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ION-BEAM MILLING OF SILICON CARBIDE EPITAXIAL LAYERS.(U)
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Ion milling of photolithographically processed silicon carbide hetero-epitaxially grown layers on Si is feasible using an aluminum mask which is produced by standard photolithographic procedures and techniques.

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Final Report

July 1st, 1981 to June 30th, 1982

# ION-BEAM MILLING OF SILICON CARBIDE EPITAXIAL LAYERS

Contract No. N00014-81-C-0758

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## ABSTRACT

Funds provided under this proposal were intended for the purchase and assembly of apparatus for performing reactive ion etching experiments principally on epitaxially grown SiC layers deposited on Si substrates and for the apparatus to be used for electrical and electronic measurement on SiC and other large fundamental bandgap compound semiconductors. During the period of this contract a manifold gas distribution panel for the introduction of halocarbon gases into an ion sputter etching chamber was designed, built and tested. In colaboration with Dr. L.D. Flesner of the Naval Ocean Systems Center, San Diego, ion milling of SiC layers using $A\ell$ as mask for lithographic processing was demonstrated to be feasible. No impediments are expected in using electron beam lithographic procedures for submicron device processing.

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## I INTRODUCTION

Ion etching is a process by which a substrate surface is eroded by bombardment with a stream of high energy ions [1]. The erosion takes place by the transfer of momentum between the incident ions and the substrate atoms with the latter achieving sufficient momentum to escape from the surface into a gaseous environment, provided that the lattice binding energy of the atoms is overcome.

Major advantages of ion beam etching compared to liquid phase etching of semiconductors is the higher resolution obtainable, with the former in some cases using simpler processing procedures. All etching procedures require control of the relative etch rates of the mask and substrate material, particularly when preferential directional etching is desired which often leads to increased etching rates of the mask. Two types of ion etching are usually employed: r.f. sputter etching in which the specimen is placed directly on the cathode of a parallel plate ion discharge system, and ion beam milling in which the ion beam is generated in a plasma source removed from the specimen itself.

Apparatus for ion beam etching [2] usually consists of an ion gun (source), a neutralizer and a substrate stage. The gun generates ions in a spatially confined plasma discharge and accelerates them in the form of a beam towards the sample. The neutralizer is usually a hot filament which emits a flux of electrons which is intended to keep the specimen electrically neutral, and the sample stage provides tilt control, rotation and water cooling for the specimen. In this way, ion acceleration, flux and angle of incidence can be controlled independently. The system is usually placed within a vacuum chamber in which argon or reactive gases are introduced and maintained to a pressure of $10^{-2}$ to $10^{-4}$ Torr.

An r.f. sputter etching system consists of a water cooled cathode on which specimens are mounted and to which r.f. power is coupled capacitively; an ion discharge is produced between anode and cathode. A dark space across which the ions are accelerated develops adjacent to the cathode, and in view of the different mobilities of the ions and electrons in the discharge the cathode charges the coupling capacitor to the peak value of the r.f. input voltage producing, in this manner, a d.c. bias at the cathode.

The sputtering field is dependent on the atomic weight of the incident ions and etching rates have also been found to be markedly dependent on the addition of small quantities of reactive gas to the inert gas supply. This is defined as reactive ion etching.

Sputter etching has been used since 1967 in device and integrated circuit lithographic procedures for delineating fine patterns, however, reactive sputter etching has only been in use since 1974. The chemically reactive gases employed for reactive sputter etching attack the specimen at a rapid rate while the ion beam assures directional bombardment from the plasma and, therefore, directional etching of the specimen surface, provided that the gases used are not overly reactive.

The addition of a chemically active gas to the ion plasma changes the etching rates due to a chemical interaction between the substrate and the added gas [3]. Most of the work thus far has been directed towards the addition of oxygen and halocarbons to both ion beam and sputter etching processes. Oxygen reduces the sputtering yield of metals such as titanium, chromium and aluminum which oxydize readily, but it has relatively little effect on inert metals such as gold or platinum. Argon/oxygen gas mixtures are generally used to etch deep patterns into an inactive material using an active metal such as chromium as the etching masks; the chromium mask is developed photolithographically; it is etched by means of pure argon ion etching, using an appropriate photoresistance mask.

The use of halocarbon plasmas to increase sputtering yield is being investigated intensively. Hosokawa et. al. [4] has investigated the etch rates of various metals in pure oxygen, $CC\ell_2F_2$ (halocarbon 12) and $CC\ell_2FCC\ell F_2$ (halocarbon 113) and found a considerable increase in the etching rates of Si and $A\ell$ as shown in Table 1 below:

Table I

Total Pressure, $2 \times 10^{-2}$ Torr and Power Density, 1.3 $W/cm^2$

| | Etch Rate (Å/min) | | |
|---|---|---|---|
| | Argon | Halocarbon 12 | Halocarbon 113 |
| Si | 138 | 2200 | 2015 |
| $SiO_2$ | 159 | 533 | 470 |
| No. 7059 glass | 103 | 347 | 144 |
| $A\ell$ | 166 | 1624 | 637 |
| Mo | 185 | 836 | 775 |
| Photoresist (modified KMER) | 185 | 410 | 608 |
| Stainless steel | 154 | 522 | 222 |

The semiconducting compound silicon carbide, grown in the form of epitaxial layers onto single crystal silicon substrates, has material properties which are likely to be of considerable significance for high-speed and high-power transistor and integrated circuit applications. Among these are its large fundamental bandgap, its high-thermal conductivity and its high-electron velocity. Quantitative measurements on the electric field dependence of the electron velocity of silicon carbide have not been made as yet. No reliable method for making ohmic contacts to this material have been developed as yet.

Little is known about its surface properties, metal-semiconductor and dielectric-semiconductor interfaces. No reliable techniques for device fabrication and construction of suitable test structures for electrical measurements have been developed as yet.

The intent of the investigations performed jointly at UCSD and at the Naval Ocean Systems Center was to determine whether conventional ion milling, ion sputter etching and reactive ion etching on silicon carbide layers grown heteroepitaxially on Si substrates is feasible. Apparatus for reactive etching was to be assembled at UCSD and in preparation for performing electrical transport measurements on SiC, apparatus was assembled for this purpose. Assuming the availability of SiC layers being made available from other sources and a technique developed for applying ohmic contacts to them then plans might proceed for the measurement of the electron velocity dependence on electric field in silicon carbide.

## II APPARATUS FOR ION BEAM AND REACTIVE ION BEAM ETCHING

In order to perform ion beam etching experiments on silicon carbide layers grown epitaxially on silicon substrates an ion beam sputtering system available at UCSD was modified to include a gas mixing manifold system for the controlled introduction into the reaction chamber of Ar, $O_2$ and $CC\ell_2F_2$.

Funds provided under this contract were used to design, build and test the gas mixing manifold system shown schematically in Figure 1. Stainless steel tubing was used for the interconnections between the standard, commercially available components. The system was leak checked and the gas flow meters were calibrated. Preliminary indications are that, in conjunction with the ion beam system available adjusted to a base pressure of $10^{-2}$ Torr, it performs in accordance with expectations. It has not been used, as yet, for reactive ion beam etching of SiC.

## III ION BEAM MILLING OF EPITAXIALLY GROWN SiC ON Si SUBSTRATES

Two epitaxially grown SiC layers were obtained from Professor Robert F. Davis of North Carolina State University and were used for the subsequently described ion milling experiments which were performed at NOSC using a Kaufman type [5] ion beam source.

Figure 2 shows the surface morphology of such a layer as received from NCU. The granularity of the surface is evident. It was feared that this might result in preferential sputtering from the surface; however, this turned out to be a negligible problem. The procedure selected for attempting the reproduction of patterns in SiC was to use a mask of evaporated aluminum because $A\ell$ can be processed by means of standard photolithographic and lift-off procedures and because in the presence of oxygen the sputter etching rate of $A\ell$ is expected to be about an order of magnitude smaller than that of SiC.

Figure 3 shows an $A\ell$ bar deposited on the SiC in the form of a thin layer by vacuum evaporation and then processed photolithographically. The scanning electron beam micrograph looks somewhat different from that of Figure 2 because the viewing angle is 60°; however, the texture of the $A\ell$ deposit replicates that of the SiC layer shown in Figure 2.

Figure 4 shows the mesa of SiC with $A\ell$ still present on top. The region on the left side of the micrograph has had complete removal of SiC from the substrate, while the region on the right still has SiC remaining. The interface between the SiC and the $A\ell$ mask is only faintly perceptible because of a thin layer of back-sputtered material which has deposited on the side of the mesa. The viewing angle is 60°. The ion milled surface apparently replicates the morphology of the surface of the SiC layer. The nature of the Si-SiC interface is, as yet, undetermined.

Figure 5 shows the SiC mesa on Si after removal of the Aℓ etch-mask. Again, the viewing angle is 60°. The back-sputtered material forms a thin ridge around the edge of the mesa that actually projects above the top. The thickness of this ridge is roughly 0.1 μm.

The resolution achievable with ion milling techniques of silicon carbide thus seems to be satisfactory, at this stage. No inherent difficulties are likely to be encountered in the production of high resolution patterns employing electron beam lithography and subtractive patterning.

It will, of course, be highly desirable to evaluate the relative advantages of reactive sputtering versus ion beam milling in processing patterns on epitaxially grown SiC layers. This is contingent on the availability of other specimens from North Carolina State University.

## IV RECOMMENDATIONS FOR FUTURE WORK

A serious problem which remains to be overcome before making electrical measurements or galvanomagnetic measurements on such specimens is the finite conductivity of the Si substrates which produces an electrical shunt across the SiC layers. Oxidation of the Si substrates is a possibility although this might introduce problems because of the high temperatures required, reduction in thickness of the Si substrates and the difference in the thermal expansion coefficient of $SiO_2$ and SiC. It might be desirable, therefore, to attempt the synthesis and growth of Si on $SiO_2$ as a first step and thereafter grow SiC such a thin transition layer. Since Si on sapphire substrates can be grown by standard organometallic vapor phase transport procedures, perhaps SiC might grown sequentially upon such a structure. Furthermore, ohmic contacts to n-type SiC can only be made with considerable difficulty. Such contacts have beeen reported,[6] heating tungsten discs in contact with SiC to a temperature of 1900°C; this forces a metalurgical bond without any apparent intervening alloy. There is an urgent need for developing a low temperature alloying process, ion implantation or diffusion of donors into SiC in order to make reliable ohmic contacts and proceed with the electrical evaluation of this material.

REFERENCES


1. For a bibliography of ion beam etching 1954 - 1975 see D.T. Hawkins, J. Vac. Sci. Technol. 12, 1389 (1975).

2. J.A. Bondur, J. Vac. Sci. Technol. 13, 1023 (1976).

3. C.M. Horwitz and J. Melngailis, J. Vac. Sci. Technol. 19, 1408 (1981).

4. N. Hosokawa, R. Matsuraki and T. Asmaki, Jpn. J. Appl. Phys. Suppl 2, 1 (1974).

5. H.R. Kaufman, Adv. Electron. Electron Phys. 36, 265 (1974).

6. H.S. Berman, T.M. Heng, H.C. Nathanson and R.B. Campbell in Silicon Carbide 1973, edited by R.C. Marshall, J.W. Faust Jr. and C.E. Ryan, University of South Carolina Press.

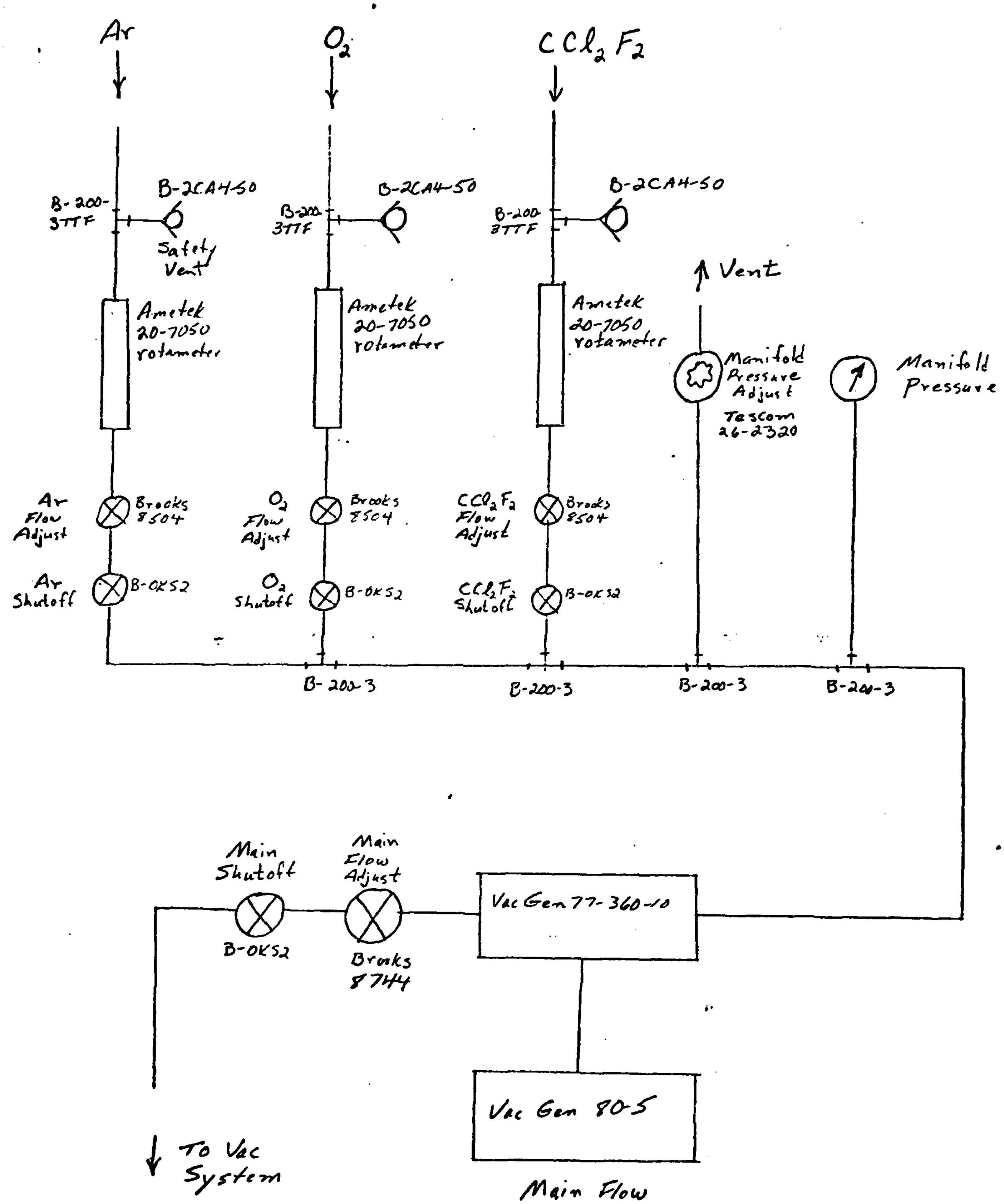


**Figure 1**

**Gas Mixing Manifold for Ion Beam Reactive Etching**


Figure 2 Morphology of SiC surface; Perpendicular view of virgin surface; Bar denotes 1 μm scale

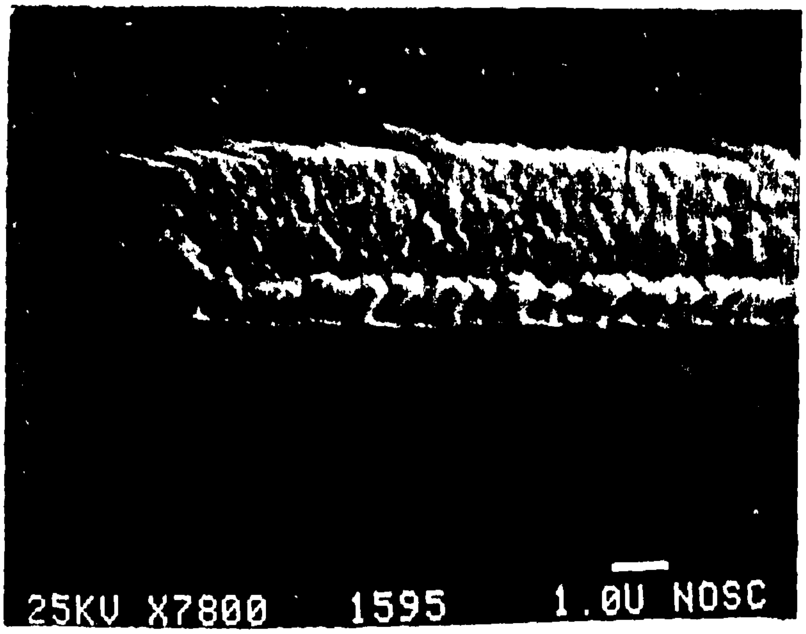


Figure 3 Photolithographically processed thin layer of $A\ell$; Used as mask on SiC epilayer surface for ion milling; Viewing angle is 60°

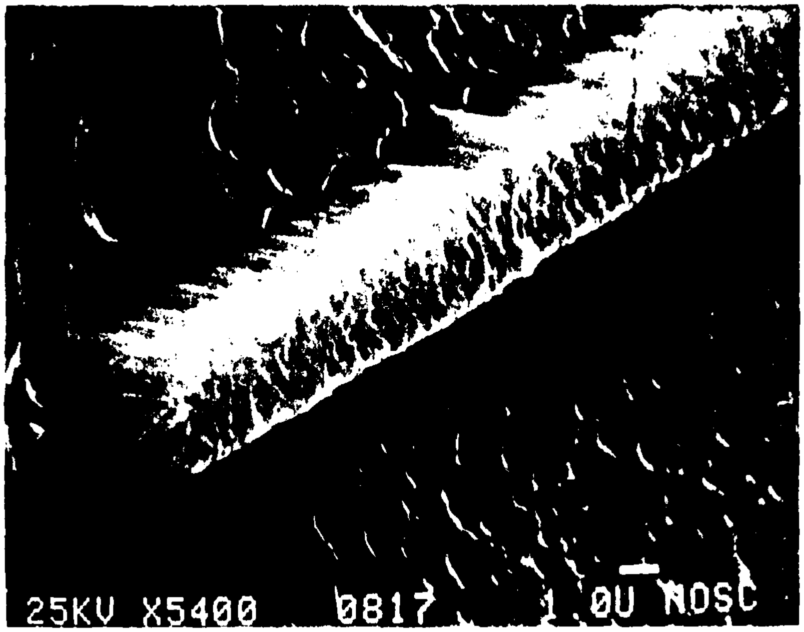


Figure 4 SiC mesa after ion milling with $A\ell$ mask still present on top. Upper left region has SiC completely removed while lower right has some remaining SiC. Viewing angle is 60°.

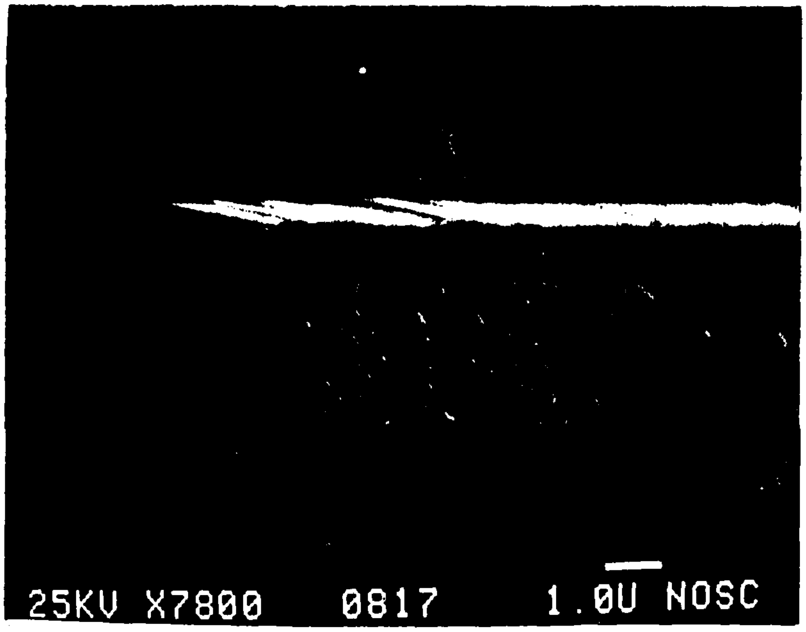


Figure 5 SiC epilayer mesa after removal of the Aℓ mask by etching in NaOH; Viewing angle is 60°