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The Martensitic Transformation in Silicon
I. Experimental Observations

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The Martensitic Transformation in Silicon

1) Experimental Observations

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ABSTRACT

Ribbons of hexagonal material are produced when silicon is indented in the temperature range 400-650°C. This series of three papers, based on two previous preliminary reports [1,2], is a detailed account of an investigation of this phase. In the first paper, the details of the experimental investigation are described. Vickers microindentations have been made in float-zone silicon in the temperature range of 400-500°C. The ribbons of hexagonal silicon are then studied by different techniques of transmission electron microscopy (TEM). Conventional microscopy using strain contrast, selected area diffraction technique, and high resolution electron microscopy (HREM) have been employed to investigate the structure of the hexagonal phase. Using the latter technique, the ribbons are investigated to within a resolution of 0.18 nm and HREM images are compared with simulated ones. HREM is specially useful for studying the faulting within the hexagonal phase and the cubic/hexagonal interface. Analytical microscopy using Electron Energy Loss Spectroscopy (EELS) has been used to show that no oxygen is present in the ribbons within the resolution of this technique.

Throughout the paper, it has been attempted to show that the present hexagonal Si is not a thermodynamically stable high pressure phase, but is related to stress relief when twins interact in silicon. In part II of this series [3], a mechanism for the formation of hexagonal silicon by double twinning is presented through a crystallographic analysis. Finally, in part III [4], a discussion of the results in the context of martensitic transformations and dislocation mechanisms is given and other recent reports of this phase in silicon which has undergone different treatments are discussed.
I. INTRODUCTION

Because of its importance in the electronics industry, silicon is currently being investigated as intensely as iron was during the height of the steel industry. Under normal pressure, silicon is a semiconductor with a diamond cubic (dc) structure which is based on the fcc lattice with a basis of two atoms at (0,0,0) and (1/4,1/4,1/4) (space group: Fd\(\bar{3}\)m). It has a lattice parameter of 0.5435 nm and a density of 2330 kg/m\(^3\). At room temperature, this structure persists up to \(-11\) GPa, and at atmospheric pressure, from 0 K to its melting point of 1683 K. Under higher pressures though, a number of other phases have been reported for silicon. These will be discussed in part III of this series [4].

In 1971/2, in a classical TEM study of defects produced by the hot indentation of silicon, two Soviet workers, Eremenko and Nikitenko, reported the observation of narrow ribbons in the plastic zone of the Vickers microhardness indentations made in the temperature range 400 to 700\(^\circ\)C [5,6]. Eremenko and Nikitenko succeeded in obtaining clear diffraction patterns from which they identified the narrow ribbons as dh silicon with a=0.386 nm and c=0.631 nm. They determined the optimum temperature range for the production of dh silicon to be between 500 and 570\(^\circ\)C. A very significant finding was that the habit plane between the two phases is \(\{511\}_\text{dc}\) with the following orientation relationship:

\[
(01\bar{1})_\text{dc} || (0001)_\text{dh}
\]

\[
[01\bar{1}]_\text{dc} || [\bar{1}2\bar{1}0]_\text{dh}
\] ... (1)

According to this interesting orientation relationship, the (0001) planes of the hcp lattice are parallel to the \(\{01\bar{1}\}\) planes rather than the expected (111) planes of the fcc lattice. The detection of the dh phase by Eremenko and Nikitenko [6] was viewed with a certain degree of skepticism because of this unusual orientation relationship, and because of extra spots in the diffraction patterns from the highly distorted region around the indentation.

Later, in a TEM study of As\(^+\)-implanted Si wafers (80keV at a dose rate of 100 \(\mu\)A), Tan, Foll and Hu [7] observed rod-like defects along \(<110>\) which were embedded in the crystalline silicon beneath the amorphous layer produced by ion implantation. These defects, typically with lengths of a few tens of nanometers and widths of 0.1-0.5 nm, gave rise to extra spots, superimposed on the amorphous ring, which were analysed by Tan et al. [7] to be dh silicon. Because of the small size of these rod-like defects, these authors were unable to determine the habit plane between them and the dc
silicon matrix. However, because of the similarity of the hexagonal/cubic orientation relationship in the two cases, Tan et al. [7] assumed that the implantation-induced defects had the same $\{115\}_{dc}$ habit plane as the indentation-induced $dh$ silicon reported by Eremenko and Nikitenko [6]. Tan et al. [7] further repeated the experiments of the Soviet workers and confirmed all their findings. Observation of hexagonal silicon in implanted silicon has since been reported by many other workers [8-11] and will be discussed in part III of the present series [4].

The first direct structural observation of hexagonal Si was made in 1986 during a HREM study of the plastic zone around Vickers indentations made in silicon at a temperature of $450^\circ C$ [1]. Clear ribbons of hexagonal silicon could be observed extending away from the indentation (similar to Fig. 5). All the findings of Eremenko and Nikitenko were confirmed in this study and it was suggested that the $dc\rightarrow dh$ transition may occur through a martensitic transformation. Later, a crystallographic analysis of the transformation, together with a dislocation mechanism, was proposed [2] which was consistent with all the findings on $dh$ silicon. The present papers are intended to give a more comprehensive description of the results presented in [1] and [2]. In part I, the experimental results on hexagonal silicon will be presented. In part II, a fuller description of the crystallographic analysis will be given. Finally, in part III, the transformation will be treated in terms of possible dislocation reactions and other reports of $dh$ silicon, obtained through processes such as ion implantation and electron irradiation [7-11], compression of Si under high hydrostatic pressures [12], and annealing of Czochralski grown Si [13], will be discussed.

2. EXPERIMENTAL

Two ingots of float-zone single crystal silicon, manufactured by Wacker Chemitronics, were used in the present experiments. Both crystals were intrinsic with resistivities of 1900 and 2000 $\Omega$.m. They were oriented along the [011] direction using the back-reflection Laue technique, and a few slices, with faces parallel to the (011) plane, were cut by means of a diamond saw. Specimens, with diameters of 2.3 mm (for the JEOL 200CX microscope) and 3mm (for the JEOL 4000EX) were obtained by cleaving the Si slices. These were polished by different grades of diamond paste. One side of each TEM specimen was given a final finish of Syton. The first series of specimens were prepared at the University of Oxford by Dr. Jayne Samuels using a Matsuzawa MHT1 microhardness tester fitted with a hot-plate capable of reaching about $500^\circ C$. For this series, a 10x10 array of 10g Vickers
indentations were made at 450° C in air on the Syton polished face of each specimen. The second series of specimens were prepared at CWRU using a Nikon High-Temperature Microhardness Tester (Model QM). In this case, 10x10 arrays of 50g Vickers indentations were made in vacuum at three different temperatures: 400, 450 and 500° C.

The specimens were subsequently prepared for TEM by mechanical polishing and dimpling to 30 μm followed by ion-beam thinning to electron transparency; all from the back face. The conventional and analytical TEM were carried out in a Philips 400T equipped with Energy Dispersive Analysis (EDS) and Electron Energy Loss Spectroscopy (EELS) facilities. For HREM investigations, a JEOL 200CX microscope, with a point-to-point resolution of 0.24 nm, and a JEOL 4000EX, with a resolution of 0.18 nm, were employed. Seven- and seventeen-beams (including the kinematically forbidden 200 and 222 reflections) in the [011] zone were used respectively to form the high resolution images in the two microscopes. Most images were taken close to the optimum defocus (~63 nm for the 200CX and ~50 nm for the 4000EX) with through-focus series in a few cases. One of the specimens was vacuum encapsulated in a quartz tube and annealed at 900° C. This specimen was also subsequently back thinned to electron transparency and examined by TEM.

3. RESULTS

3.1 The Plastic Zone

A low magnification multi-beam bright field (BF) micrograph of the general area around one of the indentations is shown in Fig. 1. The indentation site (i.e. the area directly underneath the indenter) is the square-shaped region in the center of the figure which shows dark contrast. It consists of a heavy tangle of dislocations the density of which is so high that it is not possible to resolve individual dislocations. Clarke et al. [14] have recently shown that room-temperature indentation of silicon results in a crystalline-to-amorphous transformation directly underneath the indenter. However, at the temperatures under investigation here, i.e. 400-500° C, there is no evidence of the generation of amorphous material. It is interesting to note, however, that when specimens which have been indented at room temperature are annealed at high temperatures, ribbons of twins and of hexagonal Si appear [15]. Explanation of this fact will be left until later (see part III [4]).

It should be recalled that the indentation site is a region of high hydrostatic pressure. Just
outside this region, however, shear stresses are predominant and it is here that it becomes possible
to resolve individual defects. The triangular region of contrast surrounding the indentation site
in Fig. 1 is the projection of one of the tetrahedra defining the plastic zone [16]. In this region
individual dislocations may be observed although with difficulty. These dislocations lie on \{111\}
planes and have a/2 <110> Burgers vectors which are inclined to the surface [16].

3.2 The Elastic-Plastic Zone

Outside the plastic zone, the defect configuration is much clearer. Here the defects are
basically of three types:

1) Arrays of rosette dislocations on inclined \{111\} planes which, at these low temperatures, lie
along the <110> Peierls valleys. These are the hexagonal dislocation loops denoted by R in Fig.
1 and have a/2 <110> Burger vectors which, in this orientation, are parallel to the surface
[17]. A close-up view of this region is shown in Fig. 2. The indentation site is to the bottom
right (outside) of the figure and, as may be seen, the density of dislocations decreases rapidly
going away from the indentation center. To the left of the figure, the dislocation density is
sufficiently low that dislocation interactions are relatively weak. Here, because of the high
Peierls energy, the dislocations lie in Peierls valleys along the <110> directions at this low
indentation temperature. Closer to the indentation site, however, strong interactions cause
dislocations to move out of the Peierls valleys and become curved.

2) Profuse twinning on \{111\} planes. Twins on those \{111\} planes which are normal to the (011)
surface appear in the form of narrow bands lying along the <112> directions in the micrograph.
Some of these are denoted by T in Fig. 1 and are parallel to (111) and (111) planes. A high
resolution image of one of the microtwins parallel to (111) is shown in Fig. 3 and is denoted by
T\(_1\). A parallel microtwin is visible at the upper right hand corner of the figure. There are
also two microtwins on (111) planes which intersect T\(_1\); one of these is denoted by T\(_2\) in Fig. 3.
The diffraction pattern, shown in the inset to Fig. 3, shows the expected streaking of the spots
along the <111> directions because of the narrow width of the twins. Along these directions,
and superimposed on the streaks, there are also rows of extra spots. These arise from inter­
ference effects from multiple twin boundaries; the spacing between the extra reflections is
equal to the reciprocal of the distance between the twin planes [18]. The crystal lattice of
the twin intersection is unclear in this image due to the difficulty of accommodating the shear
strains of T\(_1\) within T\(_2\). Mahajan and Chin [19] have suggested secondary twinning as a possible
accommodation mode, but as shown in the crystallographic analysis in part II of this series of
papers [3], the plane strain alternative is reverse twinning on \{511\} (indices with respect to
the matrix).

3) Ribbons of hexagonal silicon having habit planes of the type (511) and (511) parallel to the
beam. These appear as straight dark lines along <552> directions, some of which are denoted by
H in Fig. 1; a series of these bands is shown in Fig. 4. Except for the direction of their traces, the twin bands and hexagonal ribbons look quite similar; though, in general, the hexagonal ribbons are slightly wider than the twin bands. A fuller discussion of the hexagonal ribbons is presented in the following section.

3.3 Hexagonal Silicon

3.3.1 Interaction with Twins

Fig. 5 shows a HREM micrograph of one of the hexagonal ribbons originating from the vicinity of the plastic zone and extending by about 3-4 μm into the virgin silicon. In general, the width of a hexagonal band is not constant, but can vary slightly from one part of the band to another. As seen in this micrograph, there are invariably a large number of twins which intersect a hexagonal ribbon. It appears that some of the twin bands have formed subsequent to the growth of the hexagonal ribbon; in this case, the twins which originate from the plastic zone or vicinity, have stopped propagating on encountering the hexagonal ribbon; examples of this are shown in Fig. 6a.

On the other hand, there are also twin bands which may have formed prior to the growth of a hexagonal ribbon. In this case, shown in Fig. 6b, the hexagonal ribbon shears the twin bands as it crosses them. From the figure, the shear is on the (511)$_{dc}$ plane along the [255]$_{de}$ direction. The shear strain has been measured for a number of bands of different widths and, in each case, found to be -0.7. This is approximately the same as the shear strain introduced by a twin, i.e. 0.707, the strain produced by a twinning partial $\frac{1}{6} <112>$ ($=\frac{1}{6}|b|/d_{111}$). This is an important point which suggests a simple relationship between hexagonal silicon and twinning and forms the basis for the crystallographic analysis given in paper II [3].

3.3.2 Orientation Relationship

A higher magnification image of hexagonal bands is shown in Fig. 7. The upper part shows the central section of a band which is well developed. The lower part shows the tip of another hexagonal ribbon. The two ribbons are connected by a microtwin, T, at the center of the micrograph and a fault, possibly a dislocation line, running across the upper band may be observed. Some of the crystallographic information in the hexagonal and the cubic phases is noted on Fig. 8. The orientation relationship is clearly seen in this micrograph and is in good agreement with eqn. (1) as determined by Eremenko and Nikitenko [6] from their diffraction study. The direction of observation is [011]$_{dc}||[\overline{1}2\overline{1}]_{dh}$. The basal planes of the hexagonal phase are inclined about 4° to the (011)$_{dc}$ planes of the cubic matrix and the [100]$_{dh}$ direction is nearly parallel to the [100]$_{dc}$. The deviation between the two directions can vary from one part of the ribbon to another: from exact parallelism up to about 5.5°. Measurement of the fringe spacings on this, and similar micrographs, together with diffraction analysis, confirm the findings of Eremenko and Nikitenko [6] for the lattice parameters of the hexagonal silicon. Based on these lattice spacings the volume per atom is the same in both phases. The cubic/hexagonal habit plane is not atomically flat because of the high density of faulting. On average though it is very close to $\{511\}$, in agreement with the findings of Eremenko and Nikitenko [6].
In the areas where the hexagonal silicon is free of faulting, one set of the \( \{111\} \) planes in the matrix are continuous (coherent) with the basal planes of the hexagonal ribbon. An example is shown in Fig. 8 where one set of these planes is traced through the micrograph and the one-to-one correspondence between the \((1\,\bar{1}\,1)_{dc}\) planes of the matrix and the \((0001)_{dh}\) planes of the hexagonal material is clearly seen. The angle between these two sets of planes is \(141^\circ\) and the \((5\,\bar{1}\,1)_{dc}\) habit plane bisects them. The simple shear may be measured more clearly now: \(39^\circ (=180^\circ -141^\circ)\) on the \((5\,\bar{1}\,1)_{dc}\) plane along the \([2\bar{5}\,5\,]_{dc}\) direction giving a magnitude of \(2\tan(39^\circ /2)=0.707\) as determined before.

Figs. 5 and 7 show the high density of faulting on the basal planes within the hexagonal ribbon. Only in limited volumes within a ribbon is the material in the form of defect-free \(dh\) structure; such a region can be clearly seen in Figs. 7 and 8. Sometimes the faulting is so frequent and regular that the structure is closer to a different polytype, e.g. \(12H\). Laser diffractograms from some areas of the ribbons, not shown here, show these higher periodicities.

Fig. 9 shows the diffraction pattern with the beam parallel to the \([0\,1\,1]\) zone of the cubic silicon and the \([\bar{1}\,2\,\bar{1}\,0]\) zone axis of hexagonal silicon. The heavy faulting within the hexagonal ribbon causes strong streaking of the hexagonal spots in the diffraction pattern. There are also numerous extra spots due to primary twinning, secondary twinning and double diffraction. The \((0001)\) reflection, which is forbidden in the diamond hexagonal structure, is present because of double diffraction. Diffraction patterns from a few other zones are shown in Fig. 10; the inset stereogram shows the different diffraction zones in the \(dc\) \(Si\) relative to those in the \(dh\) \(Si\). Indexing of all these patterns is complicated by the presence of multiple twinning and double diffraction.

### 3.3.3 Microanalysis

A few of the hexagonal ribbons were analysed by EELS at liquid nitrogen temperature using a cold stage. An example is presented in Fig. 11, where the \(Si-L_{2,3}\) edges are shown from the hexagonal ribbon and also from the cubic matrix. The inset is a reference for pure elemental \(Si\) from Ref. [20]. There is no noticeable difference between the three spectra; in particular no pre-ionization edges, which are characteristics of other chemical states of \(Si\) (oxide, carbide, ...), can be detected. Also, no noticeable edge from oxygen, or any other element, was detected in the hexagonal ribbon or the cubic matrix.

### 3.3.4 Image Simulations

A number of image simulations have been carried out on the hexagonal phase using the multi-slice formulation, for comparison with experimental HREM images. Fig. 12a shows the simulated image for a 400 kV microscope with the electron-optical parameters typical of the JEOL 4000EX at the optimum defocus of \(-50\ \text{nm}\). It corresponds to a crystal thickness of \(-10\ \text{nm}\) and, at this value of under-focus, atom pairs appear as dark spots. The contrast changes at a defocus value of \(-80\ \text{nm}\) when the dark spots correspond to tunnels. In the experimental images, there is a slight asymmetry in the sense that the basal planes are not of equal intensity but have alternate strong and weak contrast. Two effects can give rise to this asymmetry: a slight misalignment of the incident beam, or a tilt of the hexagonal band so that the beam is not exactly along the \([\bar{1}\,2\,\bar{1}\,0]\) zone axis. At extreme cases,
with the beam misaligned by more than 10 milliradians with the optic axis, or with the hexagonal band tilted more than 3°, a doubling of the periodicity of (0001) fringes occurs. Figs. 12(b) and 12(c) show simulated images of hexagonal silicon with these two effects taken into account. In Fig. 12(b), the beam is tilted by about 3 milliradians with respect to the optic axis, while Fig. 12(c) shows a simulated image for a crystal tilted by ~1.6°.

3.3.5 Annealing Experiments

Focusing the incident beam on a hexagonal ribbon by means of the second condenser lens in order to heat the specimen did not result in any noticeable change. However, examination of an indented specimen which was annealed at 900°C in vacuum for about one hour showed that the hexagonal ribbons had disappeared. Precipitates with a faceted morphology had appeared within the plastic zone mostly decorating the dislocations and twins. Diffraction patterns and high resolution microscopy indicated that these precipitates were not hexagonal Si and EELS showed them to have a high carbon concentration. It is believed that annealing for 1 hour in the rather poor vacuum gave rise to contamination by carbon from the mechanical pump and the formation of SiC.

4. DISCUSSION

4.1 Phase Stability

Since dh silicon forms during indentation of the material, at first it might be presumed that, under the high pressure generated by the indenter, silicon undergoes a phase transformation to one of its high-pressure forms. However, as was mentioned before, the hexagonal ribbons do not form directly underneath the indenter where the pressure is highest, but rather, they occur at the periphery of the plastic zone where shear forces are prominent. Further, there is a ~30 to 40% volume change in the high-pressure transitions, whereas there is practically none in the indentation-induced phase transformation. Also, the experiments of Wentorf and Kasper [21], and the theoretical calculations of Yin and Cohen [22,23], have clearly shown that dh silicon is a metastable phase under all conditions and not obtainable by the application of high pressures.

4.2 Temperature Dependence of Accommodation Modes

At high temperatures, the shape changes necessary to accommodate the indenter can be taken care of by thermally activated processes of dislocation generation and glide. In this case, the applied stress is high enough to nucleate dislocations and there is enough thermal energy available for dislocation glide to respond quickly to the rapidly applied external stress. Although the dislocations may be dissociated, the mobility of both partials is sufficiently close that plastic deformation of the material through slip takes place [24].

On the other hand, at low temperatures, although local dislocation nucleation may take place by the very high applied stresses underneath the indenter, dislocation glide is very difficult and plastic deformation through slip is negligible. In this case, the accommodation of the indenter takes place by the fracture of the specimen under the indenter, i.e. the material simply tears apart to accommodate the indenter. An alternative accommodation mode is the decrease in the atomic volume
of the material underneath the indenter through high pressure phase transformations [25] and/or crystalline-to-amorphous transitions [14].

At intermediate temperatures, where bond rupture is not favorable, because of the increasingly different mobilities of the partial dislocations, and the complicated stress field around an indenter, extensive twinning takes place [24]. In addition to different mobilities of the leading and trailing partial dislocations, primary glide as well as cross-slipping of screw dislocations are essential for twinning to occur [24]. High resolved shear stresses on the primary glide plane result in a thickening of the twin plates, whereas high resolved shear stresses on the cross-slip plane (the twin plane) result in an extension of the twin plates. It is possible that, in the complex stress field of the indentation, at some locations a secondary twin nucleates within the primary one. This may take place by a pre-existing screw dislocation [2]. The extension of the secondary twin into the matrix will then result in the formation of hexagonal silicon. It is this aspect of the overall deformation that is treated in detail in parts II and III [3,4].

Thus, as a whole, it seems that under the huge stresses applied by the indenter, the accommodation of the indenter may take place by three different mechanisms, each of which is predominant in a particular temperature range:

1) High temperatures ($T > -700^\circ C$): dislocation glide and formation of a large plastic zone and extended rosette patterns.

2) Intermediate temperatures ($-400^\circ C < T < -700^\circ C$): martensitic transformation, in the form of twinning and through the formation of hexagonal phase.

3) Low temperatures ($T < -400^\circ C$): fracture.

The driving force for the martensitic transformation which is provided by the indentation stress should supply not only the energy required to transform the lattice and the associated energies for the elastic and plastic deformations which accompany the transition, but also nonmechanical terms such as interfacial energy between the matrix and the martensite plates. In the case of silicon, a large energy term will also be involved if the transformation results in the formation of dangling bonds, say at the interface. However, Tan et al. [7] have proposed a \{511\} interface between diamond cubic and hexagonal silicon which consists of five- and seven-membered atomic rings without dangling bonds (also see [26]). This is consistent with the results of the crystallographic analysis which shows \{511\} to be a coherent interface and a low-index plane in the 59 coincidence site lattice. Further details of this will be presented in part II [3].

4.3 Martensitic Nature of the Transformation

As pointed out in [1], there are a number of features in the HREM micrographs which indicate that the cubic-to-hexagonal phase transformation is martensitic. Firstly, the mode and temperature of formation indicate that the mechanism does not involve diffusion. A salient feature of martensitic transformations, which is at least approximately true, is that the interface between the transformed and the untransformed regions remains planar, undistorted and unrotated. Because of the constraints imposed on the transformation, the habit plane and the orientation relationship are usually irrational. In the present case, the orientation relationship is such that the close-packed
directions are exactly parallel, (0001)_{d_h} deviates by \(-4^\circ\) from (011)_{d_c}, and the habit plane is the high-index \{511\}_{d_c} plane. There is extensive faulting within the hexagonal plates which is also a typical feature of martensite transformation. It should also be noted that in the temperature range where hexagonal ribbons form in the present experiments, twinning is a prominent mode of deformation. Twinning, of course, may be considered as a form of martensitic transformation albeit with identical lattices for the two phases.

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Figure Captions

Fig. 1. Low-magnification multi-beam BF micrograph of the area around a 10 g indentation made at 450°C on (011) silicon. The dark square at the center of the micrograph is the site of the indenter impression; T and H denote the twin and hexagonal bands respectively, with composition planes normal to the foil surface; R denotes the hexagonal dislocation loops in the rosette structure.

Fig. 2. Rosette dislocations at higher magnification. Note the straight nature of dislocations lying in the <110> Peierls valleys further away from the indentation site. Closer to the indentation center, the high density of dislocations results in strong interactions which moves the dislocations out of the Peierls valleys.

Fig. 3. An HREM micrograph of a twin band intersected by microtwins, a few layers thick, on other \{111\} planes. The inset shows the diffraction pattern.

Fig. 4. Low magnification HREM micrograph of a series of parallel hexagonal ribbons.

Fig. 5. A higher magnification HREM micrograph of one of the hexagonal ribbons. Note the numerous microtwins intersected by the hexagonal ribbon.

Fig. 6. (a) Microtwins formed subsequent to growth of the hexagonal ribbon, (b) Microtwins formed prior to growth of the hexagonal ribbon. Note the shearing of the twins along the [255] direction due to the crossing of the hexagonal ribbon through them.

Fig. 7. A high magnification HREM micrograph of two hexagonal ribbons, denoted by H. The lower ribbon, which is connected to the upper band by a microtwin T, shows the tip region of the hexagonal band. Note the high density of planar faults in the upper band. A defect, possibly an inclined dislocation, which has locally disturbed the atomic arrangement in the upper hexagonal band, can be also seen.

Fig. 8. The cubic/hexagonal silicon interface in a region which is free of faults. Coherency (continuity) of \((1\bar{1}1)_{dc}\) planes of the matrix with the \((0001)_{dh}\) planes of the hexagonal ribbon is shown by the tracings.

Fig. 9. A \([011]_{dc}\parallel[12\bar{1}0]_{dh}\) diffraction pattern of cubic/hexagonal silicon.

Fig. 10. Diffraction patterns of the cubic/hexagonal silicon from six other zone axes. The stereogram shows the different zones in dc Si relative to those in dh Si.
Fig. 11. An EELS spectrum from (a) the cubic Si matrix, (b) a hexagonal Si band. The inset in (b) is a reference spectrum for pure elemental Si from Ref. [20].

Fig. 12. Effect of incident beam misalignment and crystal tilt on the simulated HREM images of the hexagonal phase at the optimum defocus (~50 nm) with the electron-optical parameters typical of JEOL 4000EX (Coefficient of spherical aberration = 1 mm; convergence semi-angle = 0.75 mrad; chromatic half-width = 8 nm; vibration halfwidth = 0.05 nm). Crystal thickness = ~10 nm, (a) No crystal tilt and incident beam along the optic axis, (b) ~3 mrad beam tilt, (c) ~1.6° crystal tilt. Note the asymmetry in the intensity of the basal planes (horizontal).
SI-L2, 3 FROM CUBIC SILICON MATRIX

Figure 11a
SI-L2, 3 FROM HEXAGONAL SILICON

Figure 11b