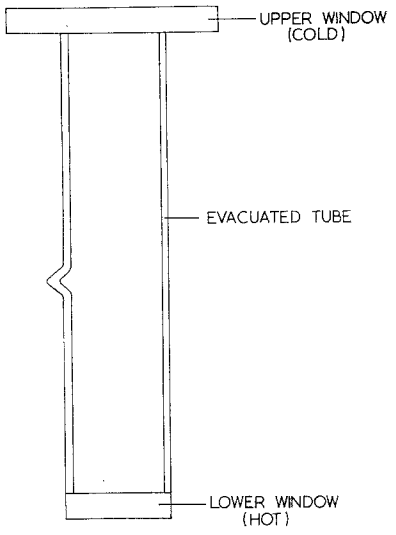

Figure 2 Clear-view window assembly.

sists of a standard silica window, to which a silica tube of smaller diameter has been sealed. The other end of the tube is fitted with a silica window of the same diameter as the tube. The system is then evacuated and sealed off to ensure that at high temperatures there will be no danger of any pressure build-up in the tube. This unit is then fitted into the growth chamber in place of the usual simple window so that the smaller window is near the hot crucible, but is not so near that it interferes with the pulled crystal. The lower window is heated by radiation from the crucible and condensation upon it is thus prevented.

Both window systems have been in general laboratory use for some time, in the growth of PbTe and GaAs, and have proved satisfactory. In general it is possible to use either type of window system, and in these circumstances the window-wiper system is generally better because it has a larger field of vision, is more rugged, and is much easier and cheaper to make. In very corrosive atmospheres the metal components of the system could be replaced by silica components, with some subsequent loss of ruggedness and ease of construction. However, in cases where liquid encapsulation is not used, or there is a possibility of contamination of the melt by material wiped off the window falling back into the melt, the use of the double window assembly is recommended, even though this results in a more restricted field of vision, and the crucible and window positions have to be carefully chosen.

This paper is contributed by permission of the Director, Royal Radar Establishment, and published by permission of the Controller, Her Majesty's Stationery Office.

References

1. J. CZOCHRALSKI, *L Physik Chem* **92** (1917) 219.
2. E. P. A. METZ, R. C. MILLER, and R. MAZELSKY, *J. Appl. Phys.* **33** (1962) 2016.
3. J. B. MULLIN, B. W. STRAUGHAN, and W. S. BRICKELL, *J. Phys. Chem. Solids* **26** (1965) 782.
4. J. B. MULLIN, R. J. HERITAGE, C. H. HOLLIDAY, and B. W. STRAUGHAN, *J. Crystal. Growth.* **4** (1968) 281.

Received 12 October and accepted 24 October 1972

H. C. WEBBER
S. E. R. HISCOCKS
Physics Department,
Royal Radar Establishment,
Great Malvern,
Worcestershire, UK

Luminescence of deformed p-type GaAs

Several papers have been published concerning the influence of crystal defects on the luminescence of GaAs. Casey [1] and also Shaw *et al* [2] used the scanning electron microscope to study the luminescence of n-type crystals doped with Se, Te, and Si. Cathodoluminescence maps were compared with pictures of the etch-pit patterns for the same crystal areas, in an attempt to relate defect structure to the patterns of luminescence. Dislocation etch pits on the etching pictures were found to correspond on the luminescence pattern to a dark spot, usually surrounded by a bright halo of light. Most of the dislocations observed in these studies would have been grown in and it seems possible that the observed effects were due at least in part to the existence of an excess "Cottrell atmosphere" of

dopant in the vicinity of a dislocation. In another investigation, on zinc-doped material [3], various surface preparations were employed on GaAs slices of the same doping, and the photoluminescence signal measured for each one. The samples were then treated with a dislocation etch. It was found that mechanical polishing severely reduced the luminescence, and a correlation was found between number of dislocation etch pits and reduction of signal. This agreed with the finding of the cathodoluminescence work in which scratches showed up as dark lines on the luminescence map.

The work involving mechanical polishing was not unambiguous, however, since it is not at all well established what the metallurgical effect of mechanical polishing is. It was not certain, therefore, that the reduction in luminescence efficiency was due solely to the introduction of dislocations into the surface by the mechanical polishing process. In this note, work is described in which dislocations were introduced into p-type GaAs by the much simpler process of bending. A dislocation etch was used to establish the number of dislocations introduced, and photoluminescence measurements were carried out over a range of dislocation densities.

The samples were zinc-doped and taken from a number of adjacent slices on the same ingot. The slices were cut with {1 1 1} faces. The doping chosen was 7×10^{18} cm^{-3}, since this is close to the optimum doping for photoluminescence [4], and sample dimensions were approximately $25 \times 5 \times 0.5$ mm. A simple technique, previously used by Abrahams and Ekstrom [5] was used to do the bending. A sample was first chemically polished to remove cutting damage and then placed across a pair of electrical contacts, as shown in Fig. 1. A force was applied to the centre

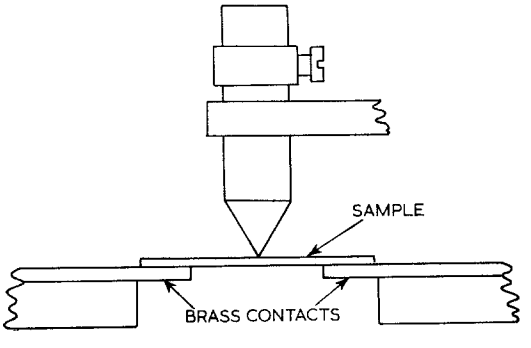

Figure 1 The bending apparatus.

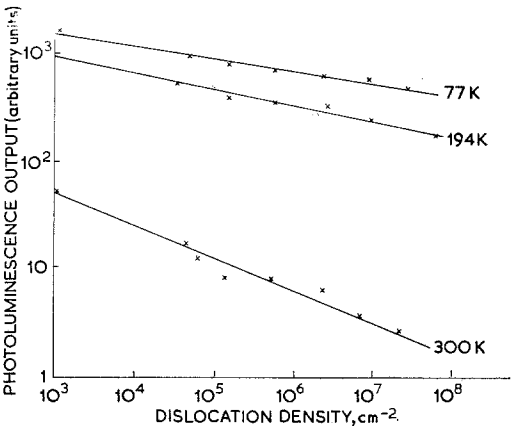

Figure 2 Photoluminescence output against density of dislocation etch pits.

of the sample using a knife-edge arrangement. A current of the order of 10 A was passed through the sample, causing it to glow red and become hot enough to be plastic. The sample then bent under the action of the applied force. The whole process took only about 30 sec. This was quite long enough for a thick oxide film to develop if the bending was carried out in air, however, and it was found necessary to carry out the process in an atmosphere of nitrogen.

Each specimen was then cleaved into a number of smaller pieces, and etched for 3 min in Schell's etch ($2H_2O$:$1HNO_3$) [6]. This etch develops pits on the A {1 1 1} faces of GaAs and a 1:1 correspondence between etch pits and edge dislocations has been established using polygonization experiments [5]. Etch pit densities over an area of a few mm^2 were counted using the optical microscope, and these same areas were tested for photoluminescence signal output. The luminescence was excited by a quartz-iodine lamp, filtered to remove any infra-red radiation (the photoluminescence from GaAs doped with zinc to 7×10^{18} cm^{-3} is approximately 8850Å at room temperature [4]). The experimental technique has been described in some detail in an earlier paper [7]. A masking technique was used to ensure that the same areas were used for etch-pit counting and luminescence measurements, which were taken at 300, 194 and 77 K.

A set of results is shown in Fig. 2 covering the range of dislocation density 10^3 to 5×10^7 cm^{-2}. At low densities, the luminescence at liquid nitrogen temperature is a factor of about 30 above that at room temperature. This compares

quite well with previous work on material with a low dislocation count [4]. The graphs show a clear decrease in luminescence efficiency with increasing dislocation density, with the effect stronger at 300 K than at the lower temperatures. This would seem to confirm the view expressed above concerning the scanning electron microscope pictures. The dark spot at the point of emergence of the dislocation is a real dislocation effect, but the bright halo surrounding it is due to the secondary effect of the doping in the vicinity of a dislocation being different to that in the bulk of the material.

The results at 300 K can be compared to the mechanical polishing work of ref [3]. In Fig. 2 the reduction in luminescence for the range 10^3 to 5×10^7 cm^{-2} is about 30. In the mechanical polishing work, over a similar range of etch-pit densities, the reduction was a factor of about 100. It would appear, therefore, that a major part of the reduction in luminescence in a mechanically-polished specimen is due to dislocations introduced during polishing.

References

1. H. C. CASEY, *J. Electrochem. Soc.* **114** (1967) 153.
2. D. A. SHAW and P. R. THORNTON, *J. Mater. Sci.* **3** (1968) 507.
3. B. TUCK, *Phys. Stat. Sol.* **36** (1969) 285.
4. *Idem, J. Phys. Chem. Solids* **28** (1967) 2161.
5. M. S. ABRAHAMS and J. EKSTROM, "Properties of Electrical and Compound Semiconductors", ed. H. Gatos (Interscience, New York, 1960).
6. M. A. SCHELL, *Z. Metallk.* **48** (1957) 158.
7. B. TUCK, *Phys. Stat. Sol.* **18** (1966) 541.

Received 23 November
and accepted 29 November 1972

B. TUCK
R. M. STURT
Department of Electrical and Electronic Engineering, University of Nottingham, UK

Electron-microscopic evidence of transformation-induced lattice defects in grey tin

Electron-microscopic observation of grey tin (α-phase, diamond structure) has never been reported because of the difficulty of preparing a specimen thin enough for transmission electron microscopy, in contrast with the ease of preparing thin films of white tin (β-phase, tetragonal structure) by vacuum evaporation or electrolytic polishing of massive samples. We tried several preparation techniques for the purpose of electron-microscopic observation of the $\alpha \leftrightarrow \beta$ transformation process, the atomic mechanism of which has not yet been established [1].

Transformation in thin vacuum-evaporated films of white tin to the grey form was not observed, but in foils electrolytically polished following mechanical thinning of pure white tin* the transformation was successfully observed by keeping the white tin in contact with grey tin granules at about $-20°$C as usual [1]. A mixed solution of perchloric acid and acetic acid (1:4) and 10 V d.c. were used for the polishing of the beaten foils of white tin. The thin parts of the polished white tin foils were sufficiently transparent to 100 kV electrons and many dislocations, sometimes piled up, were visible in it, in addition to many extinction contours due to equal thickness and equal bending. In the electron diffraction pattern, however, neither streaks nor extra spots appeared.

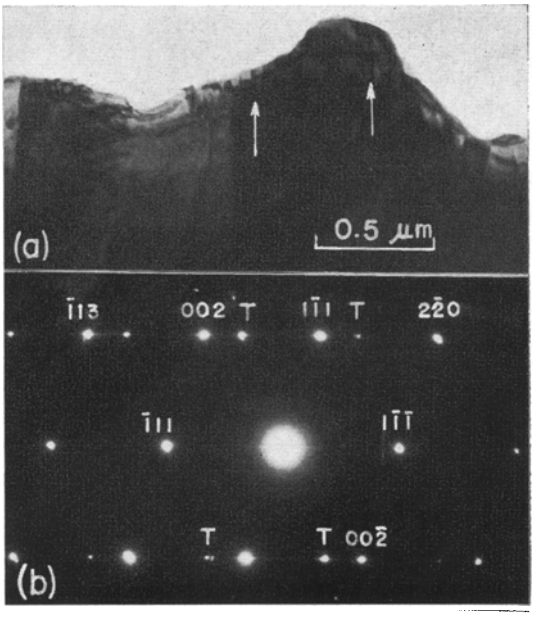

Figure 1 Transmission electron micrograph of grey tin showing lattice defects (a), and the corresponding electron diffraction pattern (b), at 100 kV.

*Zone-refined by Materials Research Corporation, New York.