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**Procedures for the Preparation of Samples for
Cross-Sectional Transmission Electron Microscopy**

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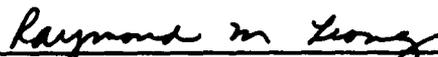
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19. ABSTRACT (Continue on reverse if necessary and identify by block number) Cross-sectional transmission electron microscopy (XTEM) is an important technique for studying the morphology and local chemistry of the interfaces of thin film materials. The preparation of samples for XTEM analysis can be summarized as follows: <ul style="list-style-type: none"> • Samples are cleaved or sawed into 2 mm x 7 mm strips. • A sandwich consisting of four strips is glued together. • 50-mil-thick slices are cut from the sandwich. • The slices are ground and polished to a thickness of 100 μm. • The thin slice is glued to a TEM grid and is dimpled until a perforation is achieved. • The dimpled specimen is thinned to electron transparency with an ion-milling machine. A detailed account of the procedures used to prepare XTEM samples of Si-Ge superlattices is given. Selected micrographs of these samples show how the XTEM technique can be used to study interfaces.				
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PREFACE

The authors thank R. C. Bowman and H. Kanter for providing support for this work, and S. Gyetvay, H. Kanter, D. Smith, and E. Watts for sharing their experiences with sample preparation and operation of the various pieces of equipment. Special gratitude is expressed to C. Ahn of the California Institute of Technology for demystifying the sample preparation procedure, ion-milling of the samples, and providing the transmission electron micrographs. The Si-Ge superlattice samples were grown by V. Arbet of the Device Research Laboratory of the Electrical Engineering Department at UCLA.



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I. INTRODUCTION

The characterization of the morphology and local chemistry of interfaces is an important aspect of contemporary materials science. This is particularly critical for thin film materials where the volume of material that is near the interface becomes a larger fraction of the total. Few analytical techniques have the resolution necessary to study interfaces in detail. Cross-sectional transmission electron microscopy (XTEM), combined with energy dispersive x-ray spectrometry or electron energy loss analysis, is a technique that can image interfaces with very high resolution (several angstroms) and provide chemical information about areas as small as 40 Å in diameter. As a result it is one of the most powerful methods for studying the interfaces of thin film materials. A number of similar methods have been described for preparing XTEM samples (Refs. 1-6).

The following is a brief summary of the basic XTEM sample preparation procedure:

- Approximately 2-mm x 7-mm strips of samples (plus blanks) are cleaved or sawed.
- A sandwich of four specimens is glued together and cured with M-bond 610 adhesive for 1 hr at 100°C.
- 50-mil-thick slices of the sandwich are cut with a wafer saw.
- One side of a 50-mil-thick slice is ground and polished to a final thickness of about 100 μm.
- The 100-μm-thick slice is epoxied to a TEM grid.
- A dimple is ground into the 100-μm-thick slice until a small perforation is achieved.
- The dimpled specimen is thinned to electron transparency (1/2 to 1 hr) using an Ar ion-milling machine.

This procedure seems simple and straightforward. However, attempts by numerous individuals have been unsuccessful, in part, because as stated in Ref. 1 "... that mundane, but critical, steps in the specimen preparation

may be omitted in the conventional journal articles." Conversations with C. Ahn of the California Institute of Technology indicated that this XTEM sample preparation procedure can be modified such that, after some initial practice, it should be possible to routinely prepare four XTEM samples a day (ready for ion milling) by processing several samples at a time during some steps, such as glueing and initial grinding. With this encouragement, we reattempted XTEM sample preparation: Our efforts resulted in the first successful XTEM samples prepared using equipment in the Aerospace laboratories.

A detailed account of sample preparation is outlined in the appendix. Its purpose is to document what we learned in developing this capability, and serves as a starting point for others who are interested in preparing XTEM samples. As noted in Ref. 2, there is no one correct way of preparing XTEM samples. The intention of this outline, along with subsequent XTEM micrographs, is to give a degree of confidence to anyone who is interested in preparing XTEM samples. With additional experience, it should be possible to modify the procedure by eliminating nonessential steps, thereby making the process less time consuming.

II. XTEM APPLIED TO Si-Ge SUPERLATTICES

In spite of the 4.2% lattice mismatch between Ge and Si, device quality $\text{Ge}_x\text{Si}_{1-x}/\text{Si}$ strained laser superlattices (SLSs) have been grown by molecular beam epitaxy (MBE). Among the interesting and useful properties of $\text{Ge}_x\text{Si}_{1-x}/\text{Si}$ heterostructures is the predicted quasi-direct optical transition in superlattices consisting of alternating monolayers of pure Ge and Si. However, to achieve this transition, it is necessary to have abrupt interfaces between the layers. Careful control over the growth conditions and subsequent thermal treatment are therefore significant factors influencing the quality of the SLS. As a result, it is important to characterize the quality and perfection of the SLS prior to measuring and interpreting its optical properties.

Six Si-Ge SLS superlattice samples were prepared for XTEM analysis. Figure 1 describes the superlattice and tunneling structures of the samples that were prepared. As an example, sample H56 consists of a Si(100) substrate upon which a 200-Å Si buffer was first grown. This was followed by 2000 Å of a relaxed $\text{Si}_{.6}\text{Ge}_{.4}$ alloy buffer. A strained layer superlattice (SLS) was then grown on the alloy buffer. The SLS consisted of 40 pairs of pure Si and pure Ge layers, 24 monolayers and 16 monolayers thick, respectively. For a (100) substrate, a monolayer of Si or Ge equals approximately 1.4 Å. The XTEM samples were analyzed at the California Institute of Technology using a Philips Electronics Instrument Model EM430 scanning transmission electron microscope with a LaB_6 filament and operated at 300 kV.

The six samples prepared for XTEM were also analyzed by x-ray diffraction (XRD) to determine the overall quality of the structures and measure the superlattice period. For superlattice samples the layered structure, with a repetition length (period) d , acts as artificial Bragg planes for x-ray diffraction. Low angle XRD scans will record the $(000)_n$ x-ray reflections of the superlattice structure, and the period of the SLS can be determined from Bragg's law:

$$n\lambda = 2d \sin \theta$$

H56

SiGe (24 x 16) SLS
40 PERIODS

2000 Å Si_{0.6}Ge_{0.4} BUFFER

200 Å Si BUFFER

Si (100) SUBSTRATE

VA19

SiGe (32 x 8) SLS
60 PERIODS

20,000 Å Si_{0.8}Ge_{0.2} BUFFER

Si (100) SUBSTRATE

VA25

SiGe (20 x 20) SLS
55 PERIODS

2000 Å Si_{0.5}Ge_{0.5} BUFFER

300 Å Si

Si (100) SUBSTRATE

VA30

SiGe (5 x 5) SLS
217 PERIODS

2000 Å Si_{0.5}Ge_{0.5} BUFFER

300 Å Si

Si (100) SUBSTRATE

VA35

4000 Å Si CAP

400 Å Si

12 PERIODS

SiGe (6 x 4) SLS
7 PERIODS

400 Å Si

4000 Å Si BUFFER

Si (110) SUBSTRATE

R255

5000 Å Si_{0.7}Ge_{0.3} CAP

150 Å Si

50 PERIODS

30 Å Si

100 Å Si_{0.5}Ge_{0.5}

150 Å Si

10,000 Å Si_{0.7}Ge_{0.3} BUFFER

Si (111) SUBSTRATE

Fig. 1. Schematic Representations of Si-Ge Superlattice Structures Prepared for XTEM

where λ is the wavelength of the x-rays, d is the superlattice period, θ is the angle between the incident x-ray beam and the Bragg planes, and n is an integer. The interface quality of the SLS can be estimated from the full width at half maximum (FWHM) and the number of orders ($n=1,2,3,4 \dots$) of the $(000)_+$ reflection. An example of a low angle XRD scan of a good quality SLS is shown in Fig. 2. A large number ($n=1-9$) of higher order reflections are observed, and the peaks have very narrow FWHMs. This sample (VA21) consists of 60 periods of a Si-Ge (8×32) SLS grown on 8000 Å of a relaxed $\text{Ge}_{.8}\text{Si}_{.2}$ buffer layer on a (100) Si substrate.

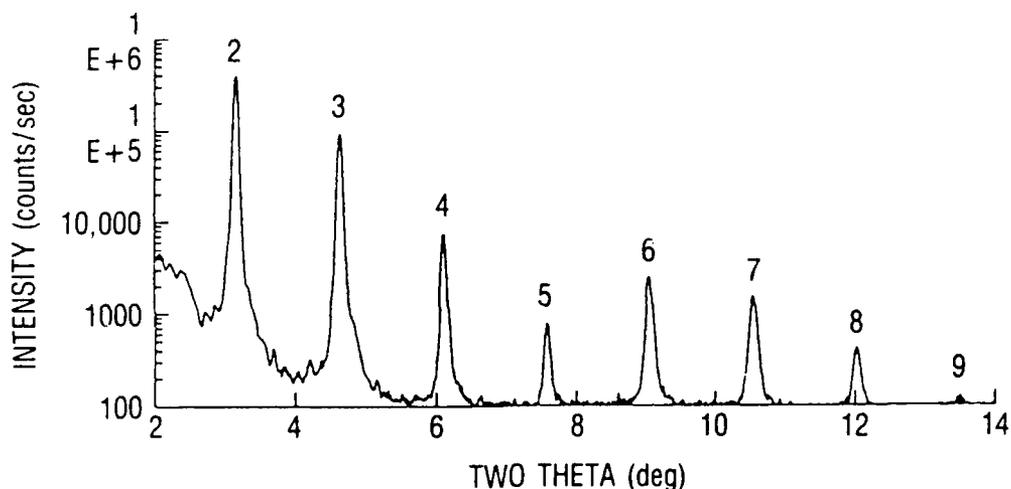


Fig. 2. Low Angle XRD Scan of SLS Sample VA21

The low angle XRD scans of the six samples prepared for XTEM analysis are shown in Fig. 3. Clearly, a wide range in quality exists in these samples as compared to that in Fig. 2. The lack of higher order superlattice reflections is usually explained in terms of non-abrupt interfaces, that is, the composition profile has a sine wave rather than a square wave form. Other defects are expected to affect the low angle XRD profile but the exact effect is often uncertain. For this reason, XTEM analysis is critical in determining the exact cause of degradation of the superlattice. By direct imaging of the interfaces, defects in the SLS can easily be identified and quantified.

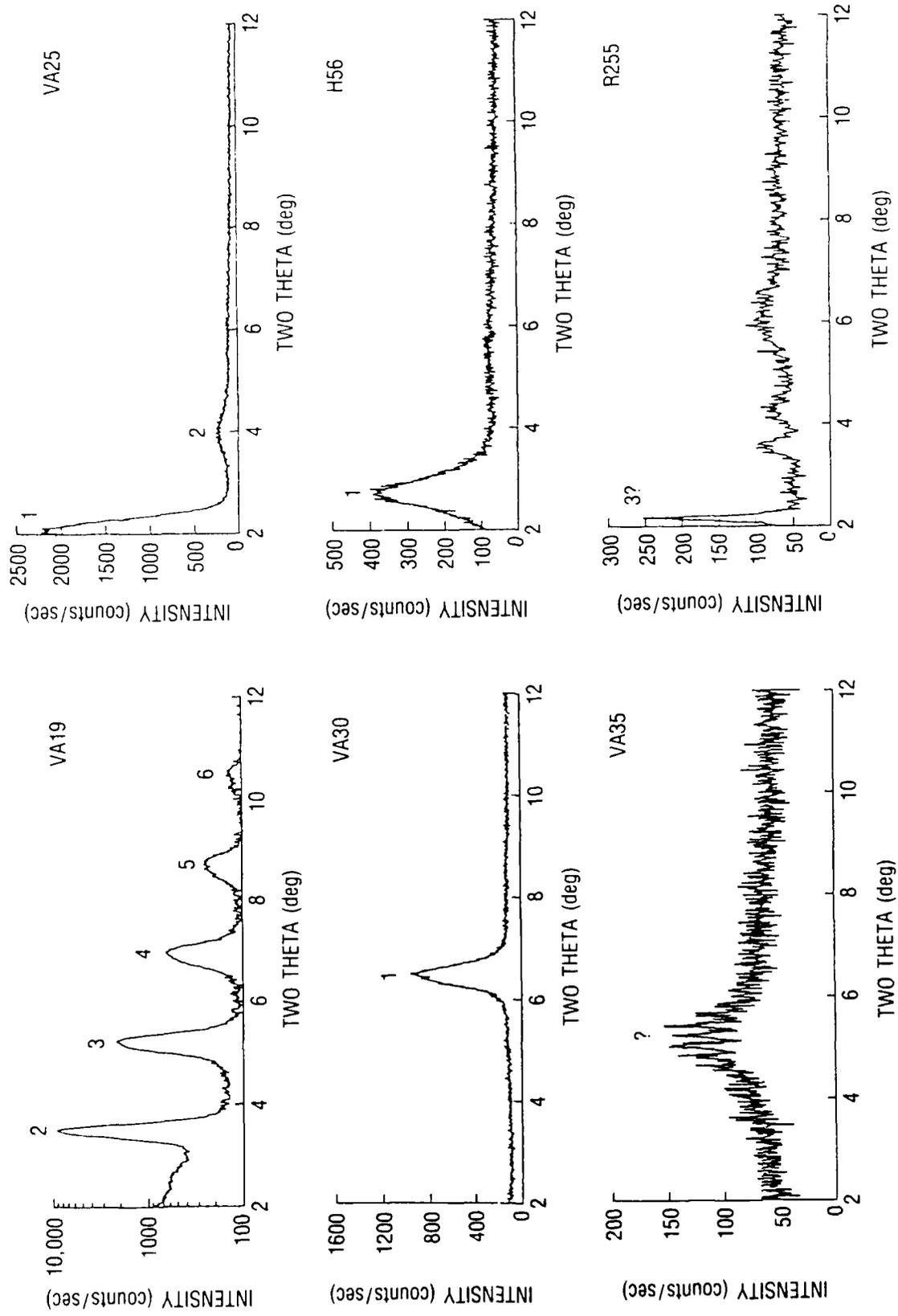


Fig. 3. Low Angle X-Ray Diffraction Scans of Si-Ge Superlattice Samples Prepared for XTEM

III. XTEM MICROGRAPHS

Figure 4 represents six XTEM micrographs obtained from five Si-Ge SLS samples that we prepared. Figure 4a is an XTEM micrograph of sample H56. The superlattice layering is clearly visible, and it is obvious that the layers in the superlattice are not flat, but wavy, and are not of constant thickness throughout the structure. These defects are responsible for the poor low-angle XRD pattern from this sample, since the superlattice structure is far from ideal. Sample VA19 produced one of the better XRD patterns that we observed from an SLS. Two XTEM micrographs of this sample are shown in Figs. 4b and 4c. In Fig. 4b the buffer layer is generally free of dislocations, except at the interface with the substrate, and the buffer-SLS interface is very smooth. Dislocations, however, are generated into the SLS beginning at the buffer interface and, while the SLS does not exhibit wavy layers, there are unusual thickness variations within the structure. Comparing the XRD pattern of VA19 (Fig. 3) with VA21 (Fig. 2), it is evident that VA19 does not have quality comparable to VA21. The defects that are responsible for the slightly degraded XRD pattern are clearly visible in the XTEM micrographs.

An XTEM image of sample VA25 is shown in Fig. 4d. In this sample loop-like dislocations are visible, in addition to wavy superlattice layers and an irregular SLS-buffer interface. Figure 4e is an XTEM micrograph of sample VA30. The SLS structure (14-Å period) is faintly visible, and dislocations in the buffer have propagated into the SLS. An XTEM micrograph of sample R255 is shown in Fig. 4f. Twins (diagonal lines) are present in this sample which were not observed in the other samples. This sample differed from the others in that it was grown on a $\langle 111 \rangle$ substrate and was highly doped. This may have been responsible for the different type of defects that were observed.

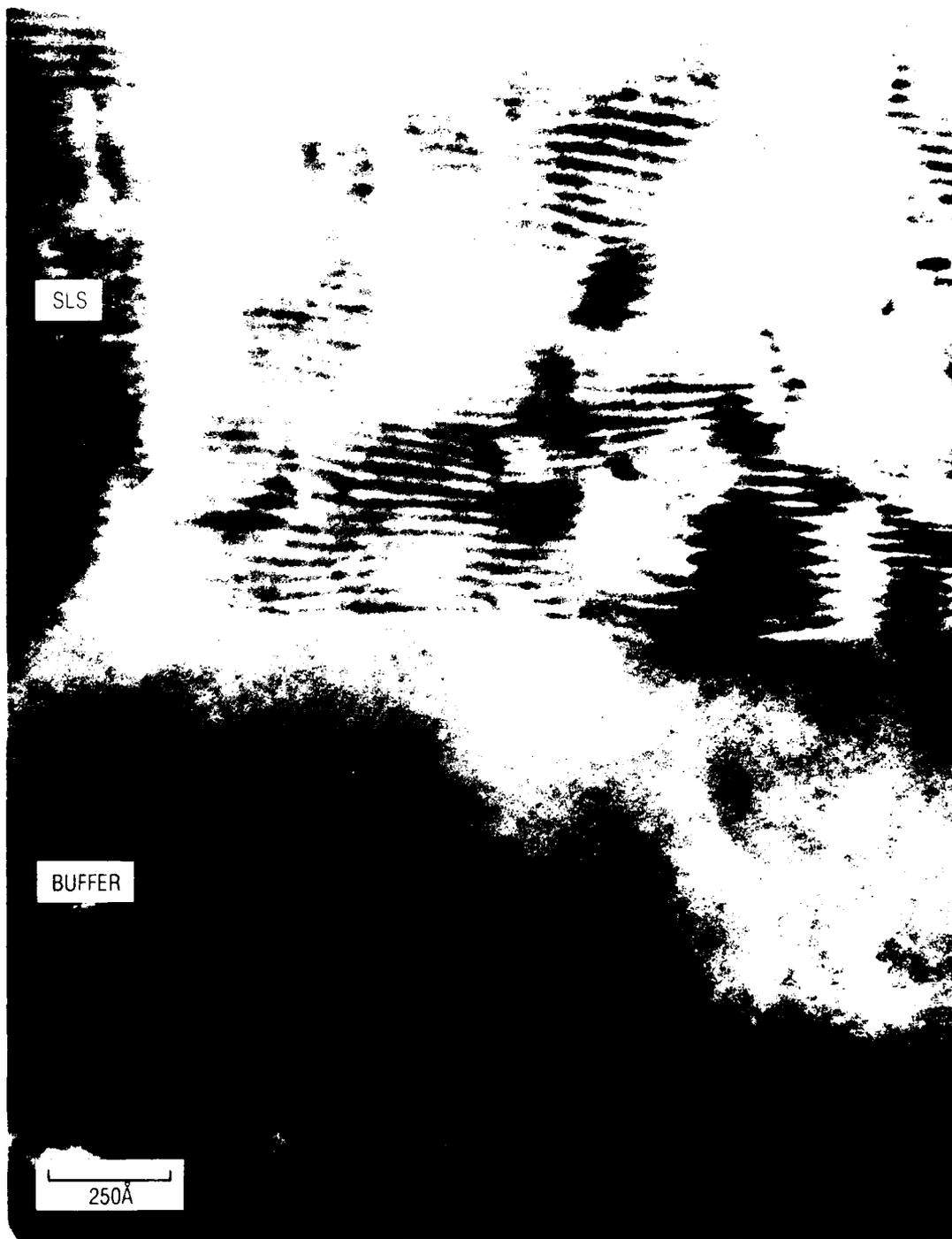


Fig. 4a. Cross-Sectional Transmission Electron Micrograph of Sample H56. H56 = 40 periods of Si-Ge (24 × 16) monolayers SLS on 2000-Å Si_{0.6}Ge_{0.4} buffer. SLS period = 33 Å.

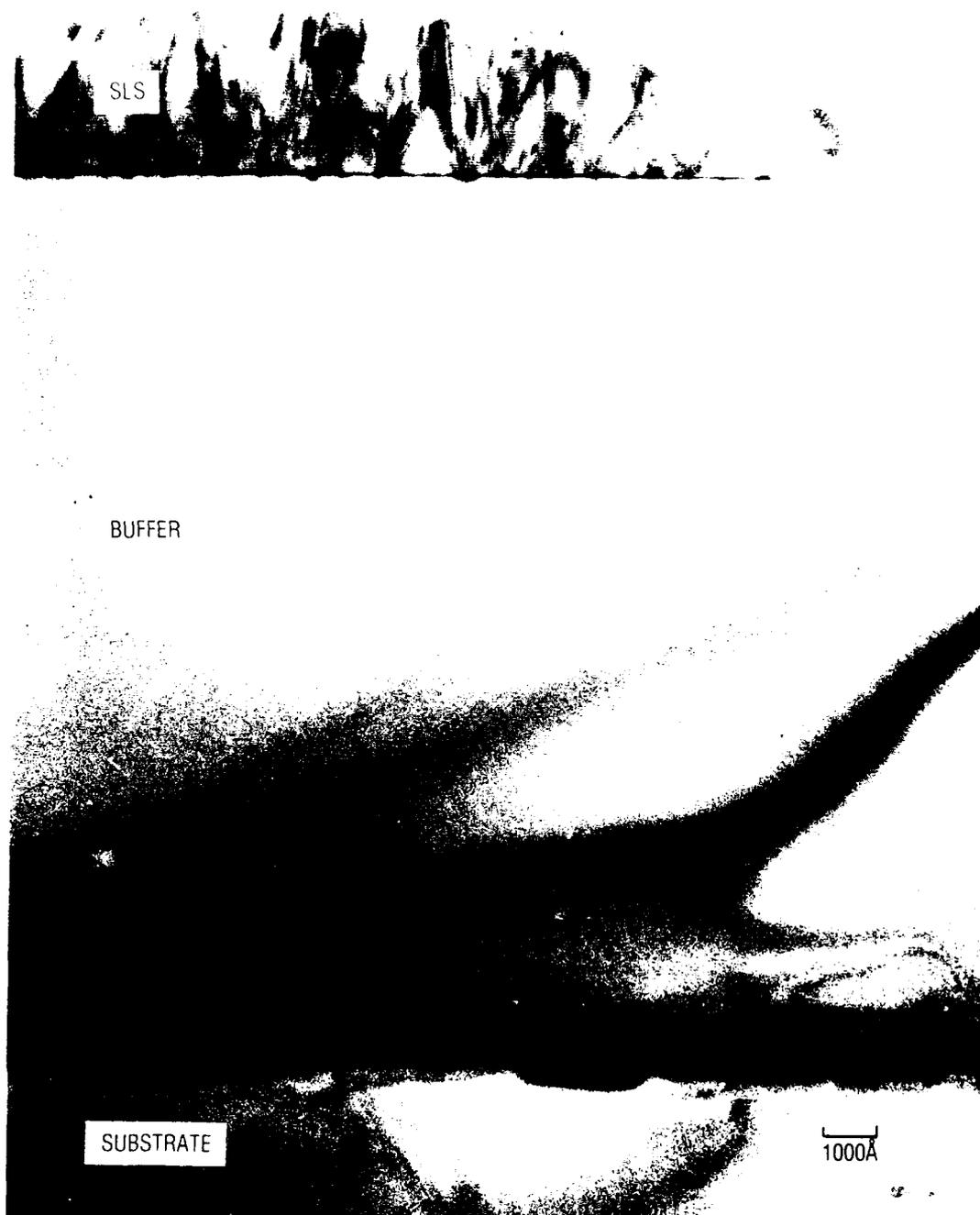


Fig. 4b. Cross-Sectional Transmission Electron Micrograph of Sample VA19. VA19 = 60 periods of Si-Ge (32 × 8) monolayers SLS on 20,000-Å $\text{Si}_{.8}\text{Ge}_{.2}$ buffer. SLS period = 54 Å.



Fig. 4c. Cross-Sectional Transmission Electron Micrograph of Sample VA19. VA19 = 60 periods of Si-Ge (32 x 8) monolayers SLS on 20,000-Å Si_{0.8}Ge_{0.2} buffer. Close-up of SLS. SLS period = 54 Å.

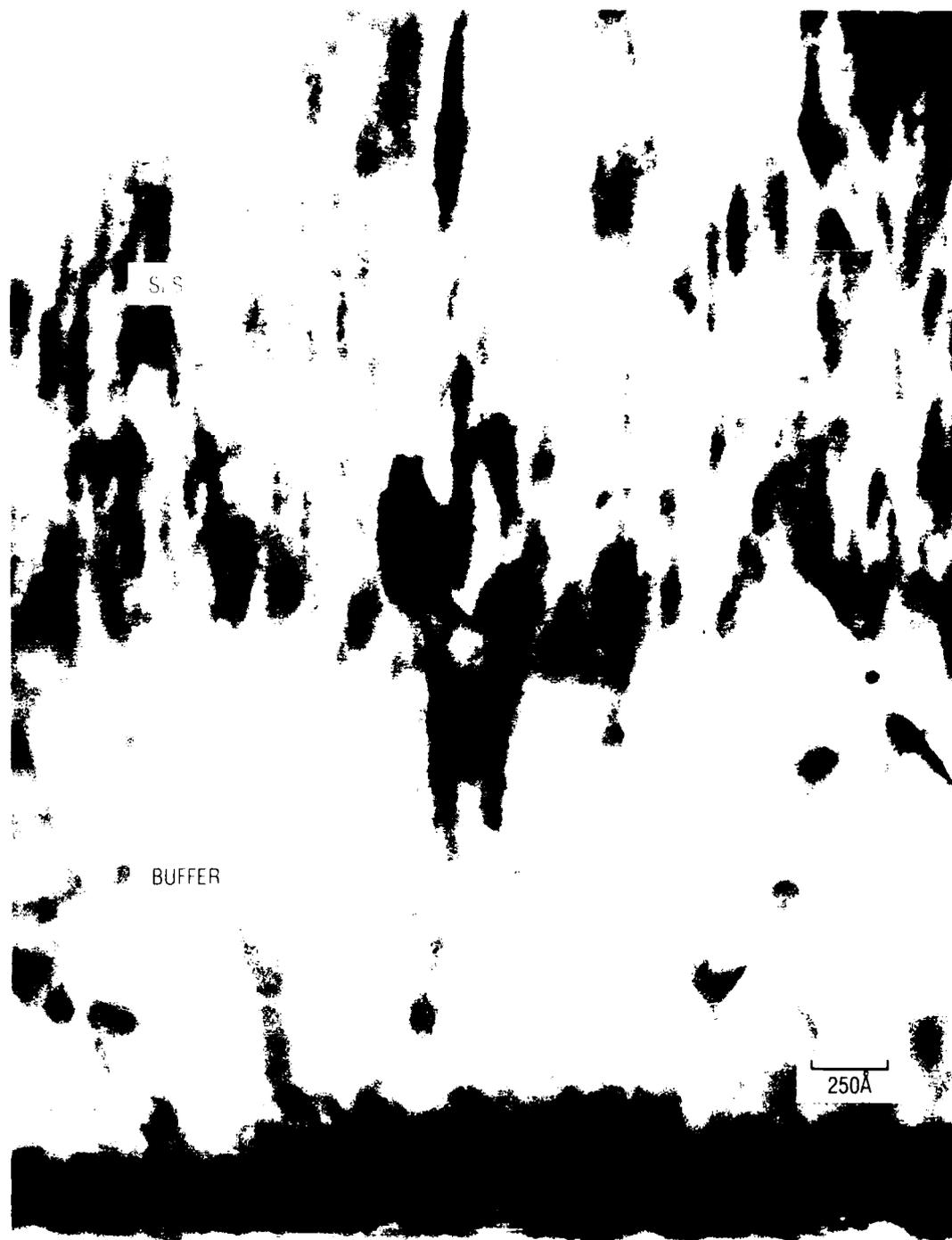


Fig. 4d. Cross-Sectional Transmission Electron Micrograph of Sample VA30. VA30 = 217 periods of Si-Ge (5 × 5) monolayers SLS on 2000-Å Si_{0.5}Ge_{0.5} buffer. SLS period = 14 Å.



Fig. 4e. Cross-Sectional Transmission Electron Micrograph of Sample VA25. VA25 = 55 periods of Si-Ge (20 x 20) monolayers SLS on 2000-Å Si_{0.5}Ge_{0.5} buffer. SLS period = 50 Å.



Fig. 4f. Cross-Sectional Transmission Electron Micrograph of Sample R255. See Fig. 1 for SLS structure. SLS period = 130 Å.

IV. SUMMARY

A procedure has been outlined for the preparation of samples for cross-sectional transmission electron microscopy (XTEM), and successful XTEM images (Figs. 4a-4f) have been obtained from samples prepared in the Materials Sciences Laboratory using this method. Defects in the strained laser superlattice (SLS) samples are clearly visible in the XTEM images. In comparison, x-ray diffraction (XRD) is an easy nondestructive method for qualitatively estimating the overall quality of the SLS, but specific defects cannot be identified. With XTEM, however, the type and distribution of defects can be identified, and it may be possible to better understand how they are generated and, therefore, modify growth procedures accordingly.

Although sample preparation may be more time consuming compared with other analytical methods, the strength of XTEM analysis lies in the ability of the observer to identify individual defects and examine direct images of interfaces at resolutions that no other technique can match.

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APPENDIX. XTEM SAMPLE PREPARATION FOR Si AND GaAs

The following is a detailed account of the XTEM sample preparation procedure that was used for Si and GaAs:

I. Si (or Si/Ge) Samples

A. Sandwiching

1. Cleave or cut at least two samples, approximately 2 mm wide by 5 mm to 10 mm in length. Scrap pieces of silicon should also be cut to these dimensions and used to bring the total thickness of the sandwich to the appropriate thickness (~ 2 mm) when there is insufficient sample material.
2. Clean sample strips in acetone with an ultrasonic cleaner for approximately 5 min and follow by rinsing with Genusolv D solvent. Blow samples dry with compressed air. Samples should be handled with a clean set of tweezers that have been rinsed with acetone and Genusolv D.
3. Glue four strips together, polished sides toward center, using M-bond 610 adhesive that is at room temperature. The number of strips glued together can be adjusted, depending on the thickness of the material, as long as the total thickness of the sandwich is approximately 2 mm. Scrap pieces of silicon may be substituted for the outer layers of this sandwich when sample material is at a premium, but the two sample-to-sample interfaces should be positioned at the center of the sandwich. Constructing the sandwich completely from the sample of interest, if it is abundant, has the advantage that if the perforation formed by dimpling is far off center it may still intersect a suitable interface. The adhesive should be stored in a refrigerator following mixing of the two components and allowed to reach room temperature before using. Shelf life of the unmixed adhesive is approximately 1 yr, while the shelf life of refrigerated mixed adhesive is about 5-6 weeks. To apply the adhesive, make sure that the brush is completely wet with adhesive and use one brush stroke to apply adhesive to the strip (or two strokes for the outer interfaces). Apply the adhesive to only one of the surfaces between each strip. Cure the sandwich for 1 hr at 100° Celsius in a finger-tight vise on a hot plate. Monitor the temperature of the hot plate with a hot plate thermometer.

B. Sawing with the Buehler Isomet Wafer Saw:

1. Place an aluminum bar on a hot plate (approximately 100°C) and melt Crystalbond 509 on the bar. Mount the sandwiched sample on the bar when the adhesive is liquid. Remove and cool.
2. Cut three or four 50-mil-thick cross sections with a 6-mil-wide, 3-in.-diameter diamond blade using a low speed setting of about 3-4, and using water as a lubricant. While cutting, carefully watch the blade and manually shut off the saw when the blade starts cutting the aluminum bar. Attempts to cut sections thinner than 50 mils usually resulted in fracturing of the thin slice being cut.
3. Melt the Crystalbond by placing the aluminum bar on a hot plate. Remove the slices, and soak samples in acetone to clean off the adhesive.
4. Measure the thicknesses of the samples with a micrometer capable of measuring ± 0.1 mil.

C. Grinding with the Buehler Minimet Polisher

1. Mount the samples with Crystalbond adhesive onto a cylindrical block (as in step B.1.). Up to three duplicate specimens of as many as four different samples may easily be ground simultaneously on one cylindrical block. Also mount scrap pieces of Si at the edges of the cylindrical block to prevent the edges of the samples from rounding. Check that the pieces of scrap Si are all the same thickness so that the block grinds evenly.
2. Measure the thickness of the mount with each sample and calculate the desired thickness of the mount plus the sample for each sample to have a final thickness of approximately 100 μm (4 mils).
3. If the upper sample surface is irregular, start with 320-grit paper at a speed of 3, with no load. Use Metadi fluid lubricant with time settings of about 1-3. The time settings of the Minimet polisher correspond to the following approximate elapsed times: (1) = 30 sec, (3) = 1 min, 10 sec, (5) = 2 min, (9) = 3 min, 30 sec.
4. Once the exposed edges are smooth, increase the speed to 6 and the load to 1/2 to 3/4, using time settings of 3-5.
5. If there is a 3-mil or more difference in sample thicknesses, hand grind to even out.

6. When the samples are about the same thickness (~ 15 mils) as the outer scrap pieces, switch to 400-grit paper. Use a fresh piece of abrasive paper each time, since the removal rate will depend on amount of abrasive grit on the paper.
7. Gradually vary the load and speed until an appropriate removal rate is obtained, usually found at a speed of 6, 1/2 load, and a time setting of 3-5.
8. When the thickness of the mount plus each sample reaches the desired amount or within 1-2 mils of the desired amount, begin polishing.
9. To polish, use a speed of approximately 5, a load of 1/2, and a time setting of 3 with the Texmet pad and 1.0- μm alumina-water slurry. Repeat until most large scratches are removed (usually once.) Check for the removal of scratches with a reflected light microscope (Lietz Ortholux or equivalent) with a magnification of approximately 200X.
10. Switch to 0.3- μm alumina-water slurry on a separate Texmet pad at the same settings and repeat until most scratches are gone (once usually suffices).

[OPTIONAL STEP(S)] additional polishing with 0.05- μm alumina and/or Syton on Texmet pads is recommended by some experimenters, but it was not necessary for Si-Ge samples.

- D. Dimpling on the South Bay Technology Dimpling Machine. (For grooving, follow the same procedure as dimpling except do not rotate the base.)
1. Dismount one of the polished samples by the method described in B.1. The sample may be removed from the block with a toothpick to avoid damaging it with metal tweezers. Using Devcon 5-min epoxy, glue the polished side of the sample to the rough side of a nickel or copper grid that has an oblong hole in its center. Use a broken toothpick to apply a thin layer of the epoxy to the part of the grid that will be in contact with the sample. Wipe excess epoxy away with a clean toothpick. A vacuum tweezer was used to position the sample on the grid. The center interface of the sample should be centered along the length of the hole. Take care not to get epoxy on the polished surface that is visible in the grid aperture.
 2. Use Crystalbond to mount the grid to a glass coverslip. View the grid through the coverslip to make sure that the Crystalbond completely fills the aperture of the grid and that bubbles or voids are not present. Mount the coverslip

to the dimpling mount with melted Crystalbond, taking care to see that the grid is exactly centered on the cross hairs of the mount.

3. With a clean 10-mm-diameter \times 0.8-mm-wide steel dimpling wheel mounted on the shaft, center a clean rotation stage by adjusting the stage micrometer. This is best done by lowering the dimpling wheel to the stage and sighting down one of the stage crosshairs that is back-lit by the lighting attachment. A small beam of light will appear underneath the dimpling wheel along the crosshair. Adjust the stage micrometer such that the beam of light bisects the thickness of the dimpling wheel. Note the position on the micrometer in order to check whether movement of the stage has occurred during dimpling.
4. Adjust the load on the dimpling arm to 30 gm. It is not possible to measure the exact load on the arm, but the following procedure is used for consistency: with the arm released, rotate the counterweight until the arm balances freely. This is assumed to be a load of zero. Each rotation of the counterweight then supposedly increases (or decreases) the load by 5 gm.
5. After cleaning off the excess Crystalbond from the surface of the sample with an acetone-soaked cotton swab, dimple for 2-3 min at a speed of 8 with several drops of 3- μ m aqueous diamond slurry, using the 10-mm \times 0.8-mm steel wheel. Numerous problems were encountered during the dimpling stage. In many cases dimples had a high spot in the center, and as a result, when a perforation was achieved it was not centered on the interface of interest. Improper centering of the dimpling wheel on the rotation stage may have been responsible in some cases, but a too watery consistency of the grinding slurry may have played a major role in this problem. For this reason it may be important to achieve the right consistency of the slurry by adding dry diamond powder on a regular basis to maintain/recharge the slurry. The slurry should have a thin paste-like consistency. Excess water can be removed from the slurry already on the sample by soaking part of it up with a portion of a paper towel. Since this part of the procedure is rather subjective, an alternative is to use a commercially available diamond paste (Diafin/Dupont diamond compound). This compound is described as being water soluble, but warm soapy water did not easily remove it without wiping with a cotton swab. This contact is extremely undesirable if the sample is very thin. Methanol and, to a lesser extent, isopropyl alcohol easily removed the compound, but their effects on M-bond 610, Crystalbond, and Devcon adhesives are not known. A

second alternative for controlling the point of perforation is to produce a groove rather than a dimple by not rotating the base while grinding and polishing. This has been successful in some instances, but may increase the amount of ion-milling that is necessary.

6. Rinse the sample clean with running water and blow dry with compressed air. Hold the sample at an oblique angle to the running water to minimize impact to the sample if a faucet is used.
7. Monitor the depth of the dimple with the Lietz Ortholux microscope using 200X magnification. The fine focus knob of this microscope is marked with divisions indicating 1- μ m height differences. The depth of the dimple is determined by comparing the focus knob reading obtained by focusing on the flat ground/undimpled surface on both sides of the specimen, and at the bottom of the dimple. If both flat surfaces are at the same height, the depth of the dimple can be measured to $\pm 5 \mu$ m. The dimpling machine also has an automatic system that uses a micrometer for automatically terminating the dimpling operation at a prescribed depth. This system lacks the precision for automatically dimpling to exact depths because it measures the distance between the sample and dimpling wheel. Since the amount of slurry and surface tension on the slurry may vary, this distance measurement is never exact. This system may be useful for automatically removing 60-75% of the sample without routinely monitoring the dimple depth, but the final measurements are best made by the Ortholux microscope and transmission characteristics.
8. Continue dimpling. Periodically remove the sample mount to optically check the depth of the dimple (usually after every 2-3 min). Also check for transmission of light through the specimen using a fiber optic illuminator and the Wild-M400 stereomicroscope.
9. Eventually a red transmission should be visible with the Wild-M400 (samples containing Si only). Continue dimpling, but check the sample every 1/2 to 1 min until a red transmission is visible using the back lighting attachment on the dimpling machine and a stereomicroscope. This light source is less intense than the fiber optic illuminator, and, at the point where a red transmission is visible, the sample is approximately 10 μ m thick. At this point decrease the load on the arm to 20 gm. Continue dimpling at a speed of 6 and time intervals of 15-20 sec until a small perforation appears, or the thinnest portion on the sample begins to crack. Small cracks are easily visible using the fiber

optic illuminator on the Wild-M400 microscope. Note that certain highly doped substrates may be more highly absorbing and that backlighting may not be useful in determining when the dimple has reached the right depth. In these cases the Ortholux microscope will be used to determine that 10-15 μm of the sample remains at the bottom of the dimple.

10. Switch to a separate Texmet-covered 10-mm \times 0.8-mm steel wheel and 1.0- μm alumina slurry. Cut thin (< 1 mm wide) Texmet strips with a paper cutter and glue to the dimpling wheel with super glue. An angle joint, rather than a butt joint is preferred. Care should be taken to ensure that the ends of the Texmet strip do not overlap, since this will cause the dimpling wheel to bounce up and down on the sample as it rotates. Testing a wheel with a newly applied Texmet strip in a blank piece of silicon is recommended to avoid damage to an already critically thin dimpled sample. To apply the alumina slurry, place one drop to cover the sample. With a toothpick, add powdered 1.0- μm alumina. Make sure that the alumina solution does not thicken so much that it will dry up during dimpling. Dimple for 4-8 min until the sample is polished and a perforation intersects the interface of interest.
11. (OPTIONAL) With a separate Texmet-covered wheel, finish polishing with 0.3- μm alumina slurry for about 2-3 min, applying slurry in the same manner as with the 1.0- μm alumina. This step may not be advisable since additional contact of the sample by the wheel may fracture critically thin portions of the samples if a perforation already exists.
12. Remove the coverslip from the base by heating on a hot plate. Do not remove the grid at this time, since the coverslip is much easier to handle and transport than the free standing grid.
13. Remove the grid and sample from the coverslip by soaking in acetone in a watchglass or Petri dish. Dissolve any remaining Crystalbond adhesive on the grid with acetone. This is preferably done at the site of the ion-milling apparatus.

II. GaAs Samples

- A. Sandwiching (same as Si).
- B. Sawing (same as Si).

C. Grinding with the Minimet polisher.

- 1.-2. (Same as Si).
3. For irregular upper sample surfaces, start at a speed of about 3, no load, using 400-grit paper with Metadi fluid lubricant. Check progress at time setting intervals of 1-3.
4. Once the top edges are smoothed out, gradually increase the speed and load until an appropriate removal rate is obtained (approx speed of 5, load of 1/4, and time setting of 5).
5. (Same as Si).
6. When sample thickness is about the same thickness as the outer scrap pieces, wash grit off sample and switch to 600-grit paper.
7. Once again vary the load and speed until a reasonable removal rate is achieved (approx speed of 7, load of 1/2, and time setting of 5).
8. (Same as Si).
9. To polish, use a speed of 4 and a load of 1/4 for a time of 3 with a Texmet pad and 1.0- μ m alumina. Repeat until most large scratches are gone (usually once).
10. (Same as Si).

D. Dimpling on the South Bay Technology dimpling machine (for grooving, follow the same procedure as dimpling except do not rotate the base.)

- 1.-2. (Same as Si).
- 3.-5. After cleaning off the excess Crystalbond with acetone, dimple for 3 min at a speed of 8 with 3- μ m diamond slurry and a Texmet-covered 10-mm \times 0.8-mm steel wheel. In some cases, dimpling with a bare steel wheel produced very deep scratches that did not adequately polish out in the last stage of dimpling (D.10).
- 6.-7. (Same as Si).
- 8.-9. When the thickness of the sample reaches about 40 μ m, decrease the load on the arm by 10 gm and continue the same dimpling process, but check the sample every

15-30 sec until a perforation is formed at the center interface. While it is not possible to use transmission characteristics of the GaAs as an indication of when the sample is reaching a critical thickness (~ 10-15 μm), backlighting can still be used to judge if the sample is approaching this thickness. As the sample becomes thinner, it will be possible to observe light transmitted along the adhesive bandline at the center interface. When light becomes visible along the bondline with the backlighting attachment of the dimpling machine, it is advisable to decrease the time interval between inspection of the sample.

10. Then dimple at a speed of 4 using a Texmet-covered 10-mm \times 0.8-mm steel wheel and 1.0- μm alumina until most large scratches are removed. Apply the alumina in the same manner as with Si.

11.-13. (Same as Si).

LABORATORY OPERATIONS

The Aerospace Corporation functions as an "architect-engineer" for national security projects, specializing in advanced military space systems. Providing research support, the corporation's Laboratory Operations conducts experimental and theoretical investigations that focus on the application of scientific and technical advances to such systems. Vital to the success of these investigations is the technical staff's wide-ranging expertise and its ability to stay current with new developments. This expertise is enhanced by a research program aimed at dealing with the many problems associated with rapidly evolving space systems. Contributing their capabilities to the research effort are these individual laboratories:

Aerophysics Laboratory: Launch vehicle and reentry fluid mechanics, heat transfer and flight dynamics; chemical and electric propulsion, propellant chemistry, chemical dynamics, environmental chemistry, trace detection; spacecraft structural mechanics, contamination, thermal and structural control; high temperature thermomechanics, gas kinetics and radiation; cw and pulsed chemical and excimer laser development, including chemical kinetics, spectroscopy, optical resonators, beam control, atmospheric propagation, laser effects and countermeasures.

Chemistry and Physics Laboratory: Atmospheric chemical reactions, atmospheric optics, light scattering, state-specific chemical reactions and radiative signatures of missile plumes, sensor out-of-field-of-view rejection, applied laser spectroscopy, laser chemistry, laser optoelectronics, solar cell physics, battery electrochemistry, space vacuum and radiation effects on materials, lubrication and surface phenomena, thermionic emission, photosensitive materials and detectors, atomic frequency standards, and environmental chemistry.

Electronics Research Laboratory: Microelectronics, solid-state device physics, compound semiconductors, radiation hardening; electro-optics, quantum electronics, solid-state lasers, optical propagation and communications; microwave semiconductor devices, microwave/millimeter wave measurements, diagnostics and radiometry, microwave/millimeter wave thermionic devices; atomic time and frequency standards; antennas, rf systems, electromagnetic propagation phenomena, space communication systems.

Materials Sciences Laboratory: Development of new materials: metals, alloys, ceramics, polymers and their composites, and new forms of carbon; nondestructive evaluation, component failure analysis and reliability; fracture mechanics and stress corrosion; analysis and evaluation of materials at cryogenic and elevated temperatures as well as in space and enemy-induced environments.

Space Sciences Laboratory: Magnetospheric, auroral and cosmic ray physics, wave-particle interactions, magnetospheric plasma waves; atmospheric and ionospheric physics, density and composition of the upper atmosphere, remote sensing using atmospheric radiation; solar physics, infrared astronomy, infrared signature analysis; effects of solar activity, magnetic storms and nuclear explosions on the earth's atmosphere, ionosphere and magnetosphere; effects of electromagnetic and particulate radiations on space systems; space instrumentation.