers to tens of nanometers will be collected from the identical sample and the data can be comparatively and complementarily evaluated.

One of the solutions for the problems faced during TEM tilt series in tomography applications is the preparation of samples with special geometries that do not cause any projection, thickness variation, and shadowing problems during tilting. This might be a problem for lamellar samples; however, pin-like TEM structures are proper for tilting without any projection or shadowing, as well as this type of sample can represent the three-dimensional nature for the nanocomposite systems or porous materials. Especially, nanoparticles can be kicked out of the matrix during final thinning of ultrathin lamellae and the pores in nano-size can be enlarged with respect to ion bombardment via FIB processing in thin samples. Hence, pin-like structures are found to be more stable both physically and chemically when compared

to lamellar samples. An example for pin-like sample preparation of human dentin using dual-beam instruments is given in Figure 14.

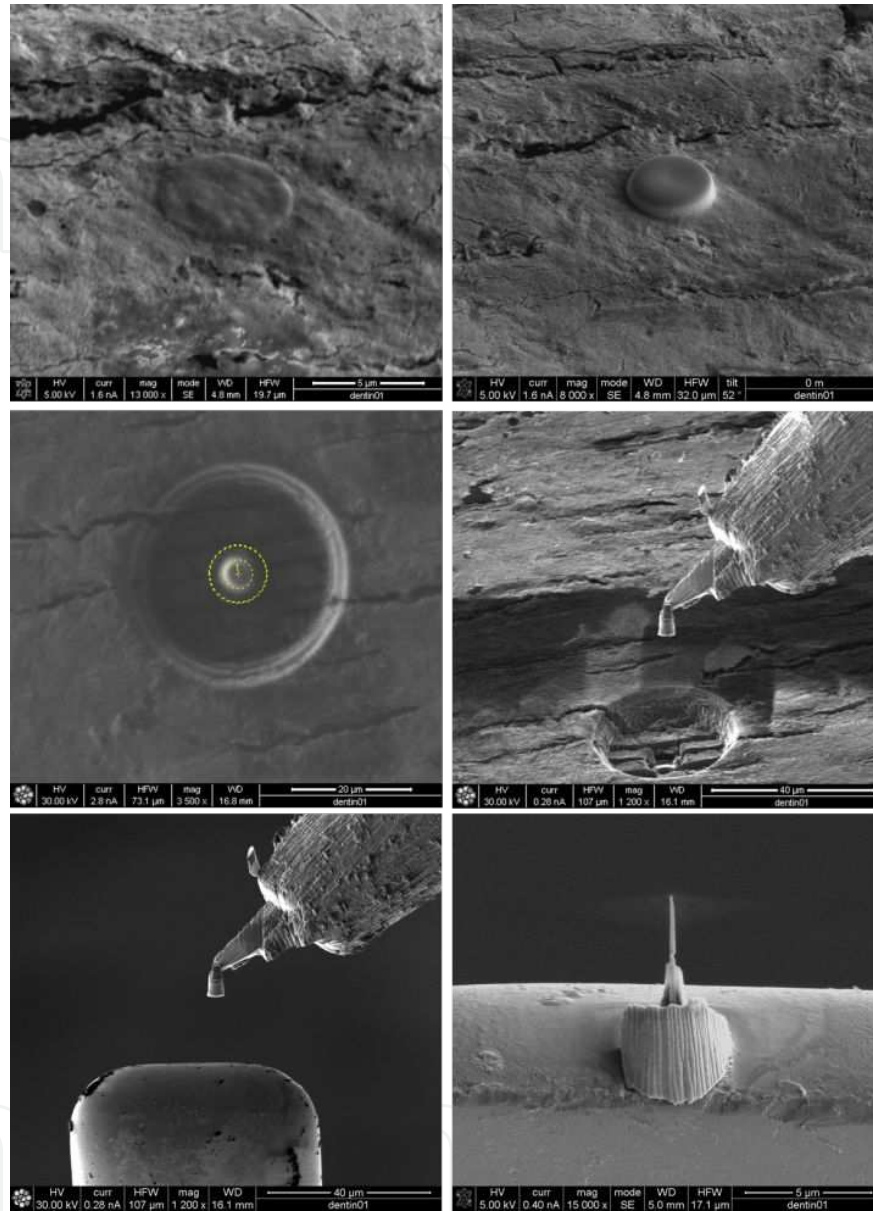


Figure 14. The steps for preparation of pin-like TEM sample using the dual-beam instruments: (a) deposition of electron beam-assisted Pt layer, (b) deposition of ion beam-assisted Pt layer, (c) ion beam milling via annular patterns, (d) lift-out of the pre-section, (e) mounting of the pre-section onto the grid; (f) final thinning and polishing (reprinted from [7] by permission)

Pin-like structures investigated by TEM allow for revealing structures in 3D at the nanoscale and below, and when combined to FIB tomography data, the overall results help in observing materials' features at different scales. An example again on human dentin is given in Figure 15 for tracking micro-sized dentinal tubules, and nano-sized collagen fibrils are investigated using FIB slicing/SEM imaging and TEM bright field imaging, respectively.

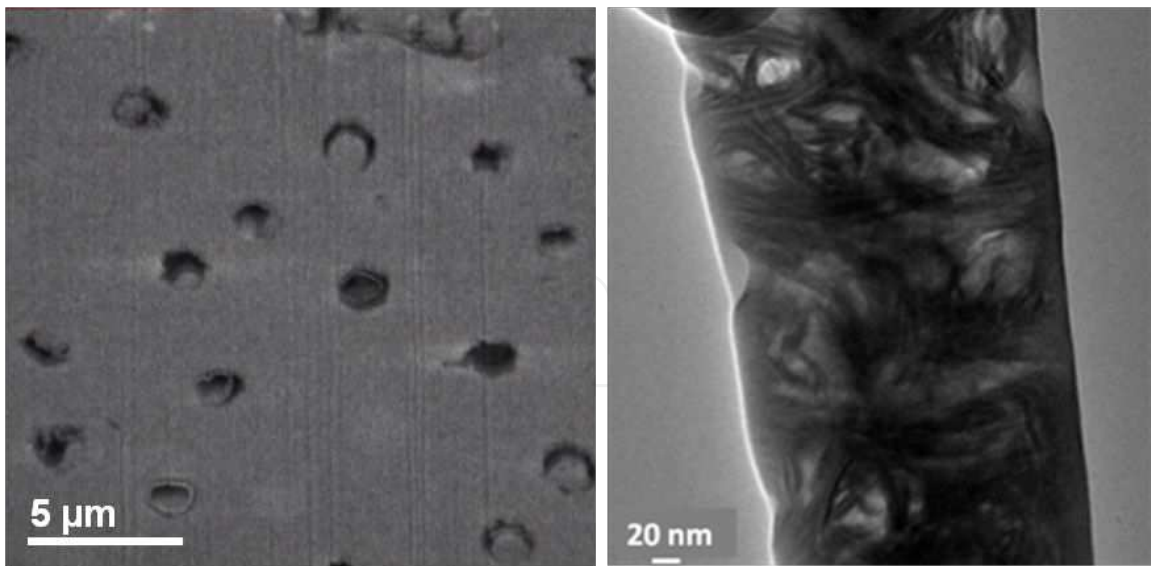


Figure 15. FIB slice showing the cross sections of tubules elongated in y -axis in human dentin and the TEM bright field images showing the 3D distribution of collagen fibrils within the human dentin. The micrographs show the nanostructures within the dentin structure [7].

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