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Failure Analysis of Electronic Material Using Cryogenic FIB-SEM

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Abstract

Two-beam systems (focused ion beam (FIB) integrated with a scanning electron microscope (SEM)) have enabled site-specific analysis at the nano-scale through in situ "mill and view" capability at high resolution. In addition, a FIB-SEM can be used to cut away a lamella from a bulk sample and thin it for transmission electron microscopy (TEM) imaging. We studied the temperature dependence of FIB milling on compound semiconductors and thin films such as copper that are used in integrated circuits. These materials (GaAs, GaN, InN, etc) react chemically and physically with the gallium in the FIB and change chemical composition and may also change morphology. Copper metallization of IC's has been difficult to mill without undesirable side effects. FIB milling for analysis of these materials becomes difficult if not impossible. Since temperature can be a big factor in chemical and physical reactions we investigated this and report here the effect of cooling the sample to cryogenic temperatures while milling. In addition, we report on the development of a process to prepare TEM lamellae with FIB entirely in a cryogenic environment.

Introduction

Cryogenic Electron Microscopy (EM) for water-containing samples (mostly organic materials) has been possible for many years [1]. One way to fix a hydrated sample for a vacuum environment is to freeze it at cryogenic temperatures (< 150 K). Rapid freezing can preserve the structure of water-containing samples by converting the water to vitreous ice without any crystal formation (water crystals can damage cell walls or the overall structure of a sample) [2]. Two-beam systems (focused ion beam (FIB) integrated with a scanning electron microscope (SEM)) have enabled site-specific analysis at the nano-scale through in-situ "mill and view" capability at high resolution. However, FIB and FIB-SEM cryogenic work is relatively new and has mainly focused on organic material [3]. We have developed a process based on a minimally modified commercially available system to address a class of inorganic materials that are adversely affected by the gallium-based FIB irradiation (30 keV gallium beam) at room temperature. While this process will also work for organic material, this paper is concerned with electronic materials only. Many compound semiconductors (GaAs, GaN, InN, etc) and some metals used for interconnect in circuits react chemically and physically with the gallium in the FIB and change chemical composition and structure [4]. FIB milling for analysis of these materials becomes difficult if not impossible (Figure 1). Copper used for integrated circuit (IC) interconnect and other electronic systems is also problematic to FIB milling. The effect of milling copper at cryogenic temperature is also reported here.

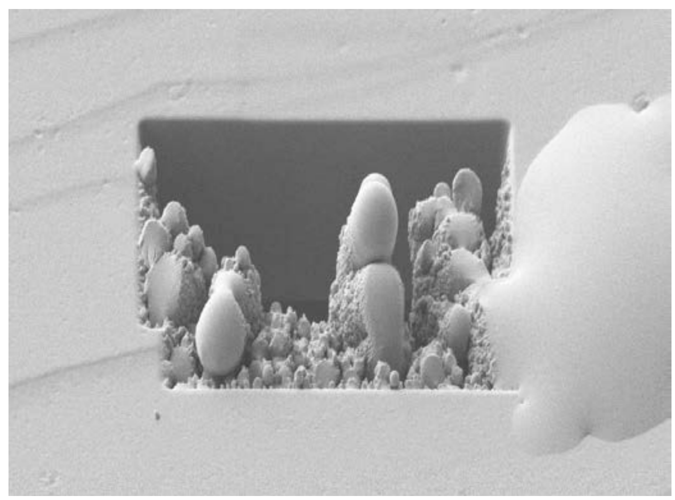


Figure 1. Result of FIB Exposure of GaN at room temperature (30 keV Ga^+, Dose: 5.5nC/μm^2).

We have developed hardware and processes that allow us to work on gallium-sensitive materials with significantly reduced side effects. The temperature control required by the class of materials described in this paper is much less stringent that required for artifact-free EM of hydrated samples. For GaN, for example, the chemical reaction slows down significantly at about 223 K and is barely visible at 173 K. The last part of the paper describes the development of a cryogenic process for TEM sample preparation using FIB and a modified tip for lift-out in a FIB-SEM with a cryo-stage (Fig. 2). A vacuum cryo-transfer module was used in the transport of the sample between equipment as shown in Figures 3 and 4.

Hardware

Minor modifications were made to existing commercial equipment to enable cryogenic FIB-SEM sample preparation for TEM cryo-imaging. The equipment used consists of a Zeiss CrossBeam retrofitted with a Leica cryo-stage (Fig. 2) and docking station for the Leica EM VCT 100 shuttle (Fig. 3). We also used the Leica EM VCT100 shuttle (Fig. 4) and a Leica cryo-loading chamber for sample transfer.

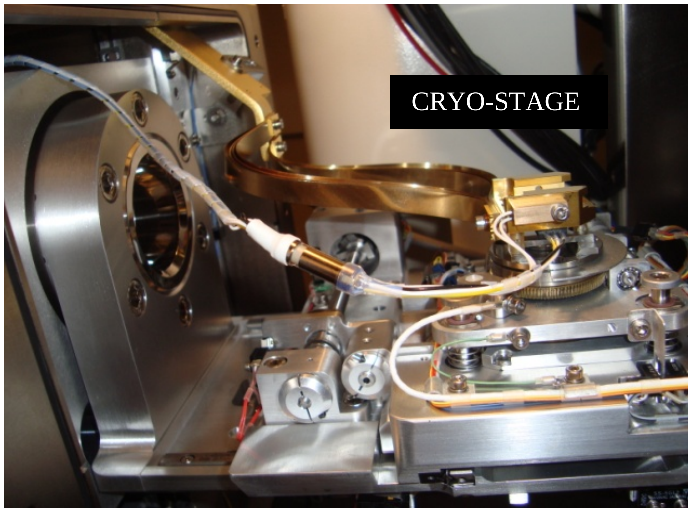


Figure 2: Cryo-stage mounted on the Crossbeam stage with copper cooling bands and wiring in place.

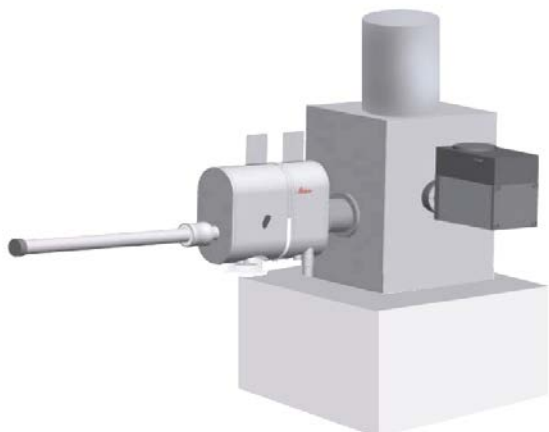


Figure 3: Drawing of shuttle docked to FIB-SEM for sample load and unload.

Figure 4: Shuttle with cryogenic and vacuum capability for transfer to and from FIB-SEM and LN_2 chamber.

To aid cryo-lift-out, custom modifications were made to an existing Omniprobe nano-manipulator. Figure 5 shows the modifications, which consist of a custom probe shaft equipped with a thermally isolated copper "probe tip gripper". A probe tip of the type normally used for the Omniprobe AutoProbe 300 in situ tip exchange is firmly held in the probe tip gripper jaws. The probe tip gripper is affixed to a copper coupling, the other end of which is attached to the main probe shaft body via a small, rigid, ceramic tube. The ceramic tube offers thermal isolation from the heat sinking mass of the stainless steel probe shaft.

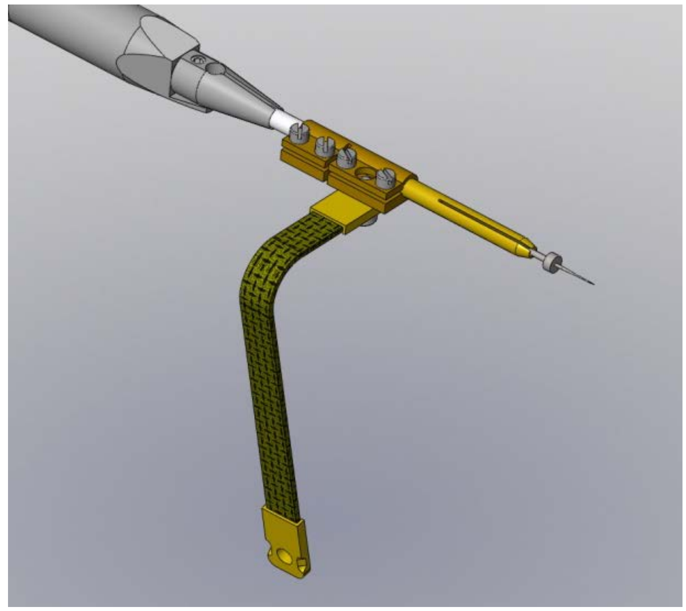


Figure 5: Drawing of modified probe tip for cryo-lift-out.

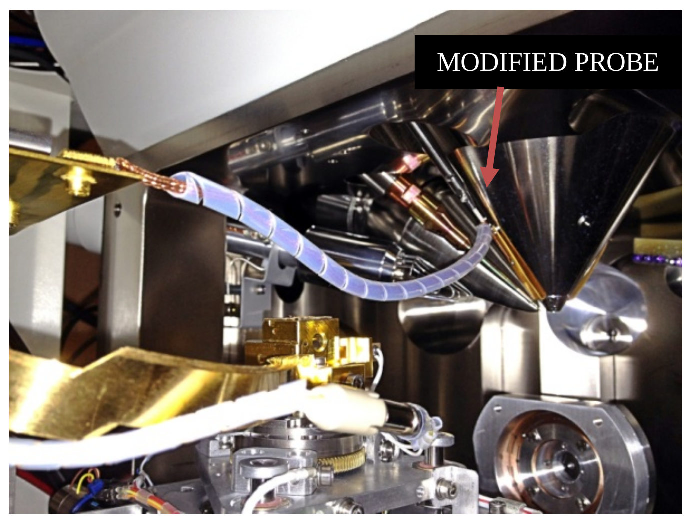


Figure 6: Modified probe tip installed along with cryo-stage

A flexible copper braid tethers the copper coupling to a cryogenically cooled "cold finger" (Fig. 5). The flexible copper tether allows free movement of the probe tip while proving a thermal path for the cold finger to cool the probe tip to cryogenic temperatures. The tether is electrically and thermally isolated from its surroundings by a TPFE spiral wrap (Fig. 6).

Effect of Temperature on Compound Semiconductor Milling

It has been reported that gallium in the FIB reacts with certain compound semiconductor materials and alters them chemically and structurally [4]. Specifically, III-V compound semiconductors undergo preferential sputtering after Ga^+ FIB exposure that removes the group V element leaving behind almost pure group III material. The low melting point of these group III metals is why the observed result appears as droplets of material (Fig. 1).

A 5 µm thin film of GaN on sapphire was used to evaluate the temperature effect of FIB milling on this semiconductor material. Other compounds have been studied and behave similarly. Although our hardware is capable of near liquid nitrogen sample temperature, for throughput and the possibility of using a simpler cold stage (such as a thermoelectric system) a warmer range of temperatures was chosen. In Table 1, we show the effect of temperature as a qualitative measure of material alteration as severe, intermediate, minimal and none observed.

Table 1: Effect of temperature on FIB milling of GaN.

Temperature (K)	Exposure ($nC/\mu m^2$)	Side Effect (Qualitative)
300 K	5.5	Severe
248 K	5.5	Severe
223 K	5.5	Intermediate
173 K	5.5	Minimized
128 K	5.5	None Observed

At room temperature, the effect is so pronounced that the area to be milled is full of spheres of post-exposure material (Fig. 1). At just 25 degrees below freezing, the undesirable effect is only present in the area of initial exposure, the start of the scan (Fig. 7 (a)). The exposure operation consisted of a single pass of the beam over the area to deliver the entire dose (single layer milling).

Finally, the effect of allowing the cryo-FIB sample to warm back to room temperature is of interest as TEM imaging or subsequent analysis is simplified if it can be done at room temperature. We performed this analysis and others have reported [5] that some of the undesirable side-effects of RT-FIB milling are exhibited after cryo-FIB if the sample is warmed up to room temperature. We found some of the Group III droplets appear when the sample is brought back to room temperature after cryo-exposure (Fig. 9) and additional surface effects are noticed (Fig. 10).

For best results the analysis should be completed at cold temperature or the milling performed at the coldest temperature possible. It is for this reason that the development of a cryogenic TEM sample preparation process that keeps the sample cold throughout all the steps including the TEM imaging was pursued.

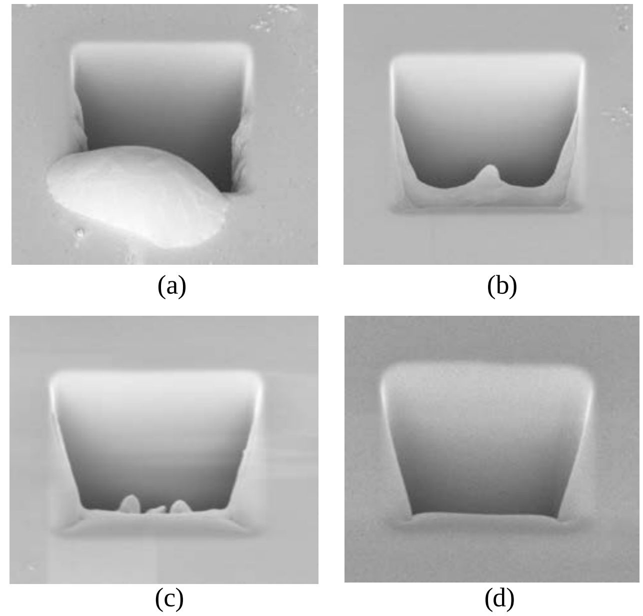


Figure 7: GaN Exposed to 30 keV Ga^+, Dose: $5.5nC/\mu m^2$ (a) at 248K, (b) 223K, (c) 173 K and (d) 128 K.

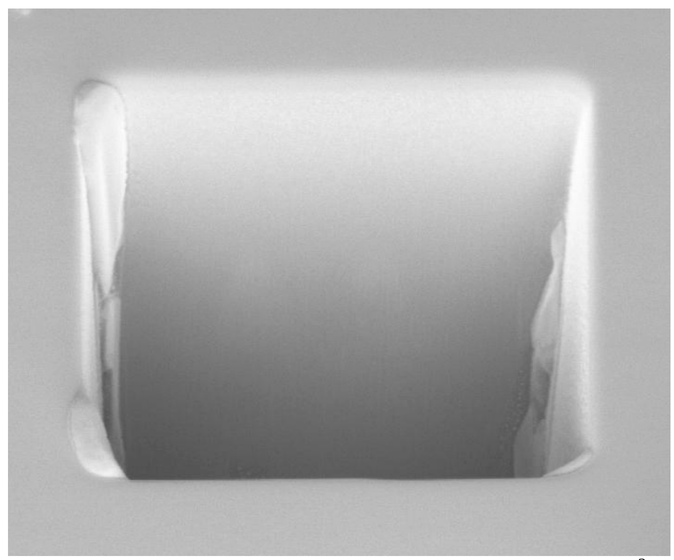


Figure 8: GaN Exposed to 30 keV Ga^+, Dose: $5.5nC/\mu m^2$ at 248 K with multiple passes of the beam.

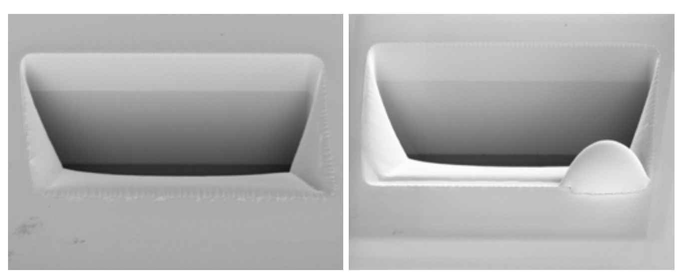


Figure 9: Effect of warm-up post cryo-FIB. Sample milled at 128 K (left) and after warming to room temperature (right).

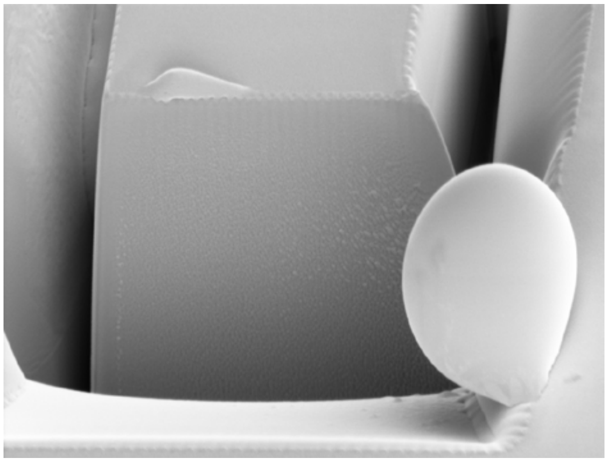


Figure 10: Effect of warm-up post cryo-FIB showing surface droplet formation that was not visible after milling at 128 K.

Effect of Temperature on Copper Milling

The replacement of aluminum interconnect with copper has been problematic not only in the production process but also in the failure analysis and debug of semiconductors. Gallium FIB milling of copper exhibits a grain orientation sputter rate difference that results in uneven removal of material (Fig. 11).

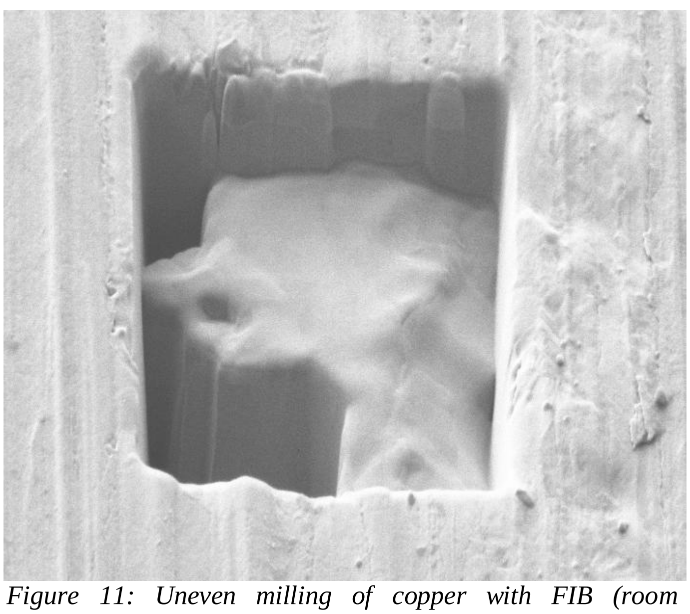


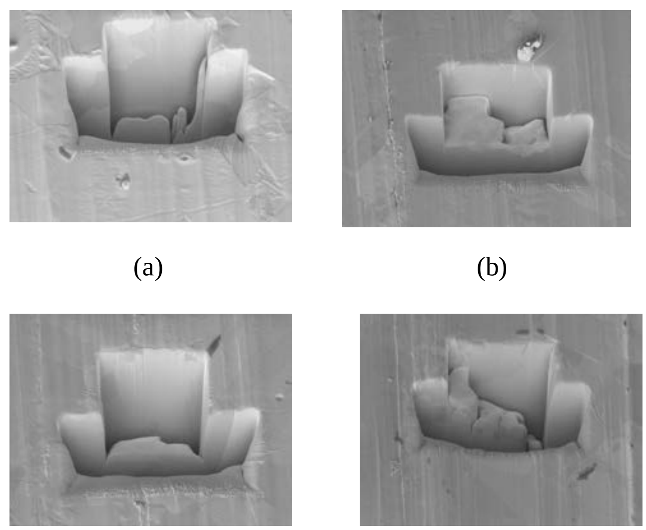
Figure 11: Uneven milling of copper with FIB (room temperature).

The rate of milling of the different grains found in copper can vary more than 3 times between the 110 crystal orientation and the 111 orientation [6]. Many efforts have addressed this phenomenon but have had varied levels of success [7] [8]. The application in the references concern circuit edit primarily but other devices also use thin layers of copper sandwiched in thin films (i.e. photovoltaics). Additional issues with copper milling in multi-layer thin-film structures are migration of material from one layer to another, milling artifacts that appear as smearing of one layer into the other and more. It was therefore of interest to investigate the effect of cryogenics on FIB-milling of copper in these sandwich structures. As shown in Table 2 and Figure 12, no observable change was realized by cooling the sample even to as low as 128 K as far as the grain orientation effect is concerned.

Table 2: Effect of temperature on FIB milling of copper

Temperature (K)	Exposure ($nC/\mu m^2$)	Side Effect (Qualitative)
300 K	5.5	Strong Grain Dependence
248 K	5.5	No improvement
223 K	5.5	No improvement
173 K	5.5	No improvement
128 K	5.5	No improvement

Therefore, no benefit is realized for copper milling in the application of circuit edit. However, copper migration and smearing have been observed at room temperature but not at temperatures of 173 K and below. Samples of complex films structures including copper have been prepared for TEM in cryo-milling without migration or smearing.



(a) (b)

(c) (d)

Figure 12: Lack of temperature dependence of copper FIB exposure. (a) 248 K, (b) 223 K, (c) 173 K and (d) 128 K.

Cryo-TEM Sample Preparation Using FIB-SEM

There is a growing interest in cryogenic FIB-SEM sample preparation and several techniques have been developed. One such technique works well with liquid samples that are frozen on a TEM grid and FIB-milled at an angle to form a trapezoidal lamella [9]. Another technique uses a cooled tip that has a notch milled in it and a notch is also milled in the grid for wedging the lamella in-place [10]. Although these techniques work well with many types of samples and can be used with our equipment set we wanted to develop a universal cryogenic sample preparation technique that is a close to our room temperature process as possible.

The universal process we developed for cryogenic TEM sample preparation using FIB-SEM is described here. Once the process is started the sample is maintained at cryogenic temperature until the TEM image is taken.

First, a standard Omniprobe grid is attached to the cryo-sample stage (Fig. 13). The sample is then mounted on the same holder and loaded into the FIB-SEM with the cryo-stage in place. For anhydrous samples there is no cryo-plunging required prior to loading the sample. It can be loaded at room temperature. However, if a protective coating is needed, the sample may be coated with platinum or a similar metal in an ex-situ sputter coater.
Then the tip of the Omniprobe is replaced with the cryogenic design shown in Figures 5 and 6. Once the sample and grid are in the system, the cryo-stage is cooled to about 128 K. The cooling process also cools the tip. Then typical milling is done to release the sample. Due to significant time savings, we prefer the Total Release process where a wedge is cut out and sitting free in the bulk sample.

Figure 13: FIB TEM grid wedged in cryo-sample holder.

A novel approach we have developed for attaching the sample to the tip uses water vapor from our gas injection nozzle to freeze the tip to the sample and subsequently to the grid (Figures 14 and 15). Our cryo-FIB is equipped with tungsten and carbon deposition and both of these materials do not deposit controllably in the cryogenic environment. Tungsten is especially problematic because the process is very sensitive to temperature and usually runs at about 350 K. It has been reported that platinum deposition can be made to work in a cryo-FIB environment but this is not presently available on our cryo-FIB [11].

Once on the grid the ice on the tip can be milled away and the sample is left attached to the grid with ice. Then the sample is thinned (Fig. 16) and cryo-transferred in the shuttle to the cryo-loading chamber (Fig. 17). In this chamber, the sample is immersed in liquid nitrogen and is transferred to a cryo-storage box (Fig. 18) in which it can be stored in a Dewar flask or kept in a container or liquid nitrogen and transferred to the TEM cryo-loading system (Fig. 19) and loaded into the TEM for cryo-imaging.

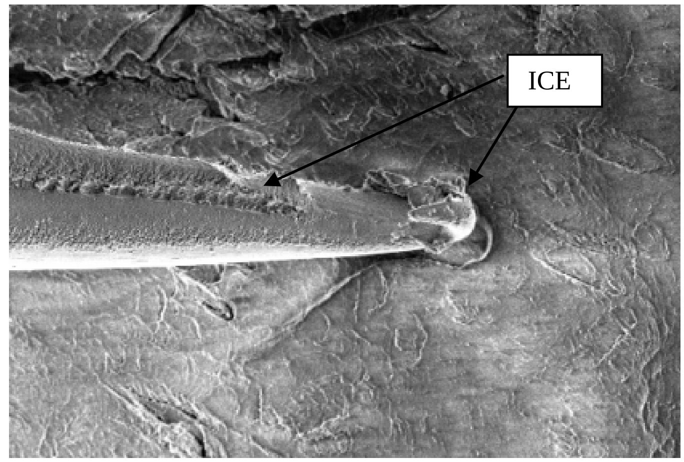


Figure 14: Ice used to attach tip to sample.

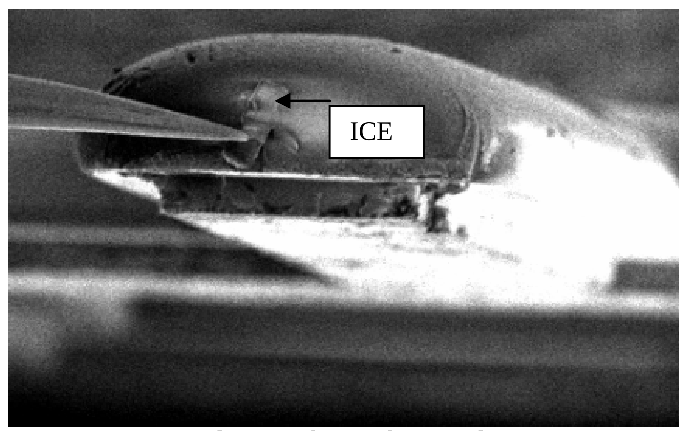


Figure 15: Ice used to attach sample to grid.

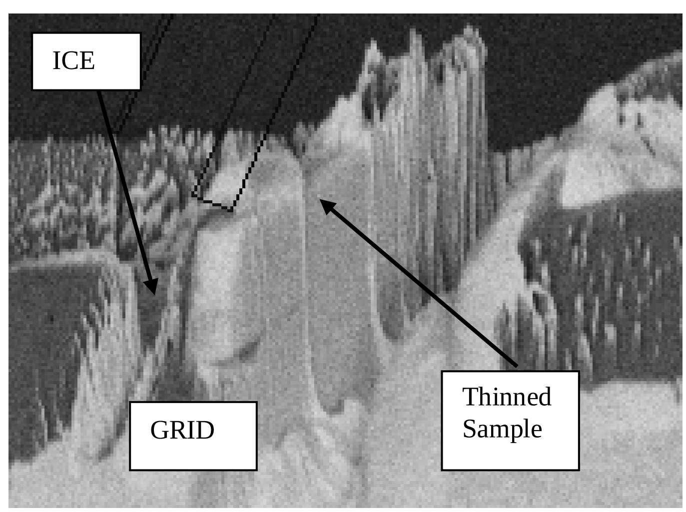


Figure 16: Thinning sample on grid (dark areas are ice).

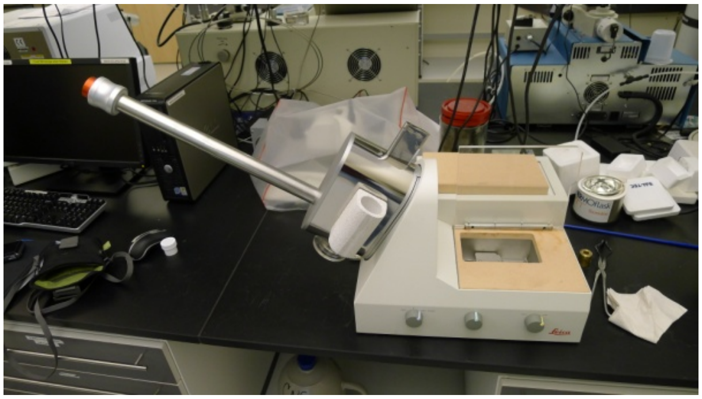


Figure 17: Cryo-loading chamber with shuttle attached

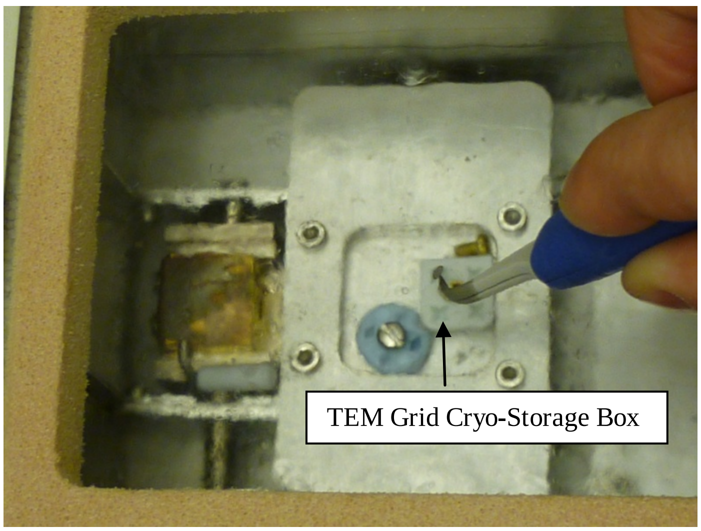


Figure 18: Transfer of grid from FIB-SEM holder to TEM grid holder designed for cryogenic storage.

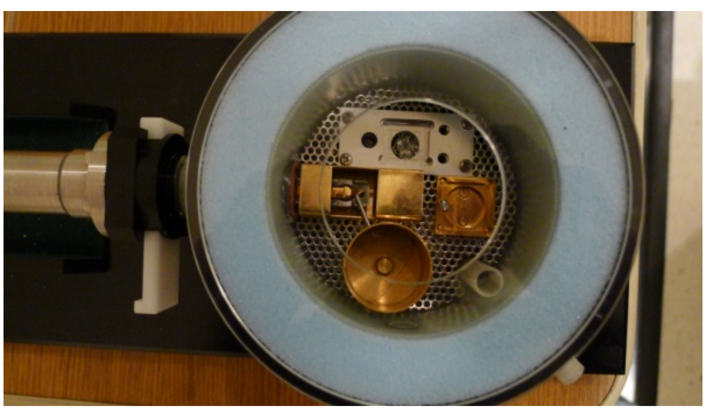


Figure 19: Cryo-loading chamber for cryo-TEM

The final steps in this process (transfer from FIB to TEM) have been successfully carried out for biological samples but have yet to be tried on electronic materials.

Conclusions

Cryogenic FIB-SEM has been proven to be useful for analyzing compound semiconductors and some multi-layer materials where migration and smearing are of concern. Specifically, compound semiconductors can be cleanly and evenly milled using gallium FIB at temperatures below 173 K. It was also discovered that warming up these milled samples post cryo-FIB introduces some of the artifacts. In order to preserve samples prepared in this fashion without return to air and without any temperature rise, a cryogenic TEM sample preparation and transfer process was developed using novel techniques such as attaching the sample to the grid with ice formed from the gas injection water system.

While it is with some disappointment that we report no improvement in bulk copper milling at cryogenic temperatures as envisioned for the application of circuit edit, there is a benefit in TEM sample preparation using cryo-FIB for other copper thin film applications.

Acknowledgments

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