Focused Ion Beam – Fundamentals (Part 1)

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Understanding the Scanning Electron Microscopy (SEM) technique gives also a knowledge about Focused Ion Beam (FIB) instrumentation as they operate according to the same principle. A beam of particles rasters over a sample surface and from the signal obtained at each position the image is created. However, instead of electrons applied in SEMs the positively charged ions are used. The implementation of the ions instead of electrons brings few consequences. First of all, there is no X-ray emission as the large ions are not able to reach the inner shell electrons of the target and cause the inner shell excitation. They are easily trapped within the atoms in the sample. Therefore the material is doped with the gallium ions. However, the gallium K-line can be easily separated from the other K-lines and there is almost no overlapping with the L-lines. As a result of the electron – atom interaction, the high energy backscattered electrons are coming out of the sample, which bring chemical composition information from the Z-contrast. Contrary to SEM, the ion (beam) – atom (sample) interaction in FIB causes removal of the atoms called milling. Further, the electro-static lenses are applied in FIB to focus the beam instead of magnetic ones which were not enough effective for heavy ions. The control of the beam current in SEM is realized by the lens control, while in FIB by selecting different apertures.

The focused ion beam instruments can be single or double column. In the second case the ion column is positioned as the side attachment to the microscope chamber. Despite the fact that sample microstructure can be observed using gallium ions of low currents and small spots, the presence of the electron column enriches the spectrum of instrument possibilities. The electrons can be used not only to image the sample but also to neutralize the charging effects and enhance for example the deposition process. Due to the beam – sample interaction three types of signal can be generated: neutral atoms of the milled material, ions and secondary electrons. The neutral atoms are removed by the pumping system. On the other hand, the charged particles can be used to create an image. The FIB system is equipped with ion detector and secondary electron detector, and if it is double-column apparatus also in back scatter electron detector and X-ray detector.

The ions used for imaging cause the cleaning of the surface when removing atoms from it during the scanning, but also can visualize the channeling contrast. The secondary ion image can be very useful giving additional information about the crystallographic orientation in the

metallic samples. The orientation contrast, which is the dominant contrast effect, originates from the channeling of the beam ions between the specimen lattice planes. It is also possible to apply secondary ion mass spectroscopy and analyze the elemental distribution during sputtering. In the FIBs blanking of the signal for the ion beam column which is not used is automotive and prevents the undesirable milling of the material.

The choice of the gallium ions as a primary beam particles were made due to several reasons. Most of all, it is characterized by a very high brightness in comparison to another possible material – argon and therefore able to produce the high current density beam. This feature comes from the flow properties of the gallium and its surface potential. Another important factor is the simplicity in design of the microscope gun – gallium can be compacted in a small volume and possesses a very low melting point of 29.7 °C, so it is referred to a liquid metal ion source (LMIS). Also, it is sufficient to mill the large spectrum of materials due its positioning in the middle of the periodic table.

Another modern solution introduced in FIB instruments is so called Gas Injection System (GIS) which allows to introduce small quantities of the gas near the surface of the sample which combined with the beam scanning results in deposition of the material such as platinum, tungsten, carbon or preferential etching of the sample. The GIS system comprises of the needle (made of tungsten) with the reservoir containing the precursor material. This needle can be introduced over the desired location in the controlled manner (around 100 μm over the surface). The operator can choose the position, area and shape of the pattern, which can be even imported by the user as image. Moreover, it is also possible to impose the time during which the beam dwells at each point, beam overlapping between the neighboring points and focus of the beam. The adsorbed molecules coming out of the GIS are bombarded by the secondary electrons. Their energy which is less than 50 eV is close to the bonding energy of the molecules. The secondary electrons can appear because of the either the electron – sample (Electron Beam Induced Deposition - EBID) or ion – sample interaction (Ion Beam Induced Deposition) which differ in a sense of deposition speed at given current. Under standard modes that are 30 kV of accelerating voltage for the Ga ions and 5 kV for the electrons the IBID is 10 times faster. It should be kept in mind that during deposition the sputtering still keep going, however it is over taken by the deposition rate. The important factor which guarantee the successful deposition process is the appropriate current density, for the example platinum deposition requires 2-10 $pA/\mu m^2$. If the current density is too low it can result in the presence within the sample of both the unreacted precursor material and the

carbon. In case of too high current the surface milling can take place instead of the deposition process. In general, IBID is preferentially used for the depositions which should be conductive while EBID for the insulating materials, but also in a cases of very sensible surfaces or for very small and precise deposition. GIS is also applied for the preferential etching by using gases such as iodine or xenon difluoride (XeF_2) which are adsorbed to the surface. They can react right away with it or it is necessary to activate the process by the SE emission from the sample.

The most commonly used material for the deposition is the platinum. It is realized by the introducing above the desired location organic-metallic molecules (methyl cyclo pentadienyl Pt(IV) tri methyl) which exposed to the ion beam will decompose and deposit platinum on the surface. It finds the application not only as the protective layer during preparation of the TEM lamella but also for making electrical contacts, obtaining thin conducting layer on the insulating sample or in nanoprototyping for creating 3D structures. Other material used widely for the deposition is tungsten, which can successfully replace platinum especially for the electrical applications. However, its deposition is more time consuming, therefore, platinum is preferred as the protective layer while making TEM lamella and tungsten can be used for further operations such as the soldering of sample to the micromanipulator or to copper grid.

Very common and useful in TEM thin foil preparation process is the carbon deposition. On one hand it can be applied as the additional protective coating before the platinum layer, and on the other hand, it can replace the tungsten in the soldering processes during preparation. It is worth to mention that the sputter rate of carbon is smaller than for the platinum which ensures even better protection of the surface and also less curtaining effects during final polishing due to the smaller grain size of the deposited carbon layer in comparison to Pt one. Finally, the gold deposition is the attractive solution in many applications such as Biology (fixing proteins) and Optics due to its low chemical reactivity and high electrical conductivity. It is possible to deposit small features in an automative manner, nevertheless, it is more expensive, and therefore less applicable to every-day preparation.

Focused Ion Beam systems were as first dedicated to the semiconductor industry and developed since late 1980s [Kir1989], however originally they acted as lithography instruments. Pre-preparation of the TEM lamella comprised of cutting of the sample into about 3 mm in length followed by mechanical polishing to 100-50 μm. Usually the polishing

was accomplished using tripod method [And1997]. The H-bar technique was the first used for the thin foils preparation where thin window is obtained as a result of FIB milling of back-to-back sections into pre-thinned bar mounted on the modified TEM grid (Fig. 1).

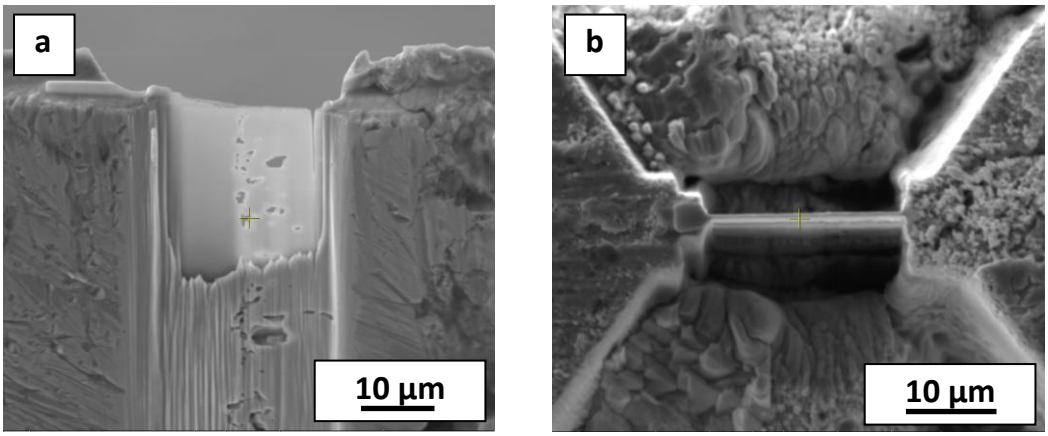

Fig. 1. TEM lamella preparation by H-bar technique: side (a) and top views (b).

The lift-out technique allowed to exclude the inconvenient preliminary preparation and obtain the TEM lamella directly from the bulk sample. Among this preparation lift-out *ex-situ* and *in-situ* variants are recognized. Both of them apply so called micromanipulator to transfer the lamella, however *ex-situ* lift out variant involves the final thinning of the lamella still connected to the bulk material followed by its transport on the special copper grid cover with the carbon membrane. The preparation by the *in-situ* manner gives better results as the observations under TEM is not interfered by the carbon membrane or edges of the copper grid, and therefore nowadays it is the most commonly used variant. As the first step, the protective platinum layer of about 15-20µm (length) x 2µm (width) x 3µm (thickness) in size is placed on the area of interest. Next, the material neighboring the both sides of the Pt layer of rectangle shape is removed using high currents (20-60 µA). The efficiency of milling (typically $\mu m^3/nC$) depends on the material - its atomic mass and orientation towards to the primary beam. Subsequently, both sides of the material under platinum layer are milled using smaller currents (5µm) until it reaches thickness of 1-2 µm. At this moment the lamella can be cut from the bulk material from almost all three sides, however from one not completely. The micromanipulator needle is soldered using tungsten or carbon to the free side of the lamella and only now it can be cut completely out of the bulk sample. The lamella is mount on a special copper grid designed for the FIB technique using as previously tungsten or carbon and the micormanipulator needle can be released. Going down with the applied currents milling of the both sides is performed until the desired thickness 100-50 µm is achieved. Milling efficiency and consequently time of preparation depends strongly on the type of material.

Modern FIB instruments allow to speed up the process by the application of higher currents during first step of preparation and designing the microscopes with smaller working distances. On the other hand, smaller accelerating voltages such as 2 kV is applied which decreases the damage depth from several tens of nanometers (for typically used 30 kV) to even 1-2 nm for such material as silicon [Rou2009].

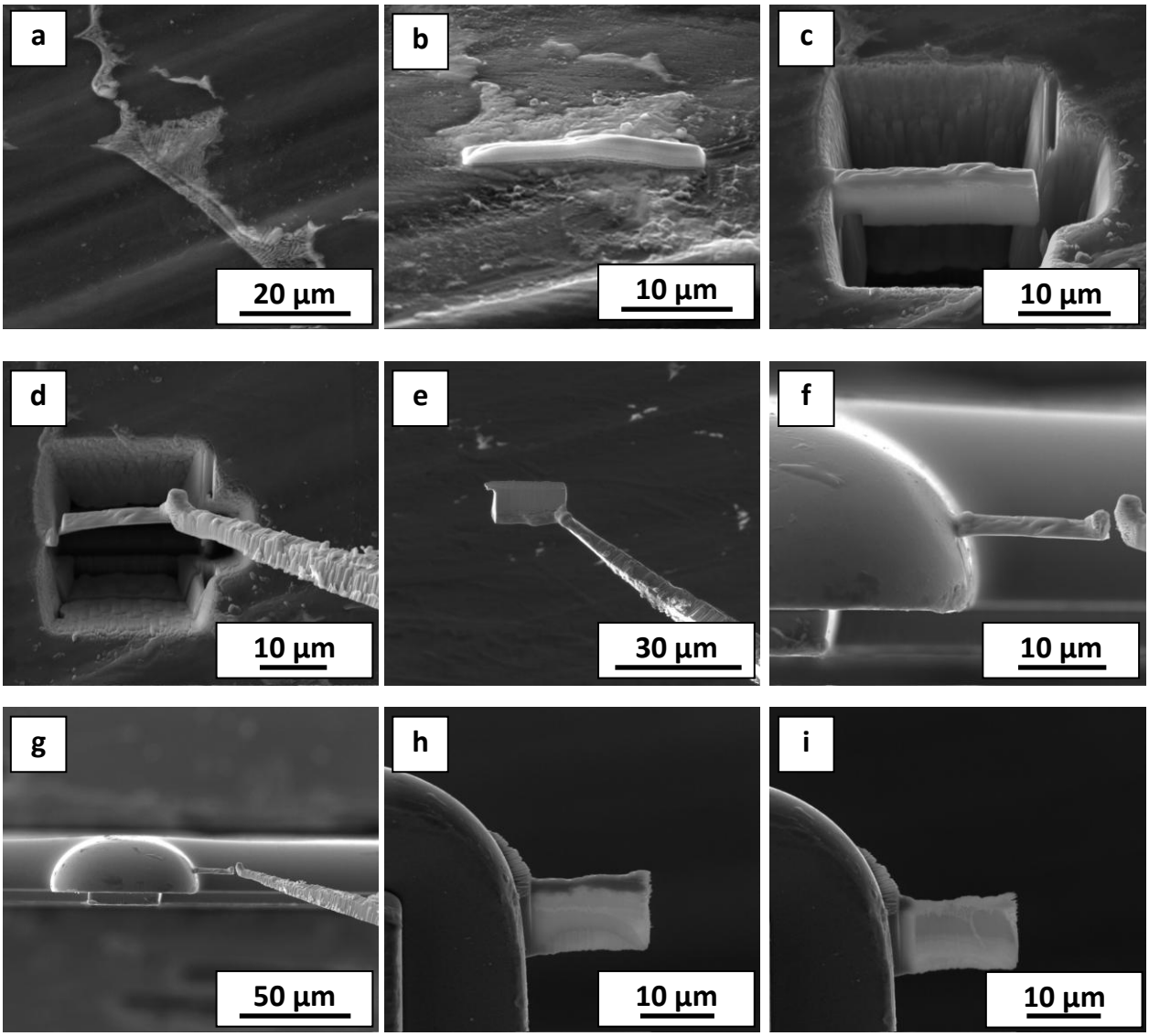

Fig. 2. TEM lamella preparation by *in-situ* lift out technique step by step: localization of the area of interest (a), deposition of the protective platinum layer on its surface (b), milling of the neighboring material using high currents followed by the cutting off the lamella from bottom and right side (c), soldering the micromanipulator needle to the lamella followed by total cutting off from the bulk sample (d), transferring the lamella to the copper lift-out grid (e), soldering the lamella to the lift-out grid followed by cutting off the micromanipulator needle (f,g), side view of the lamella soldered to the copper grid before (h) and after final thinning (i).

The Focused Ion Beam system dedicated to the TEM lamella preparation within the Accredited Testing Laboratories in the Institute of Metallurgy and Materials Science is Quanta 3D 200 (*FEI*) which is a dual beam instrument. It is equipped with the *Omniprobe* micormanipulator allowing the in-situ lift out technique, gas injection systems for platinum,

tungsten and carbon deposition as well as back scatter electron detector attached to the electron column.

References:

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