



Sample Preparation Procedure for TEM Imaging of Semiconductor Materials

by Wendy L. Sarney

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14. ABSTRACT Transmission electron microscopy (TEM) allows detailed materials characterization at high resolutions. Lattice imaging requires the preparation of electron transparent samples. Preparing such thin samples without changing the characteristics of the material is not a trivial process. The Electro-Optics & Photonics Division (EO&P) of the Army Research Laboratory uses TEM for structural characterization of MBE-grown semiconductor materials. Major projects that rely on TEM include the following structures: type-II superlattices, quantum well and quantum dot superlattices, II-VI films, and GaN-based structures.. TEM analysis provides the grower with high and low resolution images of the interfaces, surfaces, and defects. This report discusses the typical preparation procedures used to fabricate TEM samples.				
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Contents

Figure	iii
1. Introduction	1
2. Sample Preparation Procedure for Cross-Sectional TEM Samples	2
2.1 Sectioning and Assembling the Sample “Sandwich”	2
2.2 Polishing the Sample	4
2.3 Ion Milling.....	6
3. Sample Preparation Procedure for Plan-view TEM Samples	8
4. Conclusion	8
5. References	9
Distribution list	10

Figure

Figure 1. (a) Wafer before cleaving. (b) Orientation relationships of the two pieces cut from the wafer in (a).....	3
Figure 2. Schematic of a sample `sandwich.' The films sides of two pieces of the original semiconductor wafer are glued together. They are supported by two dummy pieces. The dummy pieces are usually scraps remaining from other wafers and are the same material as the substrate.	4
Figure 3. (a) Sample is mounted onto SEM stub. (b) SEM sub is mounted onto tripod polisher. (c) Sample is polished on the polishing wheel.....	4
Figure 4. Model 900™ Grinder/Polisher from South Bay Technology.....	5
Figure 5. Orientation relationships of the two pieces in the sample sandwich before (a) and after (b) polishing. The dotted line shows the direction that the electron beam will be incident on the sample when it is placed in the TEM. (c) The copper grid is mounted onto the polished sample.....	6
Figure 6. Gatan pIpS™ ion mill.	7
Figure 7. Schematic of sample after ion milling. The area bordering the hole is electron transparent. The hole crosses the film/substrate interface twice on each piece (four times total for the sample).	7

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I. Introduction

The quality of TEM images is directly dependent on the sample preparation process, a destructive technique that involves cleaving, grinding, polishing, and ion milling the material to electron transparency. It is inevitable that the sample preparation process affects the properties of the material under investigation. This, in turn, affects and limits our interpretation of TEM images. We minimize (but not eliminate) preparation-related artifacts by customizing the process for each material.

The lattice resolution of ARL's JEOL 2010F microscope operated at 200 keV is approximately 0.1 nm. Imaging at this resolution requires the TEM samples to be very thin in the region of interest. As a minimum requirement, samples must be electron transparent to an extent that the electron beam penetrates the specimen and we see intensity on the microscope's fluorescent screen. In practice, TEM specimens should be no thicker than 100 nm for low-resolution imaging, and even thinner (~50 nm) for high-resolution imaging. The required sample thinness is a function of the electron beam energy and the average atomic number of the sample. Samples made of heavy materials need to be thinner than those made of lighter materials. Higher energy incident electron beams are able to penetrate through thicker samples than lower energy beams, but the likelihood of electron beam damage to the sample increases with increasing beam voltages. Some materials commonly used in infrared devices, such as the antimonides and HgCdTe, are particularly sensitive to the electron beam. These materials degrade quickly as we examine them in the microscope; therefore, we want to minimize the time needed to examine them. Having high quality and extremely thin samples minimizes the struggle of obtaining meaningful images, and allows us to work more quickly with the TEM.

The TEM image is a two dimensional projection of a three dimensional sample. This means that the image is an average of everything that the electron beam 'sees' as it penetrates the sample. Averaging leads to difficulties when interpreting TEM images. Therefore, another benefit of preparing extremely thin samples is that it minimizes the amount of averaged information in a TEM image.

In this paper, we discuss the preparation of TEM samples from semiconductor wafers grown by molecular beam epitaxy (MBE). In the past, we have used this same procedure to prepare samples grown by metalorganic chemical vapor deposition (MOCVD) and pulsed laser deposition (PLD). The semiconductor samples usually consist of a film grown onto a substrate. Typical studies involve examining the substrate/film interface, and identifying, quantifying, and characterizing defects. We supplement TEM data with non-destructive characterization obtained from x-ray, SEM, AFM, and optical imaging.

2. Sample Preparation Procedure for Cross-Sectional TEM Samples

2.1 Sectioning and Assembling the Sample “Sandwich”

We must be mindful of which zone axis we wish to examine in the TEM before we cleave the wafer. For Si and III-V substrates the major flat usually indicates the (110) plane. It is worth verifying the orientation of the major flat with the grower or the substrate vendor, since wafers purchased outside of the United States may not use the same convention. We select the zone axis based on which crystal planes we want to image [1]. We typically prefer the [110] zone axis because the first order diffraction spots correspond to the relatively easily resolved (002) and (111) crystal planes and because the sample readily cleaves along the {110}-type planes. Occasionally we examine the [100] zone axis when we are studying a superlattice structure that does not yield sufficient image contrast. Examining this zone axis results in an image of the (002) and (020) planes. Dynamical theory suggests that imaging along the [100] zone axis allows higher contrast imaging than obtained along the [110] axis [2]. This is because the {002}-type diffracted beams are more sensitive to the chemical composition of the sample than the (111) beams. One example of an ARL-grown system requiring examination along the [100] zone axis is the AlGaAs/GaAs triple quantum well superlattice structures grown for reflection modulator applications [3–4].

It is important to wear gloves and sometimes a dust mask during the sample preparation process. Scribing and cutting the wafers can create dust and some samples leave a chalky residue when polished.

We begin the sample preparation process by cutting two rectangular (approximately 1.5 mm by 4 mm) pieces of the wafer with a diamond scribe. Figure 1 schematically demonstrates the cleaving process for a [110] zone axis wafer having a [001] growth direction. We cleave the rectangular pieces so that the longer dimension of one of the pieces is parallel to the major flat and the longer dimension of the other piece is perpendicular to the major flat. This results in one piece having a zone axis along [110] and the other having its zone axis along $[1\bar{1}0]$. This allows a more thorough characterization of the specimen, especially in the cases of certain orientation-dependent structures such as quantum wires. To examine a sample with the [100] zone axis, we would cleave the sample 45° away from the major flat. Occasionally we cut the wafer so that one piece has a [110] zone axis and the other piece has a [100] zone axis.

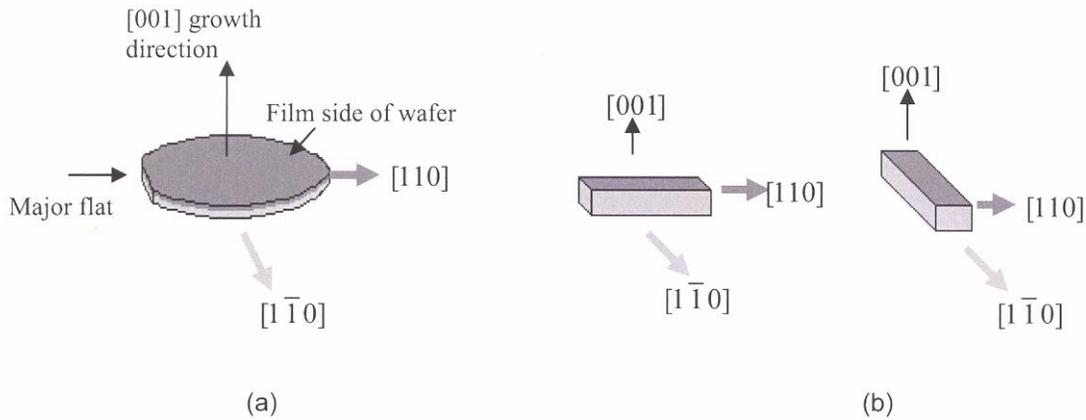


Figure 1. (a) Wafer before cleaving. (b) Orientation relationships of the two pieces cut from the wafer in (a).

After cutting the pieces, we glue the film sides together with epoxy. Usually the film side is shiny compared with the substrate side but occasionally a grower will use double-side polished substrates. In this case, we lightly scratch the substrate side with our diamond scribe. Currently we are gluing the samples with MBond, an epoxy based on bisphenol A. Periodically we must change brands of epoxy due to the manufacturer ‘improving’ the strength of the epoxy. Keep in mind that epoxy is not designed or marketed for the purposes that we use it, and for most consumers, increased strength is a desired feature! Epoxy that is too strong will not ion mill properly, and leave an undesired ‘glue line’ between the two pieces after milling. Bits of the sample’s surface will tend to stick to the glue line, and these regions are typically too thick along the beam direction. Epoxy that is too weak will ion mill quickly and leave the sample’s surface unprotected. When we image the film, we usually pick an area that still has some glue visible on the surface. This assures us that the surface is ‘intact’ and that the film is true to its original thickness. Finding proper epoxy is not a trivial issue.

After gluing the two film sides together, we attach a dummy piece to the backside of each piece. This provides more support and makes handling the sample with tweezers much easier. Figure 2 is a schematic of the sample ‘sandwich.’ The dummy pieces are usually the same material as the substrate, or at least a material of similar hardness. We place the sample into a small vice and cure it on a hot plate (at approximately 60 °C) for one hour. We then remove the sample from the hot plate (but keep it in the vice) and allow it to cool for another hour. For particularly delicate and sensitive samples, such as those containing HgCdTe, we place the vice on the 65 °C hot plate but immediately turn the heat off. We leave the vice on the plate until the plate reaches room temperature. We then remove the vice from the hot plate and let the epoxy cure for at least four hours.

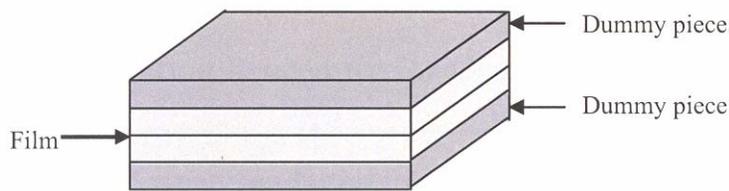


Figure 2. Schematic of a sample 'sandwich.' The films sides of two pieces of the original semiconductor wafer are glued together. They are supported by two dummy pieces. The dummy pieces are usually scraps remaining from other wafers and are the same material as the substrate.

2.2 Polishing the Sample

We mount the sample sandwich onto an SEM stub with acetone-dissolvable wax as shown in Figure 3a. The SEM stub is mounted onto a Model 590 Tripod Polisher[®] from South Bay Technology (Figure 3b). The sample is planarized by adjusting the three micrometers. We hold the tripod polisher and polish the sample (Figure 3c) with a Model 900 Grinder/Polisher from South Bay Technology (Figure 4). The Model 900 has an 8" polishing wheel upon which we place polishing disks and papers. The speed of the wheel can be varied from 0 to 1725 RPM. A constant stream of water drips onto the polishing surface whenever the wheel is rotating. Water softens the contact between the sample and the polishing medium, it keeps the sample from heating during the polishing process, and it traps the small particulates as they polish away from the sample (keeping them from becoming airborne). For environmental safety reasons, our water supply is part of a custom-built closed pumping system. As shown in Figure 4, the Grinder/Polisher machine is inside a custom-built hood, which protects the engineer from any gases that may be emitted from the sample as it is polished (such as phosphine gas from InP substrates).

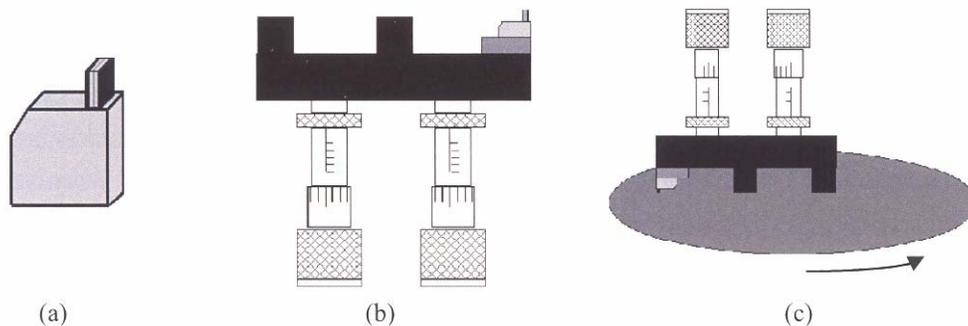


Figure 3. (a) Sample is mounted onto SEM stub. (b) SEM sub is mounted onto tripod polisher. (c) Sample is polished on the polishing wheel.



Figure 4. Model 900™ Grinder/Polisher from South Bay Technology.

The hardness and fragility of the sample dictates which polishing medium we use. For hard samples, such as GaN, we begin polishing one side of the sample with a 30 μ m metal bonded diamond grinding disk. We move through a series of progressively smoother diamond discs and then diamond papers until a shiny, scratch-free mirror-like surface appears. We then remove the sample from the SEM stub (by melting it off on the hot plate) and re-mount it on its opposite side. The sample is polished to a thickness of approximately 20 μ m with a series of progressively smoother diamond disks and papers. For softer samples, such as those based on GaSb, we use the same procedure, except that we begin polishing with 15 μ m aluminum oxide paper and move to progressively smoother papers. Figure 5 shows the orientation relationships of the sample sandwich during the thinning process. When the sample is sufficiently thin, we mount a copper grid (having an outer diameter of 3 mm) mounted onto it with M13ond (Figure 5c). The sample (still mounted on the SEM stub) is cured on the hot plate for 5 minutes and then let sit for another hour at room temperature. We then soak the SEM stub in acetone until the wax dissolves and the sample falls off the stub. At this point, we must be handle the sample with extreme care since it is very thin and supported only by the Cu grid.

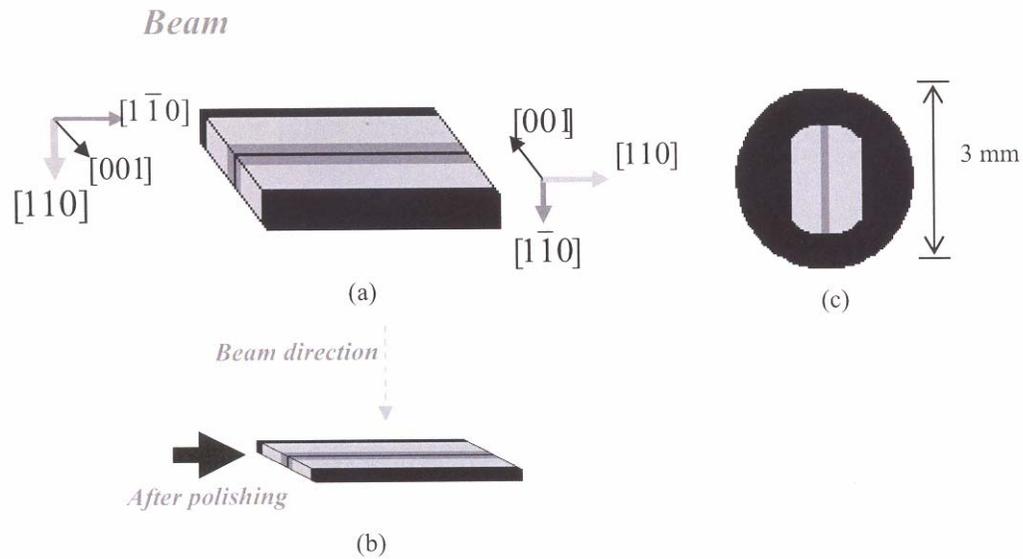


Figure 5. Orientation relationships of the two pieces in the sample sandwich before (a) and after (b) polishing. The dotted line shows the direction that the electron beam will be incident on the sample when it is placed in the TEM. (c) The copper grid is mounted onto the polished sample.

2.3 Ion Milling

We use a Model 691 Gatan PIPS™ (Precision Ion Polishing System) ion mill for final thinning to electron transparency. The PIPS (Figure 6) uses two Penning ion guns to produce a stable Argon ion beam. The system has a liquid nitrogen trap, which reduces contaminants and water vapor. Ion beam damage may affect the sample's crystalline morphology (making it appear amorphous) or introduce surface oxides. Therefore, selecting an appropriate beam voltage is critical. Using too low of a beam voltage lengthens the milling time, and increases the likelihood of contamination. Using too high of a beam voltage can cause structural damage to the sample. Because GaSb and HgCdTe-based samples are very beam sensitive, we tend to mill these samples at no higher than 3 keV. At the other extreme, GaN samples are very beam resilient and are usually milled at 6eV. We usually position the ion guns so that they are milling on opposing sides of the sample. Since the milling rates of different phases or crystal directions can vary drastically, it is difficult to obtain a cross-sectional sample that is equally electron-transparent across multiple layers. Using low milling angles minimizes differential thinning effects (the slow milling regions protect the fast milling regions from the ion beam). The PIPS allows samples to be milled at angles ranging from 1 to 10°. Rotating the sample during the milling process further minimizes preferential thinning. We align the guns so that the ion beams are incident on the center of the specimen. It is important that the thinnest area of the sample after milling be located as near to the center of the grid's aperture as possible. This allows the area of interest to stay at the same height above the TEM's objective lens (z-height) during tilting. We monitor the ion

milling process with a CCD zoom camera and video display. Thinning continues until a small hole perforates both pieces of the sample in the center. The final TEM sample then has four useful thin areas to examine—two on each side of the ion-milled hole (Figure 7).

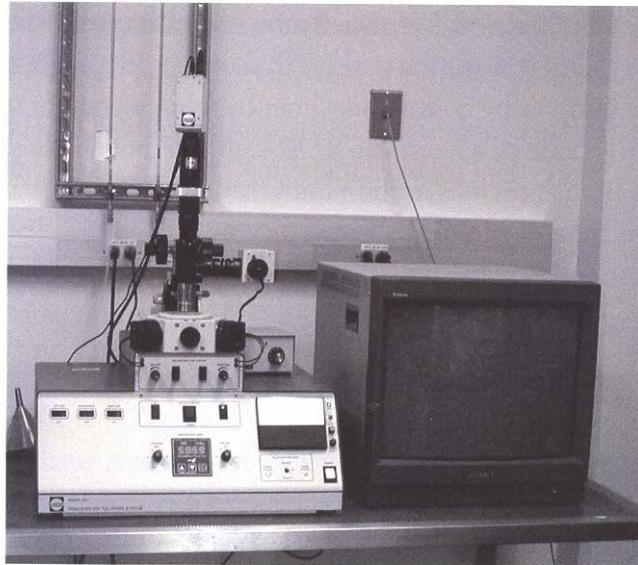


Figure 6. Gatan PIPS™ ion mill.

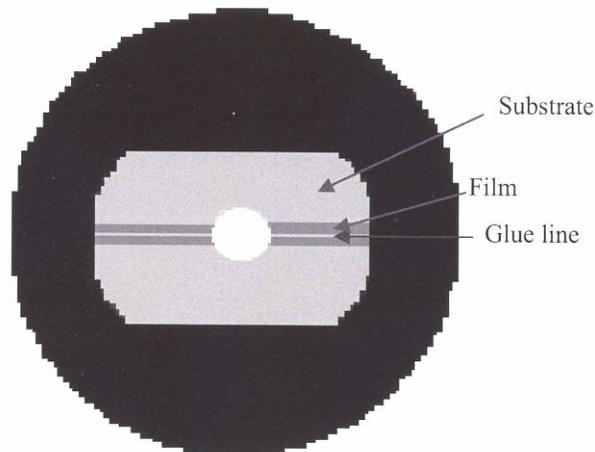


Figure 7. Schematic of sample after ion milling. The area bordering the hole is electron transparent. The hole crosses the film/substrate interface twice on each piece (four times total) for the sample).

3. Sample Preparation Procedure for Plan-view TEM Samples

Plan-view samples are prepared when we want the TEM's electron beam to penetrate the sample along the growth direction. We usually examine samples in plan-view when we need to calculate the misfit dislocation density of the film. We begin the procedure for preparing plan-view samples by cutting one 2 cm by 4 cm rectangle from the wafer. The sample is mounted film side down onto an SEM stub. We polish the substrate side of the sample as described in the previous section until the total samples thickness is 20 μ m or less. A copper grid is mounted to the substrate side of the sample. The sample is ion milled with both guns configured to mill from the substrate side only.

4. Conclusion

To take advantage of a TEM's high resolution, we must prepare high quality electron transparent samples. Although there is a wide array of known procedures used in research and commercial laboratories, sample preparation is really somewhat of an art. The best procedure is the one that the individual scientist can perform repeatably and confidently. The reward for the time invested in developing reliable sample preparation techniques is the acquisition of beautiful TEM images that provide a wealth of information for our MBE growers.

5. References

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