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Applications of the GentleMill™ To FIB Prepared TEM Samples

Introduction

Sample preparation for transmission electron microscopy (TEM) is a common analytical technique used for the investigation of virtually all materials in engineering technologies today. The high resolution and combined analytical capabilities make TEM an important tool used for materials science. Recent years have seen a large increase in the use of TEM in the semiconductor market, where the resolution of the TEM provides a clear advantage in understanding how the manufacturing process of devices can be improved and monitored.

As a result, TEM sample preparation plays an important role in today's manufacturing and research environment. For semiconductor materials the use of the focused ion beam (FIB) has become a common method used in the preparation of TEM samples. Although the FIB is extremely useful, it does have some drawbacks, especially in high resolution TEM applications. The highly energetic, tightly focused ion beam creates amorphous damage in the crystalline sample, limiting the information that can be obtained from the sample.

A low energy ion milling system, the GentleMill™, has been developed for eliminating these artifacts in FIB prepared samples. This report outlines some basic techniques used for improving the results obtained with samples prepared using FIB methods.

Sample Preparation

Sample preparation for semiconductor materials in today's production environment often involves the use of a combined process of mechanical preparation and ion milling. The added capability of the FIB has allowed for several techniques to be employed. A general discussion of two common techniques will be given below.

Mechanical Thinning

A semiconductor wafer is obtained and cut into a suitable size, typically on the order of 3mm square. The sample is mechanically polished using diamond lapping films from 30 µm down to 0.5 µm to a thickness of ~ 25µm using the Tripod Polisher® and a polishing machine as shown in Figure 2. Polishing times are generally less than 20 minutes for this procedure and can be as little as 5 minutes in some cases.

Once the sample has been thinned in this manner, it is attached to a copper grid or inserted into the FIB directly for milling. This method will depend upon the FIB setup and the FIB preparation technique used for the sample.

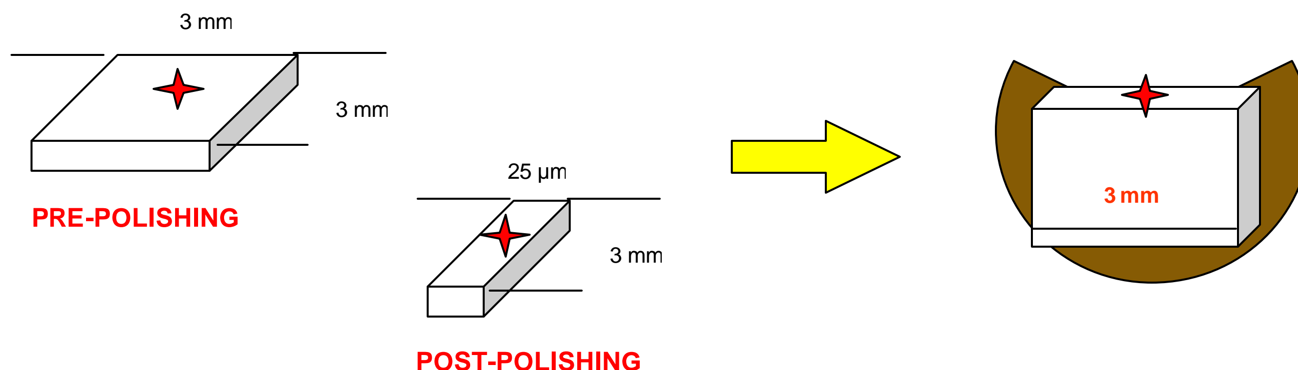

Figure 1: Illustration of the basic sample configuration. The sample starts approximately 3mm square and is polished down using the Tripod Polisher® until it is approximately 25 µm thick, with equal thickness on both sides of the area of interest. The sample can be subsequently mounted to a grid for FIB milling.

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Figure 2: Image showing the Model 920 Lapping and Polishing Machine with the Model 590 Tripod Polisher®. The sample is mounted to the face of the Tripod for thinning.

FIB Milling

Following the mechanical preparation the sample is then placed into the FIB for milling. There are several different techniques that are currently in use for sample preparation and will not be covered in this article. Two of the most common techniques currently in use are the so-called H-bar and Lift-out techniques. Both have become commonplace and are successful in their use.

For H-bar samples, the sample is epoxy mounted to a copper half grid as shown in Figure 1. The FIB is then used to mill out trenches that are approximately 5µm deep x 20µm wide around the area of interest. The resulting thin membrane in the center of the trench is supported on both ends by the remaining material from the polishing process.

For a Lift-out sample, the similar approach is used, however the resulting thin membrane is then cut away with the FIB and then lifted out of the trench, while subsequently being mounted to a carbon coated, mesh TEM grid. Recent advances in instrumentation have allowed an in-situ lift-out method to be employed, which allows the thin membrane of the sample to be attached to a grid using FIB welding techniques.

Illustrations of the basic concepts for both of these techniques are given below.

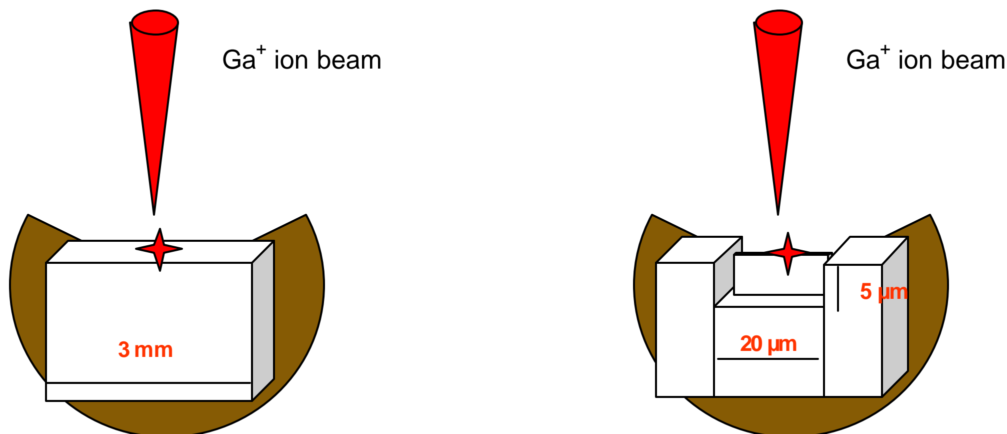

Figure 3: Illustration of the H-bar configuration used for making a sample with the FIB. The polished sample is epoxy mounted to the copper half grid and placed into the FIB. The FIB is then used to mill a thin membrane with the are of interested centered on the membrane. This assembly can then be inserted into the TEM.

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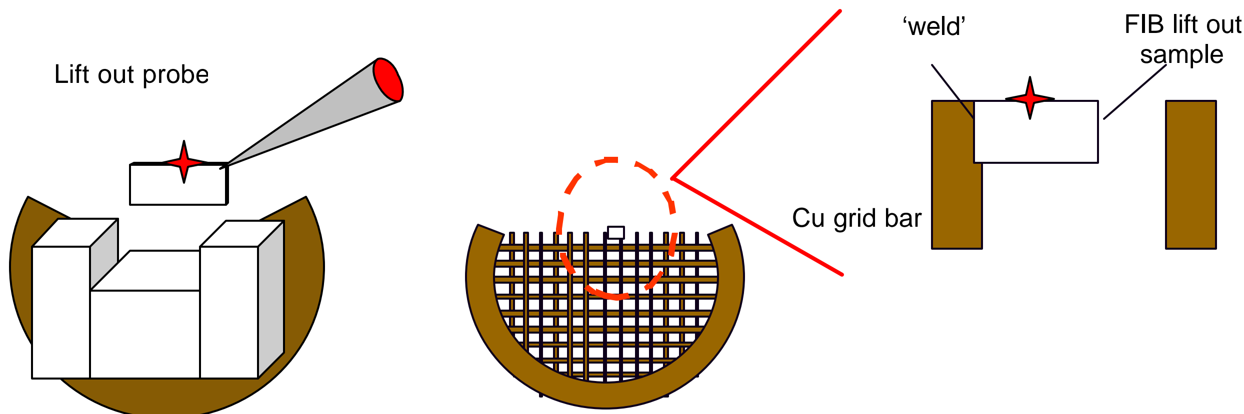

Figure 4: Illustration of the Lift-out technique. The FIB milled sample (similar to the H-bar configuration) is plucked from the sample inside the chamber using a special probe attached to the chamber. The sample can then be 'welded' to a cut 200 mesh copper grid and fixed into place for observation. Another option is to mount the thin lift out membrane onto a mesh grid with a carbon support film holding it in place.

Low Energy Ion Milling (The GentleMill™)

The GentleMill? is used as a final ion polishing system to complete the sample preparation process. The system incorporates a low energy ion source which operates at extremely low ion energies ranging from 100 eV and up to 2 keV. The ion source utilizes a hot cathode filament used to generate electrons in the rear electrode of the gun. The electrons oscillate along the anode axis between the two cold cathodes, creating a high electron density in the anode region. Argon gas is injected into this region, ionized, and then accelerated out of the front of the gun at the anode potential. An electrostatic lens at the front of the ion source is used to direct the ion beam at the sample and helps confine the ion beam to a tightly focused area, about 0.75mm in diameter at 300 volts. Figure 5 illustrates the basic construction of the ion source. This unique and patented gun design (US Patent 6,236,054) provides high milling rates at low energies, making the system flexible enough to use for sample preparation.

All ion beam milling parameters are controlled by a computer software program making selection of parameters a simple operation.

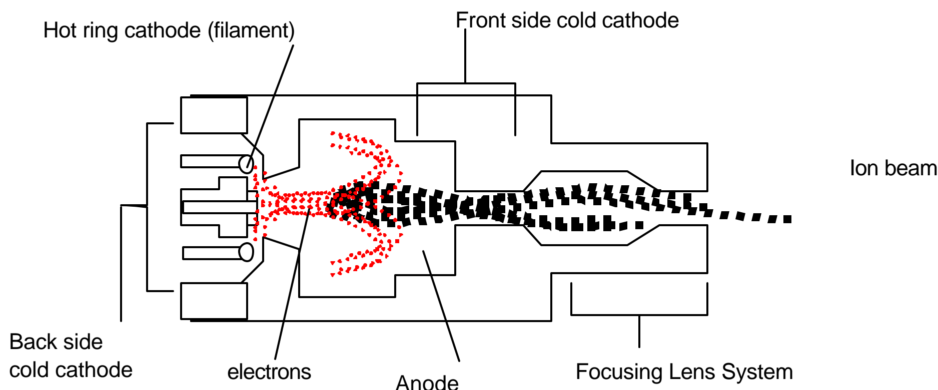

Figure 5: Schematic illustration of the low energy ion source used in the GentleMill™ system.
(Illustration courtesy Technoorg Linda, LTD.)

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H-Bar Samples

These types of FIB samples require some simple adjustments to the GentleMill™ process to obtain improved results. The sample is inserted into the stage and thinned from both the top and bottom side of the sample, with the ion beam orientation similar to that used in the FIB. Oscillating the sample is done but at small oscillation angles (20°) to minimize the possible sputtering of the wall materials of the sample. Low energy ion thinning of the sample using 1 keV for approximately 20 minutes per side is completed to reduce the remaining thickness and to remove ion damage imparted to the sample during FIB milling. Reducing the voltage of the low energy ion source to 200 eV per side and ion polishing the sample produces improved high resolution images. Generally the entire process is completed within 1 hour depending on the parameters used in the FIB and the amount of damage existing in the sample.

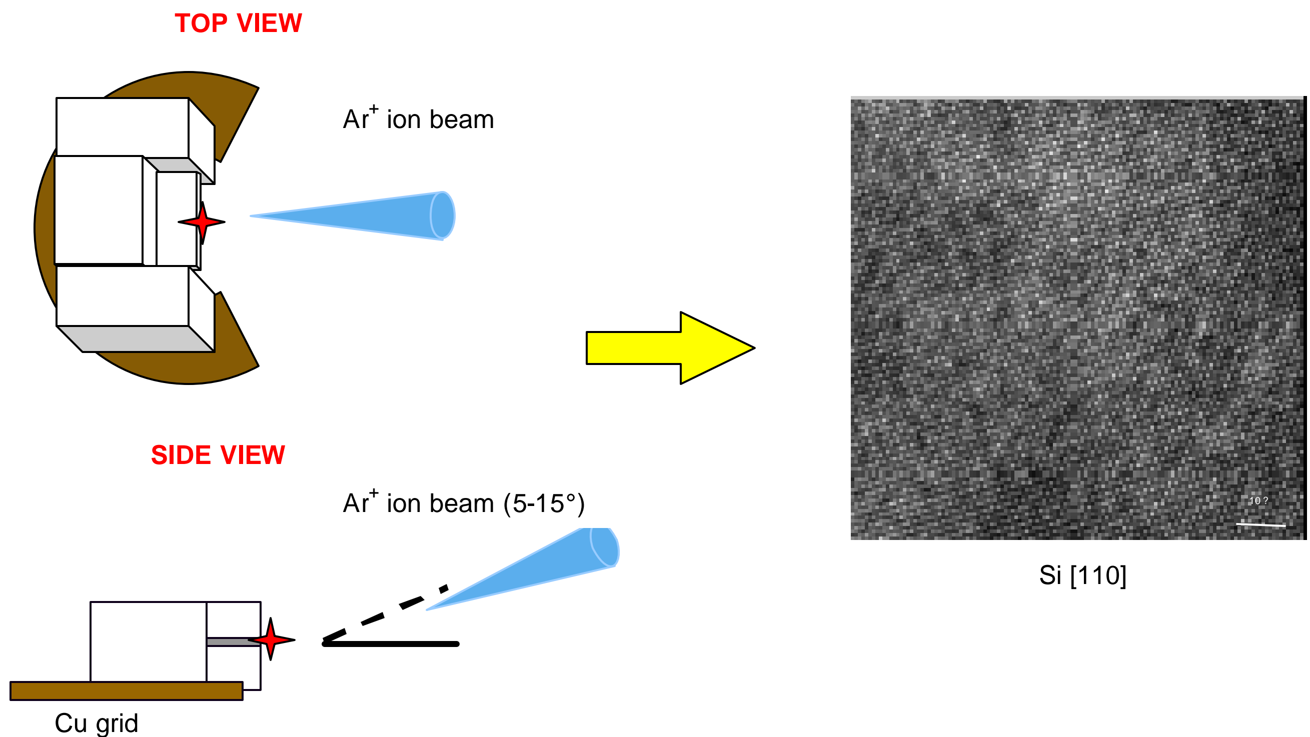

Figure 6: Example of an FIB prepared Si [110] prepared using the H-bar FIB method followed by low energy ion milling in the GentleMill™ system. The sample was ion beam thinned using an initial 1 kV Argon ion beam at approximately 10° beam incidence for 10 minutes from both the top and bottom sides of the sample. A low energy treatment of 500 volts for 10 minutes per side was completed to obtain the image seen at right. (Image courtesy B. Arnold, A. Barna, et al, Inst. Solid St. and Mat. Res. Dresden, 2000).

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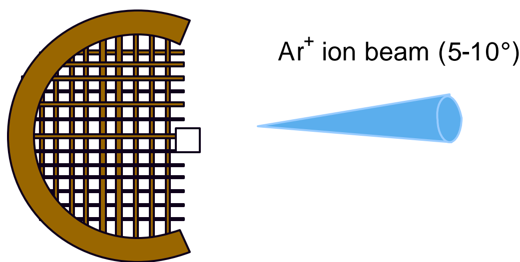
Lift Out Samples

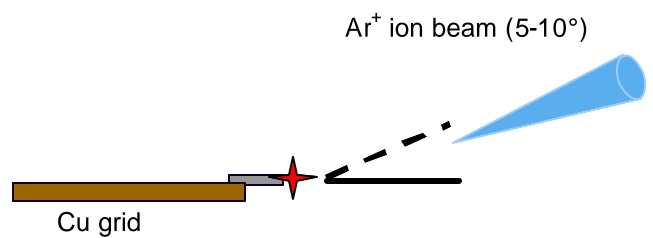
FIB Lift-out samples have also been prepared using the GentleMill™ with much success. These have required the use of an in-situ lift out probe for performing the welding process to the grids but are successful nonetheless. Once the samples have been attached to the grid they can be low energy ion milled using similar parameters as the H-bar type samples. However, these types of samples generally require only very low energy milling (below 500 volts) because the sample thickness is very low to begin with.

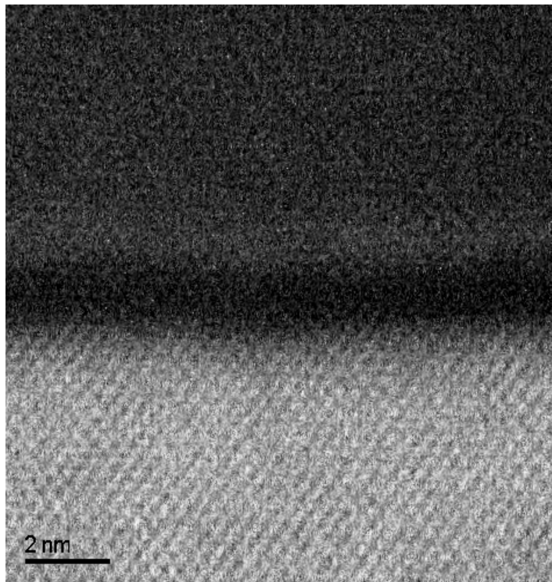
The sample is ion thinned at low angles, generally around 5° and oscillated using the standard 60° used in normal sample preparation processes. Thinning is done from both the top and bottom of the sample as with the H-bar sample case. Generally the process takes less than 20 minutes to complete and the sample is ready for observation.

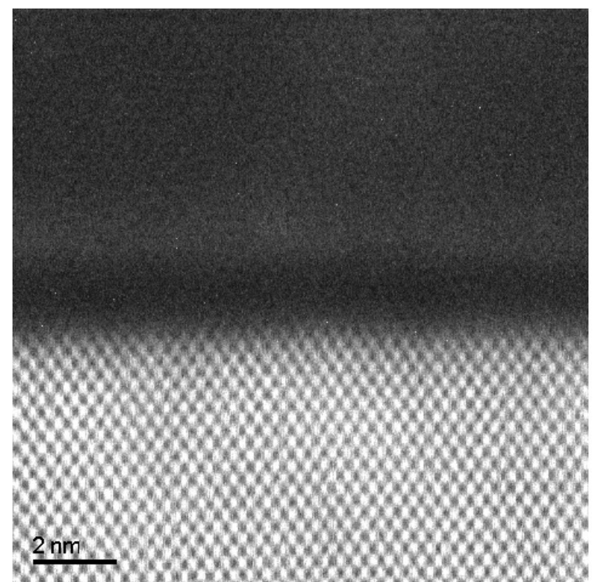
TOP VIEW



SIDE VIEW







PRE GENTLEMILL™ TREATMENT **POST GENTLEMILL™ TREATMENT**

Figure 7: Example of low energy milling treatment on FIB lift out samples. This shows a STEM image of a silicon sample directly from the FIB (PRE GENTLE MILL™) and following treatment using the low energy ion mill (POST GENTLE MILL™). A vast improvement in the quality of the STEM image is seen following treatment with the GentleMill™ and the amorphous damage has been eliminated. (Image courtesy from M. Sidorov, AMD, SALSA 2002).

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Conclusions

Low energy ion milling is an effective and efficient method of cleaning amorphous damage from crystalline materials prepared using the FIB. Applications to the semiconductor market are perhaps the most critical with the advent of gate oxide measurements becoming so difficult to measure. Amorphous damage in samples prepared by FIB can be removed using this technology and is an effective tool for improving the quality of sample preparation without increasing the bottleneck.

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