THE SMALL ANGLE CLEAVAGE TECHNIQUE: *AN UPDATE*

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Instrumentation and Supplies for SACT

- **Stereomicroscope**
  - 0.7-70x, parafocal, flexible lighting
- **Precision Grinder and Polishing Station**
  - ~±10 µm accuracy
- **Mini-Diamond Scribes**
  - 25-50 (minimum 10 on hand)
- **Sharp Tweezers**
  - 0.1 mm tips, (#5/45°)
- **Viscous Epoxy**
  - EpoTek H-22 (silver epoxy) or Super Glue Super Strength Epoxy
- **Veco 2x1 Tabbed TEM Grids**
- **Miscellaneous**
  - Teflon blocks, filter paper, lens cleaning tissue, thin flexible ruler, paintbrush, Post-its™, glass slide, hotplate

The total cost of the equipment and necessary supplies illustrated here to get started in using SACT is less than $10k. In a typical TEM specimen preparation laboratory, most of these supplies are already available, making the initial investment in the technique minimal. Non-standard items include the mini-diamond scribes, the very sharp tweezers, the viscous epoxy, and the Teflon blocks.

INTRODUCTION

The Small Angle Cleavage Technique (SACT) is a relatively simple and inexpensive method of producing superior cross sectional TEM specimens. For speed of preparation, it is unsurpassed. One major limitation is that the the substrate material must cleave or fracture. For this reason, it is readily applied to semiconductor materials. Recently, SACT has been extended to other substrates such as glass, silicon carbide, quartz, sapphire, and other brittle materials. It is particularly well suited for rapidly examining coatings and thin films very soon after they are deposited. Many multilayer films also work, but again, the limitation is adhesion. This paper is an update to the original technique developed and presented by McCaffrey. Several procedures have been added or modified to simplify the technique. For example, a method for mounting the cleaved samples utilizing a commercially available grid is presented. In addition, the advantages that the special geometry of the prepared samples have when mounted properly in a double-tilt holder will be discussed with respect to the angular range of tilting experiments that are now possible in the TEM.

OBJECTIVES

There are three objectives of this paper: (i) to present a detailed outline of the steps of SACT and convey all the new information, tips, and tricks-of-the-trade that we can remember so that others can learn the technique quickly, (ii) to illustrate some of the advantages of the geometry of SACT samples in the TEM, and (iii) present successful application of SACT to other material systems other than semiconductors that demonstrate its utility.

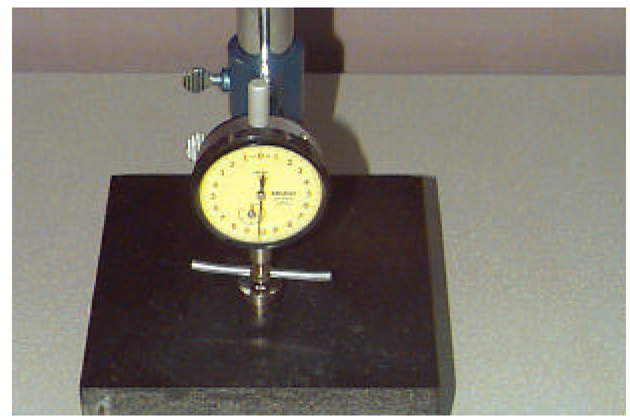
SACT Preparation Steps

Cut or cleaved samples (~4 x 6 mm) are mounted face (good surface) down on a precision hand grinder's sample stub with LT- wax.

Insert stub into the grinder and set position of sample below the grinder's polishing surface.

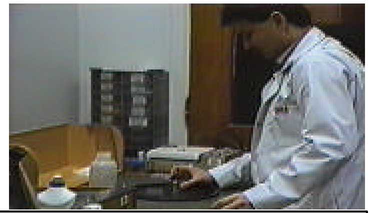
Begin polishing on a polishing wheel using 15-30 µm paper and a normal polishing motion.

An alternative to the polishing wheel is the use of a flat, glass plate arrangement using diamond film abrasives.



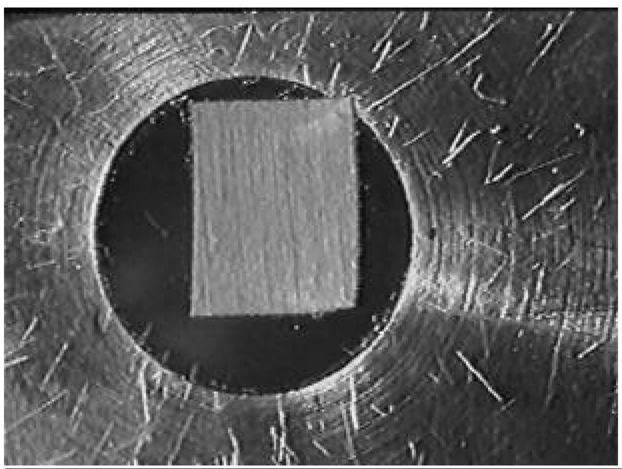
When ~50 µm away from the desired thickness, stop. Measure thickness with a "zeroed" grinder or independently with calipers or dial indicator (shown).

Set grinder to final thickness. Mentally note which mark is parallel to sample direction and estimate a line 12-15° from it. Here, each division is 18º.



Place the grinder in a stationary position so that the direction of the paper at the sample coincides with the desired 12-15º line.

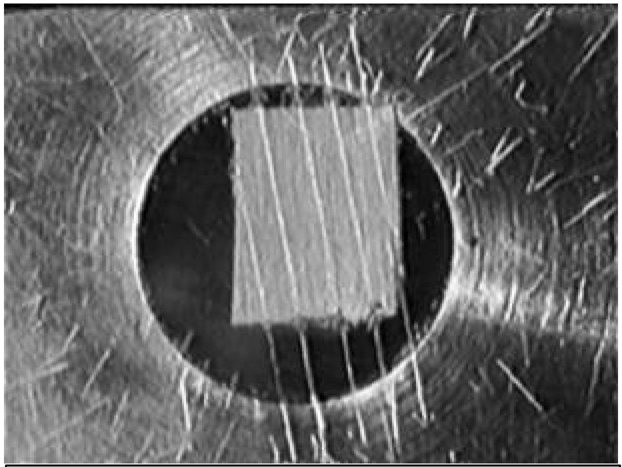
Alternative Method: The last grinding is done in a straight back and forth motion in the direction of the 12-15º estimated line on the sample.



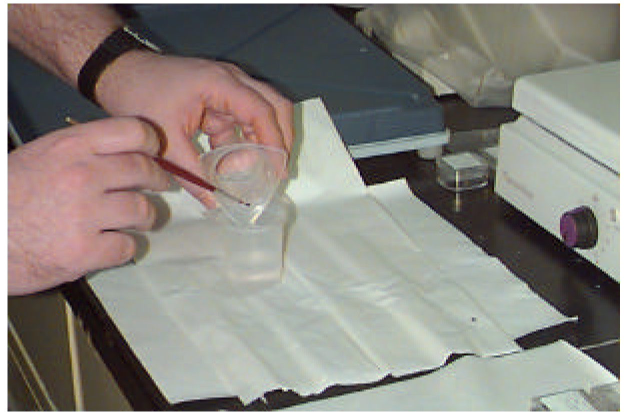
The thinned sample is approximately 100 µm thick and has scratch marks running 12-15º to the cleavage directions.

Along the scratch marks or with the aid of a protractor reticle, scribe lines with a straight edge. Multiple light passes are better than a single heavy one.

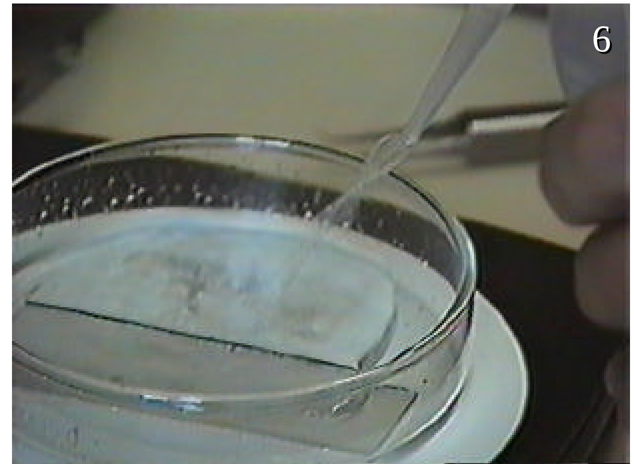
Scribe lines should be parallel and approximately 0.5 mm wide. Excess LT-wax around sample protects sample edges, diamond point, and mount.



The scribe lines should be relatively deep into the sample, and ideally, they should not be visible from the good side when sample is removed.



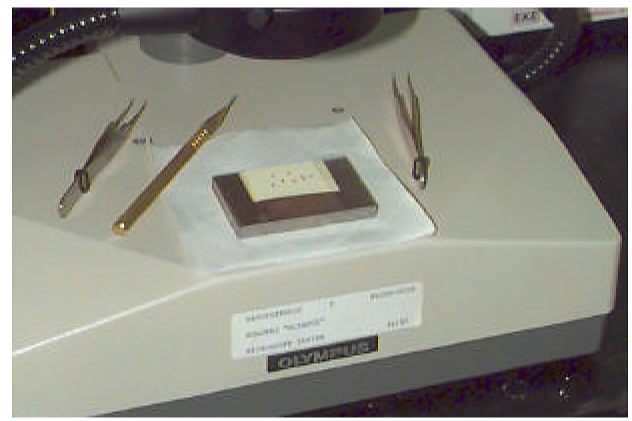
Remove the samples by heating or soaking in acetone. Use a brush to remove the delicate samples. A 2nd rinse in fresh acetone is recommended.



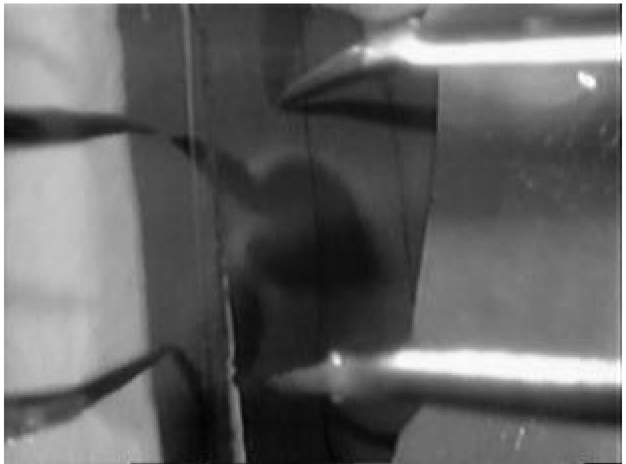
Permanently mount a glass slide in a Petri dish. Fill the dish with distilled water for each new sample. Thoroughly clean dish between uses.

Carefully place sample onto the glass slide with scribe marks facing up. Angle the sample into the water so that it does not float.

Alternative: Use no water. Some samples may be water sensitive. SACT can be performed without liquids. The water prevents the sample from flying away during the fracturing and cleavage steps. The surface tension of the water also helps clean any debris and "nano-dust" from the surface when the sample and the tweezers are withdrawn from the water. If no water is used, the samples will found somewhere in the Petri dish. In either case, after fracturing the sample along the scribe lines (2 steps away), flip them over (face up) for cleaving.



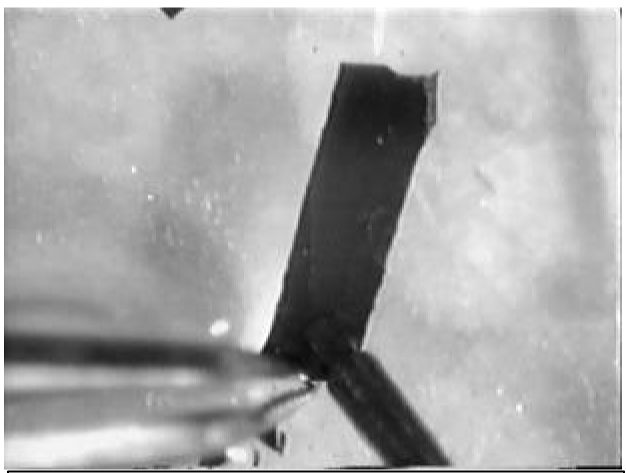
For the no-water approach, after fracturing along scribe lines (next step), place face up on the sticky part of a small Post-it™ note taped to a block.



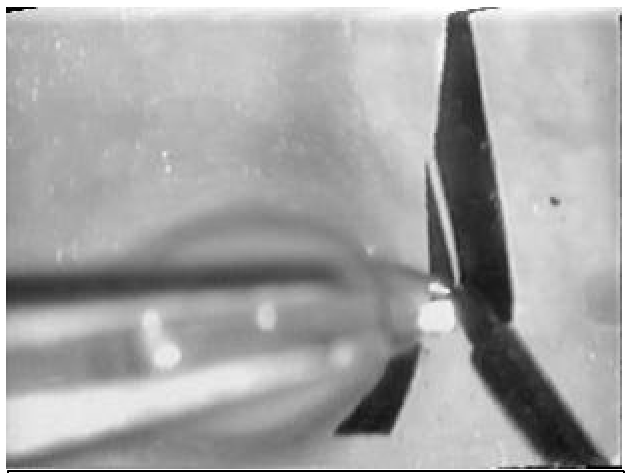
Carefully align the scribe marks along the edge of the glass slide. Gently press sample down. The tweezers should be pressing evenly on either side of line.

Carefully pick up the resulting piece, flip it over, and place it on the glass slide. Be careful of the edges of the sample. Repeat for all of the strips.

Gently brace the bottom edge of the sample and start a scribe line along normal cleavage direction. It is a trial and error process where to begin line.

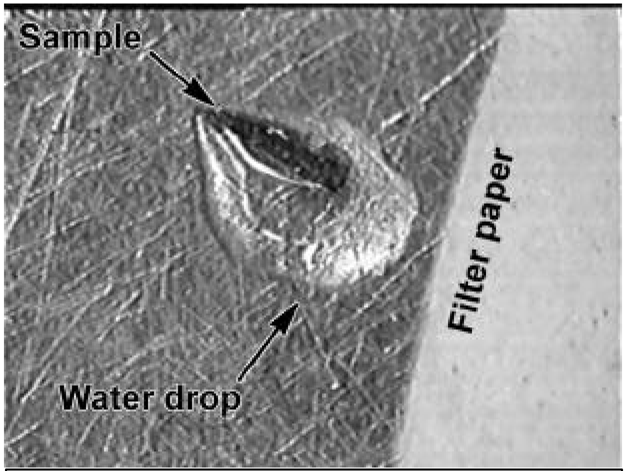


By gently applying pressure, a cleavage line will propagate up and intersect the edge at a 12-15° angle.

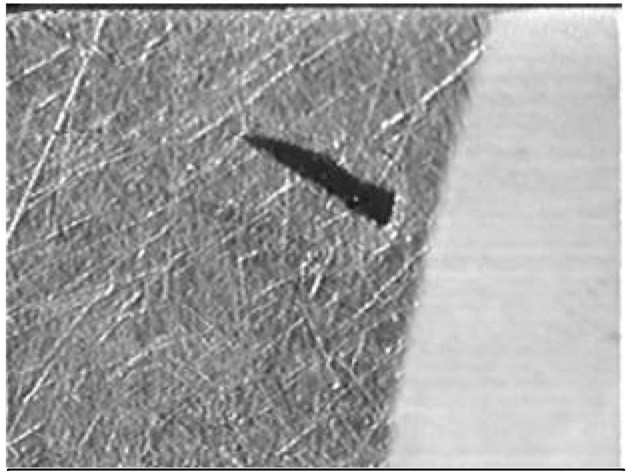


During the cleaving, a gentle sideways pressure is also exerted with the diamond scribe to push the sample away and protect the apex.

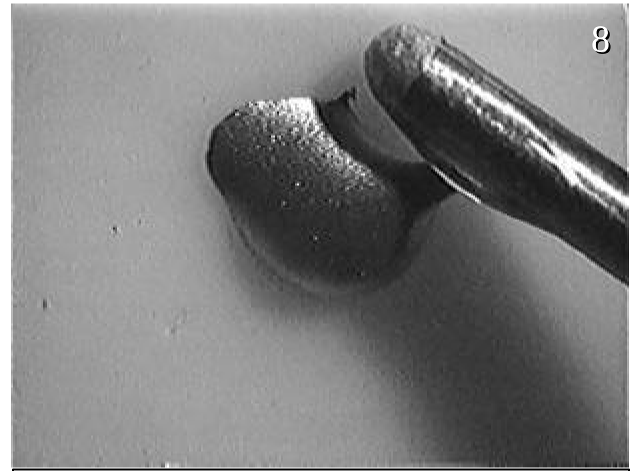
Notes: (i) Examine the fracture edge of strip before cleaving. If it is "jaggy" or not smooth, find and area that is. Try to intersect the cleavage line there.
(ii) The critical part of SACT is the determination of which cleaved samples are suitably thin, not all of them will be. Use the highest possible magnification of the microscope to image the apex of the cleaved sample. If the apex is sharp to the point where it cannot be seen, the sample is probably good. If a nice thin feathered edge can also be seen running up along the thickness to that apex, examine that sample in the TEM first.



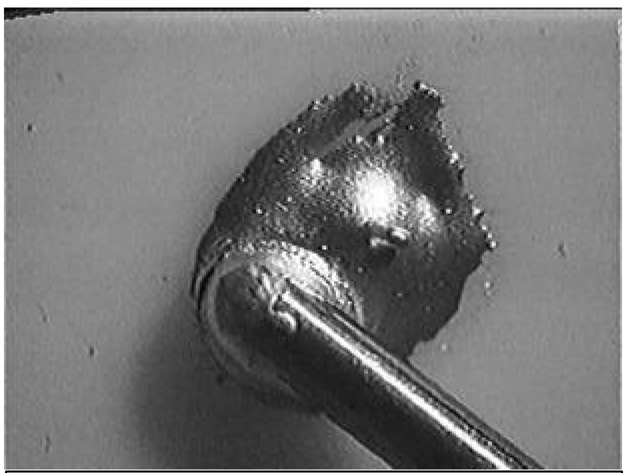
To remove the sample from water, hold tweezers vertically with points touching glass and gently tweeze sample. Place sample on Teflon block.



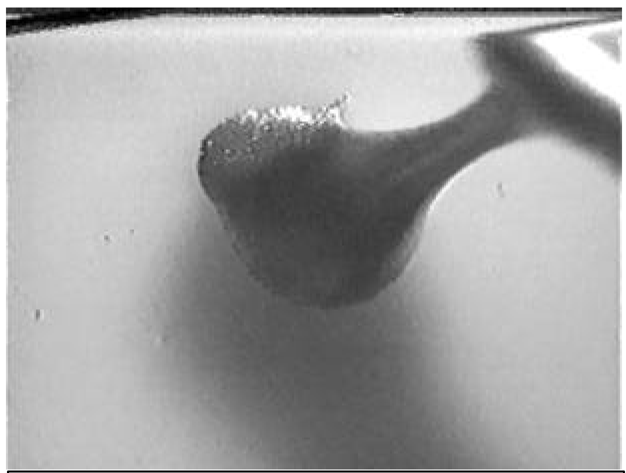
Touch the drop of water with the edge of the filter paper. The water will wick away from the sample and the Teflon block.



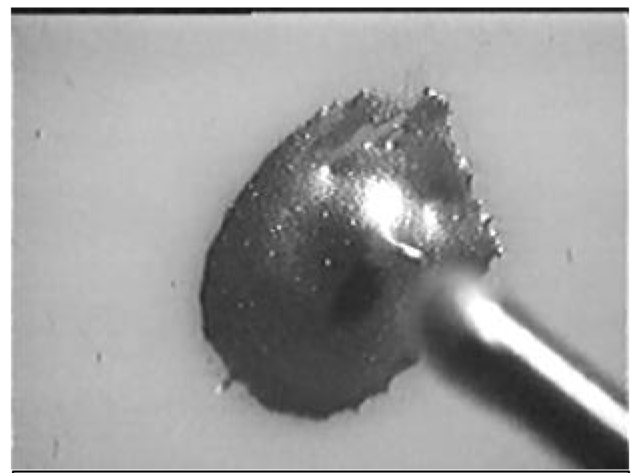
To mix the H-22 silver epoxy in the quantity needed, put a small drop of silver-filled resin onto Teflon. Touch a 1/16” rod to hardener and add to drop.



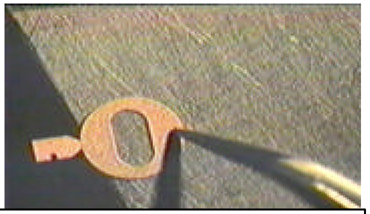
Mix well.



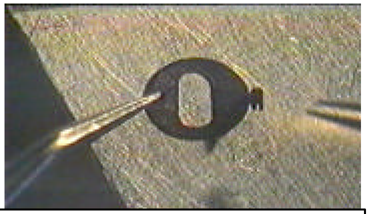
To test the epoxy for correct consistency, pull the rod up from the mix.



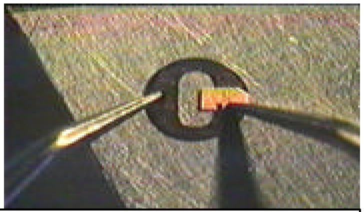
If the height of epoxy is about 1 cm when it breaks, then the consistency is just right. If not, add a little of what is needed or start over.



Position the tab of a Veco Grid over the edge of a square corner of a hard surface such as a glass microscope slide. Bend the tab up or down.

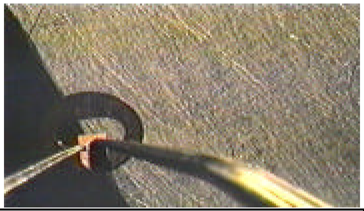


Be careful not to bend the tab more than once or it will weaken the tab to the point where it will come off.

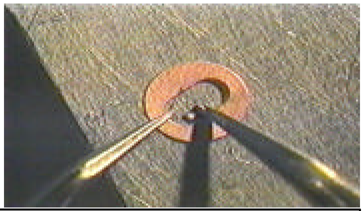


Bend the tab flat and center it in the hole of the grid.

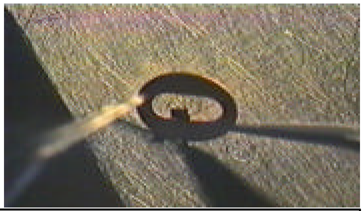
Grid Folding: Freestyle



Position the grid over the edge again along the line extending from the bottom of the cutout and bend the tab down to make the vertical tab.



Straighten and square the vertical tab by squeezing with tweezers.

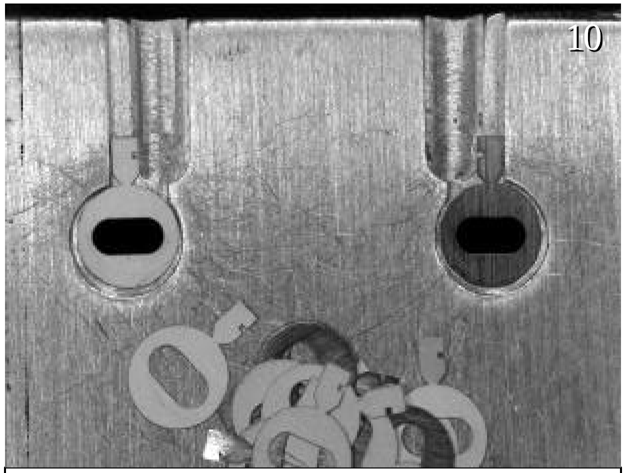


The finished grid is ready for the epoxy. Place it on a Teflon block with others of its kind.

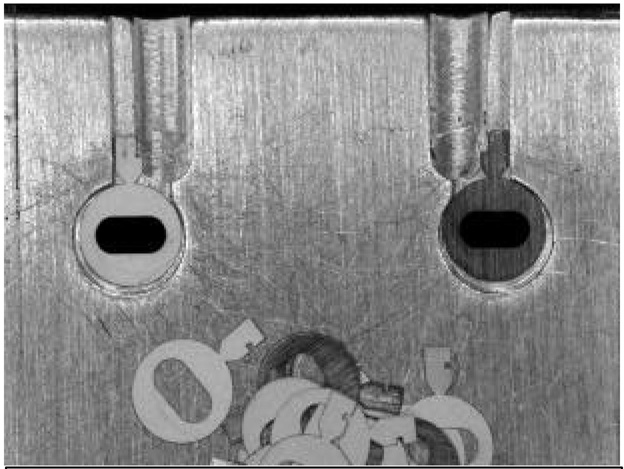
Grid Folding: Bending Jig

The grid bending jig is made by using a 1/8" end mill (a 3 mm end mill would work better) to make a seat for the grid. A ledge as wide as the bottom of the cutout to the side is made using a 1/16" end mill at the same depth as the seat for the grid. A 1/16" trough is cut in line with the bottom of the cutout area of the tab deep enough so that the vertical tab can be made.

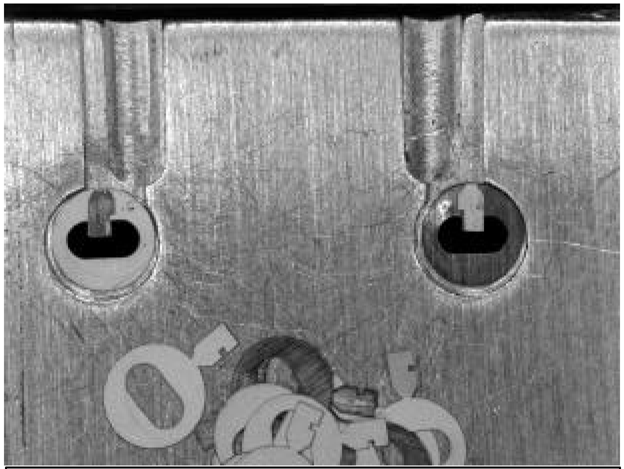
Note:
This jig can be made from the grid cutting jig designed by McCaffrey in the original SACT method.



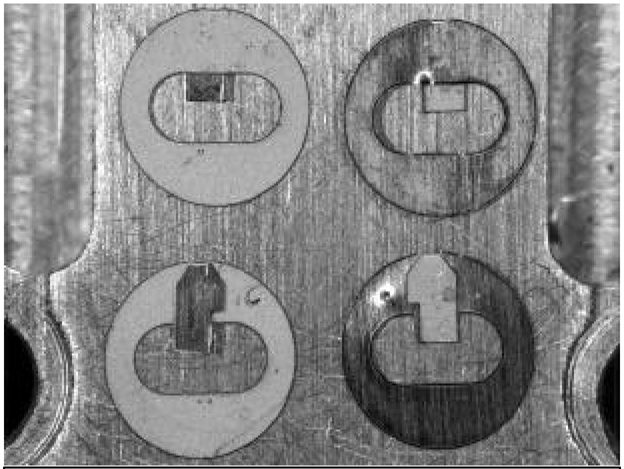
Grids are placed in the jig with the tabs on the ledge and held firmly against the wall with a pair of tweezers. The vertical tab part hangs over the ledge.



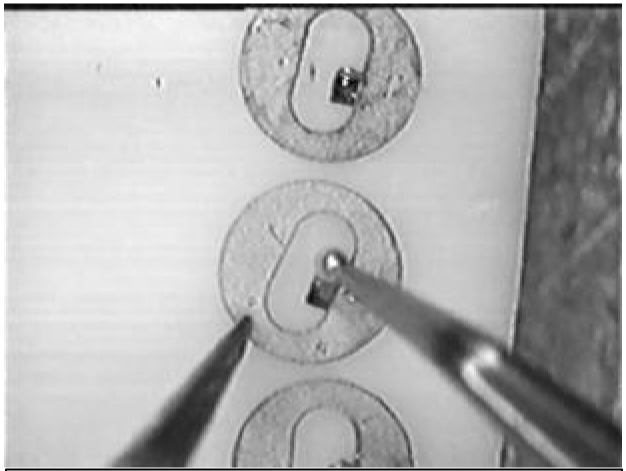
Bend the tabs down first. (This can be done first in the freestyle method, also.) Smooth the tab along the trough wall with tweezers to make a square bend.



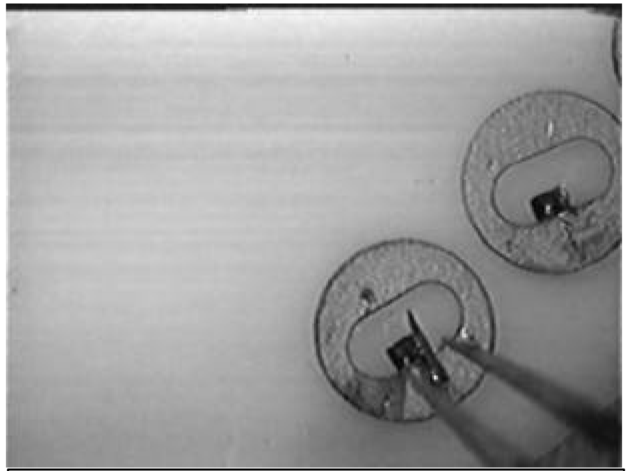
Style 1: Rotate the grid so that tweezers can get underneath the tabs and bend them up. Style 2: Flip them over and bend so that vertical tab goes thru hole.



The two sides of the bending jig can make left- and right-handed grids in both styles. Style 1 is easier to make, but is not as tight and requires epoxy.

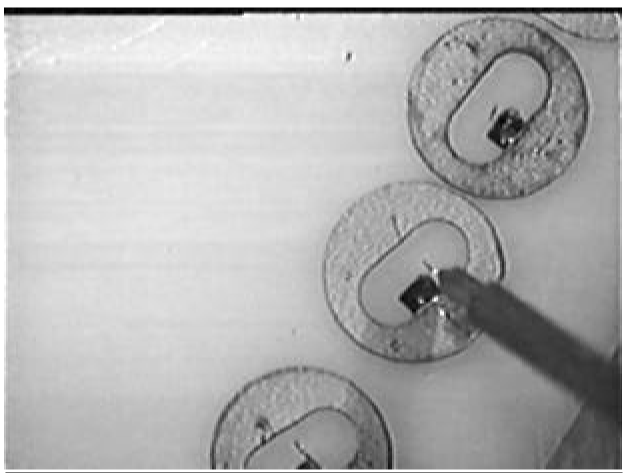


Place the folded grids on a Teflon block. Touch the end of the tweezers into the epoxy and place a small dollop of it on the vertical tab of folded grid.

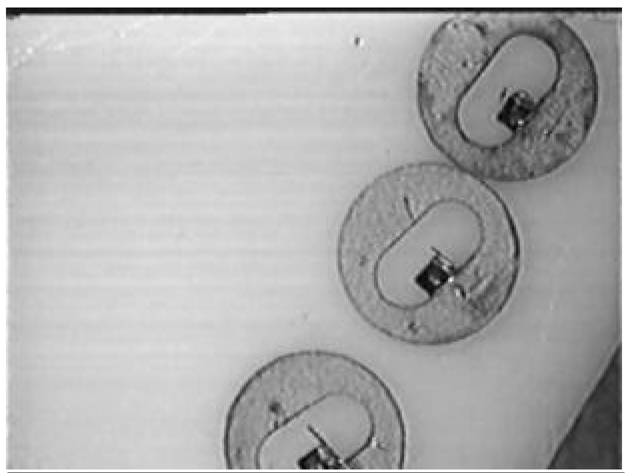


Pick up a cleaved sample and touch it to the epoxy on grid. Hold the grid down with a second pair of tweezers if needed.

Very small pieces can be picked up with the diamond tip dabbed with epoxy.



For the small piece not to be pulled into the epoxy, the dollop of epoxy on the vertical tab must be very small.

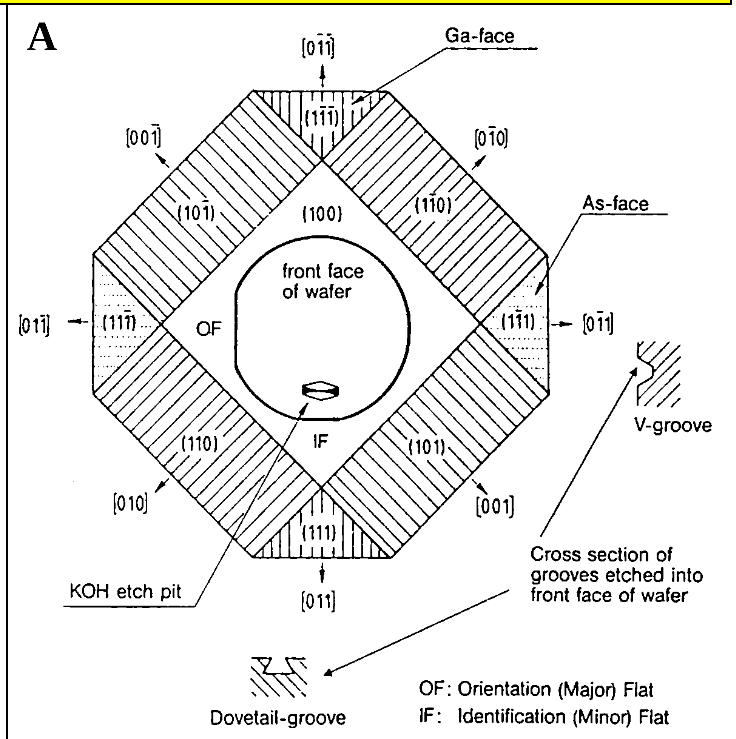


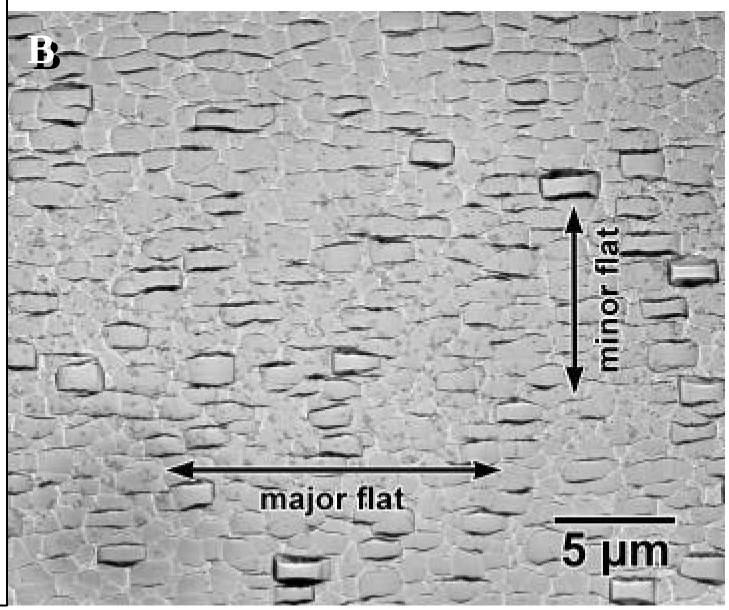
Finished samples all in a row! Cure the samples on the Teflon block with the temperature of the hotplate around 100-125°C and pop them into the TEM.

A test drop of epoxy on the Teflon block helps to determine when the epoxy is fully cured. Break off any portion of sample that will interfere with mounting the sample in the TEM holder. If the samples are heat sensitive, then On-The-Spot™ Super Strength Epoxy from Super Glue Corporation will work very well. This epoxy has a very long working time (>1½ hr), but takes more than 24 hr to fully harden. It is very viscous compared to shorter working time epoxies and will not "run" off the grid and onto the Teflon block.

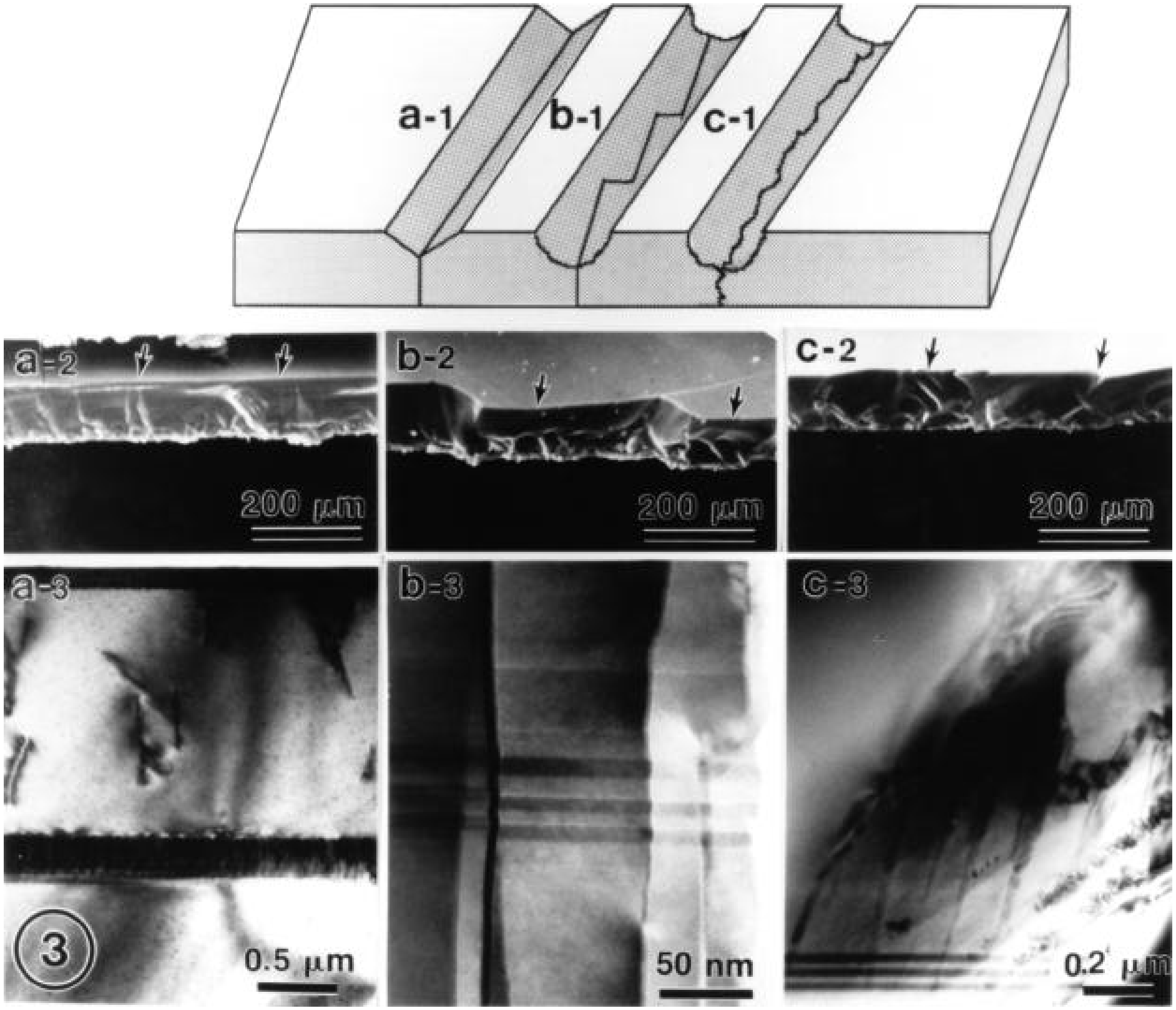
CLEAVING CONSIDERATIONS: Crystallography

There is a difference in cleavage behavior between different semiconductor materials. For example, Si substrates are a little more difficult to cleave than GaAs. For SACT, GaAs works well at 100 µm but it is a little easier on the diamond mini-scribes if Si is thinned to about 85-90 µm and SiC to 75-80µm. Some trial and error is required for different materials. If a wafer-thick sample of [100] grown Si is cleaved, most of the fracture faces along the normal cleavage direction will be either slanted in or out because it likes to cleave along {111} planes. Some of the fracture faces will be perpendicular to the surface the {022} planes. As the Si is thinned, the distribution of faces become more evenly mixed. However, for GaAs and other semiconductors having the zinc blend structure, the faces are almost always perpendicular to the surface, i.e. {022} planes. For GaAs type materials, there is also a difference in the relative ease of the two <011> cleavage directions because of the non-centrosymmetric nature of its crystal structure. A little experience with SACT of these materials is evidence enough that this is true. It helps tremendously in the final cleaving steps if the the easy cleave direction is oriented such that it is the one that is 12-15° from the scribe lines. There are several ways to achieve this. (i) Figure A shows a schematic of a GaAs wafer with the directions and the nomenclature of the flats indicated. The crystal orientations are determined by an anisotropic etch. Unfortunately, there are two specifications for determining them, SEMI and European/Japanese (EJ). Figure B is an optical micrograph of a GaAs wafer etched with an HF:H_2O_2 on the back. The easy cleave direction was determined to be parallel to the minor flat. With the special bond between the grower and the microscopist, one can always trust the grower to provide the sample referenced to the original wafer. (ii) Etch with an anisotropic etch on the back of the sample. (iii) Inspection (not always reliable): the cleavage face along the easy direction is typically flatter and more specular than the perpendicular direction. (iv) Since SACT uses so little material, mount two samples for thinning such that a mutual cleavage direction from the original wafer is perpendicular. Half of the samples will be oriented properly. The other orientation will provide some samples, but not as many per strip and at a higher frustration level.





CLEAVING CONSIDERATIONS: SACT

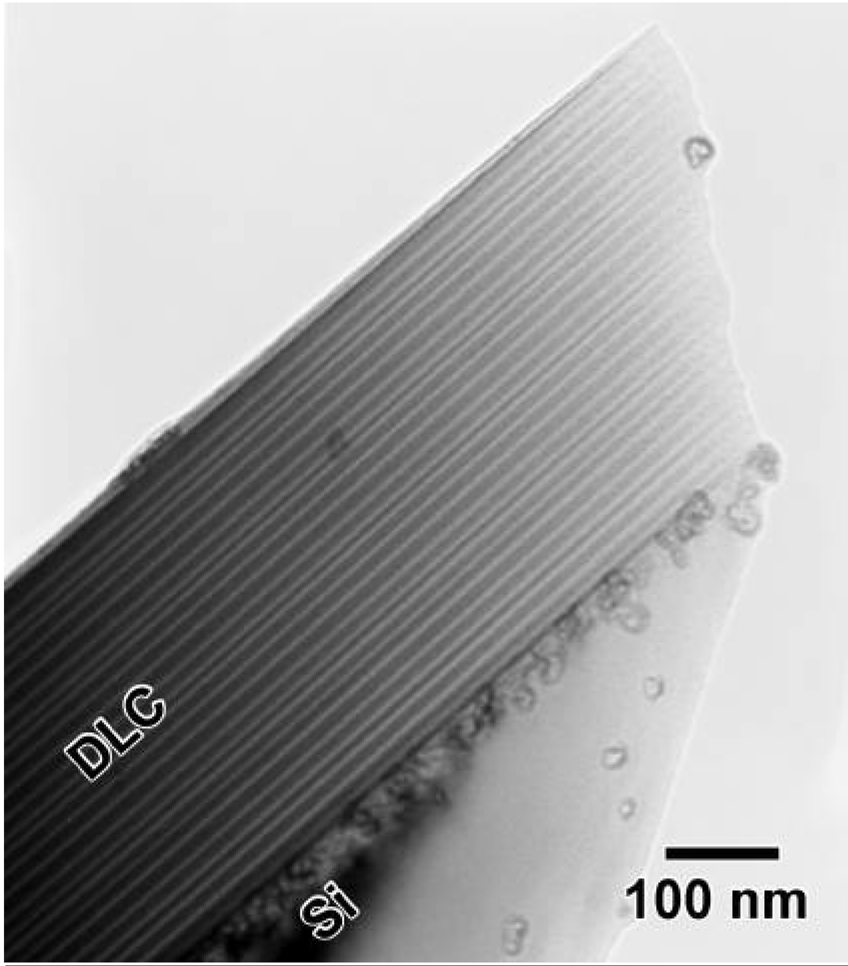


The top three drawings are schematics of possible cleaves or fractures that could occur in a single crystal material; a-1 indicates a perfect cleave along a perfect scribe line, b-1 shows a series of cleavage steps along a flat-bottomed scribe line, and c-1 shows a random fracture consisting of cleaves and tears along an uneven scribe line. Figs. a-2, b-2 and c-2 are representative SEM images of cleaved or fractured silicon wafer edges corresponding to a-1, b-1 and c-1 respectively. The arrows indicate the top wafer surface, immediately below which are the areas of interest when viewed by TEM. Figs. a-3, b-3 and c-3 are TEM images of samples prepared by SACT where the second face of the cross-sectional sample is a perfect cleave along a {110} planes, but the first face is a cleave or fracture corresponding to a-1, b-1 or c-1.

With the aid of a good stereomicroscope during the final cleaving stages, the intersection of the fracture face and the surface can be seen. If the edge is clean and specular such as it would be in b-2, then a good sample will result. If the face tucks under the surface at an angle, then the sample will have a long knife-like appearance. If it goes out, then the sample will have a short distance of electron transparent area going into the sample along the surface, but it will be a sturdy. If the fracture face is perpendicular to the surface, then the chances are high that a stepped sample will result having terraces of equal thickness for all of the layers. This is the case in b-3. Sometimes, as the samples are being cleaved, a curved edge will occur. These are very characteristic of having these good intersections with the surface. If they make a small angle with a cleavage direction, especially an easy direction, they will make good samples. An example of a "knife-like" and a "step-like" sample is shown in the next frame.

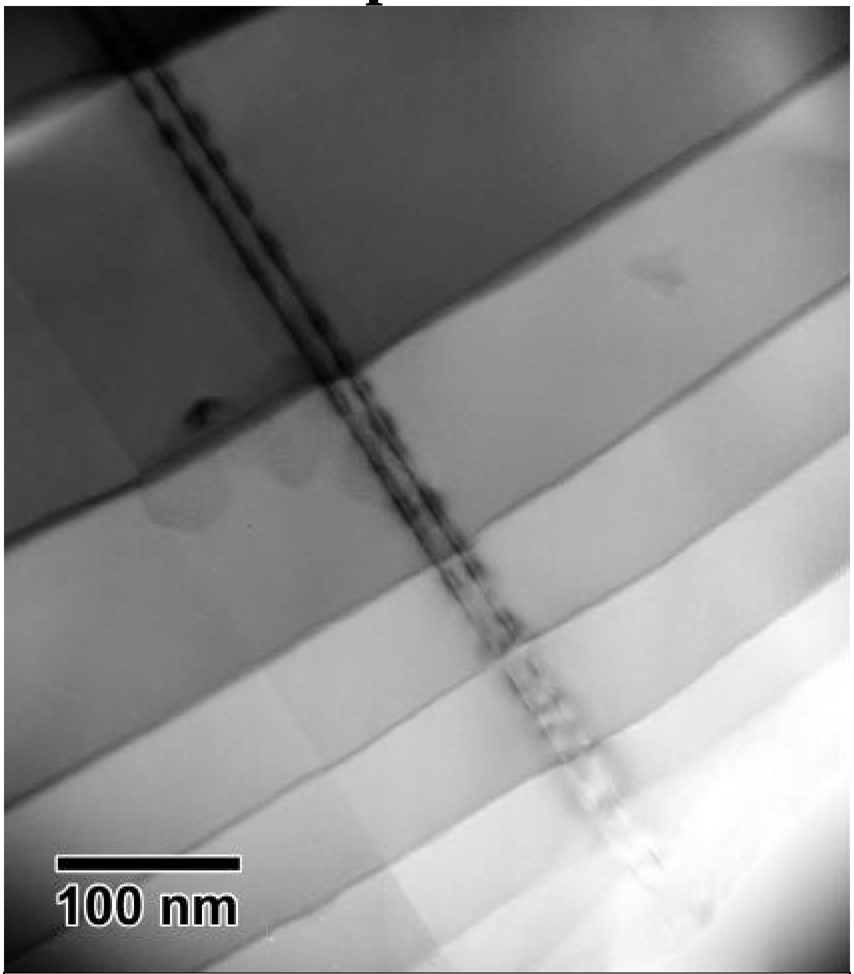
CLEAVING CONSIDERATIONS: Sample Geometry

"Knife-Like"



This figure shows a pulsed laser deposited diamond-like carbon film on a silicon substrate. The film is amorphous. The striations are density variations in the film and are caused by angular variations in the ablation plume as the laser is rastered over the target.

"Step-Like"

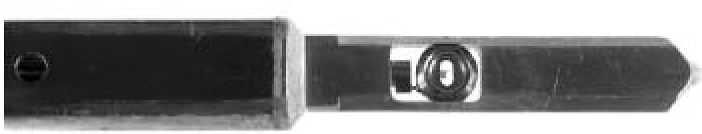


This figure shows a series of cleavage steps on a GaAs-based sample of InGaAs quantum dots. The steps range in length from approx. 70 to 200 nm. Quantum dot structures can be employed to enhance the performance of InGaAs based semiconductor lasers.

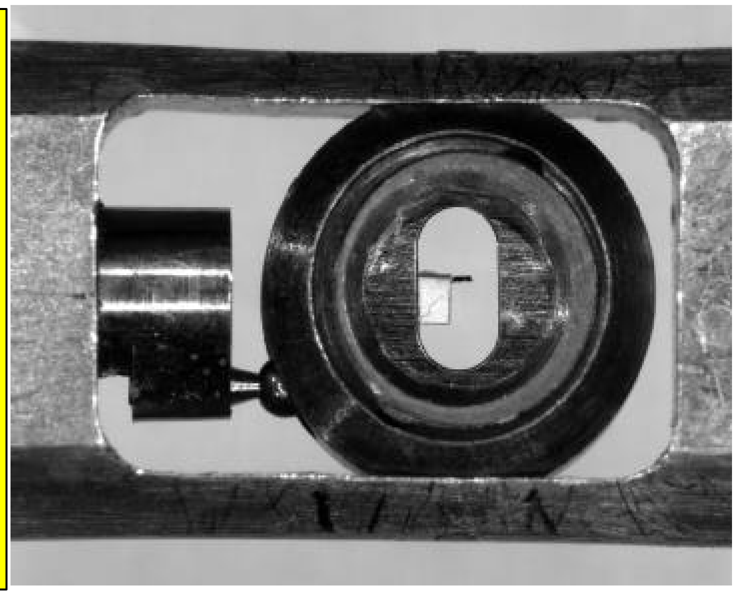
ADVANTAGES OF SACT GEOMETRY

__Quickly Examine Many Samples__: If the samples are mounted into the TEM stage in the same orientation with their apex along the center of the specimen rod, as shown in the two figures, then finding the transparent area and making it eucentric in the stage can be done very quickly. This also enables many samples to be quickly examined. First pull/pull the sample out/in along the rod axis so that the beam is in the grid hole and the front ledge of the tab is seen. Go left or right until either the sample or the side edge of the tab is reached. (Of course, go the other way if the side edge of the tab is reached.) Pull the sample back until the tip is seen. This is either the back or surface of the sample. If it is rough, it is the back side and go to the other side of the sample. If the sample's surface is not parallel to the beam, then the shadow image of the surface will have steps visible, much like viewing a staircase from above and to the side. Rotate the rod axis ("x"-tilt) until the surface is straight and the steps are not visible. (This is similar to viewing the stairs from the top of the staircase.) If a previous sample was imaged at the eucentric height, focused, and the astigmatism corrected, then adjust the height until minimum contrast is seen in the image with the objective aperture out. If the sample is mounted fairly parallel along the rod's axis, i.e. not tilted in the "y"-axis, then the sample can be judged worth of further TEM investigation. If not, take it out and put another sample in. The turn-around time required for screening samples for quality is about 5-10 min which includes mounting the sample in the TEM stage.

__"Tilt-Assist"__: In a double-tilt TEM stage, the crystallography and tilting to specific diffraction conditions of single crystal substrates become very easy since major zone axes can be placed parallel to the two tilt axes. The second tilt axis on double-tilt stages are typically limited to about 30 . The manner in which the samples are mounted on the special grids can be used to give a "tilt-assist" to the second tilt axis of the double-tilt stage by bending the tab that the sample is on. A partial Si Kikuchi map showing the extended angular range with this "tilt-assist" is shown in the next frame. This "tilt-assist" technique is particulary well suited for high resolution microscopes in which the tilt ranges are limited.

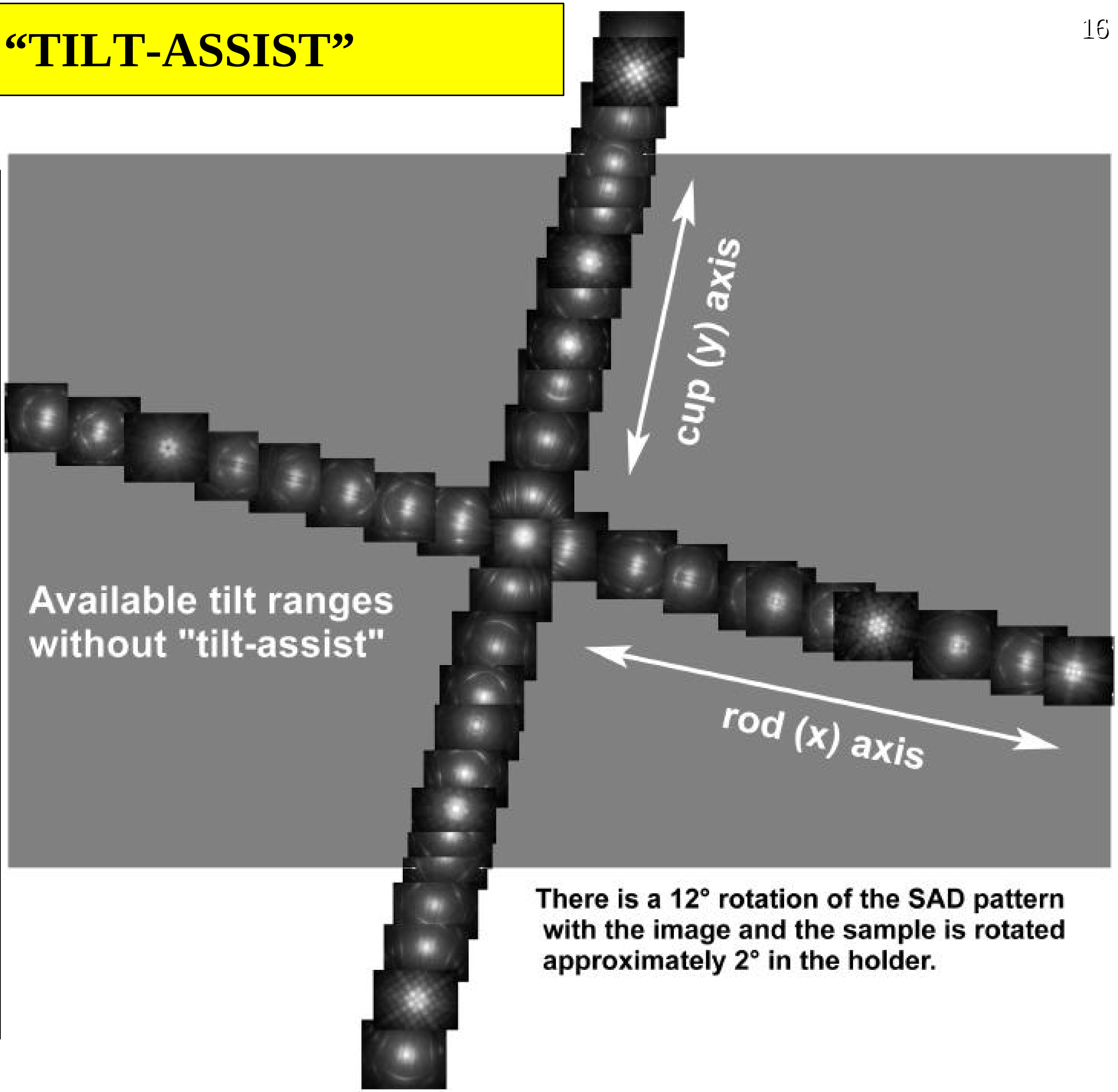


SACT Sample in a Gatan Double-Tilt TEM Holder for a JEOL 2000FX



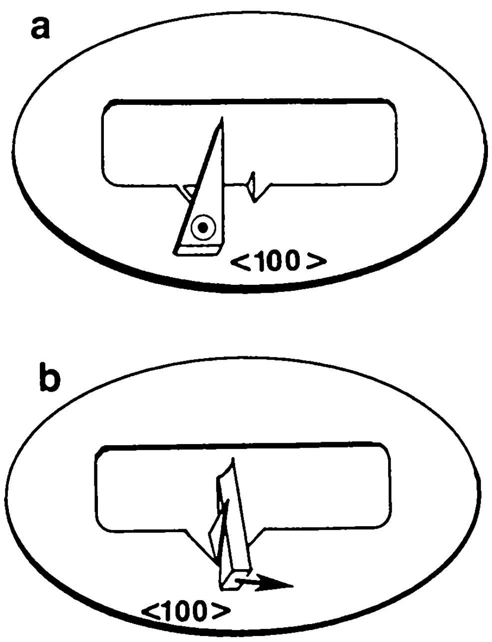
SACT "TILT-ASSIST"

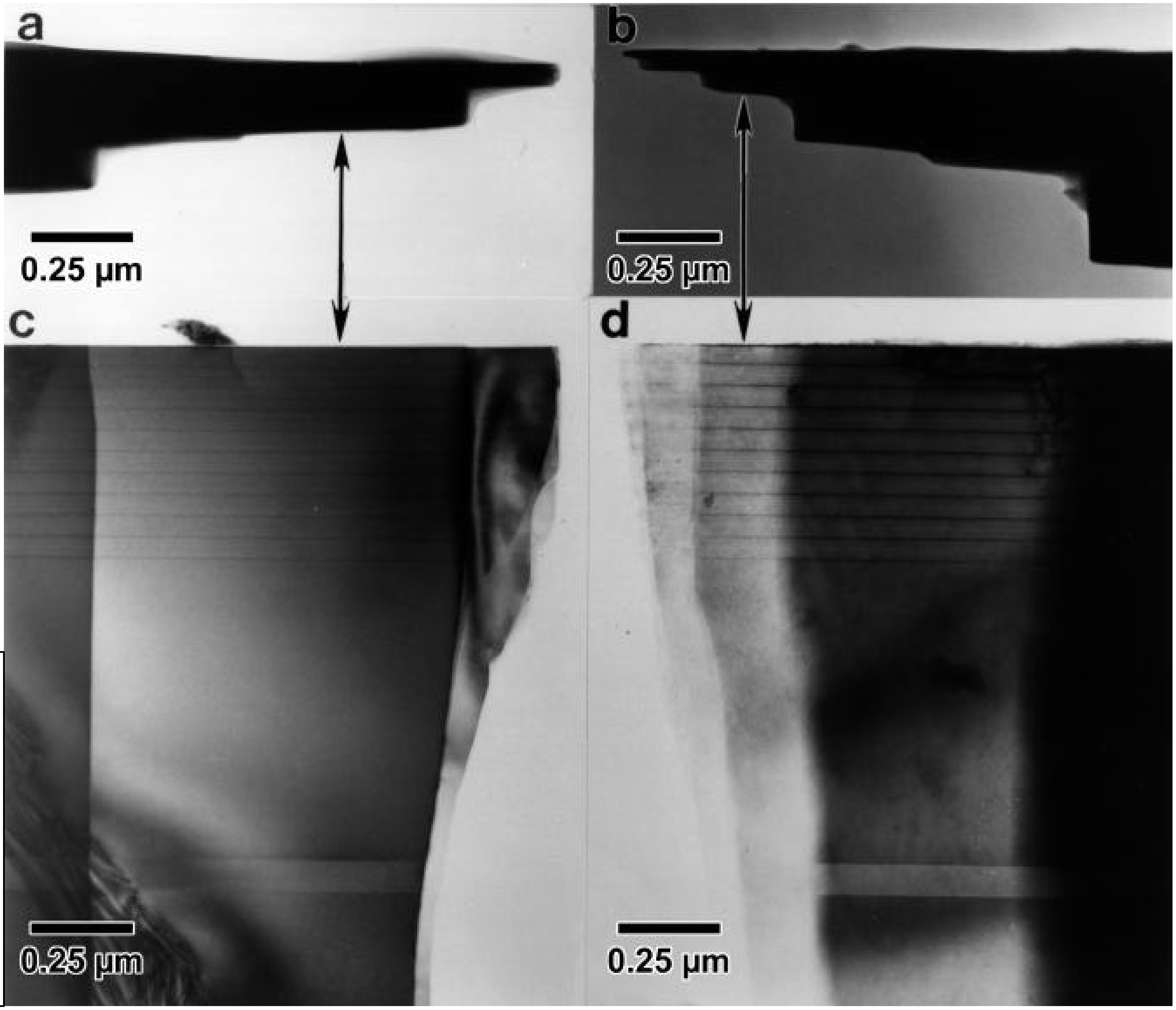
The geometry of the SACT sample is ideally suited for rapid crystallographic orientation and determination in the TEM. Furthermore, if the sample is mounted in the direction of the TEM rod, then the zone axis coincident with the surface normal is parallel to the "y"-tilt axis and the trace of the plane perpendicular to the surface is parallel to the "x"-axis. This permits rapid alignment of the sample in cross section. The sample tilt can be extended in the "y"-axis by physically bending the tab that the sample is mounted on up or down. In the figure, both the [010] and [001] of Si have been reached using the same sample. The extra tilt available with the SACT sample allows imaging contrast conditions and orientations for CBED analysis that would otherwise not be able to be performed.



There is a 12° rotation of the SAD pattern with the image and the sample is rotated approximately 2° in the holder.

"TILT-ASSIST" IN EXTREMUS

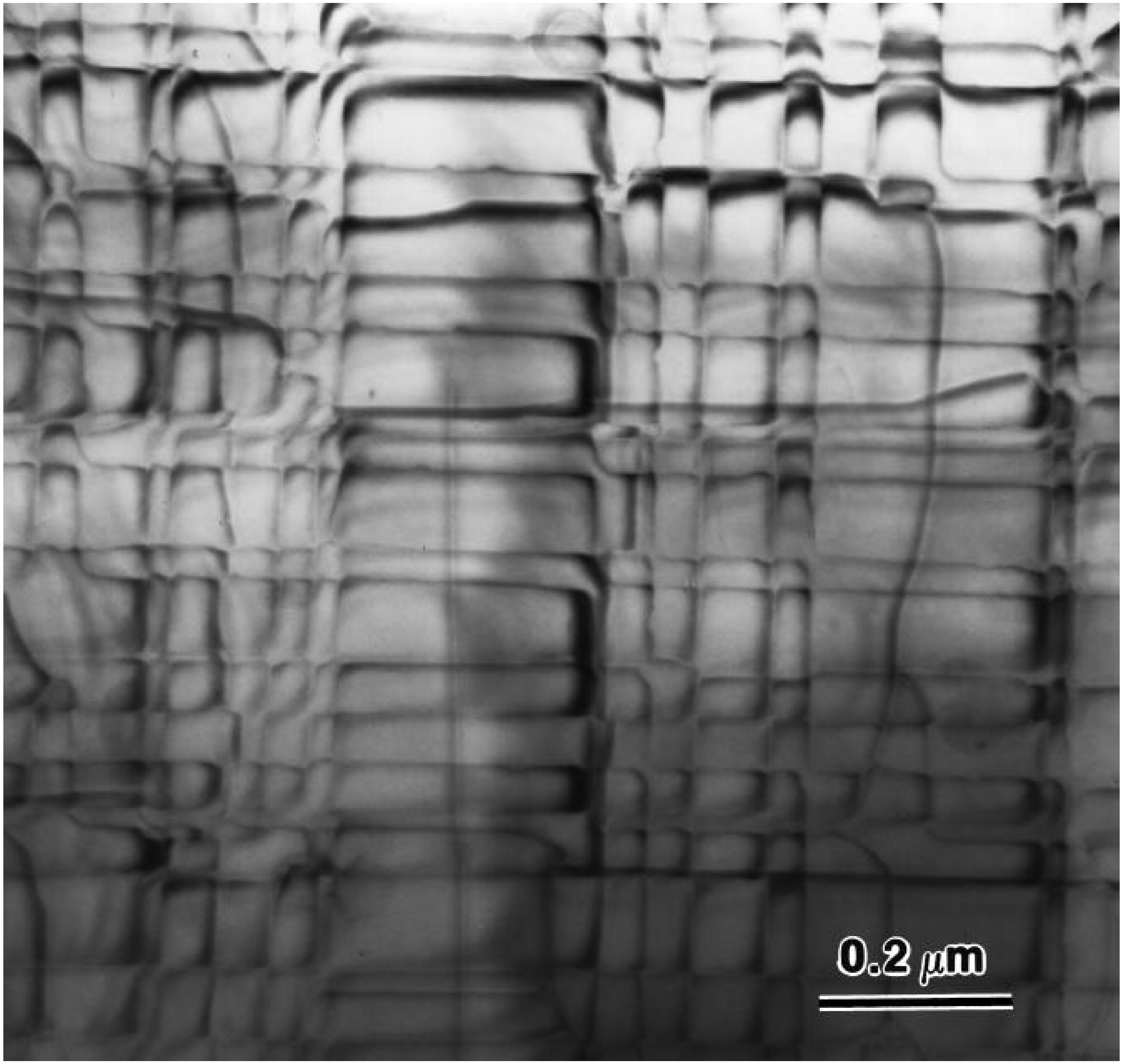




If the sample is epoxied to the vertical tab very carefully, then the tab itself can be bent over. This allows the examination of the sample in both a plan view (a) to measure thickness of the steps and XTEM view (b). The sample can also be examined in a high resolution SEM very easily in this manner.

PLAN VIEW TEM SAMPLES FROM SACT

Useful plan view TEM samples can also be made by SACT, although with much less control and predictability. The same basic procedures are used as was for XTEM samples, except, instead of looking for samples with sharp tips when observed from the surface side, a sample with a rounded tip is sought which also has a long needle shaped tip when observed in cross section. Often, when a sample is cleaved or fractured, the surface side of the fracture face will have a thin ledge of material containing the surface layer. Mounting this sample with the surface side down on a slotted grid may produce a usable plan view TEM specimen. Note that care must be taken to perform TEM observations only in areas free from cleavage tears or other preparation-induced artifacts. This figure shows a plan view TEM bright field image of a 0.16 µm thick $Si_{0.8}Ge_{0.2}$ layer on <100> silicon prepared by SACT.



JOHN AND SCOTT'S SACT TIPS AND TRICKS

- **Cleaving Technique:**
  - **It helps to have a clean, sharp diamond scribe for this step. Clean debris off of the tip of the scribe using a Post-it note.**
  - **For easily cleaved material such as the III-V materials, scribe lightly starting near the apex and draw the scribe back while gently adding pressure. A little pressure to the outside will pull the cleaved part away from strip.**
  - **For silicon, try a light scribe line, as straight as possible, and then lay the scribe along the line near the bottom of the sample and press the scribe gently down (slight rolling motion might help). This works particularly well using the Post-it note instead of water.**

- **Use a Post-It Note for Final Cleaving:**
  - **The Post-it note works very well as a substitute for the water. It has a resilience to it that helps the cleave lines propagate straight to the edge. Very little of the glue, if any, will stick to the sample and it can be cleaned in acetone after the epoxy cures. Very little damage can be done to the thin area due to excessive handling as in the case of fishing the samples out of the water, putting them down on the Teflon with a drop of water, wicking the water away with filter paper, and picking them up for final mounting. With the Post-it, the sample can be mounted directly after it is cleaved or saved in a corner of the Post-it with others. With modest care, only the bottom ground portion of the sample touches the Post-it. Originally, it was thought that there would be excessive debris on the surface in the electron transparent area, but this proved not to be the case. In addition, it is very easy to shorten the cleaved samples to the appropriate length for mounting while they are on the Post-it so that the excess overhang doesn't have to be broken off of the final sample after the epoxy is cured, further minimizing handling.**

- **Coat a Sample with Carbon:**
  - **A small price to pay for SACT samples in terms of imaging is that there are no amorphous areas in the sample if the material does not start out that way and astigmatism correction can be difficult. One trick is to put a light coat of carbon on the sample surface prior to beginning the process. This puts a little amorphous material in the region of interest that can be used to correct for astigmatism.**

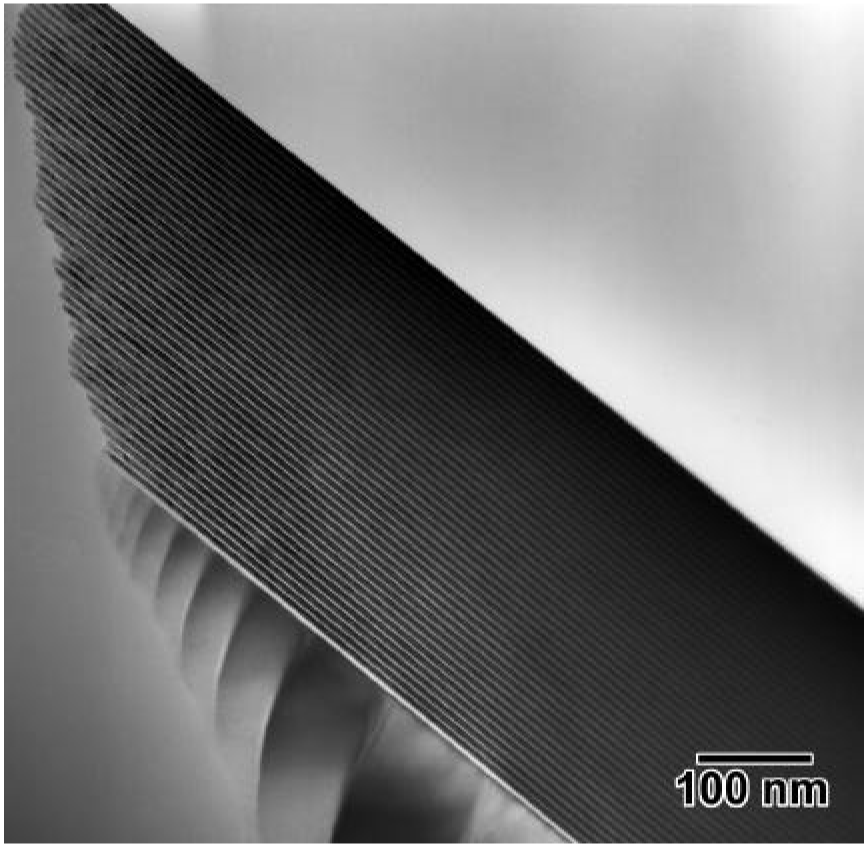
JOHN AND SCOTT'S SACT TIPS AND TRICKS

- **Fracturing Brittle Substrates:**
  - **Glass and other brittle materials can be made with SACT. The substrate should be as thin as possible because it is hard on the diamond tips. Saving broken diamond scribes for fracturing the brittle substrates is well advised. When performing the final cleave (fracture), the tip of the scribe should be as close to the edge as possible. Almost as much sideways pressure is applied as downward pressure. Patience is important here. Allow the tip of the scribe to start a crack propagating and control the speed with the pressure. Look for curved surfaces that have good clean edges and try to intersect the surface with the propagating crack.**

- **Quick Examination of Films:**
  - **Often, only the microstructure of the thin film or coating is of interest, not the substrate or the substrate/film interface. If this is the case, consider growing these types of films on No. 0 glass cover slips. They have a thickness of 80-130 µm and can be prepared directly without any pre-thinning. The No. 1 glass cover slips are 130-170 µm thick and are hard on the diamond scribes, but samples can be prepared from them. Unlike single crystal substrates with known orientations, the amorphous glass substrates are more difficult to align properly in the TEM for minimum thickness and having the surface parallel to the beam. It is recommended that amorphous substrate samples be put into the TEM perpendicular to the rods axis. It is harder to find the minimum thickness of the sample and this orients the sample with the tilt axis that maintains the eucentric condition. To set up the surface of the sample parallel with the beam, observe the thicker part of the film/substrate interface and tilt with the "y"-axis to obtain the best image of the interface.**

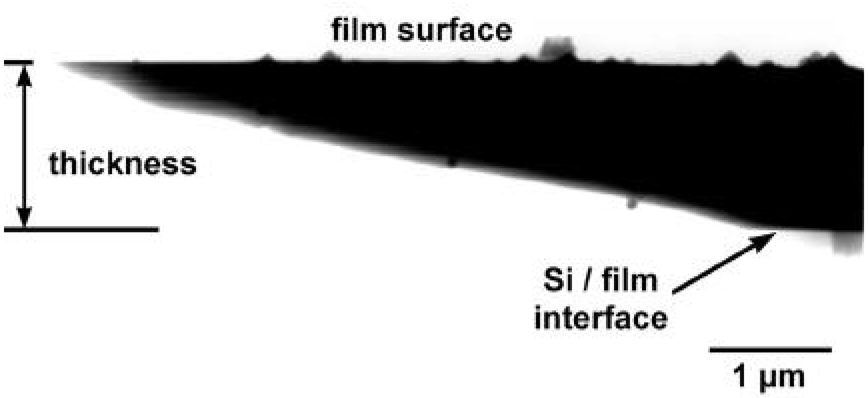
EXAMPLES OF SACT PREPARED SAMPLES

A Multiple Quantum Well structure grown by Molecular Beam Epitaxy showing the thickness terraces. The GaAs buffer, 50 periods, and GaAs cap are all seen with very small variations in thickness within a step.

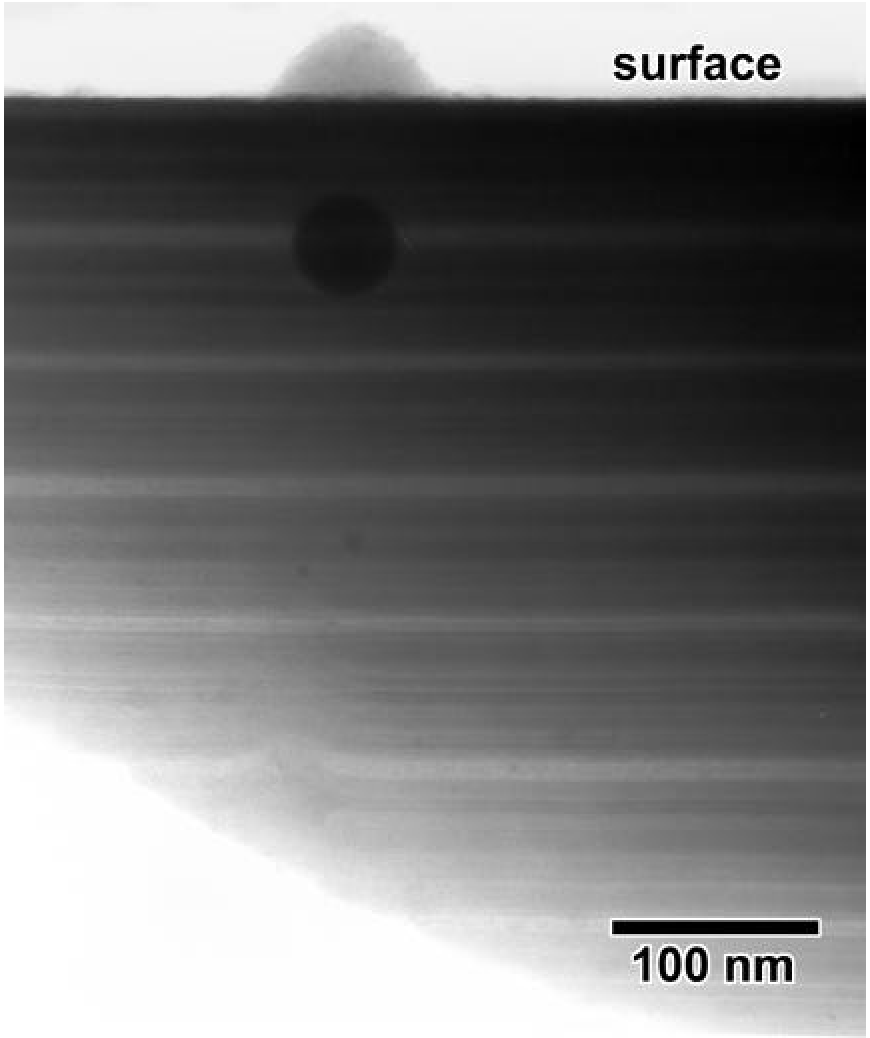


An x-ray mirror consisting of 45 layers of alternating Mo/amorphous Si (7.0 nm period) on a Si Substrate. The film was deposited using ion sputtering.

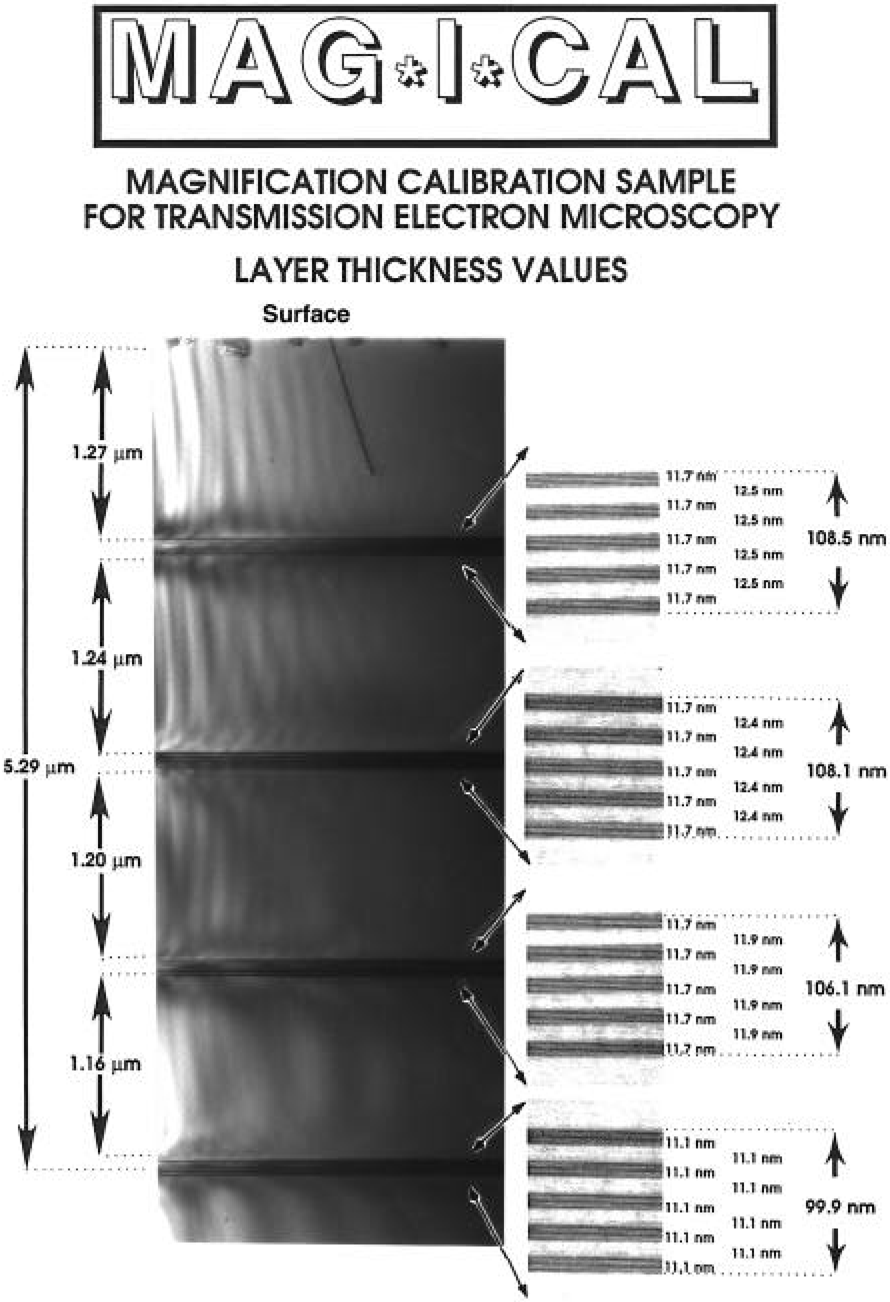
EXAMPLES OF SACT PREPARED SAMPLES



A pulsed laser deposited film of ZnO/WS_2, a high temperature adaptive solid film lubricant. The sample has the “knife-like” SACT appearance, but is electron transparent along the edge. The film is amorphous and the striations are due to differences in the W concentration because of the small angular distribution of W in the ablation plume. This sample shows very clearly how the spherical particles, inherent to the PLD process, project their presence to the surface.



WORLD’S SMALLEST RULER



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1. J. P. McCaffrey, Ultramicroscopy, 38, (1991) 149.
2. J. P. McCaffrey, Specimen Preparation for Transmission Electron Microscopy III, eds. Ron Anderson, Bryan Tracy, and John Bravman, Materials Research Society, Pittsburgh, Materials Research Society Symposium Proceedings., Vol 254, 1992 p. 109.
3. John P. McCaffrey, Microscopy Research and Technique, 24, (1993) 180.
4. Scott D. Walck, *A Simplified Method for Modifying TEM Copper Grids For Use with the Small Angle Cleavage Technique, Microscopy Today*, (96-4), (1996).
5. S. D. Walck and J. P. McCaffrey, *The Small Angle Cleavage Technique Applied to Coatings and Thin Films*, submitted to Thin Solid Films, March, 1997.

SOURCES FOR SUPPLIES

1. Precision Hand Grinder:

   South Bay Technology, Inc., San Clemente, CA (www.southbaytech.com)

2. Stackable Lapping Tray or Polishing Station, Diamond Lapping Film:

   South Bay Technology, Inc., San Clemente, CA (www.southbaytech.com)

3. Epoxy: EpoTek H-22, Epoxy Tchnology, Inc, Billerica , MA

   or On-the-Spot™ Super Strength Epoxy, Super Glue Corporation, (Wal-Mart)

4. Mini Diamond Scribes, Tweezers, Tabbed Grids, 100 count grid boxes, paint brush, Plastic Scale:

   South Bay Technology, Inc., San Clemente, CA (www.southbaytech.com)