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Applied Analytical Electron Microscopy of Semiconductors

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Applied analytical electron microscopy of semiconductors

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The role of transmission electron microscopy (TEM) in the effort to relate physical structure and elemental composition to electrical and opto-electronic performance of silicon and III-V materials and devices is illustrated. TEM is shown to have the spatial resolution to image the finest details of the internal structure of materials and from the many variations of specimen preparation and imaging conditions, combined with elemental analysis in the microscope, valuable three-dimensional characterization is obtained.

1. Introduction

The advanced technologies used for modern semiconductor devices were developed by largely empirical methods; experiment followed by observation and measurement. The primary measurement was of electrical performance, but this has always needed to be followed by physical and chemical characterization to give a two- and three-dimensional picture of topology and elemental composition. From this information, enhanced design and performance can be engineered. Linked to the engineering of devices is the need for investigative examinations to determine the causes of poor yield or the premature failure of devices. The transmission electron microscope (TEM) plays a key role in these examinations but it is only one element in a range of techniques where a multidisciplinary approach will show the maximum benefit. The understanding of the device structure that results from a thorough characterization and the linking of structural and electrical properties enables computer models of the processing to be formulated. These models enable the process engineer to understand the effects of processing changes and to design new processes for new structures with different electrical properties. An example of this application, where TEM characterization provides the input to models of the oxidation of silicon, is given by Jones *et al.* (1994). The processing of devices occurs in extremely harsh chemical, thermal and physical environments and the ultimate performance of electronic devices is only achieved by pushing the physical properties of the materials to their limits. Processing of devices with a high yield of working elements can be best achieved when the relationship between processing conditions and defects in the devices are well understood.

Transmission electron microscopy has been applied to semiconductor materials and devices since the 1960s, when the role of diffraction contrast in showing the structure of thin foils of crystalline materials was first being demonstrated (Hirsch *et al.* 1965). As device geometries have shrunk over the years, the devices have

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become more accessible to TEM examination and TEM has become essential for the detailed examination of device structures. Only TEM has the capability to give three-dimensional information of the required spatial resolution in modern fine dimensional structures.

The most common image which people associate with a silicon integrated circuit is a scanning electron microscope (SEM) image of the top surface of the chip. This shows metal interconnections and insulating oxide layers. These images are useful to show if bond wires are disconnected, or if short circuits between tracks have occurred through electromigration, but they do not reveal any information about the region in the silicon itself where the transistor action is taking place. To view this region, where the precise geometry is of maximum importance and where crystallographic defects or impurity precipitates can have a crucial effect on device performance, TEM must be used.

Throughout this discussion of impurities and defects, it must be remembered that we are dealing with some of the purest and most structurally perfect materials known to man. Tremendous efforts have gone into producing materials with metallic impurity levels below one part per billion and in producing silicon wafers which contain no crystallographic defects threading up to the top surface of the wafer where the active devices are constructed. This level of perfection is essential because a single dislocation threading through the collector, base and emitter regions of a bipolar transistor is sufficient to create a short-circuit which will destroy the transistor action. Similarly, dislocations occurring in solid state lasers and light emitting diodes act as non-radiative recombination sites which diminish the light output. Growth of these defects, either by dislocation glide under stress or by the absorption of point defects, continues the process by which the light output is diminished.

The limitations in the perfection of wafers that are in current use are determined by the crystal growth processes. In silicon, the Czochralski method is used for more than 90% of production purposes. Here, the silicon melt is contained in a silicon dioxide crucible with the result that the melt and the grown crystal become saturated with oxygen. The role of the oxygen in the processed wafers plays an important part in the structural and defect properties of the wafers. In GaAs and InP wafers, the control of stoichiometry and point defects is important and dislocation networks in the wafers are a natural result of the high stresses generated in the cooling of crystals as they are pulled from the melt (Augustus 1990).

The key to the manufacture of high performance semiconductor devices is not the complete elimination of defects and impurities from the starting wafers, as this is an impossible task and in any case other defects will be introduced during subsequent device processing. Rather, it is to create regions free from defects where the active elements of the devices are fabricated and to ensure that the remaining defects are confined to areas where they will not have a deleterious effect. Further to this, crystallographic defects can play an important role in enabling certain device structures to be achieved, such as in the role of misfit dislocations to accommodate variations in lattice parameter. Oxide precipitates in the bulk of a silicon wafer help to purify sensitive device areas by gettering metallic impurities away from the near-surface region which is denuded of these defects.

The modern transmission electron microscope is more than just an imaging machine. Electron diffraction allows crystallographic phases to be identified from their diffraction patterns and elemental analysis of micro-areas can be performed by energy dispersive X-ray analysis or electron energy loss spectroscopy.

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The scanning transmission electron microscope (STEM) usually includes a secondary electron detector in addition to the transmitted electron detector. Using this, SEM images of the top surface of the specimen can be related to STEM or TEM images from the bulk of the material. The fine electron probe of the STEM is utilized in focusing the electron beam on the selected area required for point analysis and can also be rastored for elemental mapping.

2. Specimen preparation for TEM

If we wish to see inside a material we must first strip off outer layers that might restrict our view. The TEM imaging technique relies on the transmission of electrons with negligible energy loss as once a significant proportion of the electron beam is slowed by atomic collision the image will become diffuse. We are restricted, therefore, to samples of up to 1 μm in thickness, with some proportionate increase in thickness if the energy of the electron beam is increased from the typical 100 or 200 keV to 300 or 400 keV. Microscopes of 1 MeV have been produced but with the reduction in scale of microelectronic devices the technical advantage is reduced and the financial costs cannot be justified. As a general rule, if the lack of microscope power forces the production of uniformly thinner samples then better images will result. There is, however, one 3.5 MeV TEM presently being used for *in situ* studies of electromigation in silicon integrated circuits.

Limitations in viewable specimen thickness become less of a problem as device scales are reduced, modern silicon devices made in the top few thousand atomic layers of the semiconductor wafer are readily accessible by TEM. To examine the active silicon areas of an integrated circuit it is merely necessary to strip off any surface oxide and metal layers and chemically thin the material from the back face of the wafer. This creates what is known as a plan view TEM specimen. Areas as large as many hundreds of square microns can be examined in this way and, using stereo viewing techniques, complex defect geometries can be assessed. On the other hand, the visualization of device structures consisting of multi-element materials is realized by making a cross-section TEM specimen (XTEM). Here, a slice is taken through the structure and, by using ion beam milling techniques, which will thin all materials, the layers of semiconductor, oxide, nitride, silicide and metals can be examined. The preparation of cross-sections involves delicate lapping and polishing followed by ion beam milling and can involve up to several days' work, depending upon the level of expertise employed. This is rather too time consuming when all that is sometimes needed is a thickness measurement of the active layers in a semiconductor laser structure. This need has led to the development of the cleaved edge technique, particularly suited to III-V materials which cleave on (110) planes and have a wafer orientation close to (001). The technique consists of making two orthogonal cleaves in a small chip of material and mounting this on its side such that the electron beam is transmitted through the thinnest portion of the right-angled wedge. Sufficient material is imaged for the measurement of layer thicknesses, but it must be remembered that this is only a spot measurement, information on interface flatness demands a full cross-section. The cleaved edge technique has enabled precise control of growth rate across wafers from epitaxial growth systems to be achieved. The ease of specimen preparation and fast turn around in analytical results enables samples to be taken from several positions on a wafer and measured within a few hours. The technique is also used to monitor etch back steps in device processing and can also be applied to completed laser chips.

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3. Analytical TEM

The TEM analytical methods described here are those regularly used to the benefit of semiconductor device technology. TEM has a high spatial resolution in both imaging and chemical analysis, the major challenge is to make specimens that can take full advantage of the capabilities of the analytical techniques, while taking into consideration the limitations imposed by the device geometry. Modern introductions to analytical TEM in materials science have been given by Loretto (1984) and by Goodhew & Humphreys (1988).

In studying interfaces, either by conventional diffraction contrast or by lattice imaging, the interfaces must be planar and parallel with the direction of the electron beam in the microscope or a diffuse appearance will result. This is because the sample has a finite thickness, probably at least 20 atomic layers, in the thinnest of specimens. A similar consideration applies when stepping a probe across an interface to measure either the concentration of segregants to that interface or the sharpness of the interface itself. Fortunately, the suitability of the sample can be judged by viewing the image in the microscope, the TEM image will show whether interfaces are suitable for quantitative micro-analysis. This is not an option available to other analysis techniques, such as secondary ion mass spectroscopy (SIMS) or Auger electron spectroscopy (AES), where assumptions have to be made and the interpretation of the data becomes dependant on the validity of these assumptions. The biggest problem with these profiling techniques is that it is impossible to distinguish between rough and diffuse interfaces. TEM can be used to make these distinctions but it is not always clear from viewing the sample along a single crystallographic direction.

There are two spectroscopy techniques regularly attached to analytical TEMs; energy dispersive X-ray spectroscopy (EDX) and electron energy loss spectroscopy (EELS).

(*a*) *Energy dispersive X-ray analysis (EDX)*

This is the most widely used analytical attachment to a TEM. The technique is identical to that of attaching X-ray analysers to scanning electron microscopes or electron probe micro-analysers. When these analysers are attached to a TEM and used on thin foil specimens, there are particular advantages over their use on bulk specimens. On bulk specimens, beam spreading in the sample, up to 1 μm in diameter and a similar depth, severely limits the spatial resolution of the analysis. In addition to this, most of the energy from the incident beam is absorbed in the specimen through inelastic collisions which create a high background (Bremsstrahlung) radiation. Background subtraction techniques need to be used if weak peaks are to be measured. On thin foils, such as those used in the TEM, the beam does not spread significantly before it emerges from the far side of the specimen, the background Bremsstrahlung radiation is considerably reduced and there is less X-ray fluorescence to account for.

X-rays are continuously being generated by the interaction of the electron beam and in EDX these X-rays are analysed by a solid state detector. The X-rays are generated in the full depth of a TEM sample and they are characteristic of the atoms with which the electron beam interacts. Energy dispersive detectors are available which collect the complete range of X-rays simultaneously, but detectors have traditionally been protected by using a thin Be window. This has restricted their use to elements of atomic number $Z = 11$ and above. This restrictive foil cuts out X-rays from the important lighter elements and thin film windows or windowless detectors

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are now available. However, great care must be taken not to damage these detectors when they are used on microscopes with high energy electron beams.

A typical example of EDX analysis in a conventional TEM is given by Reader (1991). Here, a sample from a typical silicon integrated circuit is examined. A TEM cross-section through a contact structure has a silicon–$TiSi_2$–polysilicon sandwich structure. A 50 nm diameter electron probe positioned on the 100 nm thick silicide layer gives an EDX signal with the correct ratio of Ti to Si, that is, with little excitation of the adjoining silicon layers. On a finer scale, but still operating with a 10 nm 120 kV probe, STEM analysis of the As dopant at the edge of an implantation window was capable of showing the variation in dopant concentration resulting from various annealing schedules. The dopant is shown in both depth and lateral measurements in the range 5×10^{19}–1×10^{21} with measurements made at 20 nm intervals. The As depth profiles obtained were in good agreement with SIMS measurements and the example demonstrated precision lateral measurement of dopant concentration.

There are competing interests in the design of a multitasking analytical electron microscope, especially when an EDX detector is added. There must be a compromise in the design of the objective lens pole piece between; the demand for the highest possible spatial resolution in TEM mode, availability of high specimen tilts for optimum phase analysis by electron diffraction and a clear take-off of X-rays to the EDX detector with a minimum chance of X-ray fluorescence from component parts of the microscope. For high collection efficiency, the X-ray detector must also be placed as close as possible to the sample and the sample must be tilted to face the detector. In this tilting it is important that interfaces to be analysed remain parallel with the direction of the electron beam. A scanning transmission electron microscope (STEM) dedicated to EDX analysis and designed specifically for this purpose is probably the best option for analysis. For high X-ray count rates from small areas the microscope should have a field emission electron gun (FEG-STEM). A standard analytical TEM with a tungsten filament has a probe size for analysis by EDX and EELS of 5–10 nm. Efforts to reduce this probe size will result in a considerable loss in electron intensity and hence X-ray intensity. With a field emission gun however, a high initial brightness from the filament allows a probe of 0.5–1 nm to be formed, still producing a workable count rate of X-rays. The main reservation in using intense probes is that care must be taken that degradation of the specimen does not occur under the impact of the powerful electron beam. The spatial resolution in EDX is not only governed by the incident electron probe diameter, some beam broadening does occur and to utilize the benefit of a small probe size the thinnest possible specimens must be used. For specimen thicknesses of less than 20 nm, a resolution of 1–2 nm becomes possible. An additional advantage of using a thinner specimen is that as the specimen thickness is reduced, absorption and fluorescence of the excited X-rays within the specimen may be neglected when correction factors are applied for quantification of the results. For a high sensitivity, a high count rate is necessary to give a high peak to background ratio. Counting for a long time gives problems with specimen drift within the microscope but automatic beam position correction can now be used where the image can be 'read' by an image analysis system. Compromises must be made; focusing to a finer spot gives a reduction in beam current and a thinner sample again means fewer X-ray counts. Also, with a very thin specimen, the near surface areas are a larger proportion of the analysed volume. Conditions may be atypical due to specimen preparation and contamination artefacts which can be significant. It is particularly important to minimize the build up of carbon

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in the area where the electron beam impacts with the specimen and breaks down hydrocarbons to give a layer of carbon on both entry and exit faces. Under optimum conditions, the minimum mass fraction detectable by EDX is about 0.1%, way above semiconductor doping levels in conventional implantations, but a level that is now occurring in shallow implants and detected where dopant segregates to interfaces and grain boundaries.

As an example of grain boundary analysis, we will consider arsenic doped polysilicon, which has many applications in integrated circuit technology and in solar cells. The proportion of arsenic in the grain boundaries, compared with the bulk of the individual grains, coupled with the size and distribution of the individual grains, will determine the electrical properties of the film. Wong *et al.* (1985) used a field emission STEM to show the segregation of arsenic to grain boundaries that occurs as the polysilicon is annealed to activate the dopant and they demonstrated the effect of arsenic segregation on the electrical properties of the thin films.

(*b*) *Electron energy loss spectroscopy (EELS)*

In this analysis technique, the energies of electrons that have passed through the specimen are analysed and elements are identified from the loss in electron energy as the beam interacts with the sample. As the electron beam passes through the sample, the majority of electrons exhibit no energy loss, any scattering of the beam is elastic. This is beneficial in TEM imaging as energy losses give rise to chromatic aberration and loss of resolution. Chromatic aberration is the principal reason for the degradation in image quality as the specimen thickness increases. Limited energy losses do occur even in the thinnest of specimens. Low losses of energy (5–50 eV) occur from an interaction of the primary beam with plasmons. Higher energy losses are produced by inner shell ionizations and appear as tiny features on a steeply decreasing background. These losses are characteristic of the material through which the electron beam has passed, but for analysis we need to analyse electrons that have suffered a single loss. The background of inelastically scattered electrons from multiple scattering events increases rapidly with specimen thickness and this means that TEM specimens which are most suitable for EELS analysis are typically less than 30 nm in thickness. To analyse thicker samples, some advantage can be gained by going to a medium voltage electron microscope of 300 or 400 kV rather than the conventional 100 or 200 kV instruments.

As previously stated, in EELS analysis the thinnest parts of the specimen are used and the electron spectrometer only accepts electrons with a small beam divergence, there are therefore no significant broadening effects as the beam passes through the sample and the spatial resolution of EELS is therefore high. Using a FEG-STEM, a resolution of 1 nm is possible and as no beam tilting is required the technique is compatible with the highest resolution lattice imaging microscopes.

Line-scans and elemental mapping can be efficiently performed in EELS. EELS imaging filters are now commercially available making electron spectroscopic imaging possible. This technique has been mainly applied to biological applications and is only just starting to find uses in materials science. Its application to semiconductor materials has been complicated by sources of strong contrast other than elemental composition, these often occur simultaneously with elemental variation and are thickness fringes and bend contours which result from the strains occurring where the composition changes. The variation in intensity from one grain to another in polycrystalline materials is controlled by the diffracted intensity which depends on

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the grain orientation with respect to the electron beam and outweighs the contrast difference that can be generated by energy filtering.

(*c*) *Dark field contrast*

The electrical properties of silicon and the III-V elements are determined by dopants evenly distributed in the lattice at a level far below our ability to detect in the TEM, unless their presence is highlighted by chemical etching (see the next section). On the other hand, opto-electronic materials based on III-V ternary and quaternary compounds have opto-electronic properties closely controlled by the chemical composition and layer thickness. This composition and thickness can best be monitored on a microscopic scale in the TEM. The most important development in TEM imaging technique for III-V materials was reported by Petroff in 1977. This involves the use of a two-beam diffraction contrast imaging condition. A (200) diffraction vector which gives zero intensity for silicon (diamond structure), has a very low intensity for GaAs, but has an intensity which increases with structure factor difference for the different elements with the zincblende structure.

In the GaAs–AlGaAs system, differences in aluminium content of less than 0.1% are clearly visible. The technique shows up the individual layers in laser and modulator structures and when used with the cleaved edge technique gives a quick and easy method for layer thickness measurement, with the bonus of a check on interface sharpness between layers and compositional stability within the layers. Using this technique on a cleaved edge sample, de Jong & Janssen (1990) attempted compositional analysis from the image intensities. They then compared the results with measurements made by SIMS. By comparing calculated and experimental extinction fringes, compositions were determined for $Al_xGa_{1-x}As$ layers with a sensitivity in x of 0.03; this was in layers with a thickness of only 3.5 nm. Analysis could be performed on layers as thin as 1.5 nm. Thoma & Cerva (1991) performed a similar analysis using high resolution electron microscopy and developed an image processing algorithm where the chemical composition of GaAs–AlGaAs interfaces could be determined with a spatial resolution of 0.28 nm parallel and perpendicular to the interface. However, the atomic interface roughness is averaged over about 20 atomic layers in the direction of projection.

Transmission electron microscopy using cleaved edge and cross-section specimens has a particular advantage compared with profiling techniques such as SIMS or Auger in that the analysis has the same accuracy and sensitivity at whatever depth the measurement is being made.

(*d*) *Etching combined with TEM examination*

As already stated above, there are features of extreme importance in semiconductor materials that are not always visible in conventional TEM images. Most semiconductor material is completely plain and featureless in the TEM image yet it may well contain interstitial elements, impurity clusters or precipitates. To be visible, the defects must strain the lattice in order to produce distortions to lattice planes which are then visible by diffraction contrast. It is rare for discoveries about materials to be made without 'signposts' from other techniques. The magnifications often necessary to see features such as small precipitates, impurity clusters and dislocation loops are in excess of five thousand times and where defect densities are low, e.g. 10^5 or 10^4 cm^{-2}, it is necessary to search for a long time. In addition to this, the search may need to be carried out under precise diffraction conditions requiring frequent checks

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against the diffraction pattern. It is therefore useful to know that there is something there worth looking for. This can often be determined by a selective etch; the etch will not show what the feature is but it acts as a marker to where and when features occur.

Another benefit of relating etch features to defects by combined etching and TEM studies is that the materials processing engineer does not have put all materials forward for TEM examination at all stages of processing, but will use etching and observation by optical microscopy to do a rough pass. He will select those features of most interest and will then characterize the etch for further use. A monograph by Graff (1995) shows how defect etching is used to check silicon substrate material for oxide precipitates and metallic impurities. The precise control of these is an important feature in the production of wafers for silicon integrated circuits.

Etching is a destructive process; where we wish to preserve crystallographic defects for examination by other techniques X-ray topography has been used to give a low magnification images of defects in silicon wafers (Schwuttke 1961).

Metallic impurities dissolved in silicon at high temperatures precipitate at the surface of the wafer as it is cooled. This precipitation concentrates the metal into small silicide particles which intersect the surface but cannot be detected in the SEM. However, a light etch is all that is necessary to produce tiny pits, termed 's' pits, in the polished surface. The effect of these pits can be seen as a haze when the wafer is viewed with grazing incidence illumination and individual pits can be distinguished when the wafer is viewed in Nomarski interference contrast in the optical microscope. A heat treatment to produce this 'haze' is given by Graff (1995), who describes a technique which has been used for many years to concentrate metal as discrete precipitates at the wafer surface. Preparing a plan view specimen then makes them available for analysis by EDX in the TEM. Figure 1 shows a plan view TEM image of the surface region of a silicon wafer which has undergone an oxidation process during which oxidation induced stacking faults (OSFs) were produced. OSFs are an indicator of metallic contamination and in this case metal dissolved in the wafer at a high temperature has precipitated out at the partial dislocation bounding the OSF. The precipitates were analysed by EDX to contain nickel had the flat platelet morphology of $NiSi_2$ (Augustus 1983). The platelets were of the order of 100 nm in size and tilting in the TEM showed that they had a thickness of 10 nm. The small 10–20 nm blobs at the edge of each platelet were shown to contain Cu. They were probably the first to precipitate and provided the nucleation points for the Ni.

Graff also describes the more recent analysis techniques of carrier lifetime measurement, a quick and easy way to show the presence of metallic impurities and total reflection X-ray fluorescence (TXRF) analysis which gives analysis of the near surface metal content. Both of these methods have the advantage of being clean and non-destructive but they cannot show where in an integrated circuit the metal has precipitated, this can only be achieved by TEM or etching. TXRF has a sensitivity of 10^{11}–10^{13} atoms cm^{-2} but when the metal is concentrated into, say, 5 nm precipitates at a spacing of 10 μm apart they are suitable for analysis by TEM with equivalent sensitivity. For this technique to work, most of the metal dissolved at the elevated anneal temperature must precipitate at the front and back surfaces of the wafer. Segregation of metallic impurities in this way will follow any of the high temperature heat treatments involved in device processing. It only needs one of these near-surface metallic precipitates to be present in the gate oxide of a MOS device to

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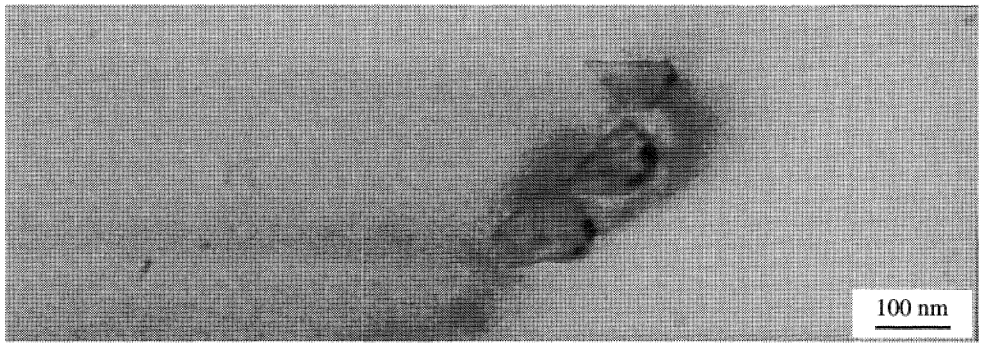

Figure 1. A plan view TEM image from the top surface of a silicon wafer. Precipitates are seen which decorate the perimeter of an OSF. The platelets are $NiSi_2$ and the dark blobs contain Cu. (Bright field $\boldsymbol{g} = 220$, parallel with the out of contrast, stacking fault.)

cause a degradation of the electrical performance of that device. TEM has the imaging capability combined with the analytical capability to demonstrate this cause of device degradation, as demonstrated by Takizawa *et al.* (1987).

(*e*) *Dopant distribution by electron microscopy of etched cross-sections*

Despite many years of study of diffusion rates, it is impossible to reliably predict the exact dopant profile that will result under device processing conditions. This uncertainty is becoming an increasing problem as device dimensions shrink. Shallower junctions and consequential finer tolerances mean that dopant distribution will need to be more accurately controlled. One way to see the dopant distribution on a completed device structure is to make a TEM cross-section through the areas of interest and then subject the cross-section to an etch which has a rate dependent upon the doping level. Such an etch is a very dilute aqueous solution (*ca.* 0.5%) of hydrofluoric acid and was developed by Roberts *et al.* (1985). Their TEM images show a series of bands, contour lines which are actually thickness fringes, the silicon is thinnest close to the polysilicon contact where the dopant concentration is highest. Of particular interest is the lateral diffusion of the dopant under the SiO_2 sidewall of the implantation window. In order to quantify the doping level, plain wafers with the identical implant and drive-in need to be analysed by SIMS. This will give the depth profile in the unpatterned areas from which the etch contours can be related to dopant concentration.

A variation on this etching technique has been used to aid the interpretation of SIMS profiles of delta doped layers of boron in MBE silicon (Augustus *et al.* 1990). An epitaxial silicon layer was grown incorporating a series of delta layers of boron grown at different growth temperatures. A cross-section TEM specimen was prepared and examined. Layers containing boron at a level of 2×10^{20} could be clearly discerned, but at the higher growth temperatures boron precipitation had occurred and only the precipitates were visible in the TEM image. The background dopant level of 2×10^{19} atoms cm^{-3} was invisible. Following etching of the cross-section, this layer of dopant was revealed in the TEM image. The image was then able to confirm the SIMS analysis data that showed that diffusion of boron towards the top surface of the growing layer had occurred. Furthermore, the TEM image was able to show that the dopant incorporation at lower temperatures was homogeneous, that is, no further precipitation had occurred.

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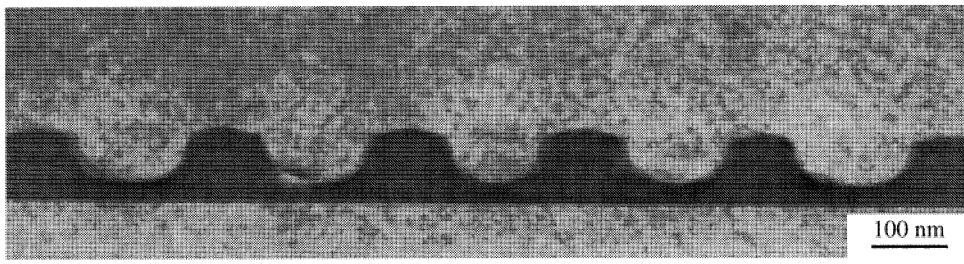

Figure 2. A cross-section TEM image of a portion of a laser structure showing a GaInAsP layer sandwiched between two InP layers. A grating structure has been etched into the GaInAsP and this has then been infilled with InP. The image was taken using a dark field [200] diffraction vector, normal to the plane of the layers.

(*f*) *Cross-section TEM of interfaces*

In the case of boron precipitation described above, it was shown that TEM can give three-dimensional information about microscopic precipitates in the bulk of a sample. More precise information on their distribution in the plane of the delta layer could have been found by preparing a plan view TEM sample at a selected depth. The granular nature of the interfaces formed at high temperatures and seen in the TEM image could not be demonstrated by the SIMS analysis which sputters over an area of many microns. Similarly, sputter profiling techniques cannot differentiate between diffuse interfaces and sharp yet wavy or rough interfaces.

Some interfaces are deliberately wavy, in fact grating structures are etched into some semiconductor laser layers and the wafer is then returned to the epitaxial reactor for an overgrowth to fill in the grating with a material of different composition. Although the result of the etching on the wafer surface can be examined by SEM analysis before the wafer is returned to the reactor, there is no guarantee that the structure will not be modified by the high temperature heat treatment that precedes epitaxial growth of the infill layer or by mixing of material during the growth of this layer. A TEM cross-sectional image of such a structure, after the overgrowth, is necessary in order to see if the integrity of the grating structure has been preserved. Such an image is shown in figure 2. The darker layer is a GaInAsP material which has had a grating etched into it and this has then been successfully infilled with InP. As the furrows will have been cut in a precisely aligned ⟨110⟩, direction the cross-section was made to view the sample along the same direction, cutting the grating at right angles. The grating could be viewed at a variety of thicknesses and the images will show sharp edges as long as the TEM sample is aligned with the ⟨110⟩ direction parallel to the electron beam. Rotating the sample in the plane of the surface made the edges of the grating appear diffuse, the thicker the viewed section, the more diffuse the edges became.

In viewing any sample where the object is to investigate interface sharpness, it is important to choose as thin an area as is practical. But there are limitations which can be imposed by surface amorphization from the ion beam thinning damage on the sample, or bending of the thin foil that occurs in the relaxation of an XTEM sample and also the lack of contrast inherent in very thin samples. A practical consideration in using lattice imaging of samples is that the sample should be at least 20 atomic layers thick and particular crystallographic viewing directions are preferred for optimum contrast in III-V materials. Furthermore, a very precise orientation, along electron channelling directions is necessary in order to create a lattice image.

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Figure 3. A cross-section TEM image showing a defective polycrystalline silicon layer. The top surface of the layer is extremely rough and voids can be seen within the layer (bright field).

If interface undulations are in any direction other than at right angles to the viewing direction the interfaces will appear diffuse.

Frequently the roughness in epitaxial layers of compound semiconductor materials occurs in only one of the orthogonal ⟨110⟩ directions. This is often because of the step propagation method of crystal growth and results from growth on a substrate deliberately misoriented from the true (001) direction by a few degrees. Stress from a mismatch in lattice parameters between the two layers is also a factor. An example from the growth of a GaInAs–InP superlattice was reported in Augustus (1986). If a cross-section is cut in, say, the [110] direction, the layers will appear diffuse and, in the $[1\bar{1}0]$ direction, will appear wavy. The answer to determining the difference between these two possibilities, where the waviness or diffuseness is in an unknown ⟨110⟩ direction, is to prepare the cross-section in a ⟨100⟩ direction and, in a high tilt microscope, tilt to the two orthogonal ⟨110⟩ directions in turn.

The measurement of very thin layers in planar structures is very important for most semiconductor technologies. If the layers are flat and parallel on an atomic scale and aligned with crystallographic directions they can be readily and accurately measured by lattice imaging in the high resolution TEM. Sub-nanometre oxide layers in silicon integrated circuit structures are an important example of a case where the layers grown are not perfect enough to enable us to take full advantage of high resolution imaging but an AES profiling technique has been used instead (Augustus *et al.* 1993). In a silicon–oxide–polysilicon structure, a well annealed sample is necessary to crystallize the polysilicon so that the oxide can be distinguished as an amorphous layer between two crystalline layers. The eventual break-up of the oxide into balls is a requirement of the technology but measurement of the oxide thickness is also required. Unfortunately, early roughening of the interfaces precludes TEM measurements and the most accurate measurement of initial oxide thickness was achieved by performing a line scan for oxygen across an Auger bevel.

As a final example, we show an area of analysis where TEM excels; the detection of voids. Here, there is nothing to analyse, only an absence of material. Figure 3 shows a cross-section TEM image of a polysilicon layer. The surface is extremely rough, a consequence of poor deposition conditions. The surface has been contaminating during growth and the growth has been restricted. This has resulted in surface roughening and deep holes in the material. In some areas, these holes have been pinched-off to leave voids in the layer. The image shown is not from a thick portion of the TEM

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foil, it is probably below 100 nm, yet the roughness through the depth of the foil has lead to multiple images, a reminder that very thin TEM foils produce the clearest sections.

4. Summary

Analytical electron microscopy is a mature technique that has been applied to semiconductor materials and devices for a period of 30 years. Basic three-dimensional characterization by imaging and diffraction techniques have been supplemented by elemental analysis techniques performed, *in situ*, in the electron microscope. New transmission electron microscopes continue to be developed which stretch the analytical capabilities and add to the range of applications available to the semiconductor industry. However, no one technique can stand alone and answer all our questions and the maximum benefit is usually derived from combining a range of techniques focused on the analytical problem.

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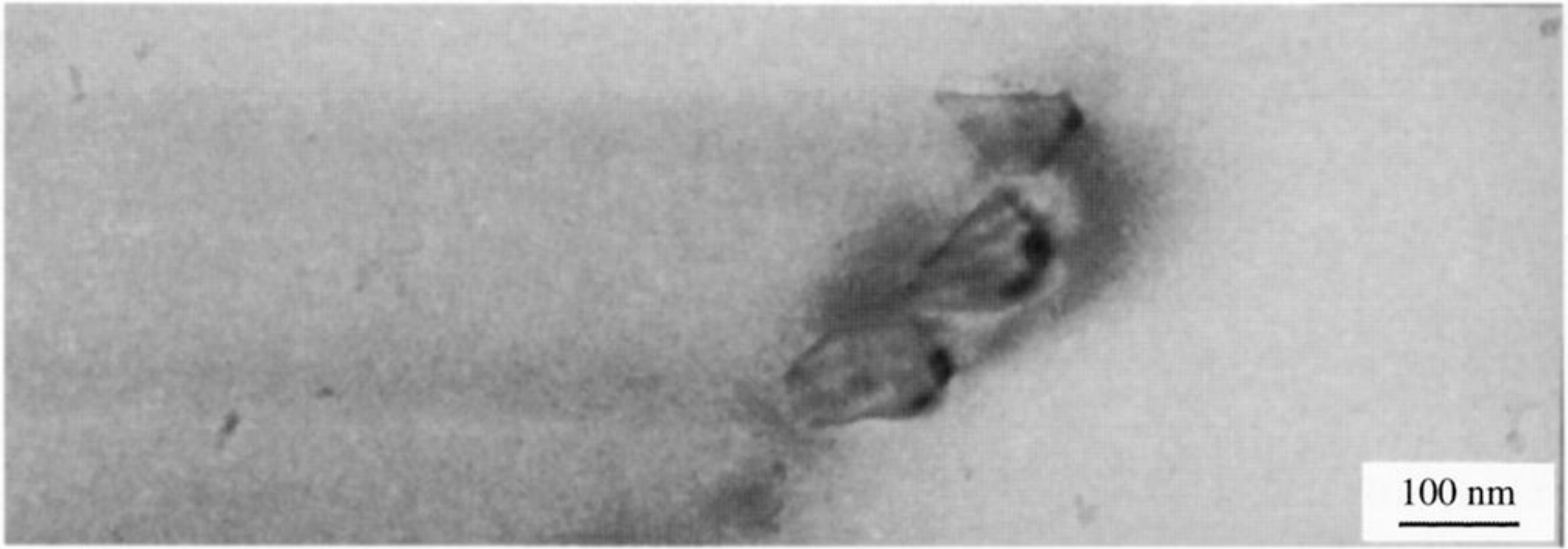
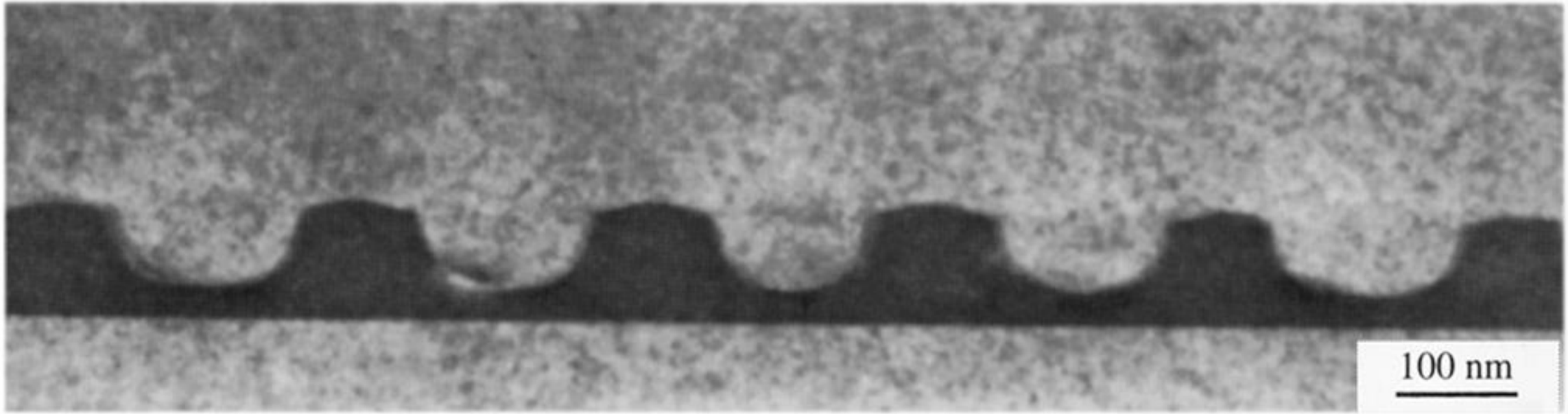
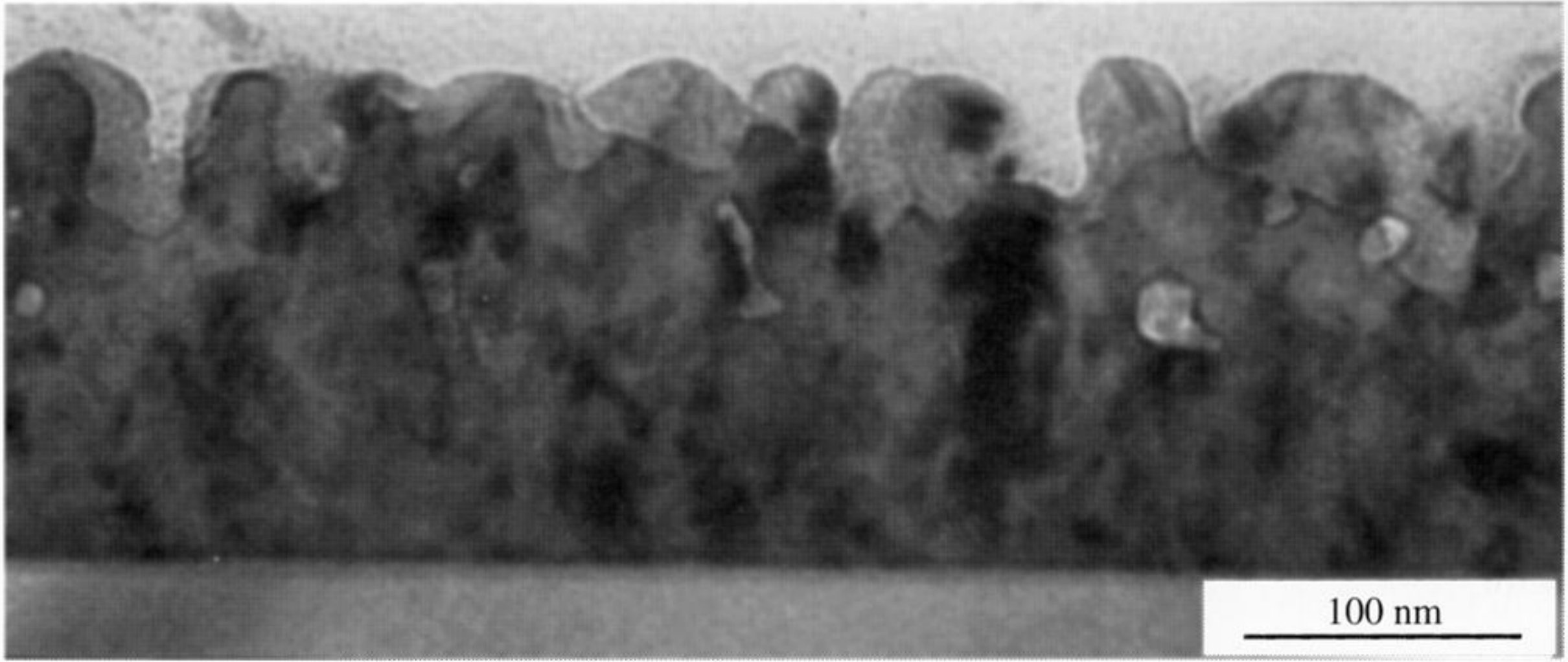


igure 1. A plan view TEM image from the top surface of a silicon wafer. Precipitates are seen
hich decorate the perimeter of an OSF. The platelets are $NiSi_2$ and the dark blobs contain Cu.
Bright field $\boldsymbol{g} = 220$, parallel with the out of contrast, stacking fault.)



igure 2. A cross-section TEM image of a portion of a laser structure showing a GaInAsP layer andwiched between two InP layers. A grating structure has been etched into the GaInAsP and his has then been infilled with InP. The image was taken using a dark field [200] diffraction ector, normal to the plane of the layers.



igure 3. A cross-section TEM image showing a defective polycrystalline silicon layer. The top surface of the layer is extremely rough and voids can be seen within the layer (bright field).