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Preparation of Semiconductor Cross Sections by Cleaving

C.J.D. Hetherington

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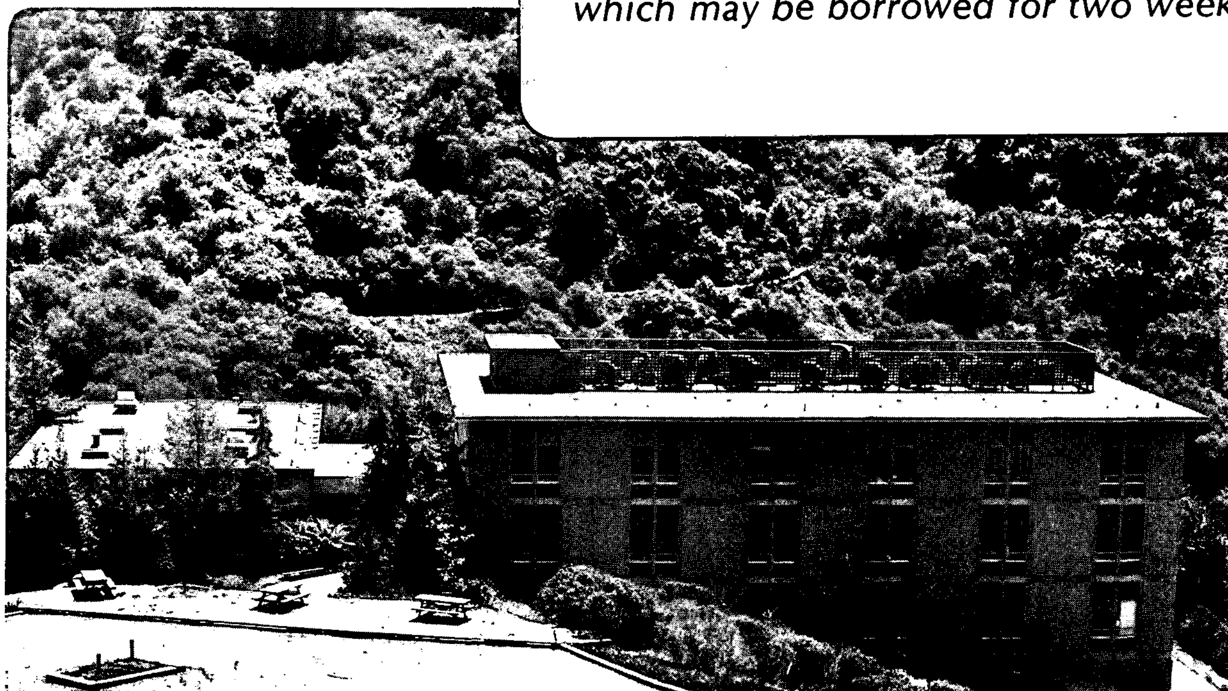

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PREPARATION OF SEMICONDUCTOR CROSS SECTIONS BY CLEAVING

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ABSTRACT

The practical details are presented for preparing cross-sectional samples of semiconductor epitaxial layers by cleaving. Examples of results are shown and a number of possible applications for the samples are discussed. A variation of the method can be used to prepare a TEM sample of a high T_c superconductor.

INTRODUCTION

Wedge-shaped samples formed by cleaving larger crystals were first used in TEM studies of MgO [eg 1] and Si [2]. Large wedge angles are also found in TEM samples prepared by other methods such as MgO smoke crystals [3,4] or chemically thinned lithium ferrite [5] and the thickness variations have been made use of in the studies of those materials. More recently, the cleaving technique was applied by Kakibayashi and Nagata to GaAs/AlGaAs multilayers [6,7]. GaAs and AlGaAs cleave predominantly on {011} planes giving the resulting wedge a precise 90° angle. This means that the sample thickness is directly related to the distance from the wedge edge when the sample is aligned with top and bottom surfaces at 45° to the horizontal ([010] axis vertical). This allows an analysis of the [010] on-axis bright field thickness fringes to give a measure of the AlGaAs composition [7,8]

In this paper, a method for preparing cleaved TEM samples of epitaxial layers on GaAs is described in detail. While the method is best suited to GaAs (100) substrates, it may also be applied to layers grown on Si and possibly other substrates. The cleaved samples have thin area running more or less along the normal to the epitaxial layers (growth direction) and are therefore well-suited to the study of these layers. Examples of some samples are shown, including a high T_c superconductor using a variation of the method. The large wedge angle and very narrow thin area running along the edge limit the samples' usefulness for typical TEM investigations. However, it will be shown in this paper that, while cleaved samples cannot replace standard ion-milled cross sections, they can make a number of contributions to the study of semiconductor epitaxial layers.

Perhaps the major contribution of cleaving as a sample preparation method will stem from its speed; one sample can be prepared in around 30-45 minutes. It is also relatively straightforward, needs no specialized equipment, and it uses only a small portion of the wafer, typically 2mm x 2mm or less. It therefore offers the possibility of an initial inspection of many wafers in a short time.

SAMPLE PREPARATION

To prepare a cleaved cross-section of a portion of a wafer, several stages are involved; initial thinning of the sample, cleaving it into pieces and mounting them onto TEM slot grids. The procedure is shown schematically in Figure 1.

The first stage thins the sample by grinding away most of the substrate. It has been found that the thinner the sample, the better the subsequent cleaving (this may be related to the force required to cleave the sample which increases with thickness). However, the sample should not be made so thin that it can no longer be handled. Typically, GaAs is thinned to 50µm and Si to 30µm. The sample can be thinned by hand, first mounting it onto a glass disc with wax (e.g., Crystalbond). Then it is ground down finishing on 600 grit sandpaper, the thickness being followed by micrometer. A helpful means of stopping at a certain thickness is to put sticky tape of that thickness onto the glass disc on either side of the sample. (Alternatively, one of the commercially available grinding or lapping devices may be used to thin the sample to the desired thickness.) It is not necessary to polish this surface. The thinned sample is removed by melting the wax and sliding the sample off the glass disc and washing in acetone and ethanol.

The next stages, cleaving the sample and mounting the pieces onto slot grids require the use of a stereo microscope (around 20X magnification) and a pair of extra fine tweezers. To cleave the sample, first place it layer side down onto a soft surface such as several filter papers or, better, one of the silicone rubber mats sold as grid "grippers" or "holders" by most EM accessory suppliers. Then apply point pressure with a toothpick or tweezers against the substrate side of the sample. This ensures that the layers underneath are in tension as they cleave and the cleaved faces are flat and perpendicular to the layers. The substrate side undergoes compression and the cleaved face through this region may be curved or stepped. A small nick in the surface on the larger side may be required to help start the cleave, although that nicked area will then be unsuitable for TEM examination. The sample should be cleaved into several pieces that, when mounted on a slot grid, will fit into the TEM sample holder.

At this point the pieces should be inspected under an optical microscope at around 100X magnification and the best of the four corners (or wedge edges) selected. The adhesive properties of the silicone rubber allow the pieces to be stood upright on a cleaved face so that the opposite cleaved face can be observed. A (100) GaAs piece should have a wedge edge that is straight right up to the top surface with flat {011} cleaved faces either side. Silicon and other materials that cleave on more than one family of planes may have curved cleaved faces; in which case, a piece should be chosen having the straightest wedge edge and the smallest wedge angle.

Finally, the piece must be mounted upright on a 3mm grid with a 2mm x 1mm slot. For the typical GaAs 90° wedge to be observed in the TEM down [010], the (011) and (011) cleaved faces must lie at 45° to the horizontal. If the microscope sample holder has 45° tilt, then the piece can sit on a cleaved face with the wedge edge overhanging the slot. If the holder has less tilt, the piece must sit on an edge (or corner) so that the selected wedge edge is already approximately aligned and situated over the slot; a height or Z control may then be required to focus the image.

A variety of methods have been tried to mount these pieces in either of the configurations, including using bent grids or small supports. However, the most satisfactory answer is simply to use the right glue -- namely an electrically conductive silver epoxy which supports the piece even before it is set and which avoids problems such as drift or charging (for example, the H2OE supplied by Epoxy Technology, Inc., 14 Fortune Drive, Billerica, MA 01821). The epoxy should

be mixed and a small amount placed on the grid. Into this can be positioned the cleaved piece in the desired orientation. Two or more pieces can be mounted on the same slot grid to increase the chances of obtaining a good sample. Figure 2 shows examples of some samples ready for TEM examination.

The handling of such small pieces during the cleaving and mounting may be found difficult, resulting in a number of failed attempts. However, very little time is wasted on these attempts and there are usually enough pieces from the original portion of wafer for at least one satisfactory TEM sample to be made.

It is worth pointing out that the initial tilting of the sample in the microscope to the [010] pole can be done in real space at a low magnification watching the silhouette of the sample. The final tilt is aided by the Kikuchi lines observed in the adjacent thick region and the absence of bending in the sample (except for samples with strained layers).

EXAMPLES

Figure 3 shows some examples of layers prepared by cleaving and imaged at [010] in bright field. Some information is readily obtained on layer and interface widths, while the thickness fringes can in some cases reveal nonuniformity of alloy composition through a layer or, with the aid of calculations [7,8], a quantitative measure of the alloy composition. In addition to these examples, cross-section samples of GaAs/GaAsP, GaAs/(In,Ga)P/InP, Si/(Si,Ge) and Si/CaF_2 layers have also been prepared by cleaving and examined in TEM.

APPLICATIONS

Two applications of this sample preparation have already been mentioned, a rapid TEM examination of semiconductor layers and a compositional analysis of alloys such as AlGaAs. A further possible application makes use of the horizontal wedge edge and absence of bending in the sample to minimize exposure of a region to the electron beam. Having set up the imaging conditions (sample orientation, beam alignment) on one region, the adjacent region can be moved into the beam and a micrograph taken immediately. As an example, Figure 4 shows a lattice image of cleaved GaAs taken after only 10 seconds in the focussed electron beam.

Another application for the uniform wedge shape is in the study of high resolution and other images as a function of thickness [e.g. 2,4]. Figure 5 shows a [110] 7 beam lattice image of a cleaved Si wedge. The high signal to noise ratio of such images has been noted by Gibson and McDonald [9]. Note that the inclined top and bottom surfaces of the sample can influence the electron scattering [3,10] and the defocus as a function of position [4].

A further application could make use of the fact that the location of the wedge edge (i.e. the intersection of the two cleaves) is accurately known; this could be important in the study of nonuniform or selective growth across a wafer. Also, the top surface adjacent to the wedge edge is still intact and could be examined subsequently by surface techniques such as X-ray diffraction.

HIGH T_c SUPERCONDUCTORS

High T_C superconductors are typically prepared by grinding and suspending on a holey carbon film or by ion milling. The first method produces very small particles and destroys grain boundaries while ion beam milling produces a thick amorphous layer and damage in the sample. Some (polycrystalline) YBaCuO was therefore prepared by i) cleaving a prethinned wafer and mounting pieces with small wedge angles onto a slot grid (see fig. 2) or ii) to avoid heating the superconductor in air, simply shattering it and clamping a fragment with sharp edges in a folding grid made from a refractory metal [11]. Figure 6 shows samples prepared by ion milling and by cleaving imaged in the ARM.

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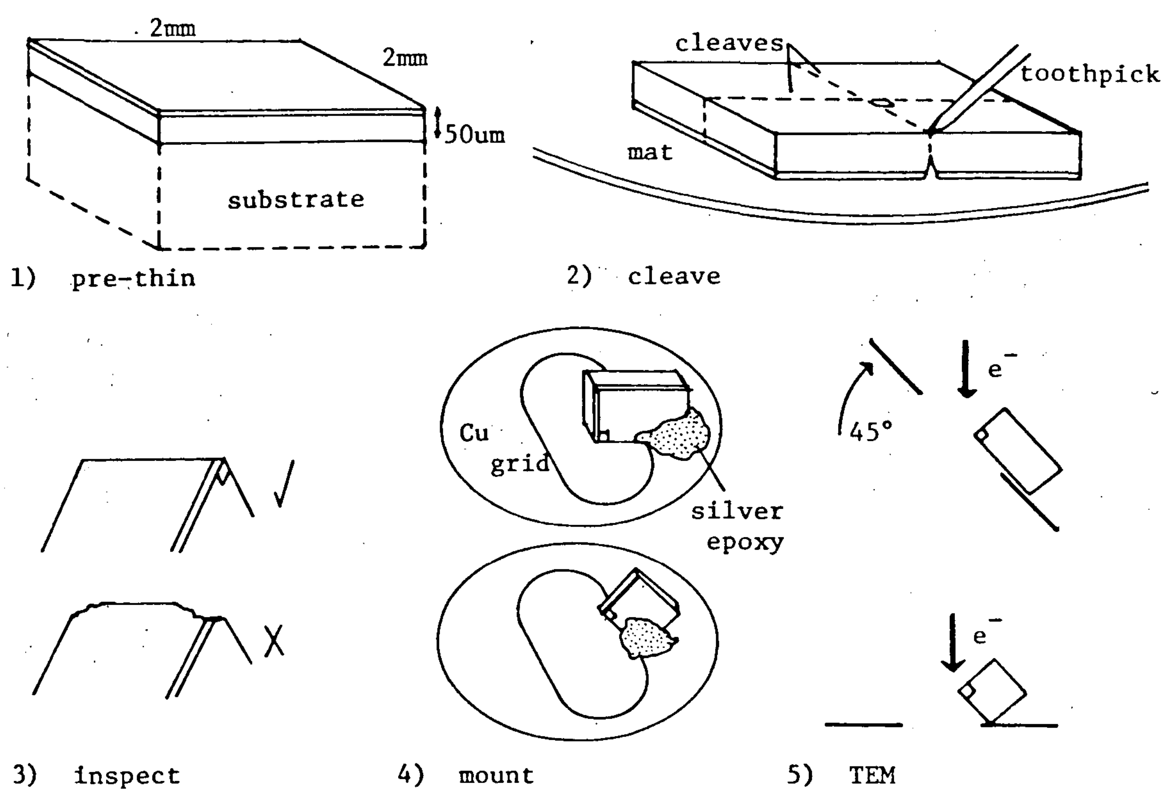

Fig. 1

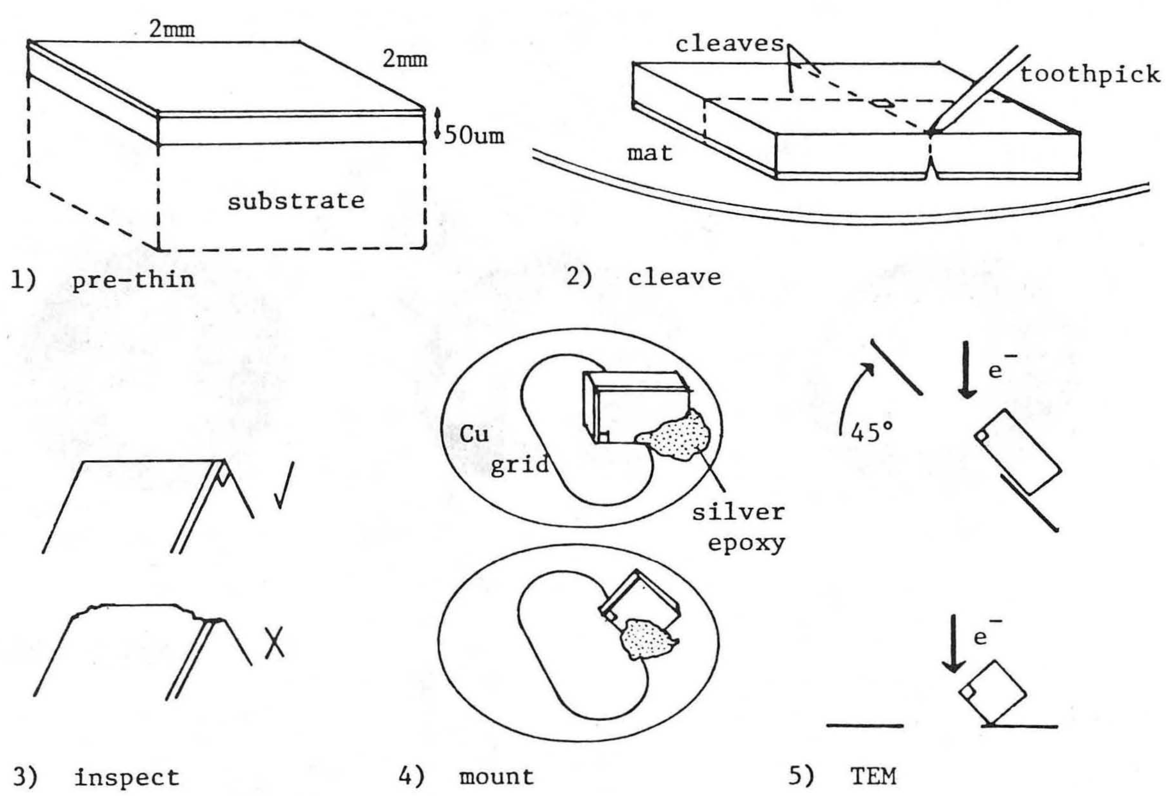

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Fig. 1

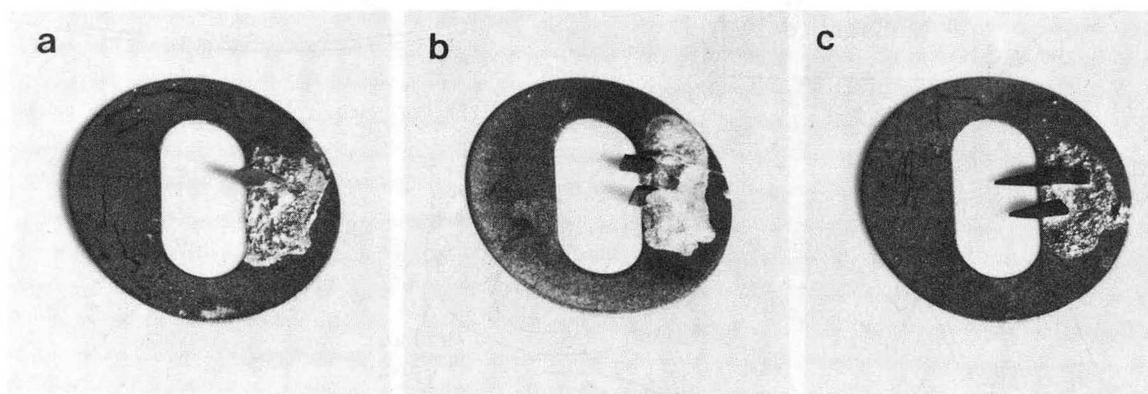

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Fig. 2

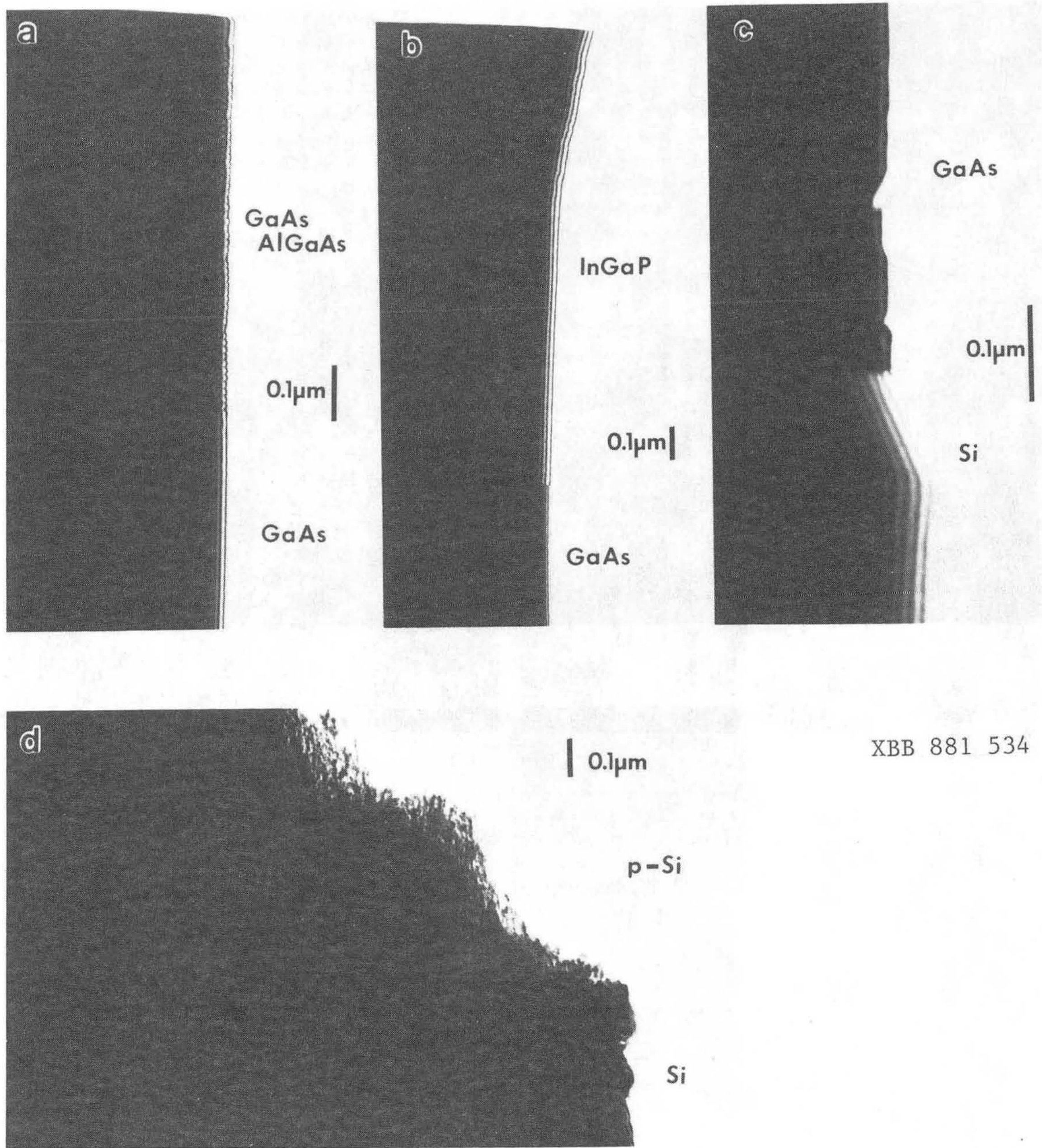

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Fig. 3

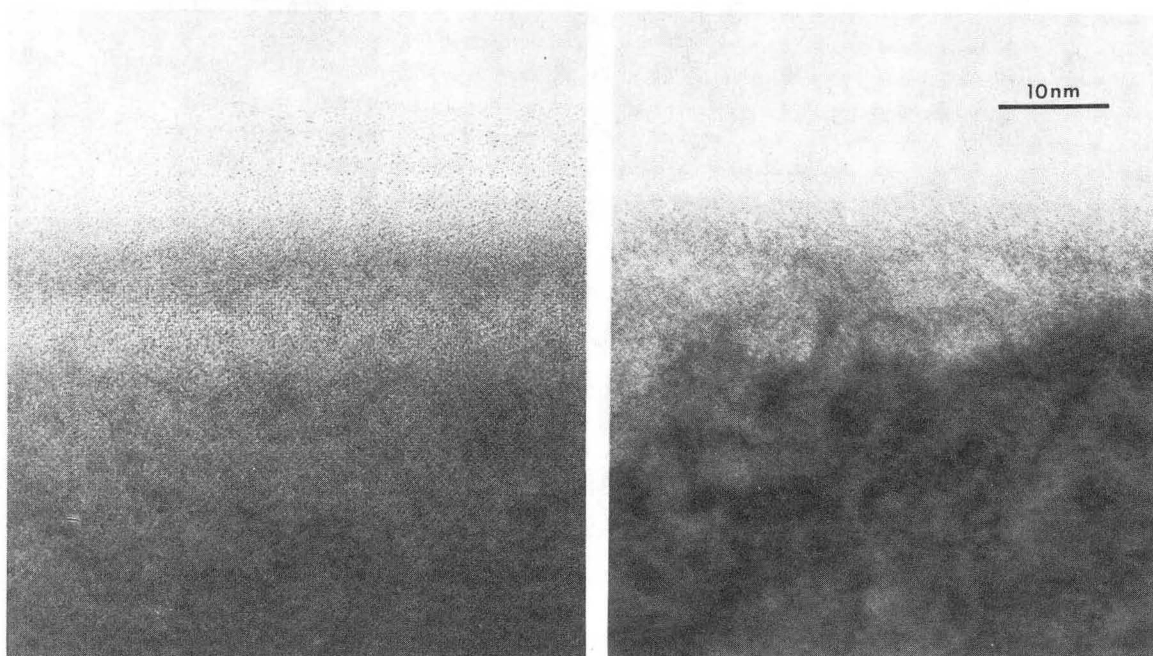

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Fig. 4

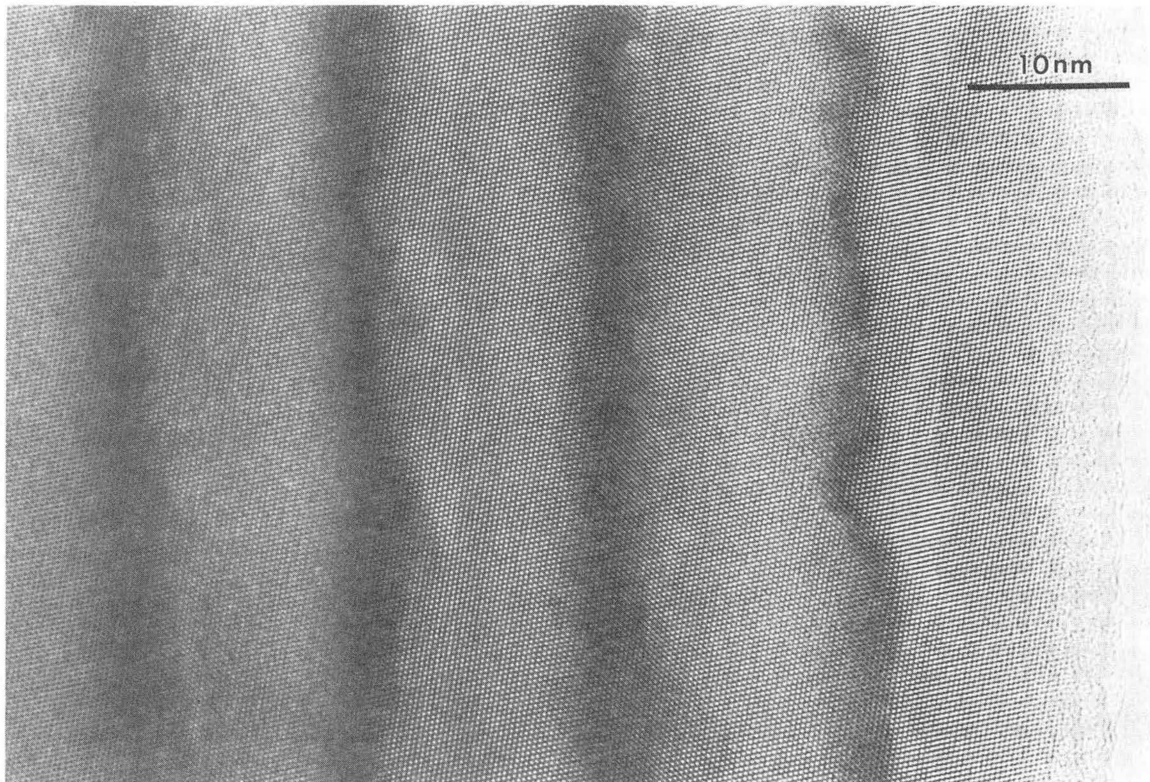

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Fig. 5

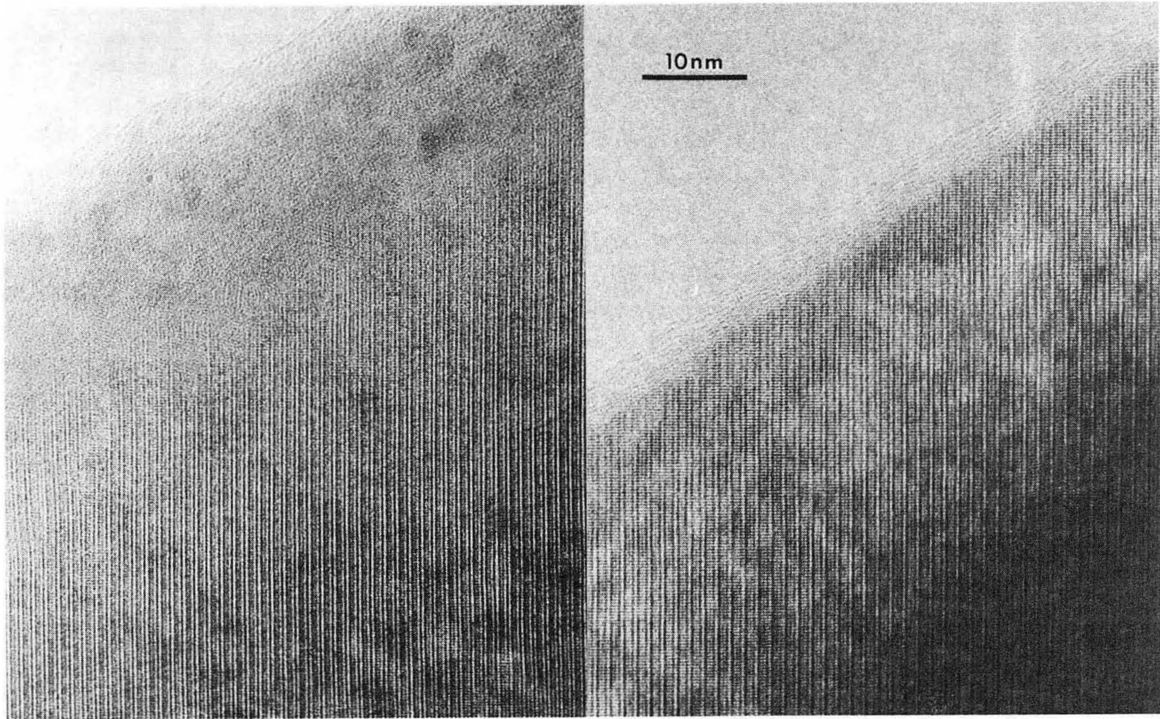

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Fig. 6

Figure 1. Schematic digram of the sample preparation

Figure 2. Cleaved samples mounted on 3mm slot grids: a) GaAs/AlGaAs mounted on an edge; b) GaAs/InGaP mouted on cleaved faces; c) YBaCuO wedges

Figure 3. a) GaAs/AlGaAs multilayer; b) InGaP layer on GaAs;
c) GaAs layer on Si; d) porous Si layer on Si substrate.
All layers on (100) substrates and images taken at 100kV in bright field at [010].

Figure 4. [010] lattice image of GaAs taken at 1000kV in the Atomic Resolution Microscope (ARM) after 10 seconds (left) and 3 minutes (right) in the beam.

Figure 5. 7-beam [011] lattice image of Si taken at 1000kV in the ARM

Figure 6. YBaCuO samples prepared by ion milling (left) and by cleaving (right).

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