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Electron Microscope studies of Ion Implanted Silicon and Gallium Arsenide after subsequent Laser and Furnace Annealing

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1. General

The present investigation is concerned with laser annealing of ion-implanted Si and GaAs specimens, and in particular with the use of the TEM and SEM EBIC methods of examination to investigate the damage structures occurring. Three separate projects are described. First, P⁺ implanted Si is laser and furnace annealed, and the resulting structures as observed by TEM are compared. Second, similar studies are made of Zn⁺ implanted GaAs, the laser annealing being performed either with or without a surface encapsulating layer. Third, As⁺ implanted Si is laser annealed, and the uniformity of the annealing process as determined by SEM EBIC studies is assessed.

For the laser annealing, specimen areas in the range 6 to 10mm across were irradiated using single pulses in the range 20 to 40ns from Q-switched ruby lasers with wavelength 0.695μm. For the TEM studies, 'plan-view' and '90° cross-section' specimens were prepared. The latter were obtained by cleaving the slices, and then mechanical polishing followed by low-energy ion-beam thinning, as described previously. The cross-section micrographs obtained correspond to the (110) plane perpendicular to the original (111) specimen surface plane. All of the TEM examinations were performed using the bright-field, strong-beam, diffraction contrast method. Transmission electron diffraction patterns were obtained to aid in the identification of the damage, using either the standard selected-area method or microdiffraction techniques. The SEM EBIC method was used by collecting the electrical signal at the p-n junction formed in the specimen by the implantation and annealing treatment.

2. P⁺ Implanted Si

p-type, 17 ohm cm, (111) Si slices were implanted with 120keV P⁺ ions using a dose of $10^{15}$ ions cm⁻². This corresponds to an LSS projected range of $1510 \pm 690\AA$. The specimen temperature during implantation was $\sim 100^\circ$C (due to ion beam heating).

For the implanted but unannealed specimen (Fig.1a), the damage consisted of an amorphous layer continuous from the specimen surface to a depth of
ν 1800Å (the broad grey band in Fig.1a). At the lower edge of this layer there was a narrow irregular zone ν 250Å thick corresponding to heavily damaged, but not amorphous, material (the narrow dark band in Fig.1a).

After this specimen had been furnace annealed at 750°C for 20 min. (Fig.1b), a first damage layer extended from the surface to a depth of ν 1500Å and consisted of twinned material, low-angle boundaries, dislocations, etc. In addition, a second damage layer was present at a mean depth of 2000Å and consisted of small dislocation loops.

After the specimen of Fig.1a had been laser annealed with an energy density of 0.7 J cm⁻² (Fig.1c), a first damage layer comprising polycrystalline material with mean grain size ν 1000Å extended from the specimen surface to a depth of ν 1500Å. A second damage layer consisting of a dense, fine structure and ν 500Å wide occurred and was in direct contact with the first damage layer.

After the specimen of Fig.1a had been laser annealed with an energy density of 1.5 J cm⁻² (Fig.1d), a first damage layer comprising single crystal material containing mainly stacking faults but also dislocations extended from the surface to a depth of ν 1800Å. The stacking fault density at the surface was ν 10⁹ cm⁻². A second damage layer consisting of a dense, fine structure and ν 200Å wide occurred and was in direct contact with the first damage layer.

For the 750°C furnace annealed specimen, the dense damage in the first layer is considered to arise from local areas in the initial amorphous band recrystallising to give small misorientated grains, twins, etc. The small loops in the second layer arise from annealing of the initial heavily damaged material present beneath the amorphous layer.

A striking feature of both laser annealed specimens is the two distinctly different damage zones in contact with one another. Our interpretation of these structures is as follows. For the 0.7 J cm⁻² specimen, a molten surface layer ν 1500Å thick forms, corresponding to ν 75% of the initial amorphous
layer. The remainder of the amorphous layer beneath the molten surface partially recrystallises in the solid phase. The molten layer then solidifies by growing epitaxially on top of the partially recrystallised material, and this produces the polycrystalline first damage layer.

For the 1.5 J cm\(^{-2}\) specimen, a molten surface layer \(\sim 1800\AA\) thick forms, corresponding to 100% of the initial amorphous layer. The heavily damaged material beneath the initial amorphous layer partially anneals. The molten layer then solidifies by growing on top of this partially annealed material, and this produces the single crystal first damage layer containing stacking faults and dislocations.

3. \textbf{Zn\textsuperscript{+} Implanted GaAs}

For most work on ion implanted GaAs, the specimens have been subsequently thermally annealed using a protective surface coating such as Al or Si\(_3\)N\(_4\) to avoid loss of As. In the present work, ion implanted GaAs has been laser annealed either with or without a Si\(_3\)N\(_4\) coating, and the results compared with similar specimens thermally annealed with an Al coating. Semi-insulating (100) GaAs slices were implanted with \(10^{15}\) cm\(^{-2}\) of 150keV Zn\textsuperscript{+} ions. A pyrolytic method was used to deposit the Si\(_3\)N\(_4\) coatings, this causing the slices to be heated to 750°C for \(\sim 8\) s. After annealing, the Al or Si\(_3\)N\(_4\) coatings were removed with warm HF.\(^2\)

TEM plan-view-specimen micrographs for the various types of specimen are shown in Figs.2 to 4. For the uncoated, unannealed specimen the damage consisted of an amorphous layer continuous from the surface to a depth of \(\sim 1600\AA\).\(^3\) The plan-view micrographs showed the damage as a fine, dotty structure (Fig.2a). For the coated but not subsequently annealed specimen, the damage consisted of a mixture of amorphous and single-crystal material and exhibited a coarser structure (Fig.3a).

For the coated thermally-annealed specimens, the behaviour was as follows. For 600°C, the damage occurred in irregular patches and comprised twins, dislocations and loops in single crystal material (Fig.2b). For 900°C,
the damage comprised mainly of loops with some dislocations in single-crystal material (Fig.2c).

For the coated laser-annealed specimens, the behaviour for 0.3 and 1.2 J cm\(^{-2}\) specimens was similar to the corresponding furnace anneal behaviour for the 600 and 900°C specimens respectively. The main difference was that for the laser-annealed 1.2 J cm\(^{-2}\) specimen, the damage was almost completely removed, only extremely small loops remaining.

For the uncoated laser annealed specimens, the results were markedly different. The damage structure immediately prior to laser annealing was similar to that shown in Fig.2a. After laser annealing at an energy density of 0.3 J cm\(^{-2}\) (Fig.4a), the material was single crystal and contained 'grey patches' often associated with dislocations. After laser annealing at an energy density of 1.2 J cm\(^{-2}\), there were local variations from area to area, but two main behaviours occurred. First, the material was single crystal but there were 'dark blobs' on the surface (Fig.4b). These blobs were shown\(^3,4\) to be Ga or Ga-rich droplets arising because of the loss of surface As. Second, the material was polycrystalline (Fig.4c). There is little doubt that in this latter case a molten surface layer formed and resolidified, the mechanism being similar to that described above for Si and illustrated in Fig.1c.

These observations indicate that for laser annealing of GaAs, better results were obtained when a Si\(_3\)N\(_4\) encapsulating surface layer was used.

4. As\(^+\) Implanted Si

p-type, 17 ohm cm, (111) Si slices were implanted with 40keV As\(^+\) ions using a dose of 10\(^{15}\) ions cm\(^{-2}\). This corresponds to a LSS range of 270 ± 120Å. The specimen temperature during implantation was close to RT. TEM cross-section and plan-view examinations showed the damage to consist of a continuous amorphous layer extending from the surface to a depth of 0.1\(\mu\)m.

A portion of this specimen was furnace annealed at 800°C for 20min. TEM examinations showed that the damage consisted of small irregular dislocations
extending to a depth of \( \sim 750 \text{Å} \). Angle-lapping and staining revealed a p-n junction beneath the surface at a depth of 0.10\( \mu \text{m} \). The junction was precisely planar and at constant depth.

Another portion of the specimen was laser annealed using an energy density of 1.5 J cm\(^{-2}\). SEM EBIC micrographs (Fig.5) showed an approximately straight, parallel fringe pattern with a dark-to-dark fringe spacing of \( \sim 250 \mu\text{m} \). Superimposed on this parallel fringe pattern were a number of circular fringe patterns. The region surrounding the laser annealed area in the EBIC image was dark because this material was still amorphous and containing no p-n junction.

The fringe patterns indicate local variations in the minority carrier signal collected at the p-n junction as the beam of the SEM scans across the specimen surface. Dark fringes in the EBIC micrographs correspond to smaller collected signals. The beam energy was 6keV corresponding to a maximum depth of the carrier generation volume of \( \sim 0.6 \mu\text{m} \). The observed signal variations could be due to either (a) local variations in damage structures, greater damage causing enhanced carrier recombination and hence a smaller signal, or (b) local variations in the distance between the specimen surface and the junction, larger distances resulting in enhanced recombination and a smaller signal.

TEM plan-view micrographs showed in general that there was no remaining damage, and consequently there was no correlation between damage and the observed fringes.

Optical microscope examination of the specimen surface using Nomarski interference contrast showed that \( \sim 10\% \) of the annealed area exhibited surface ripples. Multiple-beam interferometry showed that the maximum amplitude of these ripples when present was \( \sim 0.1 \mu\text{m} \).

A particular specimen area was selected which possessed no surface ripples but exhibited EBIC fringes. This specimen was angle-lapped and polished to give a bevel of 0.5°. SEM EBIC micrographs from this specimen area were used to study the surface topography.
(Fig.6a) showed a complex fringe system intersecting the bevelled region. As a result, on going along the edge of the bevelled region (marked S in Fig.6a), the EBIC signal intensity varied in a non-systematic manner. The SEM EBIC micrographs also showed the p-n junction located beneath the original specimen surface as a discontinuous bright line (marked J in Fig.6a) in the bevelled region.

The specimen was given a standard staining treatment which revealed the n-type material on the bevel region (the darker band running across Fig.6b). The edge between the bevelled region and the original specimen surface (S in Fig.6b) is not well defined because of the rounding of the bevel edge when preparing the specimen. However, the junction (marked J in Fig.6b) is sharply defined.

The stained specimen results showed that the junction depth varied in a non-systematic manner from a maximum of 0.23μm to a minimum of 0.17μm. (These junction depths were verified by using the standard two-beam interference examination method, which eliminates any errors arising from the rounding of the bevel edge). The EBIC results for the junction depth showed similar variations, and comparison of identical specimen areas showed that the junction depths determined by the two methods agreed extremely closely. Furthermore, the junction regions which were deeper, e.g. X in Fig.6, corresponded to EBIC dark fringes meeting the bevel edge. The converse occurred for the junction regions which were shallower, e.g. Y in Fig.6. This precise correlation was observed all along the bevelled specimen, covering a length of several mm.

These observations demonstrate that the fringes observed by the EBIC mode for this laser annealed specimen arise not from incompletely annealed damage, but from geometrical effects due to variations in the depth of the resulting p-n junction. The measurements showed that the junction depth varied by as much as 30% on going distances of typically 100μm across the specimen.
It is considered for this specimen that the laser annealing created a molten zone, and that the extent of this zone corresponded to the position of the p-n junction. The depth of this molten zone for this As⁺ implanted Si specimen would then be ~2000Å, close to that described above for the P⁺ implanted Si specimen for the same laser energy density, namely, 1.5 J cm⁻² (Fig.1d). However, in the As⁺ implanted case, the molten zone extends much deeper than the initial amorphous layer, and so no damage structures remain.

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References


Figure Captions

Fig. 1 $10^{15}$ P$^+$ 120keV (111) Si

(a) Unannealed
(b) Furnace anneal 750°C 20 min.
(c) Laser anneal 0.7 J cm$^{-2}$
(d) Laser anneal 1.5 J cm$^{-2}$

Fig. 2 $10^{15}$ Zn$^+$ 150keV (100) GaAs

(a) Unannealed
(b) Furnace anneal 600°C 20 min. (coated)
(c) Furnace anneal 900°C 20 min. (coated)

Fig. 3 $10^{15}$ Zn$^+$ 150keV (100) GaAs

(a) Not laser annealed (coated)
(b) Laser anneal 0.3 J cm$^{-2}$ (coated)
(c) Laser anneal 1.2 J cm$^{-2}$ (coated)

Fig. 4 $10^{15}$ Zn$^+$ 150keV (100) GaAs

(a) Laser anneal 0.3 J cm$^{-2}$ (uncoated)
(b) Laser anneal 1.2 J cm$^{-2}$ (uncoated)
(c) Laser anneal 1.2 J cm$^{-2}$ (uncoated)

Fig. 5 $10^{15}$ As$^+$ 40keV (111) Si, Laser anneal 1.5 J cm$^{-2}$
SEM EBIC composite picture.

Fig. 6 $10^{15}$ As$^+$ 40keV (111) Si, laser anneal 1.5 J cm$^{-2}$

(a) SEM EBIC micrograph
(b) Optical micrograph
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