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References

1. S. M. WIEDERHORN, Proceedings of the International Conference on Corrosion Fatigue, University of Connecticut, 1971 (to be published).
2. M. V. SWAIN, J. S. WILLIAMS, B. R. LAWN, and J. J. H. BEEK, *J. Mater. Sci.* **8** (1973).
3. R. N. STEVENS and R. DUTTON, *Mater. Sci. Eng.* **8** (1971) 220.
4. S. M. WIEDERHORN, N.B.S. Report 10892 (1972).
5. C. F. WILLIAMSON, J. P. BOUJOT, and J. PICARD, Rapport CEA-R 3042 (Paris, 1966).
6. J. J. H. BEEK and B. R. LAWN, *J. Phys. E: Sci. Instrum.* **5** (1972) 710.
7. A. G. MIKOSZA and B. R. LAWN, *J. Appl. Phys.* **42** (1971) 5540.
8. L. T. CHADDERTON, "Radiation Damage in Crystals" (Methuen, London, 1965).
9. P. W. LEVY, Proceedings of Symposium of Nuclear Science and Technology for Ceramicists, Washington, 1966.
10. D. I. R. NORRIS, *Rad. Effects* **14** (1972) 1.
11. T. BELL, G. HETHERINGTON, and K. H. JACK, *Phys. Chem. Glasses* **3** (1962) 141.
12. H. G. NELSON, D. P. WILLIAMS, and A. S. TETELMAN, *Met. Trans.* **2** (1971) 953.

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A contribution to etch polishing of GaAs

When etch polishing GaAs to produce mirror-like surfaces, it is obvious that the removal rate is dependent upon the pressure applied to the sample. Under certain conditions, etch pits can be produced which are caused by dislocations. Both phenomena affect the fabrication processes of GaAs devices. So far, a systematic study of etch polishing with sodium hypochlorite has not yet been published. We have, therefore, undertaken this task, particularly with regard to those parameters where no systematic data are yet available.

Etch polishing has been undertaken with such etchants as a weak bromine-methanol solution, and of 20:1 H_2O:NaOCl [1]. Owing to the chemical inertness of the latter etchant on the associated apparatus, it has often been employed. However, when metallization is to be applied to the surfaces, etch polished with NaOCl, a thin contamination layer has to be removed first.

The material used for these experiments was (1 1 1) orientated chromium-doped GaAs. Slices of this material were attached, using dental wax, to the stainless steel polishing block of the etch polishing apparatus shown in Fig. 1. The pressure exerted on the sample during etch polishing was varied by placing weights on the hollow machined-out polishing block. The etchant consisted of a 1N solution of sodium hypochlorite (low in bromine) in a 0.1N solution of sodium hydroxide and was diluted to the required strength using distilled water. Fresh solutions were used each time because it was found that the solution strength (etch rate) decreased with increasing time between solution preparation and use. After each period of polishing, the samples were thoroughly cleaned in distilled water to ensure that no further etching took place.

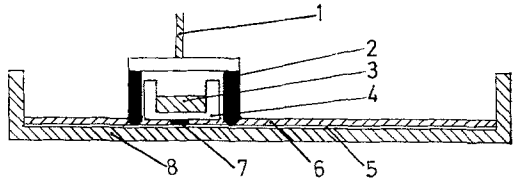


Figure 1 Etch polishing apparatus. 1. Polishing arm. 2. Stainless steel holder. 3. Additional weights. 4. Polishing block. 5. Polishing cloth. 6. Etchant. 7. Sample. 8. Perspex bowl.

Pressure dependence of etch rate. Experiments to determine the pressure dependent etch rates were carried out by varying the pressure applied to the sample and measuring the resultant etch rates, the rate of rotation of the polishing pad being the same each time; these results are shown in Fig. 2.

According to Rideout [1], the primary process here is the chemical etching of the GaAs, the mechanical motion being used mainly to distribute the etchant uniformly over the surface of the sample; but there is also a contribution made by the mechanical polishing effects. These

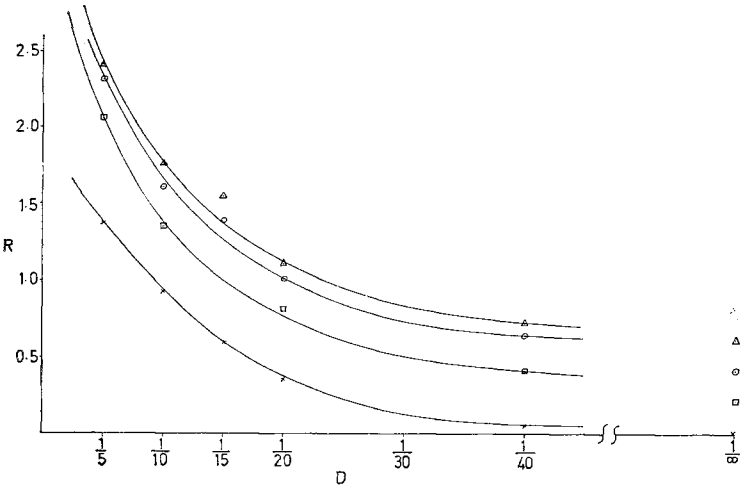
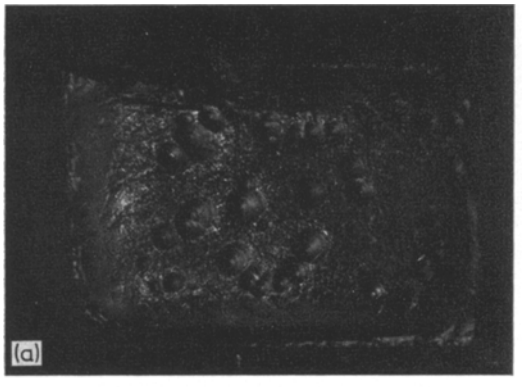
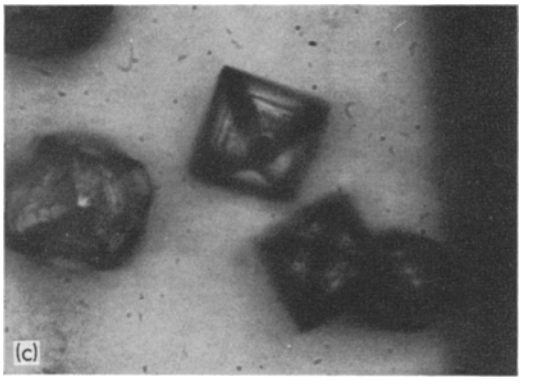


Figure 2 Graph of variation of etch rate (R) with etchant dilution for various values of applied pressure. R is etch rate in μm min^{-1}. D is volume NaOCl/volume H_2O. △ is for pressure = 12.87 kg cm^{-2}. ○ is for pressure = 8.75 kg cm^{-2}. □ is for pressure = 2.77 kg cm^{-2}. × is for pressure = 0.0.





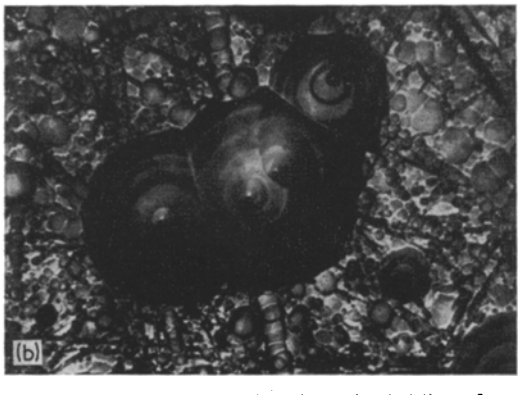


Figure 3 (a) Etch pits produced on the (111) surface of GaAs by the sodium hypochlorite etchant (× 60). (b) As (a) but (× 200). (c) Distorted etch pits produced by nitric acid on the (111) surface of GaAs in the presence of some wax.

details are verified by the above results. The line $p = 0$ in Fig. 2 represents the results for pure etching, when the rotating sample was suspended in a beaker filled with the etchant where no polishing pad was present. The removal rate of GaAs as given by Fig. 2 for $p = 0$ takes place only for about 1 min. Then the removal ceases, obviously owing to the formation of a layer which is insoluble by the etchants (see [2] for experimental evidence). It is, therefore, important to employ also some polishing action simultaneously in order to continuously remove any such layer formed.

The point at infinite dilution ratio of Fig. 2 represents the contribution of mechanical polishing. Because these latter results were strongly dependent on the condition of the surface of the

polishing pad, they were taken when a new pad was used.

The errors incurred in the calculation of these etch rates were ± 0.1 μm min^{-1} for all points except those on the line $p = 0$ which were ± 0.5 μm min^{-1}. This higher value of error range is caused by the formation of the contamination layer, so that the etch rate had to be calculated over shorter time periods.

Good mirror-like surfaces such as those required for the nitric acid dislocation etch [3], were obtained with weak etchants, the weaker the etchant, the better the surface finish.

Dislocation etch pits. On etch polishing with low pressures, of the order of 1 kg cm^{-2} and strong etchants of dilution ratio 1/10 or stronger, the surface was found to exhibit conical etch pits, see Fig. 3a and b. If the etchant strength was decreased to a dilution ratio of 1/40, these etch pits could be polished away leaving a good mirror-like surface.

To show that these etch pits correspond to dislocation etch pits, a sample was mounted on the polishing block and polished to produce these conical etch pits. The etch pit configuration was recorded and the sample was polished to give a flat mirror-like surface. The sample was then detached from the polishing block, cleaned and etched in a nitric acid solution [3], to reveal the location of dislocations. These two etch pit patterns were compared and found to have 1:1 correspondence, showing that the etch pits produced by the sodium hypochlorite solution are also dislocation etch pits.

It was found that if any wax, which was used to attach the samples to the polishing block, was present during the nitric acid etch, then deformed etch pits, as shown in Fig. 3c resulted.

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References

1. V. L. RIDEOUT, *J. Electrochem. Soc.* **119** (1972) 1778.
2. B. SCHWARTZ, *ibid* **118** (1971) 657.
3. H. C. GATOS (ed) "Properties of elemental and compound Semiconductors" (Interscience, New York, 1960) p. 226.

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