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Transmission Electron Microscopy (TEM) Sample Preparation of $Si_{1-x}Ge_x$ in *c*-Plane Sapphire Substrate

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Abstract

The National Aeronautics and Space Administration-invented X-ray diffraction (XRD) methods, including the total defect density measurement method and the spatial wafer mapping method, have confirmed super hetero epitaxy growth for rhombohedral single crystalline silicon germanium ($Si_{1-x}Ge_x$) on a *c*-plane sapphire substrate. However, the XRD method cannot observe the surface morphology or roughness because of the method's limited resolution. Therefore the authors used transmission electron microscopy (TEM) with samples prepared in two ways, the focused ion beam (FIB) method and the tripod method to study the structure between $Si_{1-x}Ge_x$ and sapphire substrate and $Si_{1-x}Ge_x$ itself. The sample preparation for TEM should be as fast as possible so that the sample should contain few or no artifacts induced by the preparation. The standard sample preparation method of mechanical polishing often requires a relatively long ion milling time (several hours), which increases the probability of inducing defects into the sample. The TEM sampling of the $Si_{1-x}Ge_x$ on sapphire is also difficult because of the sapphire's high hardness and mechanical instability. The FIB method and the tripod method eliminate both problems when performing a cross-section TEM sampling of $Si_{1-x}Ge_x$ on *c*-plane sapphire, which shows the surface morphology, the interface between film and substrate, and the crystal structure of the film. This paper explains the FIB sampling method and the tripod sampling method, and why sampling $Si_{1-x}Ge_x$, on a sapphire substrate with TEM, is necessary.

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Nomenclature

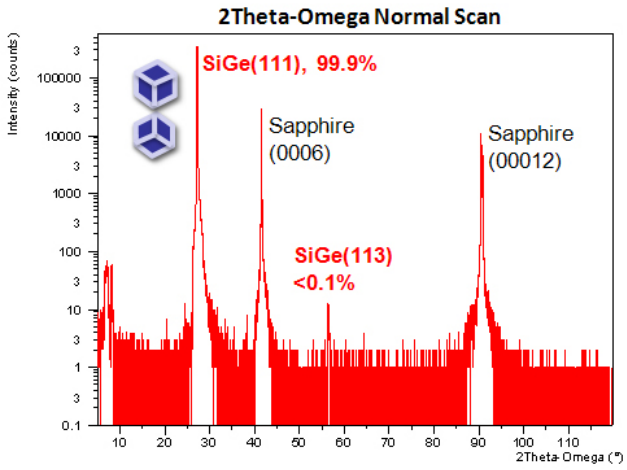
Al_2O_3	sapphire
C	carbon
Cu	copper
DI	deionized
EBSD	electron backscatter diffraction
FIB	focused ion beam
Ge	germanium
GIS	gas injector system
HRTEM	high resolution transmission electron microscopy
Pt	platinum
SAED	selected area electron diffraction
SEM	scanning electron microscope
Si	silicon
STEM	scanning transmission electron microscopy
TEM	transmission electron microscopy
W	tungsten
XRD	X-ray diffraction

Overview

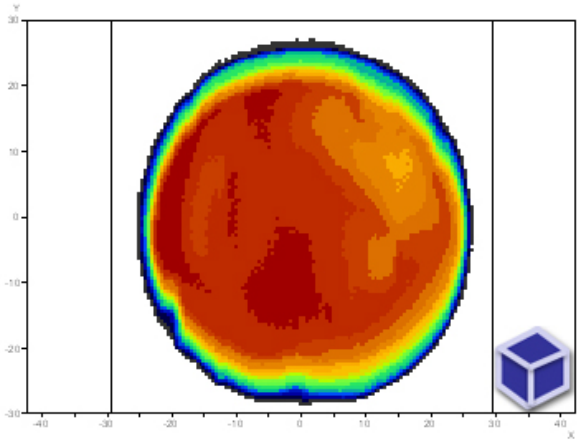
Lattice-matched $Si_{1-x}Ge_x$ opens the possibility of speed improvement, compared to the single crystal silicon itself that has its own intrinsic limit on speed. The electron mobility of germanium is 4,000 $cm^2/V{\cdot}s$ while that of silicon is only 1,400 $cm^2/V{\cdot}s$. The attainable speed of transistors made with $Si_{1-x}Ge_x$ is based on the gate length and the charge mobility as related to the film morphology, twin structure, the number of $Si_{1-x}Ge_x$ twins, and crystal structure of the $Si_{1-x}Ge_x$ on a *c*-plane sapphire substrate. If the defects in $Si_{1-x}Ge_x$ can be removed, $Si_{1-x}Ge_x$ should allow faster electron motion than single crystal silicon. Transistors with higher operational frequencies can be fabricated for a new generation of ultrafast chipsets (among other things) for numerous applications. $Si_{1-x}Ge_x$ on *c*-plane sapphire substrates also can improve the following products: ultrafast complementary metal oxide semiconductor chipsets; heterojunction bipolar transistors; thermoelectric devices; photo-voltaic solar cell devices; advanced detectors; high frequency high power transmitters, and so on. The National Aeronautics and Space Administration-invented X-ray diffraction (XRD) methods, including the total defect density measurement method and the spatial wafer mapping method, confirmed super-hetero epitaxy growth for rhombohedral single crystal silicon-germanium ($Si_{1-x}Ge_x$) on a *c*-plane sapphire substrate (ref. 1 and 2). However, the XRD method cannot observe the surface morphology or roughness, which affects the efficiency of devices, because of the method's limited resolution. For this reason, the authors have studied the structure between $Si_{1-x}Ge_x$ and sapphire substrate, and $Si_{1-x}Ge_x$ itself with transmission electron microscopy (TEM), and characterized the film morphology and twin structure of $Si_{1-x}Ge_x$ thin films sputtered on sapphire (0001) substrates using cross-sectional TEM analysis.

Figure 1 shows the differences in the results by XRD, electron backscatter diffraction (EBSD), and TEM of $Si_{1-x}Ge_x$ thin film sputtered on sapphire (0001) substrates. The XRD wafer mapping method shows spatial distribution of major single crystalline $Si_{1-x}Ge_x$ (99.9 percent) (Figure 1a and b) and twin defect $Si_{1-x}Ge_x$ (< 0.1 percent), which coexist on the same sapphire wafer. The small cube icons (and) in Figure 1 (a) and (b) represent two possible in-plane azimuthal alignments inside rhombohedral alignment view from the [111] direction. The crystal structure of the entire wafer can be characterized with the XRD wafer mapping method. However, one cannot observe the surface morphology, roughness, and density of dislocation with the XRD method. Figure 1c shows the EBSD analysis of the crystal orientation domain in a small region (20 x 20 μm^2). A few line-art cubic shapes are drawn to indicate the crystal orientation of each color domain with the same-colored domain having similar crystal orientation. The majority of the area is the blue-colored domain, a [111]-oriented majority single-crystal region. In XRD analysis, the ratio between a single crystal and a twin crystal was 99:1. Therefore, the EBSD analysis further supports the result of the XRD analysis. Currently, EBSD is the fastest and most reliable way in which to acquire data for crystalline structure and orientation in a solid crystalline phase. However, the data of EBSD are generated at very shallow depths (10 to 20 *nm*) within the sample, so the cross-section TEM study is needed to confirm the crystalline structure and orientation of the $Si_{1-x}Ge_x$ film through the thickness. Figure 1d shows the high resolution transmission electron microscopy (HRTEM) image of the $Si_{1-x}Ge_x$ / Al_2O_3 (sapphire) interface. The orientation relationships between the Al_2O_3 substrate and $Si_{1-x}Ge_x$ single crystal layer are (0001) Al_2O_3 || (111) $Si_{1-x}Ge_x$ and [01–10] Al_2O_3 || [–112] $Si_{1-x}Ge_x$. The TEM results

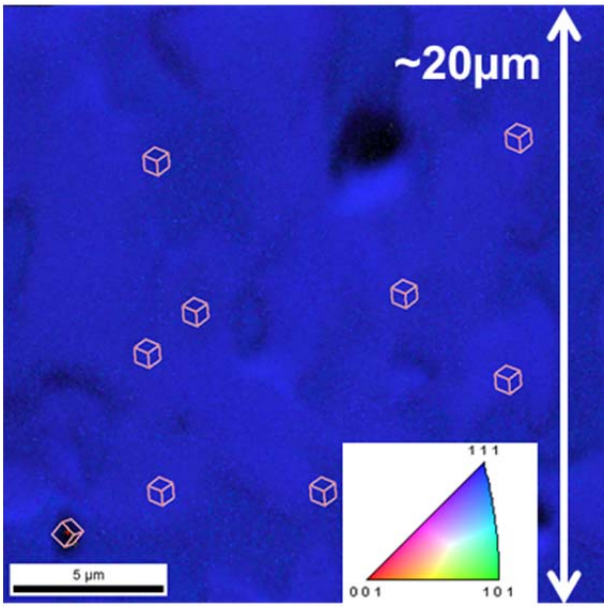
directly show crystal structure of the film, interface between film and substrate, and surface morphology.



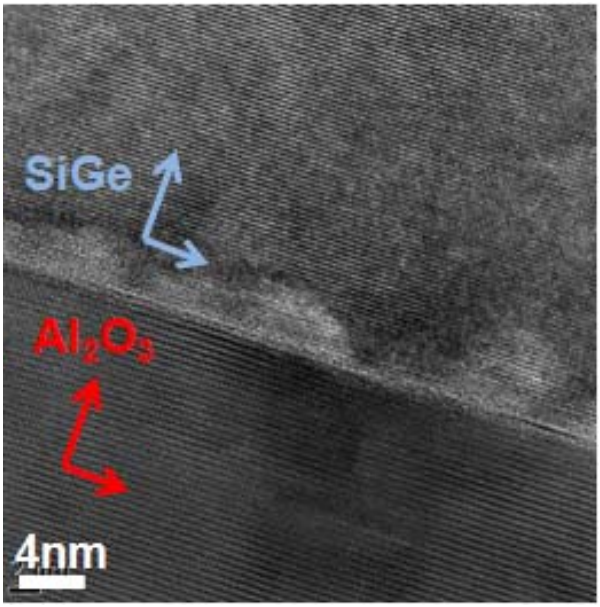
(a) Zero to 20 scan in the direction normal to surface with XRD. Small cube icons represent two possible in-plane azimuthal alignments inside rhombohedral alignment



(b) Two inch sized *X–Y* wafer mapping with majority {440} peak showing the distribution of a single crystal.



(c) Color-coded scanning electron microscope image with crystal orientation mapping as analyzed by EBSD. The blue region is the majority single crystal (111) domain.



(d) HRTEM and result of the $Si_{1-x}Ge_x$ / sapphire interface.

Figure 1. Analysis of $Si_{1-x}Ge_x$ sample.

The standard sample preparation method of mechanical polishing often requires a relatively long ion milling time (several hours), which increases the probability of inducing defects into the sample. These defects could be:

- an ion implantation that will modify the crystal structure,
- an amorphisation of the surface layers of the sample,
- a possible modification of the sample chemical composition,
- sample heating,
- an inhomogeneity of the final thickness between the different compounds,
- a redeposition, onto the sample's surface of the milled materials (especially for a sample in plain view showing the layers on the surface and milled only from the rear side).

$Si_{1-x}Ge_x$ on a sapphire substrate has problems not inherent to other silicon-based materials when using the standard preparation method. Sapphire is a difficult material to thin because of its high hardness and its mechanical instability when thinned below $20\mu m$. Delamination of $Si_{1-x}Ge_x$ can occur during polishing or ion milling. In addition, the $Si_{1-x}Ge_x$ has a markedly different ion milling rate than the sapphire substrate (ref. 3). Therefore, the sample preparation for the TEM should be completed as quickly as possible so that the sample will contain few or no artifacts induced by the preparation. The focused ion beam (FIB) method or a tripod polisher method succeeds on both counts when performing a cross-section TEM sampling of $Si_{1-x}Ge_x$ on sapphire (0001).

Focused Ion Beam Method

Whereas the initial development of focus ion beam 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 (ref. 4). Many FIBs are largely used to prepare TEM cross-section samples (ref. 5–9). FIB systems operate in a similar fashion to a scanning electron microscope (SEM), except, rather than a beam of electrons, FIB systems use a finely focused beam of ions that can be operated at low beam currents for imaging or at high beam currents for site specific sputtering or milling. A tungsten (*W*) or platinum (*Pt*) line is deposited on the area of interest to protect the top portion of the sample and to mark the position of the target area. A thin slab of the material is cut from the area of marked interest in the sample and mechanically polished as thin as possible. The sample material is then mounded on a half-grid and inserted vertically into the FIB chamber where it is then milled.

There are three advantages to this method. First, the target area can be precisely selected using a FIB scope; in that lamellae can be prepared with a spatial accuracy within ~$20\mu m$. Second, FIB preparation techniques are virtually independent of the nature of the material. Finally, FIB sample preparation can be applied to almost any material type—hard, soft, or any combination.

The main disadvantage of FIB is the ion milling process. Here, the ion collision initiating sputter removal can also lead to ion implantation and cause severe damage to the remaining bulk of the material.

Tripod Method

A tripod polisher, designed by scientists at IBM, was used to prepare micro sizes of TEM and SEM samples (ref. 10). For TEM samples, the tripod polisher has been used to limit ion milling times to less than 15 minutes and, in some cases, has eliminated the need for ion milling. It can be used to prepare both plan-view and cross-sections from a variety of sample materials, such as ceramics, composites, metals, and geological samples (ref. 11 and 12). The cut sample is mounted on the tripod polisher in order to polish the first side. This polishing is carried out with the change of different grain-sized diamond lapping films ($30\mu m$, $15\mu m$, $6\mu m$, $3\mu m$, $1\mu m$, $0.5\mu m$, and $0.1\mu m$). The final polishing, used to eliminate all the scratches from the surface, is carried out on a soft felt pad wet by a colloidal solution that contains very thin grains ($0.05\mu m$) either of silica, alumina, diamond, or other hard media. The sample is then removed from its support, turned over and reattached. The sample can be mounted on the TEM grid before polishing the second side. The Pyrex® cylinder (insert) has to be polished flat and level to insure the correct angle of the wedge shape of the sample while the opposite side is polished. Finally, the sample is mounted on a TEM grid with a diameter adapted to the sample holder of the ion-milling machine. There are many advantages utilizing this method. It allows the observation of a sample that cannot be ion milled. The surface obtained is of a better quality than the surface obtained with the mechanical polishing method. The difference in final thickness, due to the different material properties, between the different compounds of the sample is greatly reduced, as is the preparation time. Finally, this process provides the possibility of dry polishing those materials that cannot be polished using a lubricating fluid. The disadvantages of the tripod method are; it is hard to clean very large samples, it does not work well with very soft materials and may require some practice.

$Si_{1-x}Ge_x$ on Sapphire (0001) Sample Cutting for Cross-section TEM

The pole piece gap of TEM equipment limits the tilting angle of sample. If the TEM sample is cut in the proper direction, near to the referenced zone axis, one can easily find the epitaxial relationship between the film and the substrate. From the X-ray diffraction data and the selected area electron diffraction (SAED) pattern between $Si_{1-x}Ge_x$ film and sapphire substrate, the authors confirmed the epitaxial growth between the film and the substrate (ref. 1 and 2). The epitaxial relationship between the majority of the *SiGe* film and the sapphire substrate was found to be (111) $Si_{1-x}Ge_x$ // (0001) sapphire and [011] $Si_{1-x}Ge_x$ // [10-10] sapphire. To obtain the distinct diffraction patterns for two crystallographic variants rotated from each other by 60 degrees, the TEM sample has to be tilted to *SiGe* <011> zone-axis orientation. This can be readily located by finding the [10–10] sapphire zone, which is parallel to [011] *SiGe*. Usually the (0001) sapphire substrate, that is the *c*-plane, with a flat {11–20} has the crystal orientation as shown in Figure 2. If the sample for the FIB or the tripod method is cut normal to the *a*1 axis as shown in Figure 2, one can find the [10–10] sapphire zone easily.

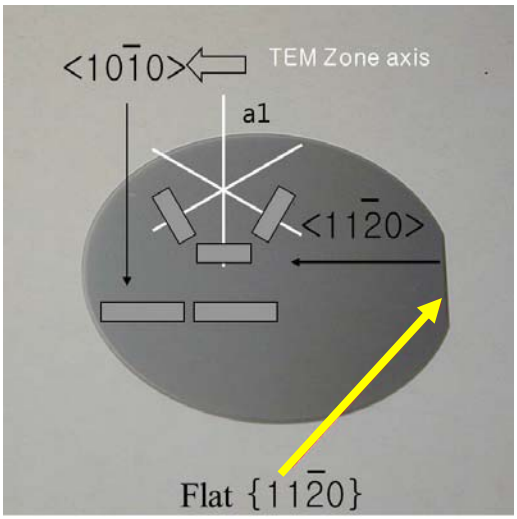


Figure 2. Crystal orientation: The three grey rectangles are all equivalent.

TEM Sample Preparation of $Si_{1-x}Ge_x$ on Sapphire (*c*-Plane, Al_2O_3) Substrate Using FIB

The preparation of good TEM samples with minimum milling damage can be complicated, especially from a specific area in the $Si_{1-x}Ge_x$ / sapphire sample. The TEM is a powerful tool for investigating the microstructure of materials, providing crystallographic and composition information at the nanometer scale. Therefore, obtaining samples that are uniform and thin (less than 100*nm*) is critical. The focused ion beam method is generally faster than other manual preparations and has exceedingly high placement accuracy.

The general process using FIB for TEM sample preparation is described in several papers (ref. 4–9). This NASA document describes specific conditions (adding the numerical value) in the process for sapphire sample preparation. Also, this document includes some extra tips such as using a felt-tip pen and cutting the TEM grid to prevent copper (*Cu*) redeposition during the ion milling.

The complete process is as follows:

Note: 1) Yellow marker in the figure: etching area 2) Green marker: deposition area

1. Mount the $Si_{1-x}Ge_x$ / sapphire sample on 12 mm sample holder.
    - To conduct between the sample and the sample holder, apply a silver (*Ag*) paste to prevent the buildup of an electrical charge on the sample surface (Figure 3).

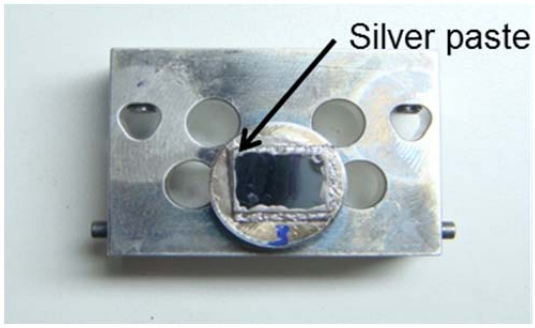


Figure 3. Image of sample with silver paste on FIB holder.

2. Mark on the sample surface.
    - Mark the necessary indications with a felt tip pen on the surface of the sample to be analyzed (Figure 4). The purpose is to protect the sample surface and to increase the contrast. The ink of a felt tip pen is amorphous, which makes marking the surface much easier and cheaper than the sputtering method. After drying the sample, coat the sample surface with a conducting material if the sample is composed of a non-conducting material.

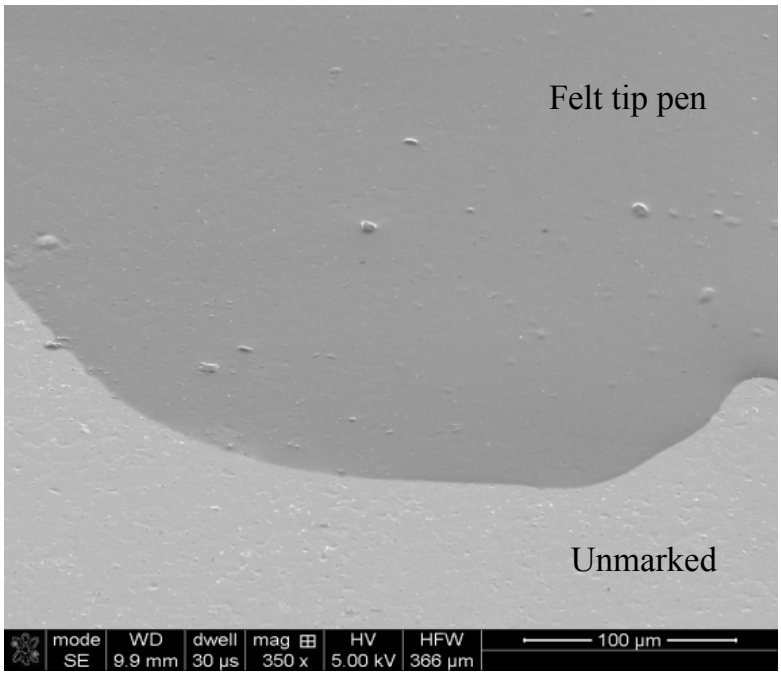


Figure 4. SEM image of sample surface (top dark area: marked with a felt tip pen).

3. Mount the sample holder in a FIB (FEI™ Company, Quanta™ 3D FEG) chamber.
    (1) Evacuate the main chamber.
    (2) Turn on beams, the SEM column and ion column.
    (3) Move the sample stage to the location where the fabrication is to be done.
    (4) After focusing the sample surface with the SEM, raise the holder to about Z = 10mm to adjust the eucentric height of the sample to a 0 degree angle. Repeat but adjust the angle by 7, 15, and 52 degrees, respectively. This is because the

FIB column has a different angle to the SEM column by 52 degrees. The 0 degree angle means that the sample is located vertically to the SEM column and 38 degrees to the FIB column. So, if that sample is to be faced vertically with the FIB column, it should be tilted by 52 degrees. For the same eucentric height, both in the SEM and the FIB mode, repeat the adjustment for the same location by 7, 15, and 52 degrees, respectively (Figure 5).

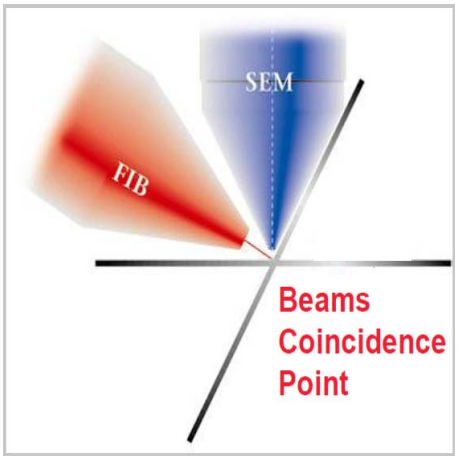
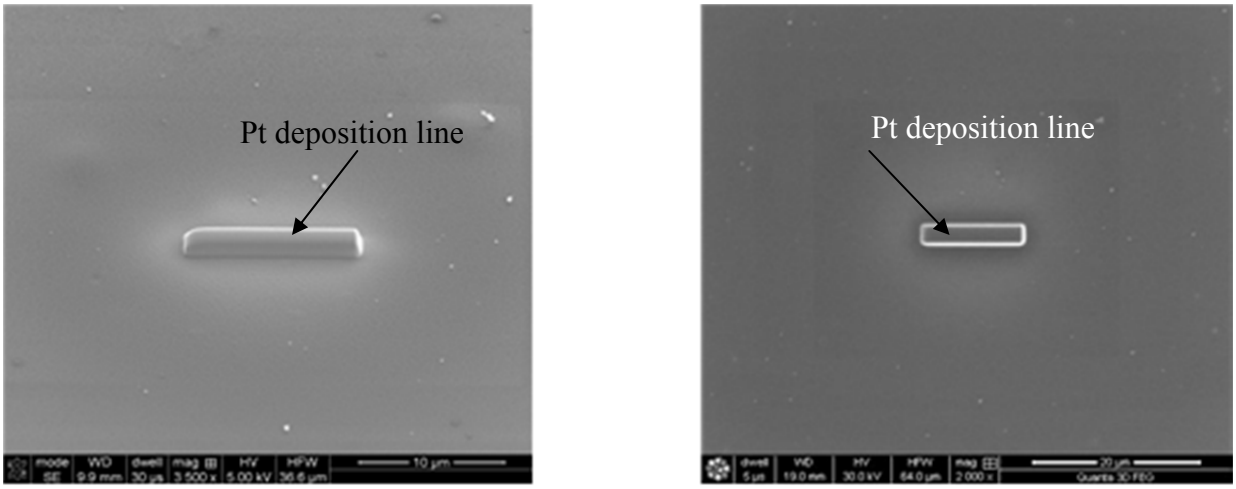


Figure 5. Schematic diagram of FIB and SEM column position.

4. Perform e-beam deposition of *Pt* (tilt 0 degrees).
    - Put a platinum line on the area of interest to protect the top portion of the sample and to mark the position of the target area. (If step 2 has been completed, skip this step.)
    - SEM condition: $2kV$, $0.47nA$, $X = 12\mu m$, $Y = 2\mu m$, with a duration of 3 minutes
5. Perform ion deposition of *Pt* (tilt 52 degrees) (Figure 6).
    - Ion condition: $30kV$, $0.3nA$, $X = 12\mu m$, $Y = 2\mu m$, $Z = 1.5\mu m$, with duration 3 to 5 minutes.



(a) Tilted view SEM image. **(b) Top-view FIB image.**

Figure 6. Images after *Pt* deposition.

6. Cut the sample.
    - Select the mode "Regular Cross Section (making a regular cross section)," which will work quickly and will roughly make the sample the shape of a "step" (Figure 7).
    - Cut the sample in ion condition: $30kV$, $30nA$, $X = 24\mu m$, $Y = 18\mu m$, $Z = 8\mu m$, with a duration of 14 minutes.
    - Figure 8 shows the images after regular cross section.

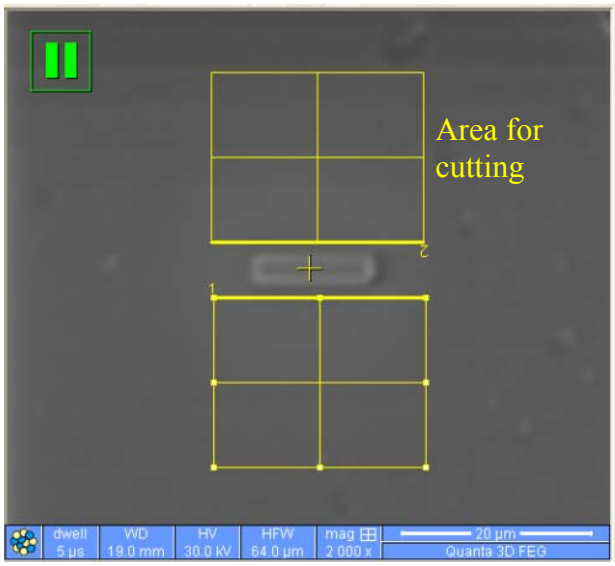
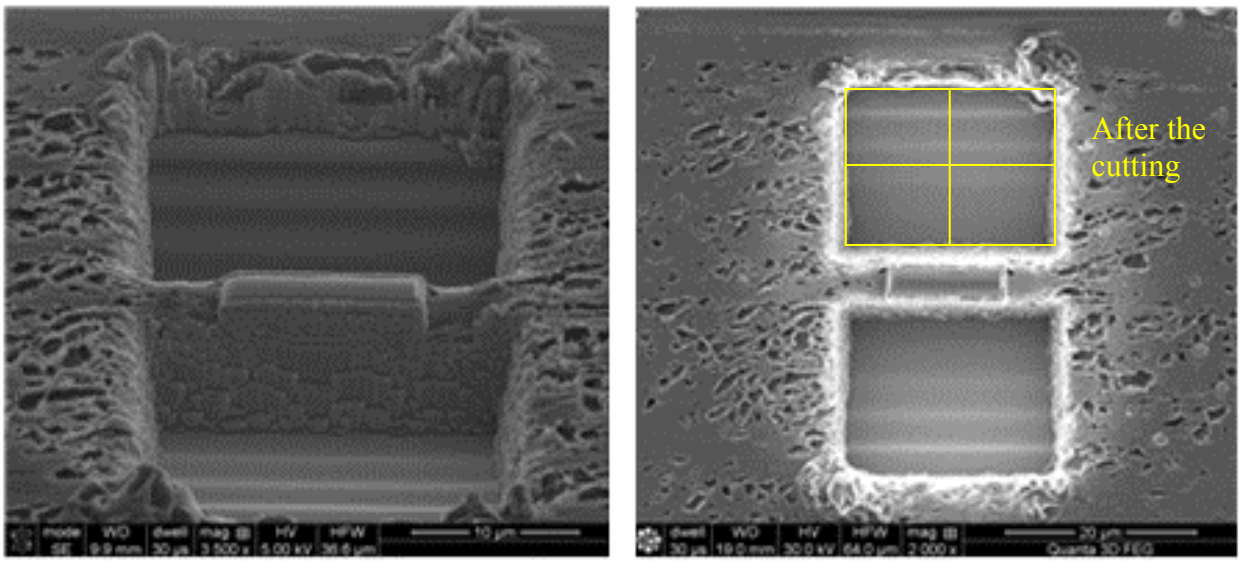


Figure 7. FIB image with regular cross-section marking.



(a) Tilted view with SEM image. **(b) Top view with FIB image.**

Figure 8. Images after cutting out cross-sectional areas (Fig. 7).

- Tilt the sample by ±3 degrees, 55 degrees for milling the bottom side and 49 degrees for milling the top side (Figure 9).
- Select the mode "Rectangular (30*kV*, 5*nA*)." This mode has the same etching rate in the yellow box (Figure 9) so the etched section of sample can be clean and smooth (Figure 10b).

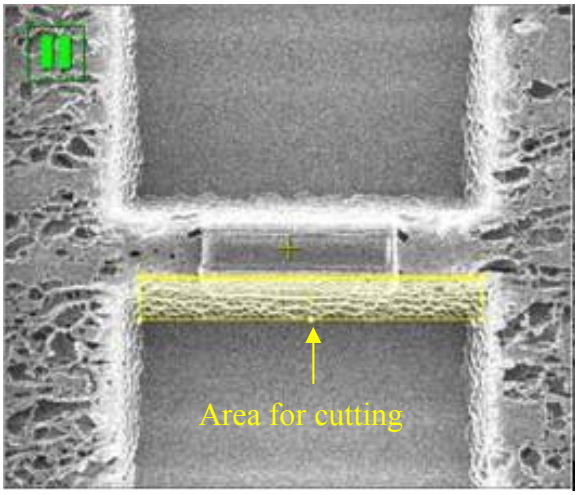
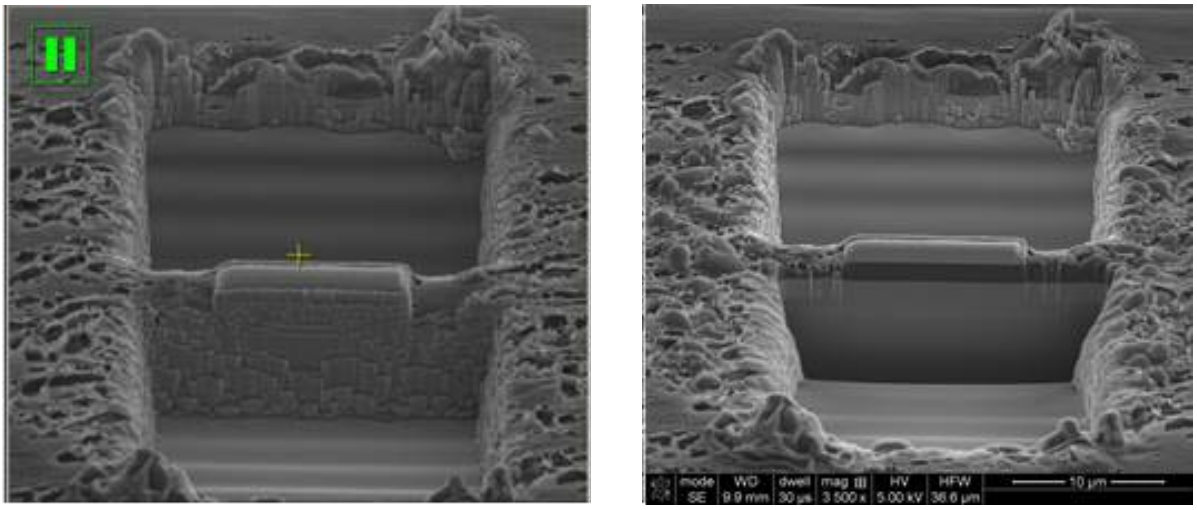


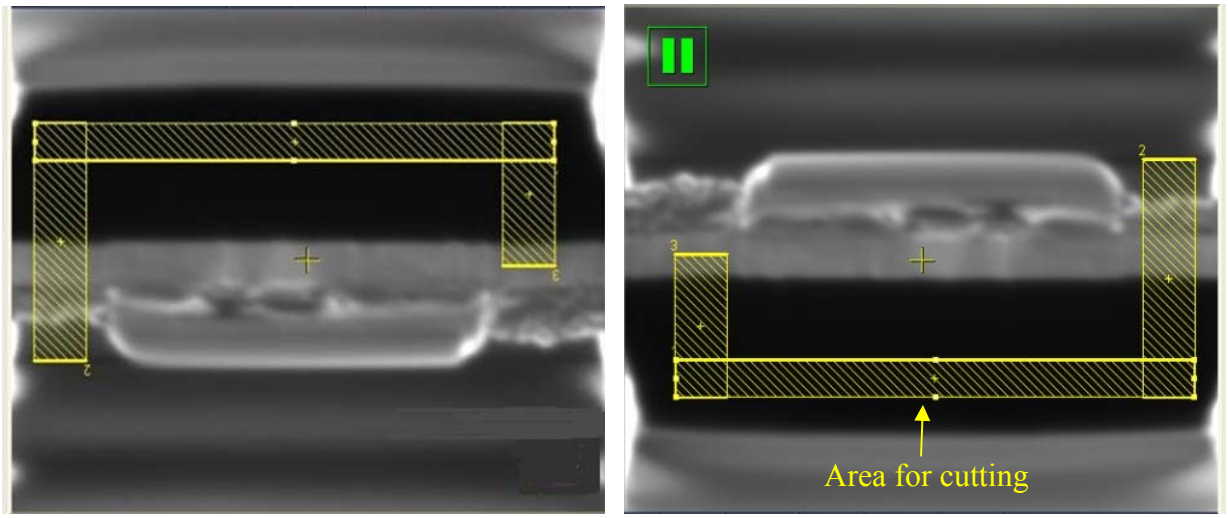
Figure 9. Milling the bottom side (tilt 55°) for smoothing the surface.



(a) Image before milling the sample. **(b) Image after milling the sample.**

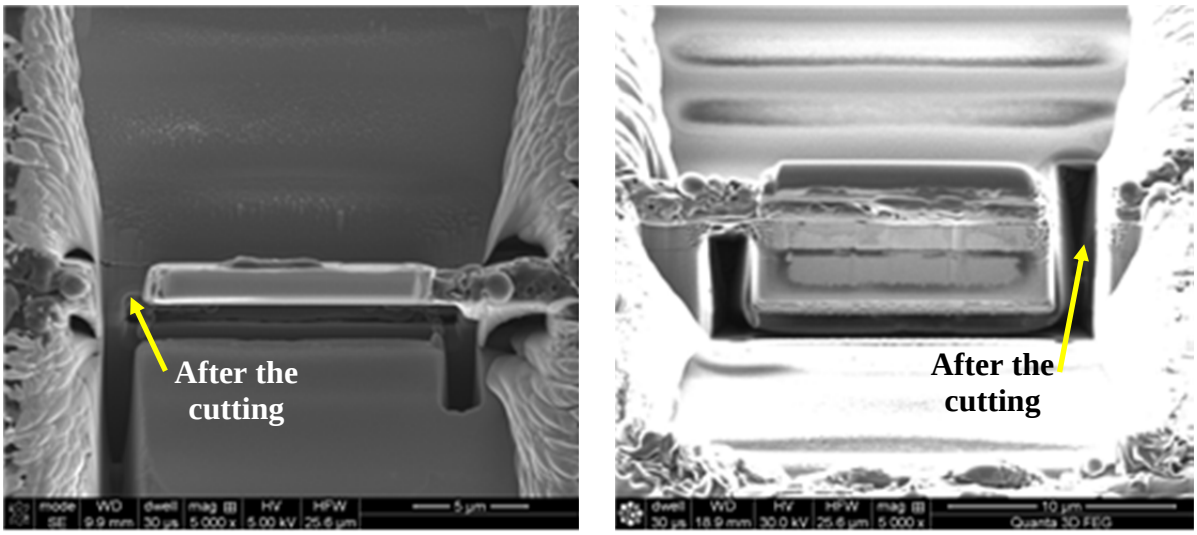
Figure 10. Image of milling the bottom side.

- Do a scan rotation ("Ion image") by 180 degrees. (This is to correct the ion image displayed upside down from (a) to (b) in Figure 11.) (keyboard shortcut for scan rotation : shift + F12)
- Select the mode "Rectangular (30*kV*, 5*nA*, tilt = 0°)" as shown in Figure 11 for cutting the bottom and side walls.
- Continue until the etched line is visible on the opposite surface with the SEM image as a reference (Figure 12).



(a) Before 180° scan rotation **(b) After 180° scan rotation**

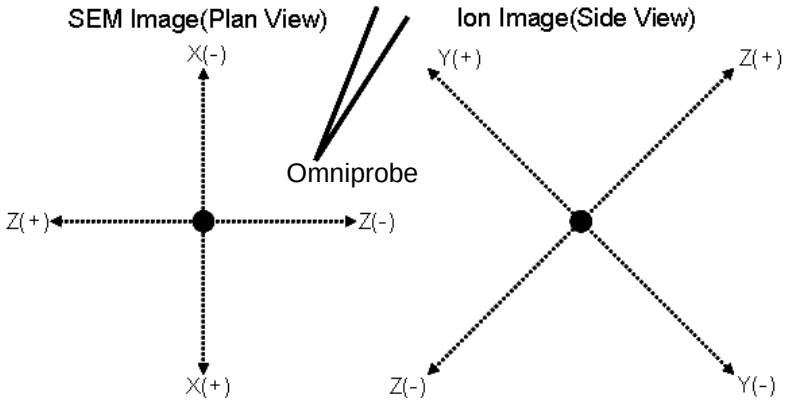
Figure 11. FIB image with a marking section for the bottom and side cuts.



(a) Top view SEM image after cutting. **(b) Tilted view FIB image.(Already 180° scan rotation done in Figure 11)**

Figure 12. Images after bottom and side cuts.

7. Lift up the sample using the omniprobe.
    - Figure 13 shows the omniprobe direction in the SEM and FIB modes. When looking at the SEM image, one can control the *X* and *Z* axes. Also, one can control the *Y* and *Z* axes in FIB mode.
    - Insert the omniprobe and gas injector system (GIS) for using *Pt* at tilt = 0° (Figure 14 and Figure 15).



(a) The SEM image. **(b) The ion image.**

Figure 13. Schematic image of omniprobe direction.

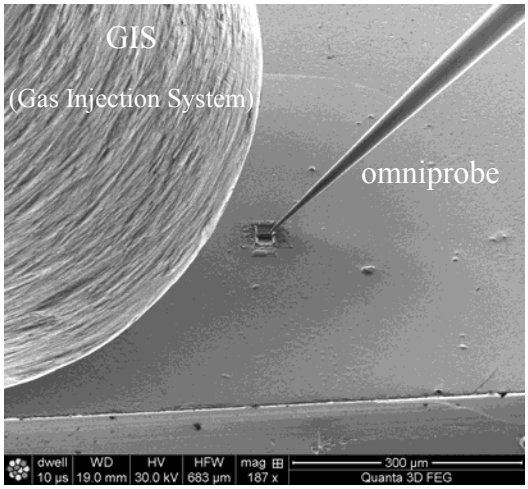
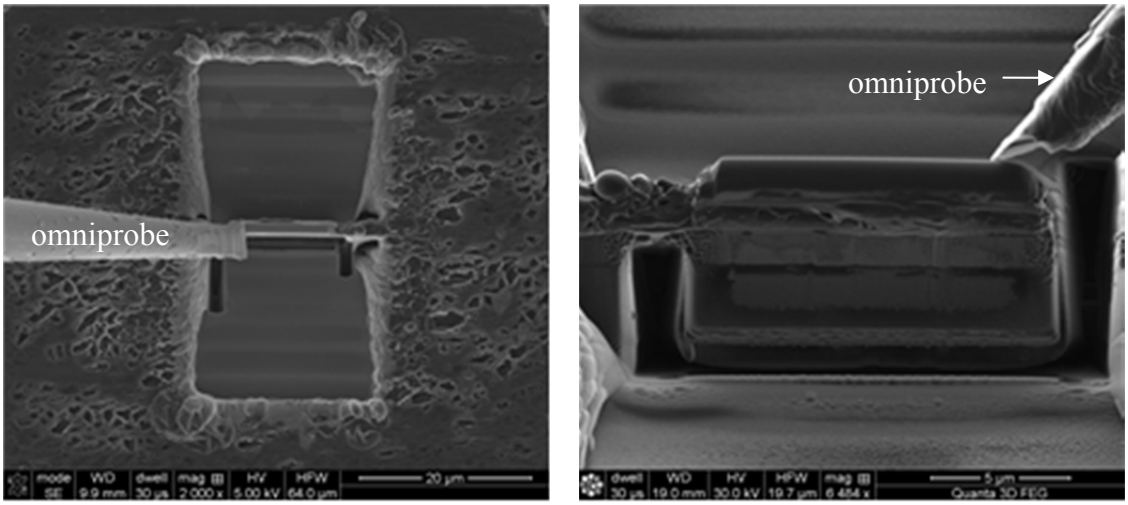


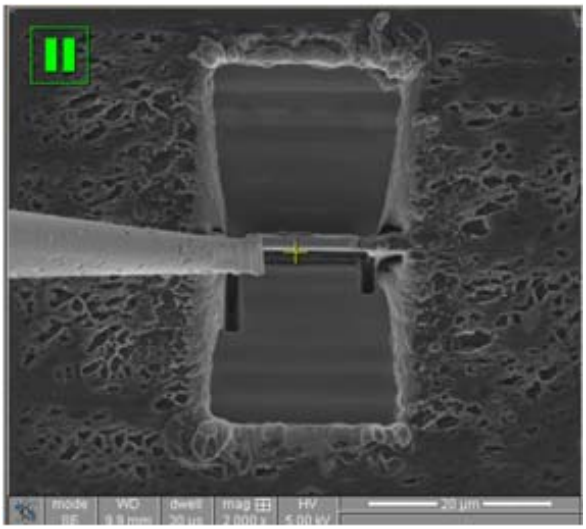
Figure 14. Image after inserting GIS and omniprobe.



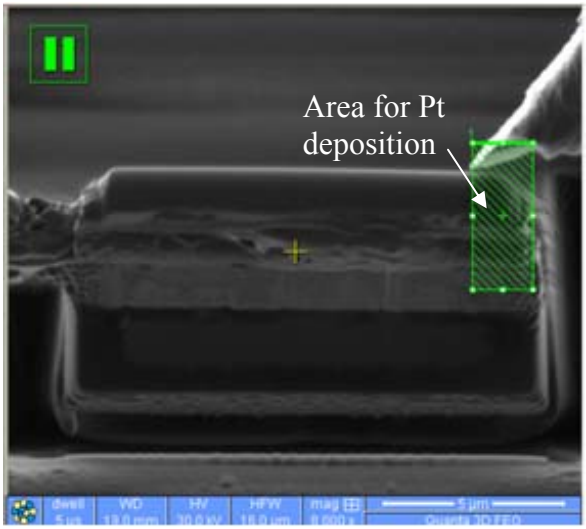
(a) Top view of SEM image. **(b) Side view FIB image.**

Figure 15. Images showing that omniprobe is well aligned with the sample.

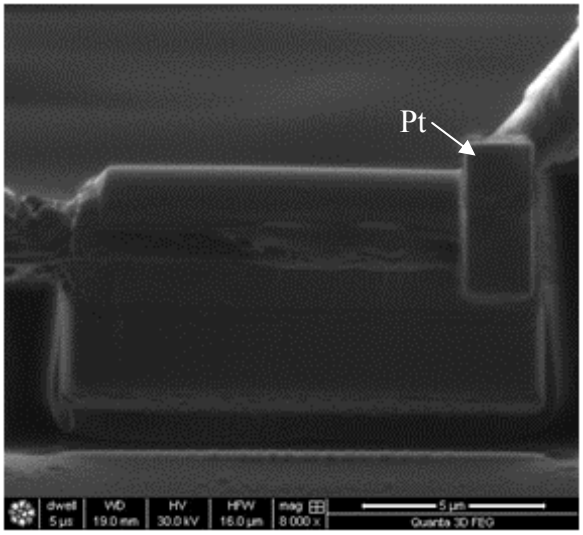
- The process of attaching the probe to the sample by Pt deposition is performed with the condition: $30kV$, $0.1nA$, $X = 1.69\mu m$, $Y = 4.09\mu m$, $Z = 1.0\mu m$ using *Pt* gas for 2 minutes (Figure 16).



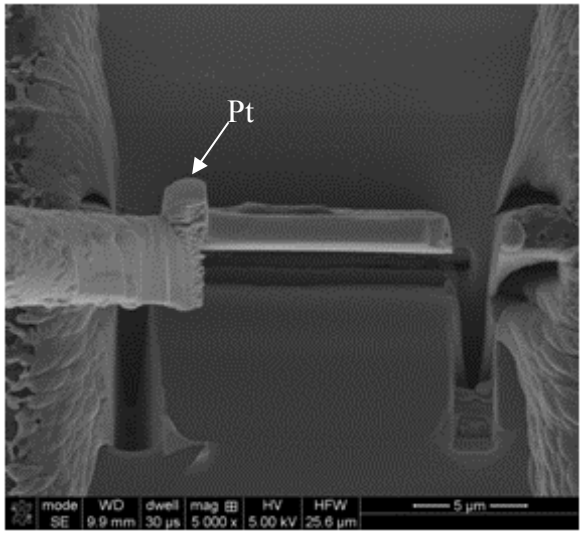
(a) Top view SEM image.



(b) Tilted view FIB image with the marking for *Pt* deposition.



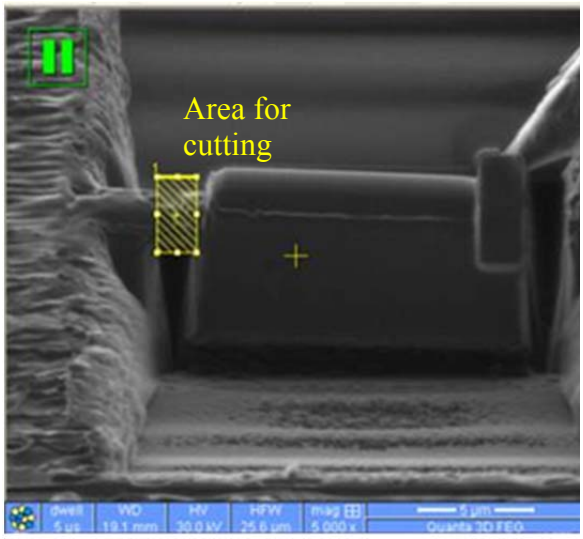
(c) Tilted view FIB image after deposition.



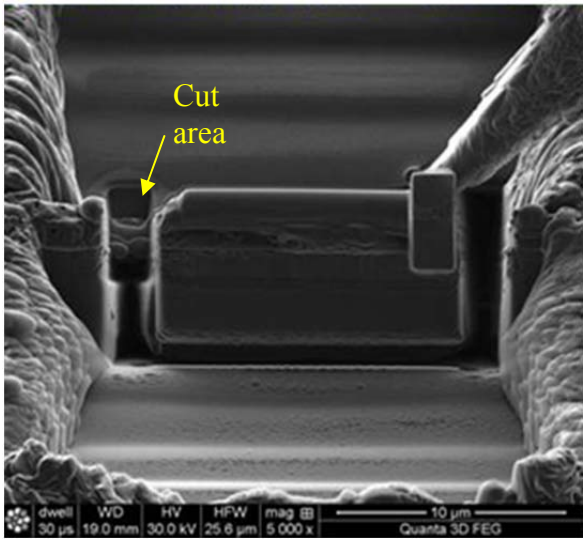
(d) Top view SEM image of figure (c) after *Pt* deposition.

Figure 16. Images show *Pt* deposition.

- Cut the remaining side wall of the sample ($30kV$, $5nA$) as shown in Figure 17.



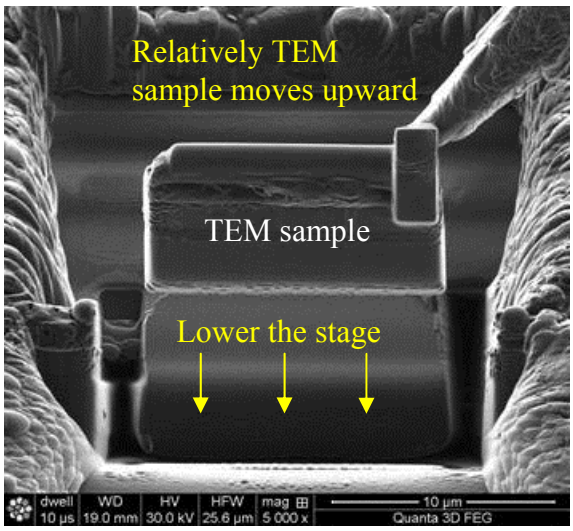
(a) FIB image with a marking for cutting the remaining side of the sample.



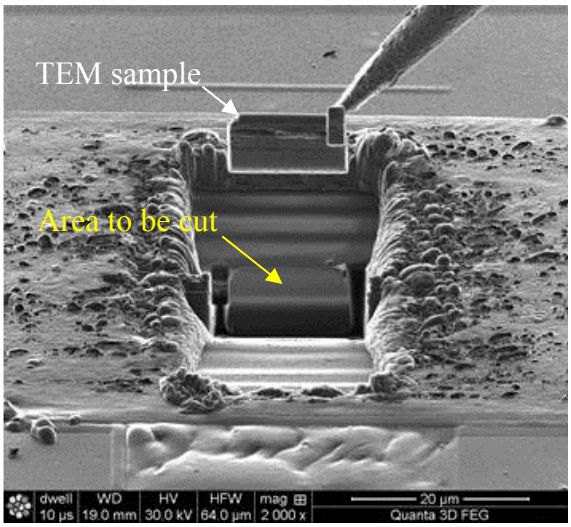
(b) FIB image after cutting the side.

Figure 17. Before and after cutting.

- Lower the stage carefully after cutting the remaining side wall (Figure 18).
- Move omniprobe to a safe location, the proper place that sample and stage don't collide with each other when stage moves. (Figure 19).



(a) FIB image.



(b) Low magnification FIB image.

Figure 18. Lifting the sample.

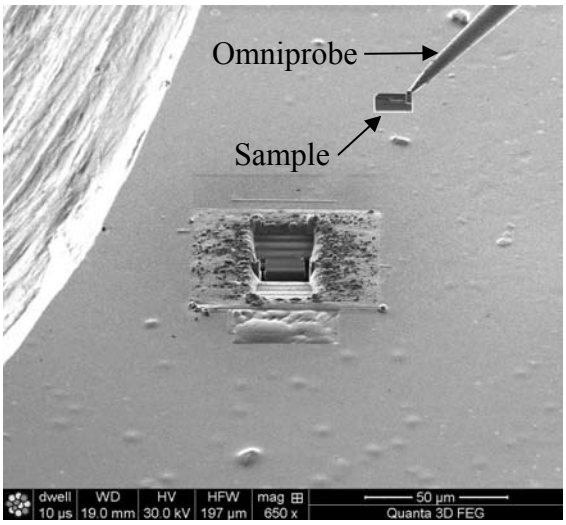


Figure 19. Image after omniprobe is moved to a safe location.

- Remove omniprobe and GIS (Figure 19). Move the stage to the place where the grid is located to attach the sample to the grid (Figure 20).

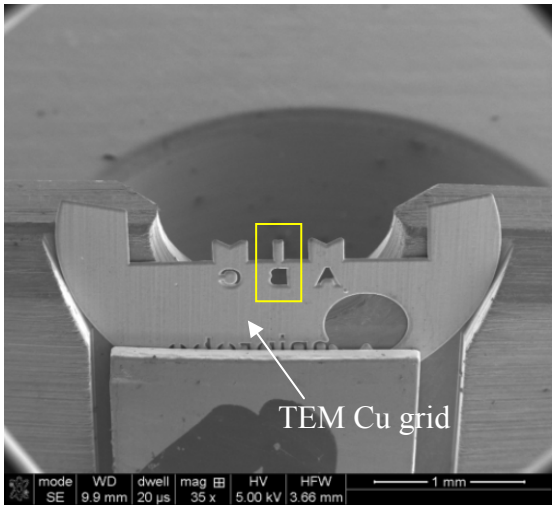
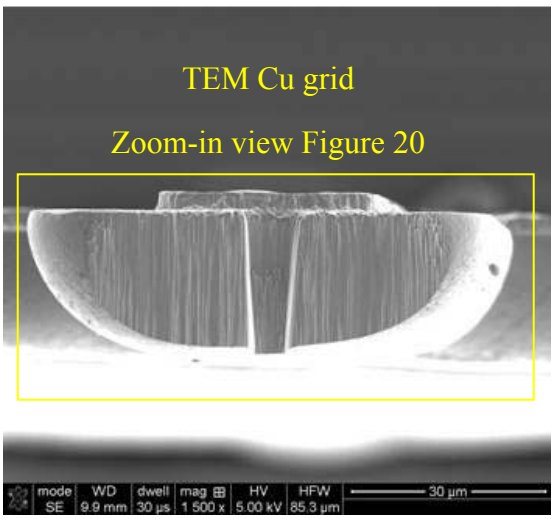
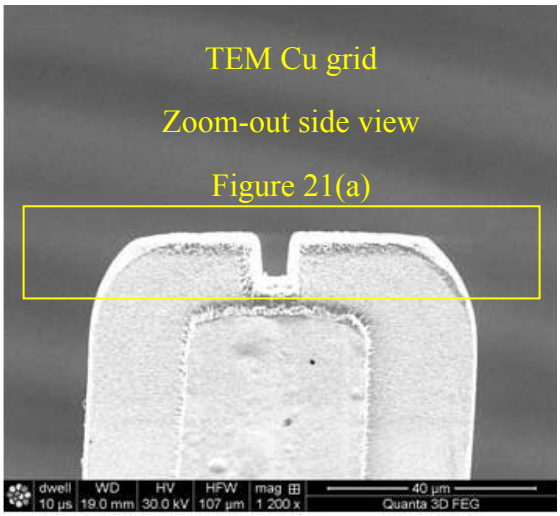


Figure 20. Image of Cu-3-post grid.

8. Remove a part of the top of the grid to prevent *Cu* redeposition from grid when milling the sample using ion-beam.
    - The etching width should be less than the lateral width of the sample (Figure 21).





(a) Top view Fig. 20. **(b) Side view Fig. 21a.**

Figure 21. Image after partially removing the top of TEM grid to prevent redeposition during the FIB sample milling.

- To attach the sample to the grid, insert the omniprobe and GIS (*Pt*) (Figure 22).

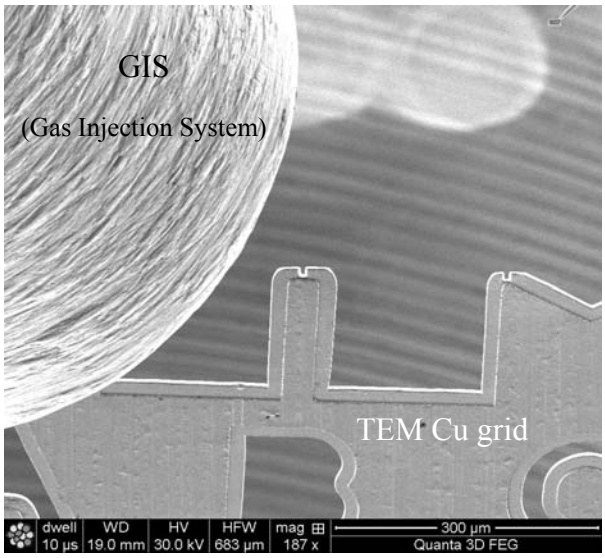


Figure 22. Image after inserting the GIS near the TEM grid for attaching the sample to the grid.

9. Mount the cut sample on top of the grid using the omniprobe (Figure 23).

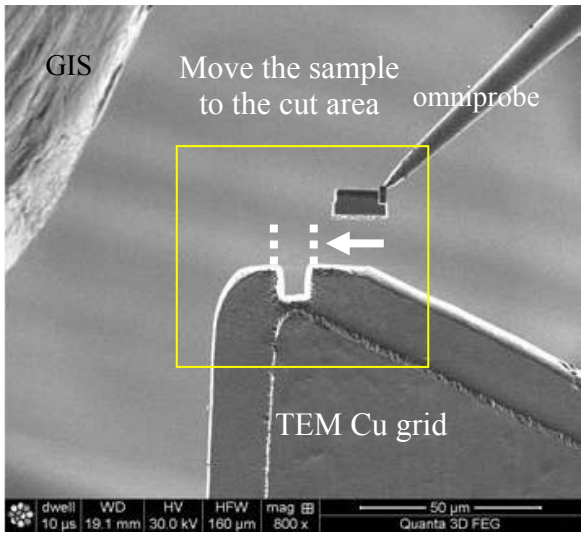
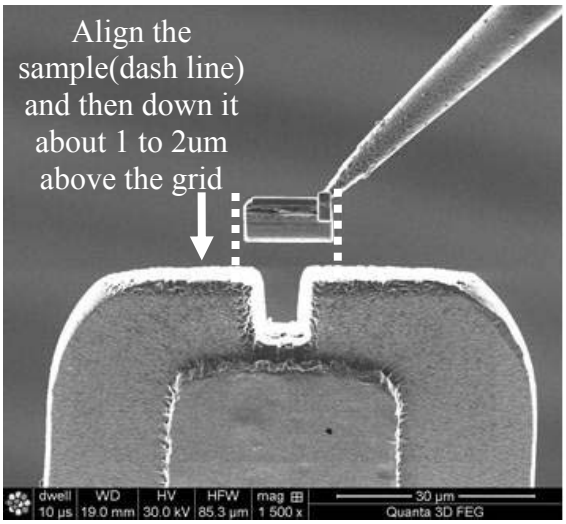
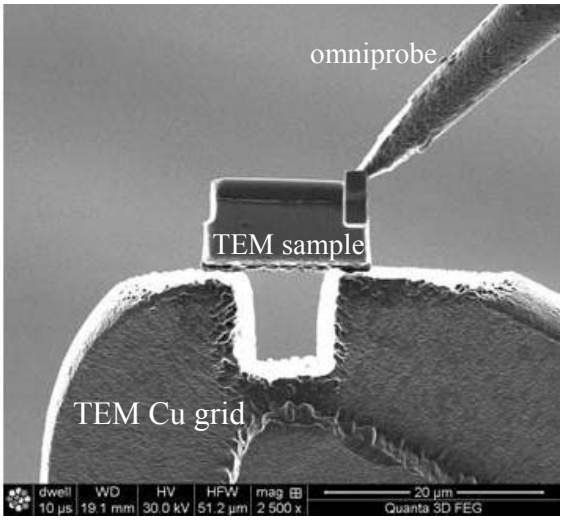


Figure 23. Image showing the sample as it is placed on top of the grid with the omniprobe.

- Locate the sample about 1 to 2μm above the grid to facilitate deposition (Figure 24).



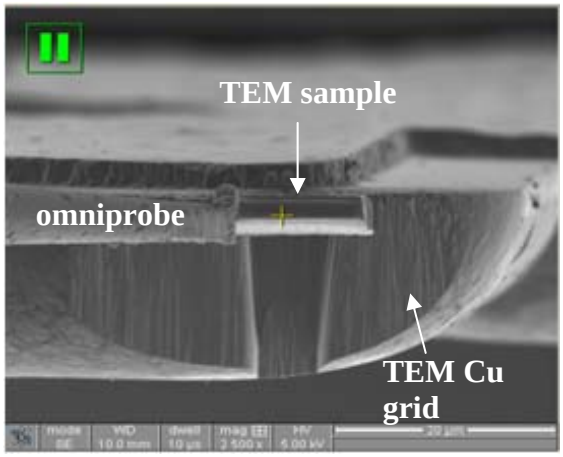
(a) The sample near the top of the grid.



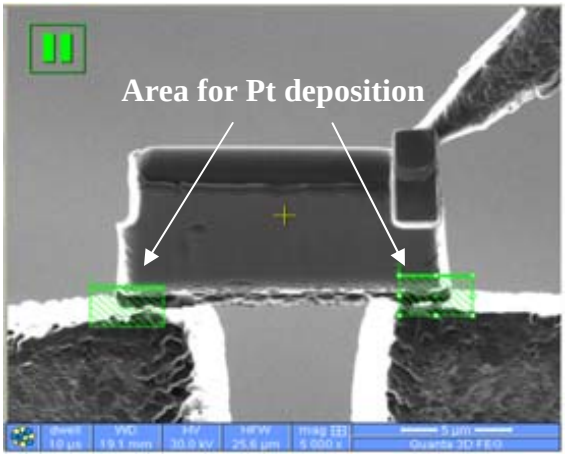
(b) The sample about 1 to 2μm above the grid.

Figure 24. Images showing the placement of the sample.

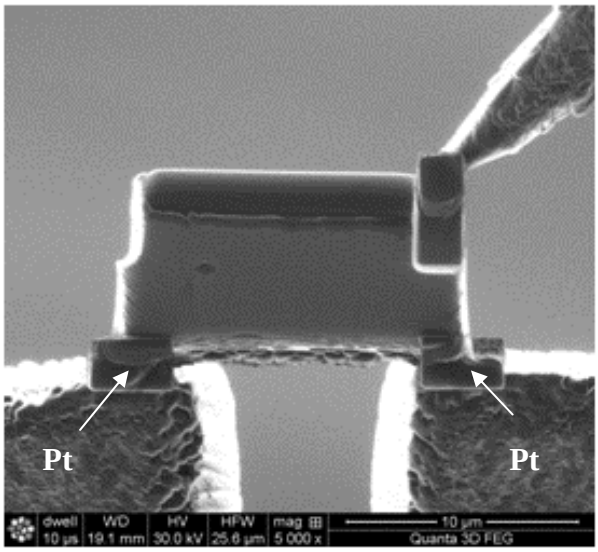
- Attach the sample to the grid by deposition (30kV, 0.1nA, $X = 3.40\mu m$, $Y = 2.10\mu m$, $Z = 1.00\mu m$ using Pt gas for 2 to 3 min). Figure 25 shows the process.



(a) Sample well aligned with the TEM grid.



(b) Markings on the sample for *Pt* deposition to attach the sample to the grid.



(c) After *Pt* deposition.

Figure 25. The process of *Pt* deposition.

- Remove the omniprobe from the sample by cutting the connection between the sample and omniprobe (30*kV*, 1*nA* for 2 min) (Figure 26).

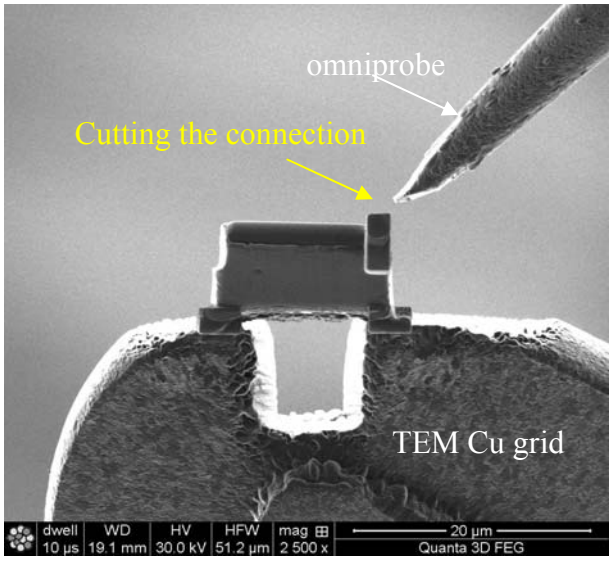
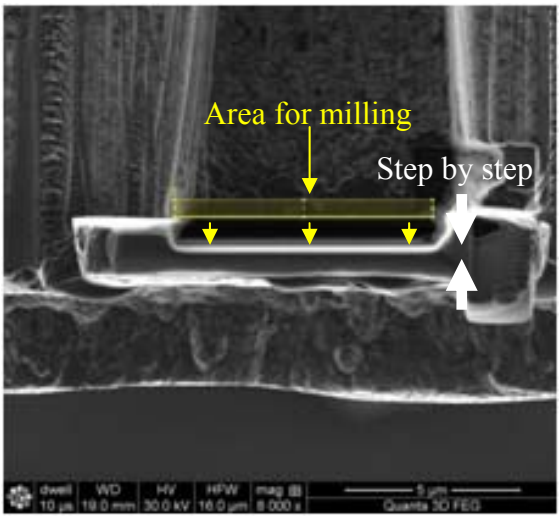
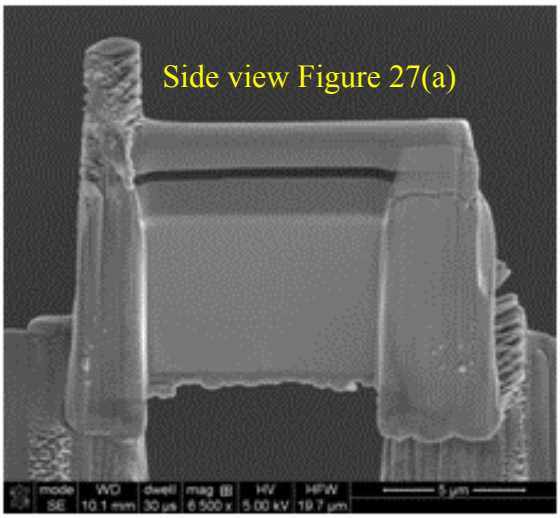


Figure 26. Image after removing omniprove from the sample.

- Move the omniprobe to a safe location. To do the final milling, the tilt = 52°. After tilting the sample ±1.3 degrees (for top side : 53.3 and for bottom side : 50.7 degrees), perform the milling at 30*kV*, 1*nA*. Continue until the thickness of the sample comes into the range of 200 to 300nm (Figure 27).

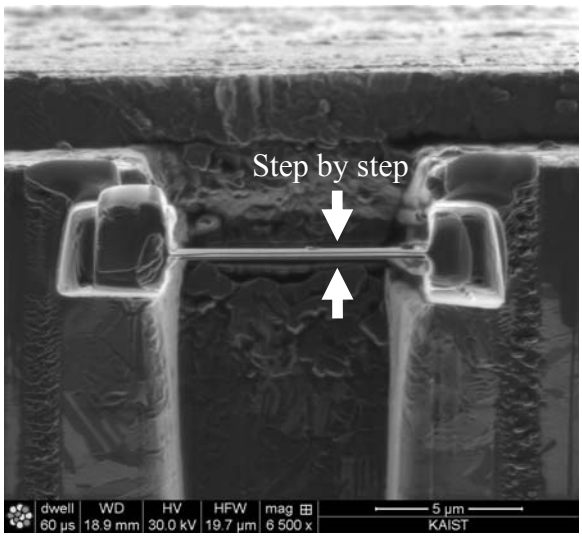


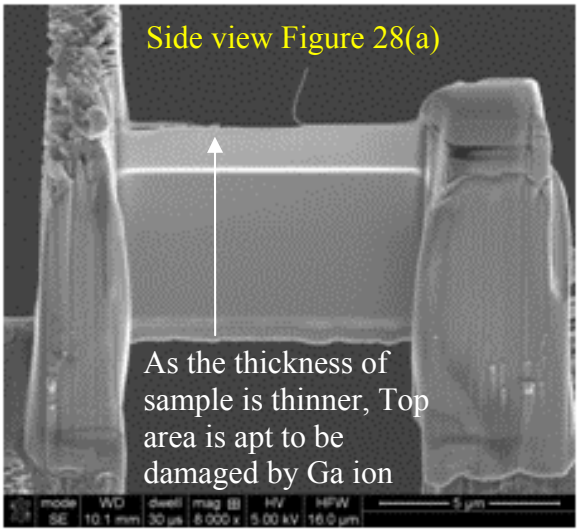


(a) Top view FIB image. **(b) SEM image.**

Figure 27. Performing the milling.

- After tilting ±0.8 degrees (for top side : 52.8 and for bottom side : 51.2 degrees), perform the milling at 30*kV*, 100*pA*. Continue until the thickness comes into the range of 40 to 70nm (Figure 28).





(a) FIB image. **(b) SEM image.**

Figure 28. The sample with less than 100nm thickness.

- Figure 29 shows the TEM low magnification image after making the sample using FIB.

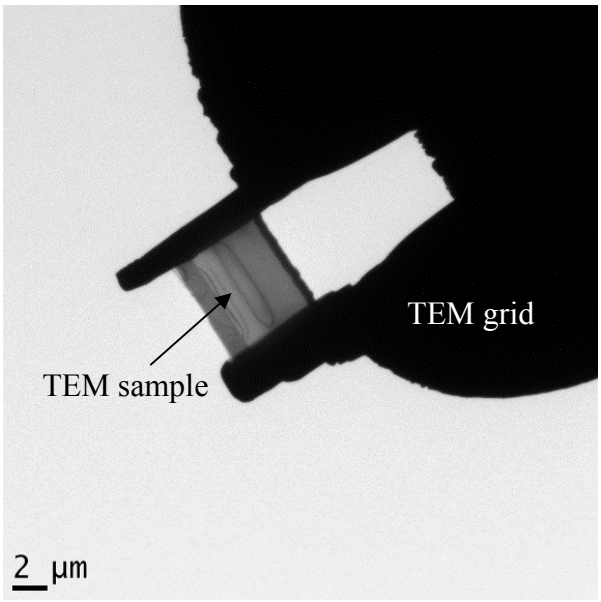
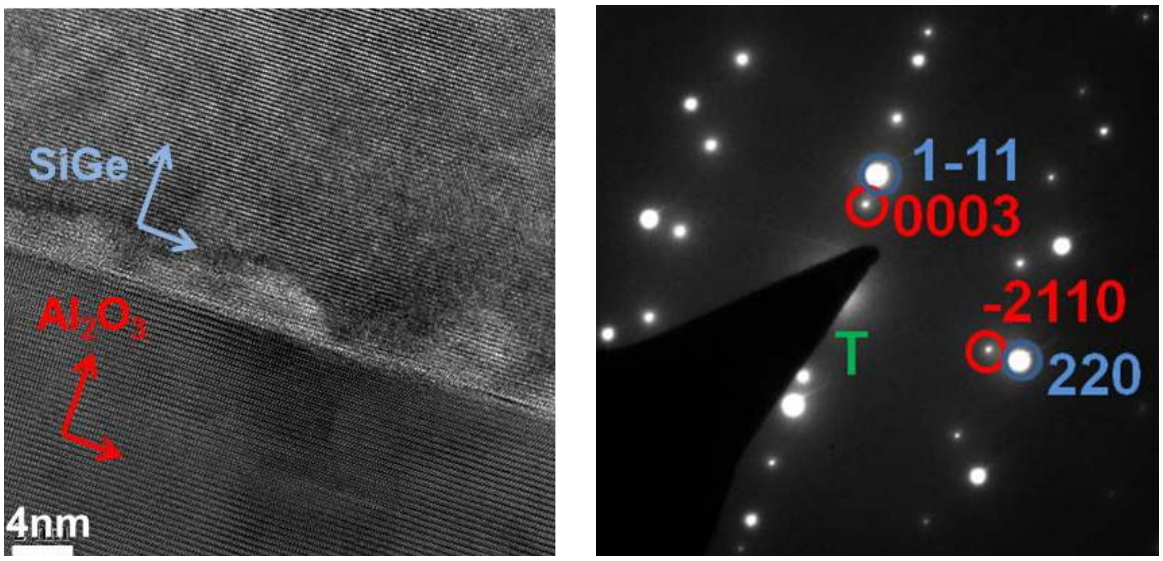


Figure 29. Low magnification TEM image.

- From the SAED pattern, the epitaxial relationship between the majority of *SiGe* film and sapphire substrate was found to be (111) $Si_{0.2}Ge_{0.8}$ // (0001) sapphire and [-112] $Si_{0.2}Ge_{0.8}$ // [01–10] sapphire (Figure 30).



(a) HRTEM. **(b) Diffraction pattern result.**

Figure 30. HRTEM and its diffraction pattern result of the $Si_{0.2}Ge_{0.8}$ / sapphire interface.

TEM Sample Preparation of $Si_{1-x}Ge_x$ on Sapphire (Al_2O_3) Substrate Using the Tripod Method

The use of the tripod allows the polishing of the sample to be faster than with the traditional preparation, with the resulting image being of higher quality. The total sample preparation time was approximately 3 hours, with the ion milling comprising over half of the time.

1. Prepare the sample.
    - Cut slices of the $Si_{1-x}Ge_x$ / sapphire into samples, each nor greater than 2.4 mm width (for a 3 mm slotted grid) and 4–6 mm length along the [01–10] directions (Figure 31).

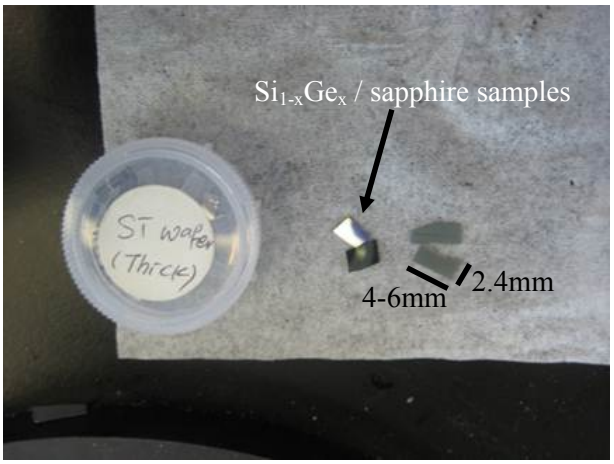


Figure 31. Cutting slices of 2.4 mm width and 4–6 mm length out of the $Si_{1-x}Ge_x$ / sapphire wafer.

- Clean the pieces with deionized (DI) water, acetone, and *iso*-propanol, then put the pieces on a hot plate at 100 degrees Celsius (C) to evaporate any solvent residue.

2. Prepare the stack.
   - Prepare the epoxy. Using G1 from Gatan which permits a very thin film of glue and becomes very hard after the polymerization. It is also relatively stable under the electron beam.
   - The standard ratio between the hardener and the resin of G1 is 1:10 (Figure 32). At 120 degrees C, the hardening time is about 10 minutes. A good way to find out when the epoxy glue has hardened is to put a small drop of epoxy next to the stack on the hot plate. The color of the epoxy will change from transparent yellow to brown when it is fully hardened.

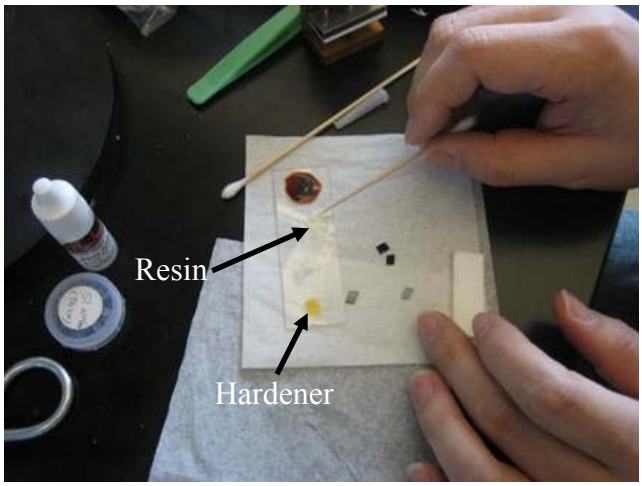


Figure 32. Mix the hardener and resin in a ratio of 1:10.

- Spread the glue on a smooth, clean surface with a wooden toothpick. Place the mating surfaces of the sandwich on the glue, taking care to obtain a very thin film of glue (Figure 33).

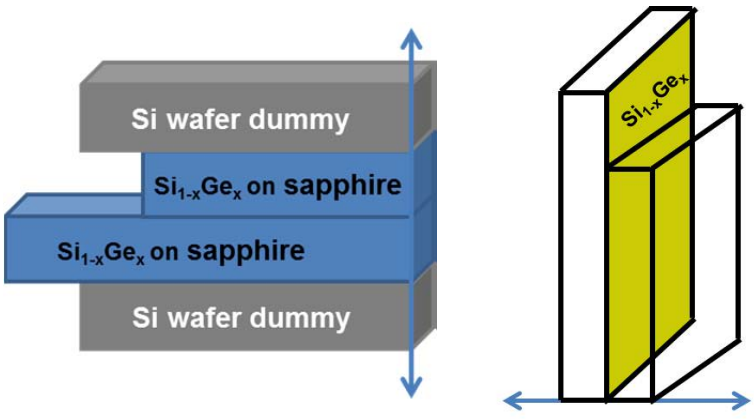
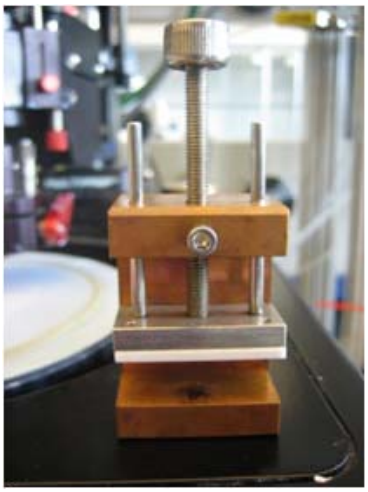
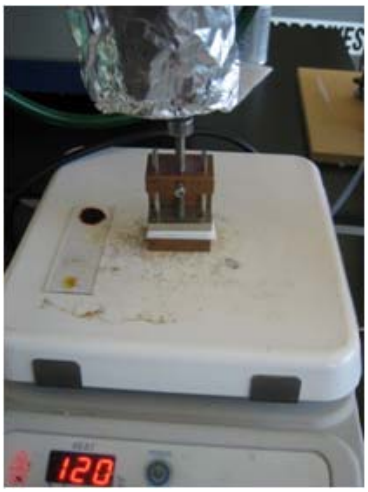


Figure 33. Schematic illustration of the forming a sandwich (stack) by slices of $Si_{1-x}Ge_x$ / sapphire and *Si* dummy slices with the same level of the one side (marked by arrow).

- Stick the cutting pieces of the $Si_{1-x}Ge_x$ / sapphire together, taking care that at least one side at each end is at the same level. A *Si* wafer dummy is needed for the front and back sides of the sapphire wafer since the sapphire wafer is transparent, one cannot estimate the thickness after polishing. The attached *Si* dummy wafer is used to measure the polishing rate.
- Place the stack in a press using two Teflon® adhesive sheets to prevent the sample from being glued to the press.
- Heat the stack in the clamping fixture for a minimum of 10 minutes (Figure 34).



(a) Clamping fixture with Teflon® adhesive sheets.

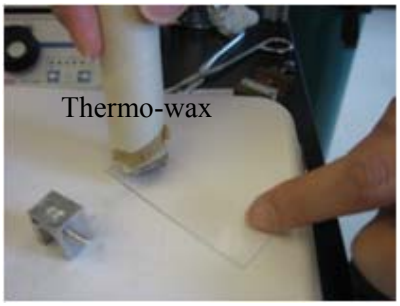


(b) Stack in a clamping fixture on hot plate at 120°C.

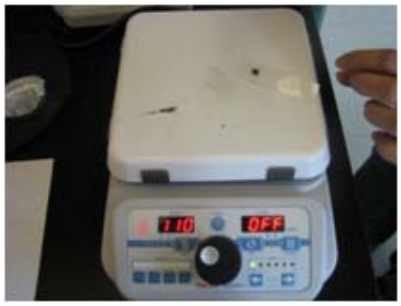
(c) The clamping fixture heated for 10 minutes at 120°C.

Figure 34. The heating process.

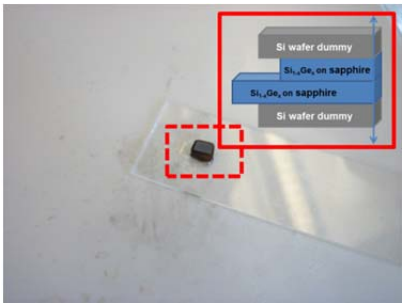
3. Mount the sample on the tripod.
   - Place the glass on a hot plate (120 °C). Once the glue is liquefied, attach the sample on the glass with a QuickStick™ 135 Temporary Mounting Wax, used to bond samples during processing (Figure 35).



(a) Applying thermo-wax.



(b) Slide on a hot plate.



(c) Stack with thermo-wax on the glass.

Figure 35. Coating stack with thermo-wax.

- When the thermo-wax liquefies (after a few seconds on the hot plate), the stack will sink into the QuickStick™ 135 Temporary Mounting Wax (thermo-wax). If

the stack does not sink into the thermo-wax, apply a small amount of pressure on the stack.

- Attach the glass, with the thermo-wax covered stack, to the aluminum holder. Using a diamond saw, cut disks of about 300–400 μm thickness (Figure 36). Apply the thermo-wax all around the glass when mounting it on the *Al* holder to ensure adhesion between the two. The speed of the diamond saw should be kept low.

(a) Mounted on the diamond saw. **(b) Disks of about 300–400 μm thickness.** **(c) Disk under the microscope.**

Figure 36. Results of cutting disks with the diamond saw.

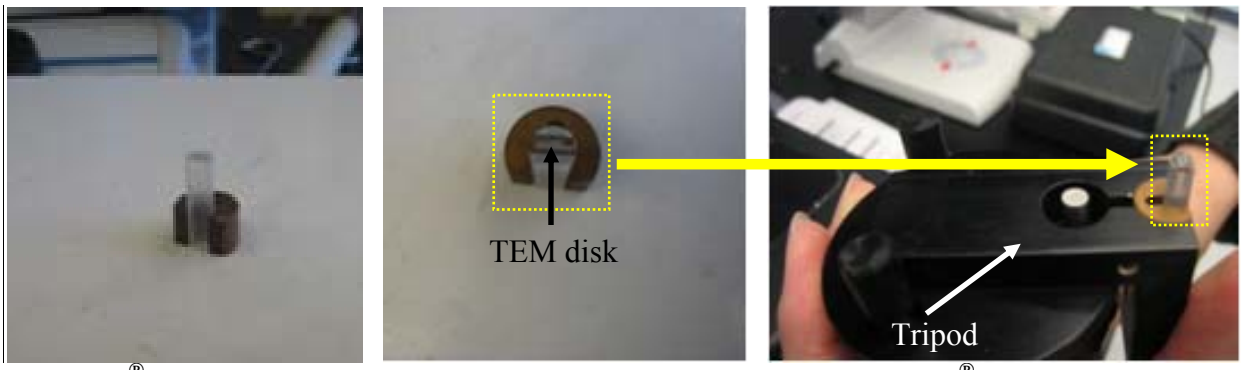
4. Polish the sample.
   - Lay down the 35μm diamond lapping film (Figure 37) on the wet polishing machine plate and remove the water from between the polishing plate and the lapping film using a squeegee until the lapping film adheres completely to the plate.

Figure 37. Diamond lapping films.

- Select a Pyrex® cylinder to use as a support for the disk, and measure its height with a micrometer.
- Attach one disk with thermo-wax on top of the Pyrex® cylinder.

Mount the Pyrex® cylinder with the stack sample (disk) in the tripod (

- Figure 38).



(a) Pyrex® cylinder to use as a support for the disk.

(b) Attached disk with thermo-wax on top of the Pyrex® cylinder.

(c) The Pyrex® cylinder with the stack sample mounted on the tripod.

Figure 38. Sample mounted on tripod.

- Put the tripod on the disc by placing the rear micrometers on it and gently lowering the sample, keeping the glue-line parallel to the direction of motion.
- Polish the sample in a spiral motion from inside to outside with the 35μm diamond lapping film until the entire face of each of the two pieces is abraded. This can be seen by the homogeneity of the scratches induced on the surface of the sample. Take care not to use the same area of the lapping film more than once. When the whole surface of the film has been used, clean it with a wet, lint free paper towel.
- Measure the thickness of the sample either by using the focus indicator of the microscope or by installing a digital thickness indicator on the microscope. Use the attached *Si* dummy wafer to measure the polishing rate since the sapphire wafer is transparent.

5. Glue the TEM grid on the TEM sample and polish the second side (Figure 39 and Figure 40).
   - Glue the TEM grid on the polished, mirror-like surface of the sample. Mount the sample with thermo-wax on the Pyrex® cylinder of the tripod for polishing the second side. Adjust the two rear micrometers to obtain the desired angle.
   - For the second side, measuring the thickness of the sample is very important and must be done periodically (once every 20 seconds). This is carried out using an optical microscope with the *Si* dummy samples. Polish with a 35μm and 30μm diamond lapping film until the the sample is 170μm thick. Polish parallel to the glue line, the thin zone being at the tapered corner.
   - Polish with a 15μm diamond lapping film until the thickness is 80μm.
   - Polish with a 6μm diamond lapping film until the thickness is 30μm.
   - Polish with a 3μm diamond lapping film until the thickness is about 10μm.
   - Polish with a 1μ, 0.5μ, and 0.1μm diamond lapping film until the appearance of interface fringes at the side of the sample.

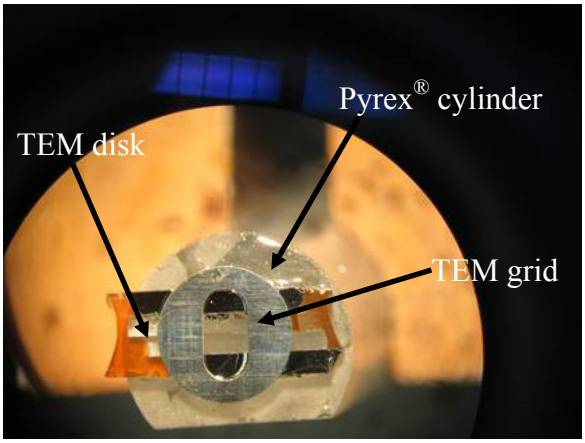


Figure 39. Microscope image of the TEM grid on the polished, mirror-like surface of the sample mounted on the Pyrex® cylinder with thermo-wax.

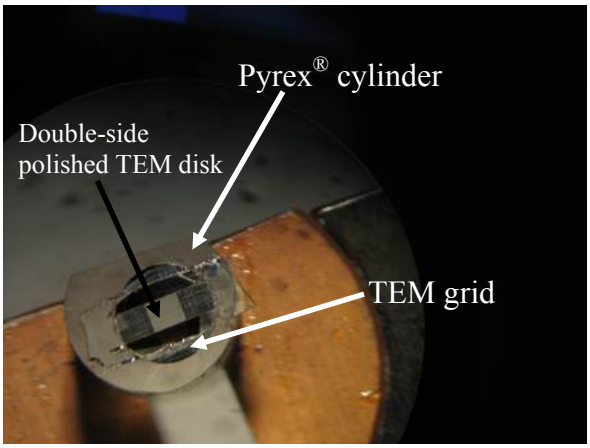
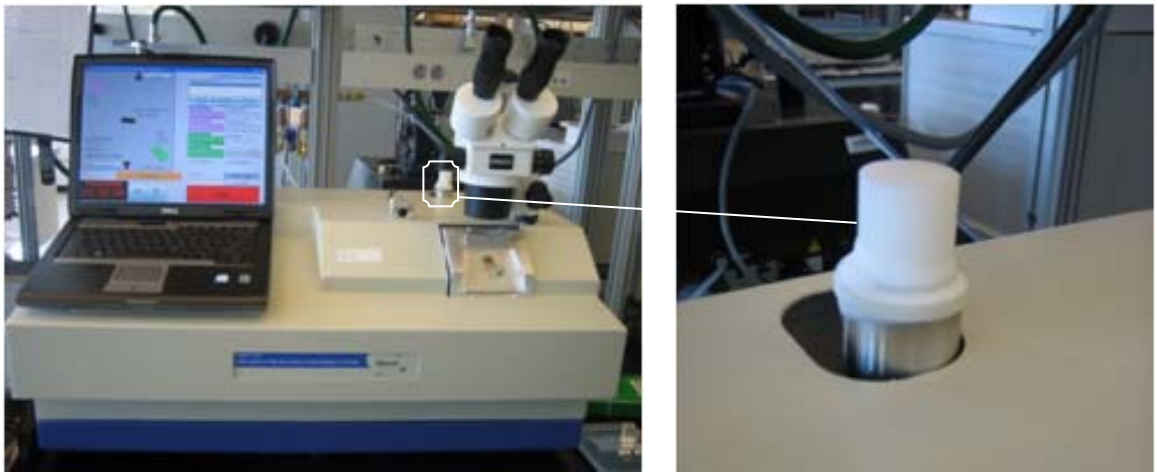


Figure 40. Microscope image of double-side polished surface of the sample with TEM grid.

6. Clean the sample.
    - Clean the sample mounted to the TEM grid with acetone, followed by an ethanol or trichlorethylene bath (all solvent must be of analysis quality) to remove the thermo-wax from the sample and separate the sample from the Pyrex® cylinder. Put the TEM sample on a hot plate at 100 °C to evaporate the solvent residue.

7. Ion milling the sample (Figure 41).
    - A low angle ion milling and polishing system (Fischione Instrument Model 1010), was used for the ion milling process. The parameters for the ion milling process are rather specific for each material and have to be optimized. As ion milling rate increases with a higher etching angle and higher etching voltage; the sample is more severely damaged. Therefore, the angle, as well as the voltage, should be kept rather low. Generally, a higher voltage combined with a lower angle is less harmful to the sample than a lower voltage combined with a higher angle.
    - Add liquid nitrogen to cool the ion miller since the sample becomes severely heated during ion milling.



(a) A low angle ion milling and polishing system for the ion milling process. **(b) Liquid nitrogen sample cooling to eliminate artifacts.**

Figure 41. Ion milling system.

- Mount the polished sample (TEM sample) on the ion milling sample holder (Figure 42).

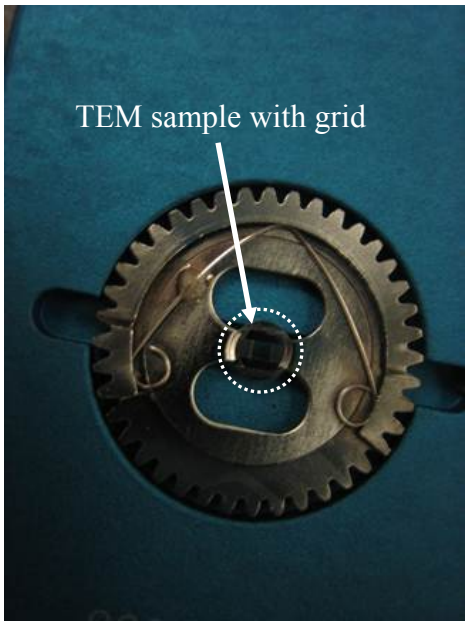


Figure 42. The TEM sample on the ion milling sample holder.

- Make sure that the ion milling machine is turned on approximately 1 hour before beginning the ion milling in order for it to reach stable, high-vacuum conditions.
- Mount the sample holder into the milling machine.
- Close the cover and create a vacuum of the chamber.
- Set the turning velocity, ion milling angle, ion milling voltage, and duration of the ion milling.
- Start the ion milling process (Figure 43).

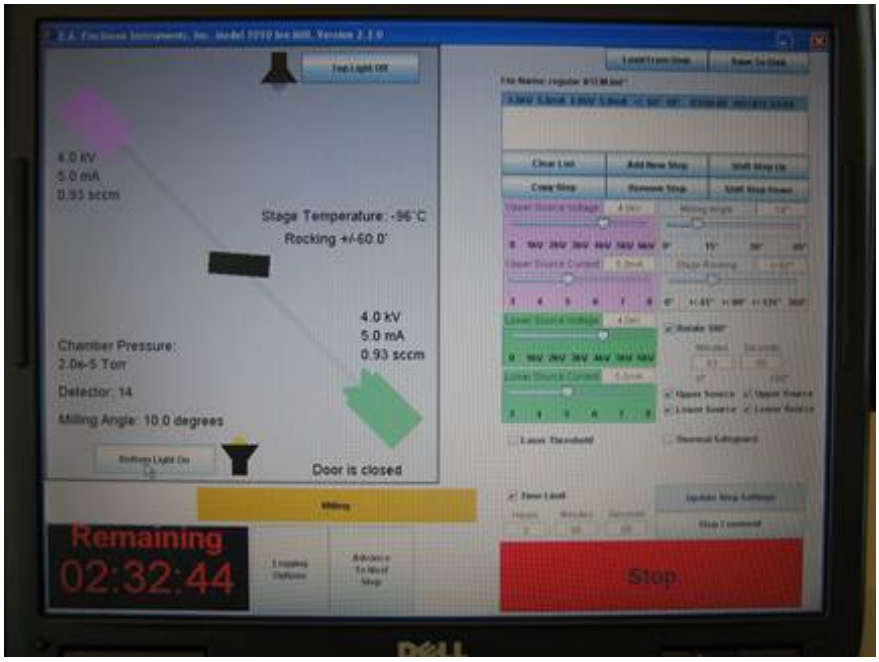


Figure 43. Screen shot of the ion milling setup.

8. Finalize the TEM sample (Figure 44 and Figure 45).
    - A TEM sample is least contaminated directly after the ion milling process. If necessary, you can remove some contamination by the short plasma-clean prior to the TEM investigation.
    - From the diffraction pattern, the epitaxial relationship between the majority of the SiGe film and the sapphire substrate was found to be (111) $Si_{0.2}Ge_{0.8}$ // (0001) sapphire and [–112] $Si_{0.2}Ge_{0.8}$ // [01-10] sapphire.

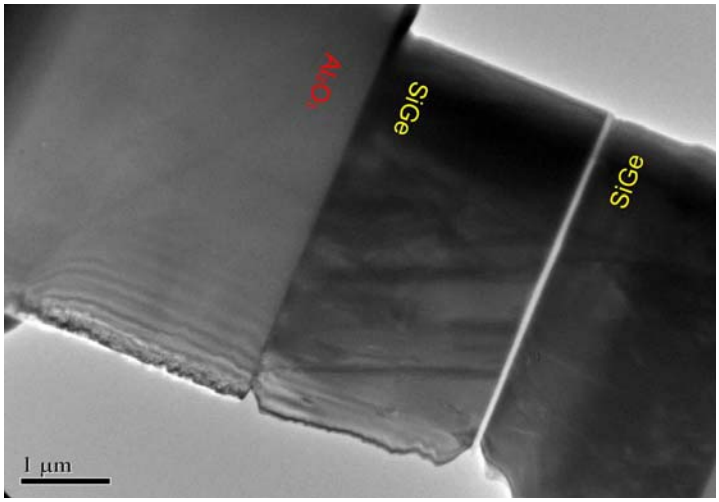
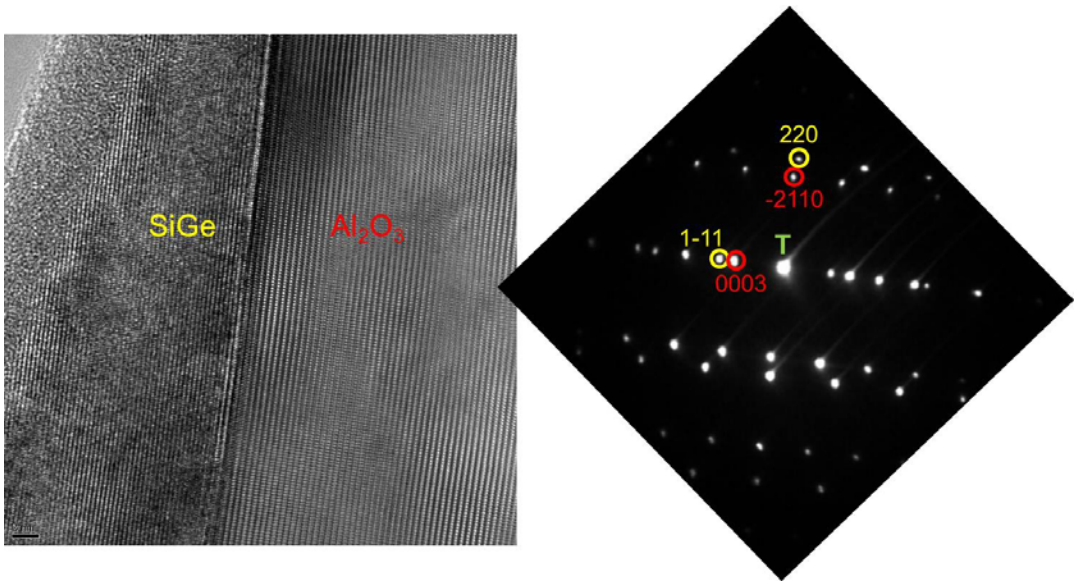


Figure 44. Low magnification TEM image after the ion milling process.



(a) HRTEM. **(b) SAED pattern.**

Figure 45. Results of the $Si_{0.2}Ge_{0.8}$ / sapphire interface.

Conclusion

In high resolution transmission electron microscopy, the quality of the TEM samples becomes a limitation factor, and, with the realization of aberration corrected TEMs, this effect is more prominent. Thus, it is crucial that the preparation does not cause any degradation of a sample used in a TEM study. The conventional argon-ion milling scheme can leave an amorphous surface layer on the sample with a thickness up to 10 nm. Such alteration leads to a deterioration of the electron channeling along atom columns and, hence, a decreased contrast in atomic resolution scanning TEM. The preparation of a TEM sample should be as fast as possible, and the specimen should contain few or no artifacts induced by the preparation. The standard sample preparation method of mechanical polishing often requires a relatively long ion milling time, which increases the probability of inducing defects into the sample and the amorphous layer on the surface of the sample. Promising approaches are to apply the FIB and tripod polishing methods in the sample preparation procedure. The use of the FIB or tripod methods allows faster polishing of the sample than with traditional preparation and obtains excellent image quality as well.

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14. ABSTRACT

The National Aeronautics and Space Administration-invented X-ray diffraction (XRD) methods, including the total defect density measurement method and the spatial wafer mapping method, have confirmed super hetero epitaxy growth for rhombohedral single crystalline silicon germanium (Si1-xGex) on a c-plane sapphire substrate. However, the XRD method cannot observe the surface morphology or roughness because of the method's limited resolution. Therefore the authors used transmission electron microscopy (TEM) with samples prepared in two ways, the focused ion beam (FIB) method and the tripod method to study the structure between Si1-xGex and sapphire substrate and Si1-xGex itself. The sample preparation for TEM should be as fast as possible so that the sample should contain few or no artifacts induced by the preparation. The standard sample preparation method of mechanical polishing often requires a relatively long ion milling time (several hours), which increases the probability of inducing defects into the sample. The TEM sampling of the Si1-xGex on sapphire is also difficult because of the sapphire's high hardness and mechanical instability. The FIB method and the tripod method eliminate both problems when performing a cross-section TEM sampling of Si1-xGex on c-plane sapphire, which shows the surface morphology, the interface between film and substrate, and the crystal structure of the film. This paper explains the FIB sampling method and the tripod sampling method, and why sampling Si1-xGex, on a sapphire substrate with TEM, is necessary.

15. SUBJECT TERMS

Focused ion beam; Sapphire; Substrates; Transmission electron microscopy; Tripod

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