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THERMAL ETCHING OF SILICON CARBIDE CRYSTALS IN A PLASMA TORCH

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Thermal treatment of silicon carbide crystals in an inert atmosphere of a plasma torch, in the high temperature range 2000-3000° was carried out. Evidence is provided that such a treatment can reveal dislocations. The treatment does not leave any reaction products and the surface of the crystal is left clean and shiny.

INTRODUCTION

Silicon carbide is one of the few wide band gap semiconductors. It is a non-conductor when pure and has a resistivity of the order of $10^3 - 10^5$ Ohm Cm. The onset of intrinsic conductivity begins at 500°. The Hall mobility of silicon carbide varies with temperature. The maximum value of 10 cm^2/V. sec. at 200°, for *n*-type falls to nearly zero at 90°K.

The usefulness of silicon carbide as non-linear resistors, varistors, electrical heating elements, cold cathodes in electron guns has been realised fairly recently. Grown p-n function are used as a detector of phonons or nuclear properties.

A great deal of effort is being devoted to the fabrication of silicon carbide which is free of structural irregularities, viz., the point defects and dislocations.

Etching is a widely employed technique for investigations such as the study of the structure of dislocations and determination of orientations of crystals. The etching method for revealing dislocations is based on the assumption that there is a one to one correspondence between the pits formed by etching and the emergence of dislocation lines on the surface.

Etching of the crystalline solids is carried out using one of the methods, viz., chemical, electrolytic; cathodic bombardment, and thermal treatment. In the chemical etching method, the material is reacted with a molten salt, at its melting point, in a platinum or gold crucible. In electrolytic method etching is carried by dissolution by the flow of current in a suitable electrolyte. This method is not suitable for certain covalent materials, such as metal carbides, ceramic oxides and non-conductors. Cathodic bombardment etching is not commonly employed as it may damage certain surfaces under study.

The extent to which dislocations, in the crystalline matter can alter both physical and chemical properties, cannot be overlooked. The density of dislocations in silicon carbide (type 11 or 6H) crystals is generally very low (1-100 cm^2) but it may be as large as 10^4 cm^2.

In view of the potential uses of silicon carbide for various purposes, especially in the fabrication of semi-conductor and charged particle detectors, there is a growing interest in the study of dislocations in this material.

Faust Jr [1] has reviewed the various etching processes employed for silicon carbide crystals. The chemical etching technique has been widely used by several investigators. Patel [2] carried out thermal etching of silicon carbide crystals in a carbon arc at an unspecified temperature. Svaitskii *et al* [3] combined the chemical and thermal etching techniques to produce well defined etch pits. During our investigations of the effects of high temperature plasma torch annealing on the mechanical properties of silicon carbide, in the temperature range from 2000 to 3000°, we found that the thermal treatment in the inert atmosphere of the plasma torch constitutes an etch for SiC.

EXPERIMENTAL

Silicon carbide (type II) crystals, measuring approximately 0.5 x 0.5 x 0.1 cm, were heated in the plasma torch, in the temperature range 2000-3000°, for fixed time in the range 5 to 10 min. The torch consisted essentially of a 8 MHz RF generator, with a coil in its tank, run at a low power. A quartz tube, having an inlet for argon, was concentric with the coil. The gas was introduced into the tube at a very low velocity at atmospheric pressure. The hot zone of the flame was estimated to be approximately 0.1 metre. The temperatre varied vertically from 1500 to 3500° from near the tip of the flame to its centre. A disappearing filament pyro-meter was used to estimate the temperature at any point in the flame. The crystal for etching was placed in a cavity in a carbon rod and lowered into the flame. The heat treatment was ended by cutting off the input to the RF generator. The etched crystal was examined by an electron microscope using carbon replica.

RESULTS AND DISCUSSION

The principal result of the etching experiments are summerised here. Fig. 1 shows a characteristic etch pattern on the (0001) faces. Large pits, with a step at the edge, correspond to the screw dislocations and small pits are believed to be at the sites of edge dislocations. The etch pits seen in Fig. 2 are interlaced spirals. It is possible that individual growth spirals collapsed during the thermal treatment, thereby forming the interlaced spirals.

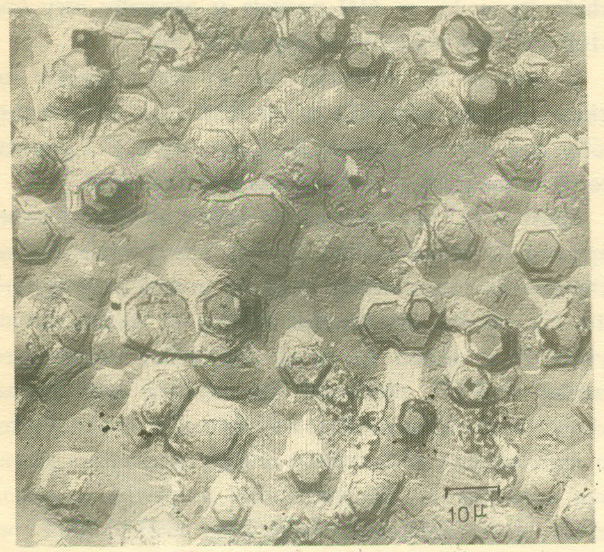

Fig. 1. Etch pits on (0001) plane. Crystal etched at 2500°, for 10 minutes.

No reaction products were left behind on the surface of the crystal because they would be volatile at high temperatures. It is well known that crystallographic etch pits are formed under the conditions of low surface energy and

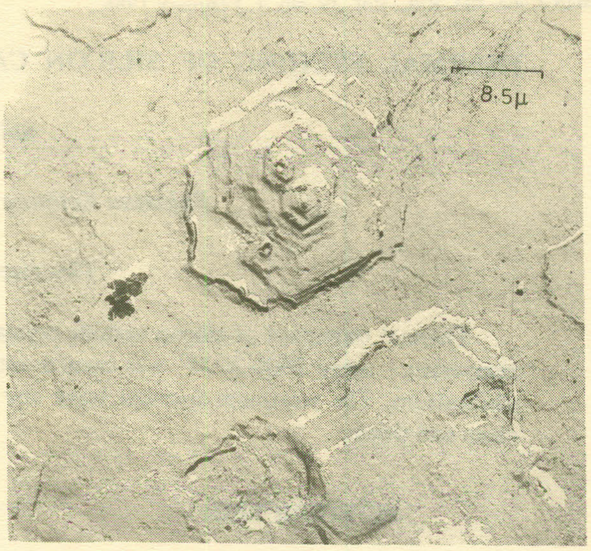

Fig. 2. Coalescence of two spirals.

low undersaturated vapour pressure. The vapour pressure of the system SiC+SiC/, in argon at normal pressure in the temperature range 2000-3000°, has been estimated by Knippenberg [4] to be of the order of 10^{-2} torr. The etching is done by silicon vapours at this low pressure when a silicon carbide crystal is heated in a plasma torch.

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