Rapid Cross-Section TEM Specimen Preparation of III-V Materials

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ABSTRACT

Cross-section transmission electron microscope (TEM) specimen preparation of III-V materials using conventional methods can be a painful and time-consuming activity, with a day or more from receipt of a sample to examination in the TEM being the norm. This article describes the cross-section TEM specimen preparation technique used at Bookham Caswell. The usual time from start to finish is <1 hour. Up to 10 samples can be prepared at once, depending upon sample type. Most of the tools used are widely available and inexpensive, making the technique ideal for use in institutions with limited resources.

INTRODUCTION

III-V materials have come to play an increasingly prominent role in microelectronics and communications technology, such as high speed signal processing used in mobile telephones, high power lasers used to send signals thousands of kilometres along optical fibres, and high brightness GaN LEDs in traffic lights. The wide variety of devices that can be manufactured depends upon the ability to deposit very closely controlled epitaxial layers. At Bookham Technology Caswell, TEM is used routinely to provide rapid and very accurate measurements of epitaxial layer thicknesses using the cleaved edge technique. Cross-section TEM is also used extensively to characterise epitaxial layer quality and device structures. The cross-section technique described here has been developed in recent years as a response to the ever-increasing number of samples in need of characterisation. The aim of this article is to give a detailed description of the sample preparation method, which should allow it to be implemented with the minimum effort. The equipment used should be available in most specimen preparation laboratories. The conventional method of cross-section TEM sample preparation for semiconductors involves the following steps. a) Sawing or cleaving several pieces of material, one of which is the piece of interest. b) Gluing them in a stack with epoxy resin and leaving to cure. c) Grinding and polishing one side of the stack to within a few microns of the site of interest. d) Turning the sample over and grinding, then polishing, to a thickness of 4 to 30 microns (dependant upon material). e) Gluing a support grid to the sample and removing excess material, followed by f) ion milling to electron transparency. It is difficult to perform this procedure in less than two hours, even using 5-minute epoxy resin in steps (c) and (e). The minimum time needed for grinding and polishing is approximately 20 minutes, with typically 20 to 30 minutes needed for ion milling. The method described here omits steps (b) and (c), saving the time needed for the epoxy to cure and leaving only one grinding and polishing step. The main steps of the procedure are: 1) selecting a region of interest and back thinning; 2) cleaving the sample next to the site of interest; 3 mounting the sample on a support grid; 4) grinding and polishing and 5) ion milling. These are described in detail below.

PROCEDURE

1. Selecting a large region of interest and back thinning Typical III-V wafer sizes vary from 100 mm (e.g. InP) to 150 mm (e.g. GaAs). The region of interest may be the whole wafer (apart from some exclusion zone around the edge), a specific site a few microns across, or part of an individual device or integrated circuit. As III-V materials cleave readily on {110} planes, the easiest and quickest way of selecting a region of interest, down to a size of ~10 microns is by cleaving it from the wafer or die. Cleaving is most accurate when the material is thin (~50 to 100 microns), and so wafers are back thinned as a preliminary step for TEM specimen preparation. It is usual to back thin fully front-processed wafers prior to dicing, in which case further back thinning can be omitted; back face processed wafers may have a thick metallisation layer present which would make cleaving difficult, in which case it is best removed. For samples in wafer form, the first step is to cleave a piece approx. 4 to 8mm square. Hardened filter paper can be used as a disposable, clean work surface. The easiest way to cleave a full thickness wafer is to make a mark 1 to 2 mm long on the edge of the material with a diamond scriber, then turn the wafer over and press on the opposite side above the mark. A crack should nucleate from the scribe mark and run across the wafer. Alternatively just use a scalpel blade on the wafer edge. The cleaved piece is then mounted - face down - on a small glass slide using acetone-soluble thermoplastic wax (e.g. glycophthalate), with pieces of grade zero glass cover slip on each side. Grade zero glass cover slips are approx. 120 microns thick. Placing one (or part of one) on each side of the sample allows the sample to be back thinned to the same thickness as the cover slips, without the chance of grinding the sample away. The glass and wax must be clean - a piece of dust will cause the sample to crack. Typical hot plate temperature is 180°C. Pressing the piece down and sliding it around with a pair of tweezers while still hot will make sure only a thin film of wax is present between the piece and the glass. Several samples can be mounted and ground at once, as long as care is taken to keep track of the different pieces. The glass slide with cover slips can be cleaned and re-used until the cover slips become too thin (~50 microns). The slide can be held on a block - big enough to hold - with double-sided sticky tape (Figure 1). It is best if the tape is not too tacky, or the glass slide will be difficult to remove. The piece is then back thinned until the surface is coplanar with the glass slides. Grade 1200 SiC grit on a rotary grinding wheel with running water cooling will grind the excess material away in a few seconds (Figure 2). The piece is cleaned and excess wax removed with a cotton bud dampened with acetone. The glass slide is placed on the hot plate, the piece gently pushed off and placed in acetone to dissolve excess wax. After a short time, the sample is removed from the acetone and placed face down on clean filter paper. All wax residues and stains are gently

Caution

Toxic fumes and/or dust are released while grinding III-V materials. Fume extraction, slurry collection and personal protection are required. Do not grind these materials without a liquid to contain the dust. Also consider contamination of grinding/polishing machines and where the effluent will end up. ALL environmental data sheets (EPA) and relevant govermental and institutional regulations governing the use of these materials and procedures MUST be followed. If you are unsure of the appropriate safety precautions to be followed, DO NOT perform these procedures! This is the users responsibility. Neither the Microscopy Society of America, nor the editors of MT, nor the author can accept legal responsibility for your actions.

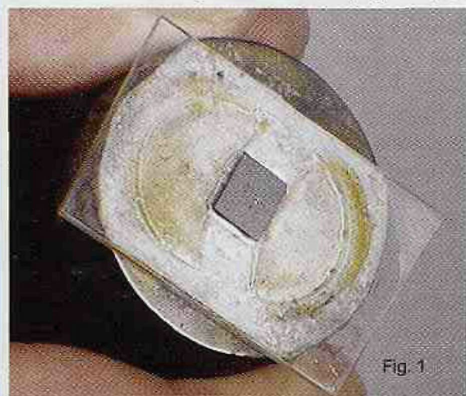
Fig. 1

Fig. 2

Fig. 3

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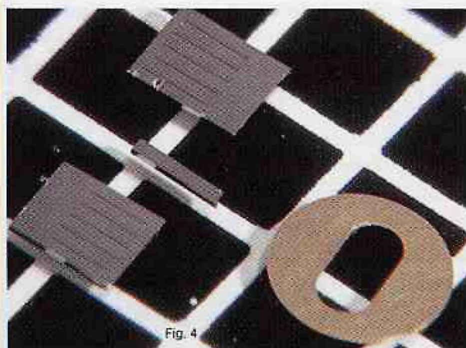


Fig. 4

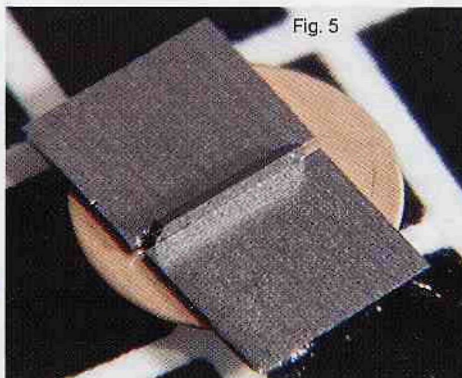


Fig. 5

Fig. 6

Fig. 7

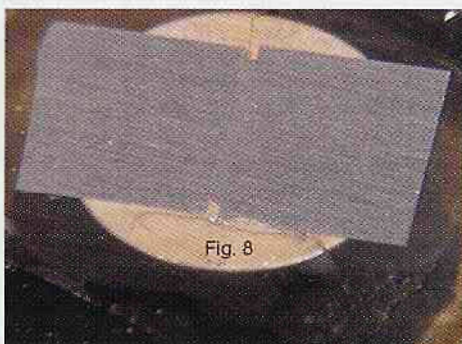


Fig. 8

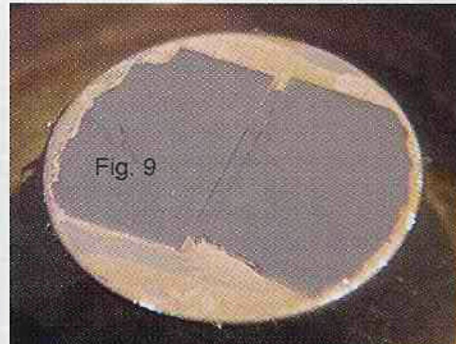


Fig. 9

cleaned away with cotton buds dampened with acetone.

2. Cleaving the sample next to the region of interest

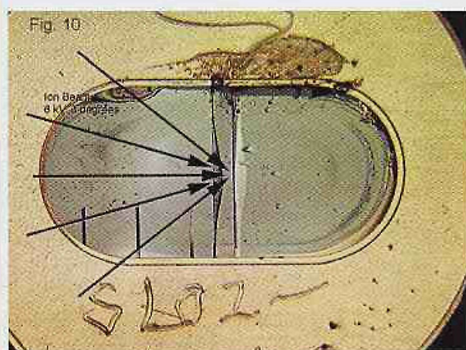
For regions of interest down to a size of approx. 10 microns, it is possible to cleave the sample such that the centre of the region of interest lies approx. 10 microns away from the line of cleavage. Even the simplest purpose-built scribing machine should be able to achieve this accuracy quite easily. For larger regions of interest, a hand-drawn scribe mark will suffice. Figure 3 shows a sample with regions of interest in 20 micron wide lines. Scribe marks have been made at the edge of the wafer aligned with one edge of the regions of interest. Turning the sample over and gently pressing on the back of each mark should nucleate a cleave along the scribe mark, which runs across the sample. A workpiece 1 to 3 mm long and 0.2 to 0.5 mm wide is needed, with the region of interest half way along the long side. The long side of the piece next to the region of interest must be straight and flat. Cleave out two more pieces (or use thin scrap material) approx. 1.5 x 2mm, each with at least one straight, flat edge (Figure 4).

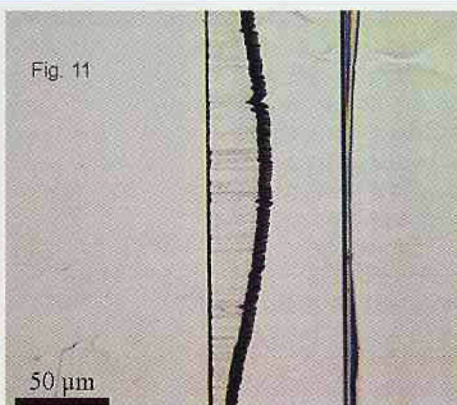
3. Workpiece mounting

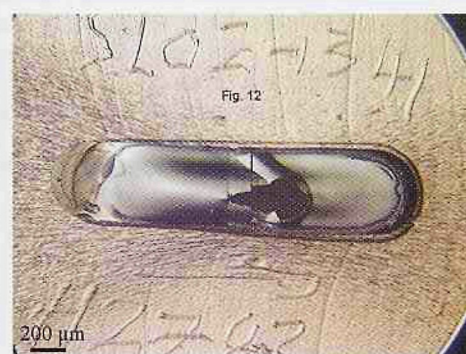
3.05 mm diameter Cu TEM support 'grids', with a slot 2 mm long and a variety of widths (0.3, 0.5, 0.75 and 1 mm) are readily available from electron microscopy supply houses. If the region of interest is small, or the piece is only 1 mm long, a small slot width is chosen (e.g. 0.5 mm). A larger region of interest and a larger piece of material allows a larger slot width to be used (e.g. 1 mm). The amount of thin material available is larger for larger slot widths; also, thinning times are marginally quicker. A variety of grid thicknesses are available. Standard thickness grids work well, since they provide sufficient support and are not so thick that they cause significant shadowing during ion milling. Figure 4 shows a 2 x 1 mm slot grid, the cleaved sample as well as two larger (scrap) pieces. The parts lie on slightly tacky silicone rubber (e.g. a Gel-Pak) which makes for easier handling. The sample ID has been scratched on the shiny side of the grid as well as an arrow that will indicate the direction of ion milling. The grid is then turned over so that the uppermost surface is the matte side. The grid surface must be flat and clean. A small amount of 5-minute epoxy is mixed and a thin layer is painted on the grid with the point of a cocktail stick. The grid is moved away from any excess glue which lies on the gel so that it does not inadvertently coat the sample. The workpiece is placed on its side across the slot. The ends of the workpiece should lie on the wider parts of the grid, i.e. the axis of the workpiece should be perpendicular to the axis of the slot. The side touching the grid should be the one cleaved to lie approx. 10 microns from the site of interest, and the site of interest should be centrally placed with respect to the edges of the slot. The top (epilayer, or device) side of the workpiece should lie such that the arrow on the other side of the grid points from substrate to device layers. (Several samples that are not site specific may be stacked up, one next to the other, at this stage. Up to ten samples may be attached at the same time, although they will usually need to be examined one or two at a time in the TEM as they become thin for the best results.). Place the two scrap pieces - face down - on either side of the workpiece, with the straight edges next to the work piece. Slide them to lie snugly against the workpiece (Figure 5). *The success of the technique relies on the perfectly flat faces of the workpiece, the flat edges of the scrap pieces and the flatness of the support grid. If they do not lie flat on the grid, thinning will be unsuccessful.*

4. Mechanical thinning

When the glue has set, the grid is placed on a platen suitable for insertion into an automatic polishing machine using thermoplastic wax (Figure 6). The platen has a glass slide on top, on top of which are four smaller pieces of glass slide (all held together with thermoplastic wax). The four pieces are ground to be co-planar using 1200 SiC grit. A small piece of Si approx. 100 microns thick is mounted on top of each of the outer three glass pieces using the same wax, as is the grid on the central piece. All parts are pushed down while hot so that the wax layers are thin. The sample is ground gently on 2400 SiC grit under running water until the pieces on the grid are 20 to 30 microns thick. (This is just about the minimum thickness InP







will stand before it begins to shatter. GaAs, and especially Si, can be taken thinner. Other III-V substrates are more brittle and may need more gentle treatment.) The sample thickness can be measured using an optical microscope at about 200x by focusing on the grid, then the workpiece, reading the difference from the focus knob (usually graduated in microns). The most reliable way of doing this crucial stage is to remove a small amount of material (a few seconds), check the thickness and repeat until the desired thickness is achieved. More grinding will be needed on the first sample done in this way; if the outer Si pieces are left in place, subsequent samples will be ground to the correct thickness very quickly (a few seconds) and more caution may be needed. This process is illustrated in Figures 7 and 8. The platen is placed on the automatic polishing machine (soft nap pad, 1 micron diamond paste with appropriate dispersant/lubricant - e.g. Struers OP-Nap, heavy distillate dispersant) and left for 10 to 15 minutes. When the sample is polished, it is cleaned with cotton buds dampened with acetone. Any material that lies outside the circle of the grid is cut away with a scalpel blade, and all traces of wax and debris are wiped away (Figure 9). The platen is placed on the hotplate and allowed to heat thoroughly. When hot, the grid is pushed gently from the glass slide and immersed in acetone for a minute or so to dissolve any traces of wax. It is then removed from the acetone and placed gently on a piece of filter paper to dry.

5. Ion milling

Low angle ion milling has the advantage that the smooth surface texture of the backing piece is transferred onto the sample during milling, as long as the ion beam only hits the workpiece when its top (epilayer, or device) edge is facing away from the ion beam. Figure 10 shows a sample after 10 minutes ion milling from both sides (6 kV, 3 degrees incidence angle) in a Gatan precision ion polishing (PIPS) machine. The sample rotates in the machine; the arrows indicate the quadrant over which the ion beam is on. The top edge of the workpiece is on the right. A higher magnification image of the sample is shown in Figure 11. III-V compounds can be damaged during ion milling due to overheating. A modulated ion beam can overcome this problem by minimising the heat generated in the sample. In systems with this capability, each ion beam is only on for a short period, allowing the sample to cool before the next ion beam is switched on. If the ion beam is not modulated, cooling with liquid nitrogen may be necessary, particularly for In-containing compounds. Shielding of the sample (e.g. with Ta sheet) from the ion beam over the three quadrants in which ion milling is not needed will also be required Differences in height between the workpiece and the backing and/or facing pieces produce a 'shadow' step which moves across the surface as the sample is thinned. The shadow moves more in the middle than the edges due to the higher intensity of the ion beam there. Eventually the shadow line will move across the workpiece completely, giving a smooth surface 'transferred' from the backing piece. As the sample nears electron transparency, it will begin to buckle and then a hole will appear, usually just in front of the shadow line. Ion milling is continued until the region of interest is electron transparent, as in Figure 12.

SUMMARY

The protocol described here allows rapid preparation of TEM specimens of III-V materials. Silicon samples can also be prepared as long as the edge of the sample is cleaved along {110} or ground flat. While some equipment is not available in every lab (e.g. scribing machine, automatic polishing machine, ion beam milling with modulated beams), it is usually possible to find an equivalent (e.g. for the above - cleaving with a scalpel, manual polishing, or making shields from Tantalum sheet to get 'one-sided' ion milling). The amount of material used is very small (typically 0.5x2 mm) and samples can be 'stacked up', allowing up to 10 samples to be prepared almost as quickly as one. The technique has been used at Caswell since. 2001, and has been developed from previous versions in use since approx. 1996. It allows the lab to cope with a very high workload with minimal effort (e.g. the sample above is No. 1341 in the year 2002). ■

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Editor's Note: One of the big pluses associated with the recent International Congress on Microscopy Meeting in Durban, South Africa was the opportunity to visit with old friends. I've known Luc Harmsen, die Groot Baas at ***Anaspec South Africa****, since the last Microscopy Society of Southern Africa's Capetown meeting sometime toward the end of the last century. Anaspec is a remarkable company that installs and services microscopes literally all over the world. They also put out a hilarious newsletter that Luc has been sending me since the Capetown meeting. Hilarious, yes, but jammed full of extremely useful material. The following is from* ***Anaspec Info****, Issue 36/02, October 2002, with Luc's permission. Check out www.anaspec.co.za for the golfing jokes in the referenced issue!*

... Ron Anderson, Editor

Scan Speed, Mag and Accuracy. That is the Question!

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In the past few weeks we have been very involved in setting up new QemSCANs, MLA's and analytical scanning systems and it is amazing how many times we had to explain the relationship between accuracy, magnification, scan speed, resolution and the resulting time taken to scan over a number of samples.

Let me explain. All the above systems are used to scan over a number of geological samples using a scanning electron microscope and EDX analytical systems. In most cases the users are simply looking for a phase/type of mineral within each sample. They then need to analyse it. The main criteria being that they scan through a large number of such samples in as short a time as possible.

Time being the critical problem here

The factors influencing this would be as follows:

* Magnification. If you run at a low magnification you end up scanning a large field of view and therefore would cover the sample in a few frames and so in theory get through the sample very quickly. The problem being that if you go very low in mag it takes a longer time to scan over that area and if you are looking for small particles, less than 10 microns, you may not see them. So low is good for speed but bad for accuracy.
* Scan speed. The faster you scan over the field of view the quicker you get through the field but again you loose on accuracy if you have smaller particles. On some SEMs you have a control called scan speed. In analytical systems this could be known as slew time or dwell time and then you select step size. This can be interpreted as the time taken for the e-beam to move from one pixel to the next, before the analysis is done. Then the step size would be the distance each pixel is from each other. On some ED system you select this by stating the resolution of the image.
* Image Resolution. On some SEMs scan speed may be known as resolution. This means how many lines in a frame. Too many and the field takes a long time, too little and you miss data.

Can you calculate the step size? Yes. Select a magnification and set up an image. Now use the SEM measuring annotation, that's if you have that option, and calculate the scanned area at that mag. Better known as the field width. For example field width at 150x mag on SEM X is 250µm. If you now set a image resolution of 256x 256 then each step is about 1µm apart because 256 x 256 means 256 pixels x 256 pixels. 256 pixels into 250 µm = ~1µm per pixel. If you select a lower resolution of 128 x 128 the scan will go faster but the step size will be about 2µm apart. If you are looking for 1µm particles you may miss them because the step size is too big.

* Dwell time or slew time. How long or short should that be? As with anything in EM it all depends! If you move the beam too fast from pixel to pixel the image is unclear. If you move too slowly you may be damaging the specimen as you are putting too much energy into one spot for too long.
* Spot size/ beam current. Now we need to analyse the image. For this we need to put in enough electrons to get back the correct information. This could be

  — SE image. To get the best SE image, we would want a small spot size to give good resolution imaging. That is basic EM knowledge!

  — BSE image. The back scatter image resolution is largely determined by the detector used, the sample and the kV of the SEM. Some BSE detectors have a better resolution at defining different "grey levels" than others. Then the combination of the energy of the beam or kV selected and the sample will give you an image that looks sharp or out of focus. This is due to the dew drop effect as demonstrated on the Monte Carlo software. Changing the spot size (probe current, beam current, resolution setting depending on what type of SEM you have.) will only increase the number of electrons going in, and so coming back out, of the sample. This means that the BSE image will only appear less noisy with a bigger spot size.

  — X-Ray acquisition. The bigger the spot size the more electrons into the sample the more the x-rays out so the faster the count rate. Again the resolution is determined by the kV selection and the sample.

From this you will note that spot size is not that important in an analytical system. However if you have the spot size too big you may end up damaging the sample as it may not be able to handle the number of electrons at that energy.

* The sample to BSE detector and X-ray detector distance. The closer the detectors are to the sample the lower the spot size to get a good collection of data. Both the BSE and EDX detectors rely on the "data" to enter the detector by chance. The closer the detector the higher the chance of the "data" entering the detectors. Fact!
* Filament setup. Then we get to the first step of setting up the scanning and that is the cathode/whenelt/gun set-up. By changing the filament saturation point, position in the cathode, cathode to anode distance and the bias, all the top conditions will change. In most cases the scanning is going to happen over a long period of time. In this time we need the beam to be as stable as possible. It would also be nice if the filament did not "pop" in that time.

Basically what we have learned over the past few months is that trying to set up a "system," any system, to do long runs, scanning samples for specific data takes a lot of trial and error testing with a good test sample. From this you will be able to set up different conditions for various samples depending on the data required.

Once this testing and trial period is over you should have a set of conditions which stipulates what mag and resolutions you would require to pick up certain particle sizes and what would be crucial in making sure you pick them all up. ■

GOLFING JOKE from Anaspec Info:

Golfer: "You've got to be the worst caddy in the world."

Caddy: "I don't think so sir. That would be too much of a coincidence."

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