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R. H. BHAWALKAR
S. K. SHARMA
A. K. SARKAR
VIPIN KUMAR
DANESHWAR SEN
*National Physical Laboratory,*
*New Delhi, India*

## *Crystal imperfections in silicon epitaxial layers grown on ion-implanted substrates*

Ion implantation is now being introduced into many semi-conductor manufacturing processes. Generally, it is extremely successful, but recently Moline *et al.* have observed that crystal defects can be generated in an epitaxial layer grown on an ion-implanted substrate [1]. They grew epitaxial layers by silane pyrolysis on substrates implanted with arsenic ions at 150 kV. The general perfection of growth was monitored by Berg-Barret X-ray topography but the individual defects were not identified. We have investigated a similar experimental system and have found the same imperfect growth. Further, we have used transmission electron microscopy (TEM) to identify the defects present as edge dislocations and epitaxial stacking faults.

2 in. diameter 5 Ω-cm p-type Si slices were implanted with arsenic ions at 100 kV and 1 mA through a thermal oxide mask. This mask consisted of fingers of oxide 650 μm wide with 650 μm spacing which was aligned parallel with the long axis of the ribbon beam of ions. The beam was held stationary while the slice was passed under the beam, moving perpendicular to the fingers of the mask. After each pass the slice was moved ~ 1 mm parallel with the fingers of the mask and then passed under the beam in the opposite direction. The total dose received by the slice was ~ $10^{16}$ ions $cm^{-2}$. After implantation the slices were oxidized at 1200° C for 2 h. We examined the slices before and after epitaxial growth using Lang X-ray topography (XRT), platinum/carbon replicas and TEM.

The conditions of epitaxial growth and the observations made by the various techniques are shown in Table I.

Fig. 1 is a transmission X-ray topograph of part of a slice with 0.5 μm epitaxial layer grown from silane at 1050° C. The ion implantation gives rise to bands of contrast running approximately parallel to the direction of movement of the slice as it passed under the ion beam. These bands are cut by the fingers of the oxide mask

TABLE I

| Material | Visual inspection | XRT | Pt/C replica | TEM |
|---|---|---|---|---|
| Substrate after implantation and oxidation | Smooth polished surface | Bands of contrast, slip, "swirls" | Very smooth | Edge dislocation loops |
| Epitaxial layer grown from silane at 1050° C | Bands showing diffuse reflection | Bands of contrast, slip | Shallow circular depressions with pits at bottom | Epitaxial stacking faults ~ $10^4$ $cm^{-2}$; edge dislocation segments ~ $10^7$ $cm^{-2}$ |
| Epitaxial layer grown from $SiCl_4$ at 1140° C | Smooth polished surface | Bands with slight contrast | Very smooth | Epitaxial stacking faults ~ $10^4$ $cm^{-2}$ |

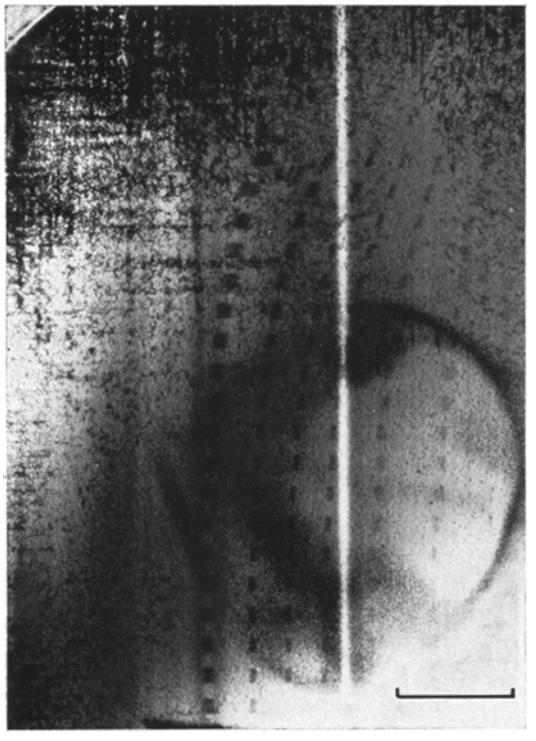

*Figure 1* 220 Mo$K\alpha_1$, transmission X-ray topograph. Bar ≡ 500 μm.

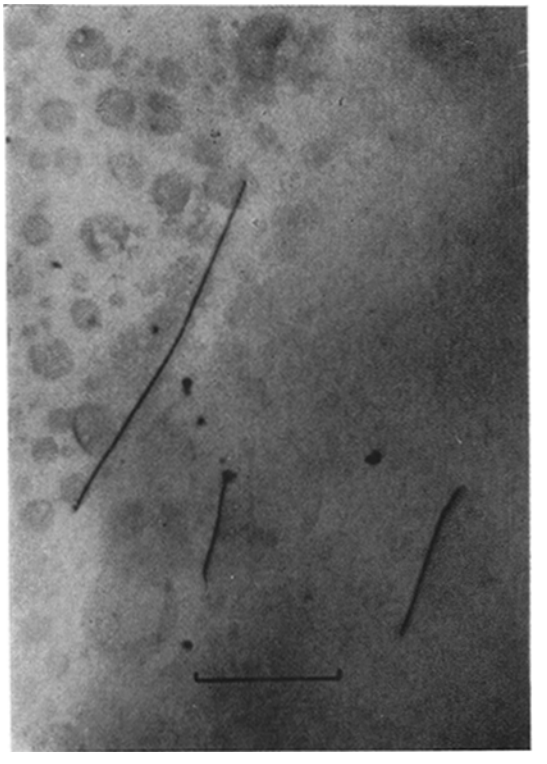

*Figure 2* Transmission electron micrograph of dislocation loops. 220 type reflection, bright-field. Bar ≡ 1 μm.

and result in the rectangles of high contrast seen in Fig. 1. Each of these rectangles contains defects too closely spaced to be individually resolved by X-ray techniques. TEM has high resolving power but requires specimens with a relatively high concentration of defects. We used XRT as a non-destructive method of selecting areas of high defect density for thinning for TEM. Fig. 1 also shows "swirling", which is believed to be due to point defects in the substrate [2] and dislocations generated by thermal slip. Fig. 2 is a transmission electron micrograph of one of the regions of high contrast seen in Fig. 1. The micrograph shows dislocation loops lying on (1 1 0) planes perpendicular to the (0 0 1) substrate plane. A complete Burgers vector analysis showed that they were sessile in nature with $\frac{1}{2}\langle 1\,1\,0\rangle$ Burgers vectors lying in the plane of the slice. Most of these dislocation loops penetrated > 1 μm into the substrate so that only the outer ends of the loop are seen in the micrograph. Similar dislocations are observed in heavily doped emitters and it has been suggested [3] that these dislocations are produced by a climb process using vacancies moving from the surface. Alternatively, Hu [4] suggested that silicon interstitials are responsible for the climb. In either process, the layer of ion-implanted damage could supply either the vacancies or the interstitials required.

Although we observed a low density of epitaxial stacking faults, no oxidation-induced stacking faults were found. It is also worth noting that the pits revealed by Pt/C replicas on grown layers (Table I) were not associated with dislocations. It is known [5] that similar depressions can be caused by crystallites of SiC formed on the substrate surface due to contamination. Glancing angle electron diffraction was carried out on an ion-implanted substrate but no evidence of additional crystalline material was detected.

The observation that a large-area beam of ions produced narrow bands with high defect density was unexpected. The area covered by one pass of the beam had a large overlap with areas covered on preceding passes. The causes of this effect are not understood but are probably related to heating of the slice by the implanting beam.

The epitaxial layers grown from $SiCl_4$ at 1150° C appeared to have a smooth shiny surface under visual inspection. Platinum/carbon replicas confirmed that the surface was free from steps of height greater than 50 Å. However, XRT and TEM showed that defects were still present, though at low densities.

It is well known [6] that ion implantation creates an amorphous layer just below the surface of the crystal. As the surface is oxidized this layer is partly absorbed into the oxide and partly annealed out. Our work shows that the damage is not completely removed but that dislocation loops propagate into the crystal by a climb process. Most of these loops propagate more than 1 μm into the crystal, a finding which is in conflict with Moline *et al.* [1]. These authors reported that the damage in their slices could be eliminated by etching only 0.2 μm from the surface.

Under some conditions of epitaxy the ends of these dislocation loops propagate with the layer where they form the majority of defects found ($\sim 10^7$ cm$^{-2}$). The density of epitaxial stacking faults was much less ($\sim 10^4$ cm$^{-2}$). Although the growth from $SiCl_4$ was much more successful, some defects were still present in those layers.

Work is continuing to elucidate the mechanisms by which the dislocations are formed. In particular we wish to investigate the effect of beam heating as both our results and those of Moline *et al.* suggest that the defect density may be related to substrate temperature during implantation.

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J. F. C. BAKER
R. OGDEN
*Allen Clark Research Centre,*
*The Plessey Company Limited,*
*Caswell, Towcester,*
*Northants, UK*

## *Crack growth in epoxide resin adhesives*

The vast majority of the structural adhesives commercially available are based upon epoxide resins and this has generated interest in the fracture properties of these relatively brittle, thermosetting materials. This paper describes some preliminary results on the application of continuum fracture mechanics to the failure of epoxide resins and although this, in itself, is not novel [1-3], by also measuring rates of crack propagation some unexpected characteristics of crack growth have been observed.

The specimen geometry employed in this investigation was a tapered double cantilever beam joint as shown schematically in Fig. 1. Detailed specimen preparation and fracture test techniques have been reported elsewhere [1, 4] and it is sufficient to note that the substrate was aluminium alloy, to specification British Standard 1476.He 30, which was machined into cantilever beams, 308 mm long, 12.7 mm thick and with a height, $h$, varying between 16.0 and 47.8 mm. The surfaces to be bonded were first subjected to a liquid and vapour degreasing bath of dichloro-ethane, then grit blasted with 180-220 mesh alumina, then degreased again and finally allowed to air-dry in a desiccator. The epoxide adhesive employed was a diglycidyl ether of bisphenol A cross-linked with either 9.4 mass per cent of a tertiary amine curing agent (tri-2-ethyl hexanoate of 2,4,6 tris (dimethylaminomethyl) phenol) or 11.0 mass per cent of a primary curing agent (triethyltetramine). In the former case the curing schedule was 23° C for 96 h, followed by 100° C for $1\frac{1}{4}$ h and finally 180° C for $2\frac{1}{2}$ h and, in the latter case, 23° C for 2 h followed by 60° C for 2 h. The specimens were then usually conditioned at 23° C and 56% R.H. for a few days prior to testing. The thickness of the epoxide resin layer was controlled to 0.50 ± 0.06 mm.