SOURCES OF MORPHOLOGICAL DEFECTS IN SiC EPILAYERS

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Transactions Third International High-Temperature Electronics Conference
Volume 1, pp. II-3 - II-8
Sandia National Laboratories, Albuquerque, NM USA
9-14 June 1996

Submission Date: 12 April 1996

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Abstract

A variety of conditions lead to the formation of morphological defects observed in SiC epilayers grown on SiC substrates. In addition to the well-known micropipe defect, SiC epilayers contain growth pits, triangular features (primarily in 4H-SiC), and macro steps due to step bunching. In epilayers grown at NASA Lewis, we have determined that factors contributing to the formation of some morphological defects include: defects in the substrate bulk, defects in the substrate surface caused by cutting and polishing the wafer, the tilt angle of the wafer surface relative to the basal plane, and growth conditions. Some of these findings confirm results of other research groups. Surprisingly, the dominant factor in the formation of growth pits appears to be the polishing of the wafer. This paper presents and discusses recent results of a continuing investigation into the sources of observed morphological defects.

INTRODUCTION

Silicon carbide (SiC) semiconductor technology is currently being developed world wide for high temperature and high power applications (Neudeck, 1995). Among the challenges facing the development of this technology is the need for improved structural quality and surface morphology of epitaxial films used in device fabrication. Typical 4H-SiC and 6H-SiC epilayers contain unwanted structural features such as line dislocations and micropipes; it is known that these defects in SiC wafers propagate into the epilayers (Powell et al., 1994). The epilayers also include unwanted surface morphological features such as growth pits and macro steps, up to 30 nm in height, caused by step bunching (Powell et al., 1995a). In addition, 4H-SiC epilayers exhibit a triangular feature not normally found in 6H-SiC (Powell et al., 1995b; Burk, 1996). This paper will focus on the sources of growth pits and triangular features.

The micropipe defect is sometimes called a “killer defect” because it leads to premature reverse breakdown in SiC pn junctions (Neudeck and Powell, 1994). Thus, the presence of this defect precludes the fabrication of high voltage devices. Bhatnagar et al.(1996) reported that degraded performance of Schottky contacts on epitaxial SiC was probably caused by localized defects at the SiC/metal interface. The nature of the defects was not determined. The purpose of this current investigation is to determine the cause(s) of morphological defects that may hinder device performance.

Commercial 4H-SiC and 6H-SiC wafers are available in diameters up to 35 mm with a polished growth surface that is tilted “off-axis” relative to the (0001) basal plane. Typically, a tilt angle of 3.5° has been used to ensure that step-flow growth is achieved in epitaxial growth. Step-flow growth, the lateral growth of atomic-scale steps on the growth surface, enables the homoepitaxial growth of α-SiC polytypes on SiC substrates. Recent development efforts for high temperature and high power applications have shifted emphasis to 4H-SiC because of its higher mobility and lower nitrogen donor activation energy. Typical wafers of both 4H and 6H polytypes contain micropipes (approximately 100 cm^{-2}) and dislocations (approximately 10^4 cm^{-2}).

The growth pits (sometimes called amphitheaters) are small micrometer-sized depressions in the epilayer. They are thought to be caused by interruptions in the desired step flow growth. Some reduction in the density of these growth pits has been achieved by altering the epitaxial growth process. Powell et al.(1995a) found that reducing the exposure of the substrate to pure hydrogen prior to growth reduced the density of growth pits. In their preferred growth procedure, the time of the pregrowth hydrogen chloride (HCl) etch was extended up to within seconds of the epilayer growth. Burk (1996) found that an over pressure of propane and hydrogen chloride prior to growth reduced the formation of "Si droplets" and growth pits.

Recent reported results on the epitaxial growth of 4H-SiC have shown that 4H has a much greater tendency than 6H for having 3C-SiC inclusions in the epilayer in the form of large (tens of micrometers) triangular features. Powell et al.(1995b) also found that this tendency of 4H to contain 3C inclusions became worse at smaller tilt angles. Tsvetkov et al. (1996) reported that using 4H wafers with a tilt angle of 8°effectively eliminated the 3C inclusions.

EXPERIMENTAL

SiC wafers used in this work were produced by Cree Research, Inc.(Cree, 1996) from sublimation-grown boules. The 30-mm-diameter wafers used in this work had been sliced off-axis from the (0001) basal plane by 3.5° and 8° in the <1120> direction and the Si face of the wafers had been polished to form the growth surface. The 4H wafers had tilt angles of both 3.5° and 8°, whereas the 6H wafers had tilt angles of only 3.5°. The wafers were cut into half-wafer pieces or into 7.5x6 mm^2 pieces to reduce substrate cost of growth experiments.

The epilayer growth experiments were carried out in a CVD system that has been described previously (Powell et al., 1995a). The system utilized a horizontal water-cooled quartz growth chamber with an inside diameter of 50 mm. The substrates were heated by an inductively-heated SiC-coated graphite susceptor. Growth was carried out at atmospheric pressure with hydrogen as the carrier gas, and silane (3% in H_2) and propane (3% in H_2) as the sources of silicon and carbon, respectively. Before loading into the CVD growth chamber, substrates were sequentially cleaned with organic solvents, hot H_2SO_4, scrubbed with liquid detergent, rinsed with deionized water, and then blown dry with nitrogen. The H_2 carrier flow was maintained at 3 liters/min throughout etching and growth. Prior to growth, the substrates were etched in 3%HCl/H_2 at 1375±25 °C for approximately 4 min; then growth was carried out at 1500 °C. Typically, the silane concentration in the chamber during growth was held at about 200 ppm and the propane concentration was varied to produce various Si/C ratios in the range 0.1 to 0.8. In other runs, both the silane and propane were varied. Epilayer growth rate was about 3 μm/h.

A series of epi growth runs were carried out with the purpose of distinguishing which morphological defects were caused by bulk defects in the substrates such as dislocations, micropipes, low-angle grain boundaries, etc. in the original boule crystal and which were caused by surface defects that had been generated by processes involved in cutting, polishing, and preparing the wafer for growth. The following approach was used. First, a typical epilayer (designated as epilayer #1) was grown on some selected commercial 4H and 6H boule-grown substrates (all 3.5° off-axis, Si-face substrates). The epilayer surfaces were characterized with Nomarski differential interference contrast optical microscopy. Second, epilayer #1 was then polished off to below the original growth surface and another epilayer (#2) was grown on the same samples. Epilayer #2 was also characterized by Nomarski optical microscopy. Specially placed laser-etched markers on the backside of the transparent Cree samples enabled Nomarski photographs to be taken of identical locations on epilayers #1 and #2. The purpose of this procedure was to compare the morphology of these two epilayers. We would expect that (a) features common to both epilayers would be caused by defects that are present in the original boule crystal, and (b) features that were common to only one of the epilayers would be caused by defects at the surface of the wafer (features caused by processes related to preparing the wafer surface for growth).

Other growth runs were carried out (1) to confirm the effect of large tilt angle (8° vs 3.5°) on the morphology of 4H epilayers, and (2) to further investigate the effect of changes in silane and propane concentration on growth pit density. Transmission electron microscopy (TEM) was used to study structural defects and to confirm the presence of 3C-SiC in the triangular -shaped features..

RESULTS

The results of the repolishing/regrowth experiments are shown in Figures 1 and 2. Nomarski photographs of epilayers #1 and #2 for each of three samples are shown in these figures. Photographs of the same locations on the 4H and 6H boule-derived samples are shown. The "dot-like" features seen in these photographs are the growth pits that we have observed in all of our SiC epilayers. The results can be summarized as follows:
(a) In the 4H epilayers (Fig. 1), the growth pit density is much less in epilayer #2 (Fig. 1b). Also, new triangular features appear epilayer #2, apparently the result of scratches that were produced in the repolishing. There does not appear to be any correlation between any features seen in these two 4H epilayers.
(b) In the 6H epilayers (Fig. 2), there are two groupings of growth pits: large and small. There does not appear to be any correlation between the large pits seen in the two 6H epilayers. Also, the density of large pits is much smaller in epilayer #2; whereas the density of the small pits is about the same. For convenience of comparison, we have put boxes around three groupings of small pits. These small pits are present in both epilayers #1 and #2.

The immediate first conclusion from Figs. 1 and 2 is that all features in the 4H epilayers and the large growth pits in the 6H epilayers are caused by surface defects; that is, from the polishing. The second conclusion is that many of the small pits the 6H epilayer are caused by bulk defects in the substrate.

Epilayers grown on 4H substrates with tilt angles of 3.5° and 8° in the <u>same</u> growth run are shown in Fig. 3. As can be seen, the triangular features are almost entirely absent in the epilayer grown on the 8°-tilt substrate. In general, the epilayer morphology of the 8° 4H samples was much better than the 3.5° 4H samples.

The result of a series of three growth runs with different silane and propane concentrations, but constant Si/C ratio, is shown in Fig. 4. In these runs, the Si/C ratio was constant at 0.19. The silane concentration for the three runs was Fig. 4a (run#2120): 170 ppm, Fig. 4b (run#2121): 200 ppm, and Fig. 4c (run#2119): 220 ppm. Note that the first run in the sequence was Fig. 4c, then Fig. 4a, and then Fig. 4b. This demonstrates that increasing the concentration of the precursors increases the probability of the formation of growth pits.

DISCUSSION

The results of the repolishing/regrowth experiments were somewhat of a surprise to us. We expected that bulk defects would be the dominant cause of morphological defects. However, the fact the large growth pits in epilayer #1 did not correlate with the pits in epilayer #2 means that surface defects rather than bulk defects are the cause of the larger growth pits. Also, the procedure of removing epilayers, repolishing the surface, regrowing epilayers, and making before and after comparisons is a very useful in tracking the source of epilayer defects. In the past, comparing epilayers grown on different samples was frustrating because of the variability of the results. The reason, in retrospect, is that poor polishing procedures can be an important contributing factor in the formation of defects.

The growth results with the 3.5° and 8° 4H samples do confirm that the triangular features (3C-SiC inclusions) can be largely eliminated by growing on 4H substrates with large tilt angles. It remains to be seen if there will be a price to be paid for using large tilt angles. For example, an electrical anisotropy resulting from large tilt angle could be a negative factor in the operation of some devices. One can speculate that perhaps such large tilt angles will not be necessary when the 4H substrates are polished and prepared in a manner that does not leave surface defects which can cause 3C inclusions.

The epilayer results shown in Fig. 4, using high silicon concentrations, demonstrate that growth conditions can also be a factor in the formation of growth pits. This is important because, in the application of site-competition epitaxy (Larkin, 1995) to dope SiC epilayers, a large range of Si/C ratios (and perhaps large concentrations of silane) may be desired.

CONCLUSIONS

A surprising conclusion of this study is that the dominant factor in the cause of growth pits in our SiC epilayers are surface defects generated in the polishing and preparation of substrates rather than bulk defects such as micropipes and dislocations. The results also demonstrate that at least some morphological defects are caused by bulk defects. Although we confirmed that using 4H-SiC substrates with larger tilt angles eliminates 3C-SiC inclusions, we speculate that adequate polishing procedures on samples with smaller tilt angles may be just as effective. Finally, we also demonstrated that growth conditions can also be a factor in the formation of growth pits.

Acknowledgements

The work in this paper was largely carried out at NASA Lewis under internal funding. Some funding support was received from ARPA (AO No. D149). Structural characterization and repolishing of substrates was carried out at CWRU supported under NASA Grant No. NAG3-1702. We thank Andy Trunek and Luann Keys for performing the epilayer growth.

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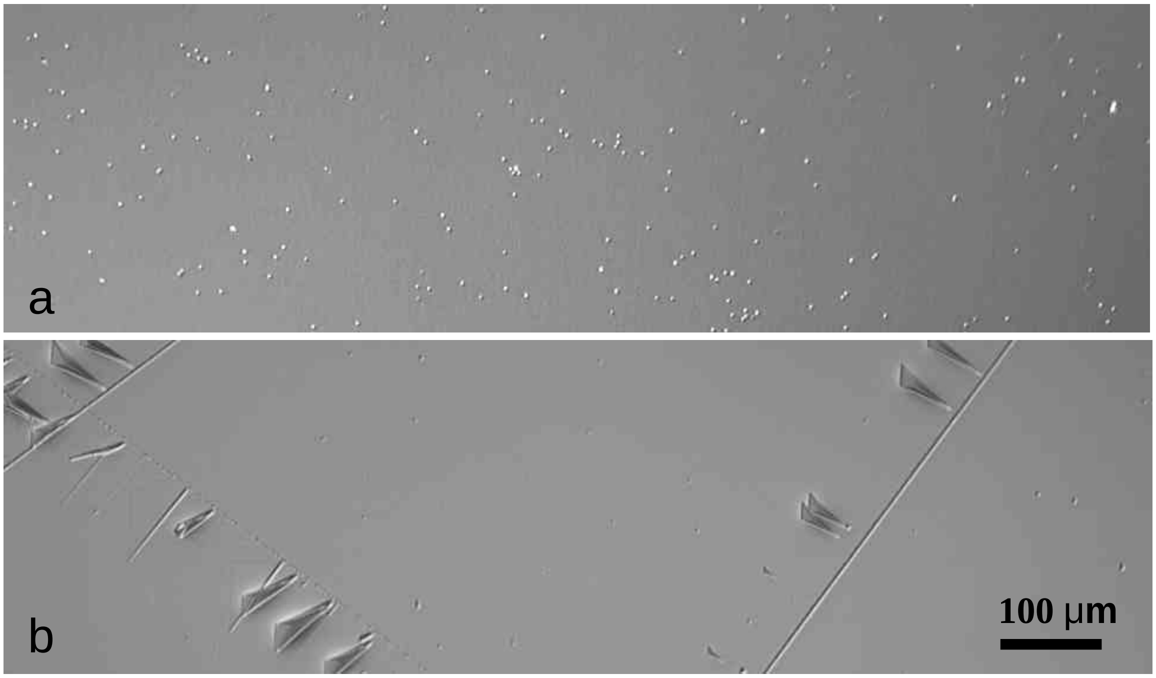

Fig. 1. Nomarski photographs of the same area of a 4H-SiC sample with two different epilayers. (a) first epilayer, (b) second epilayer, after first was removed. Note triangular features growing from scratches.

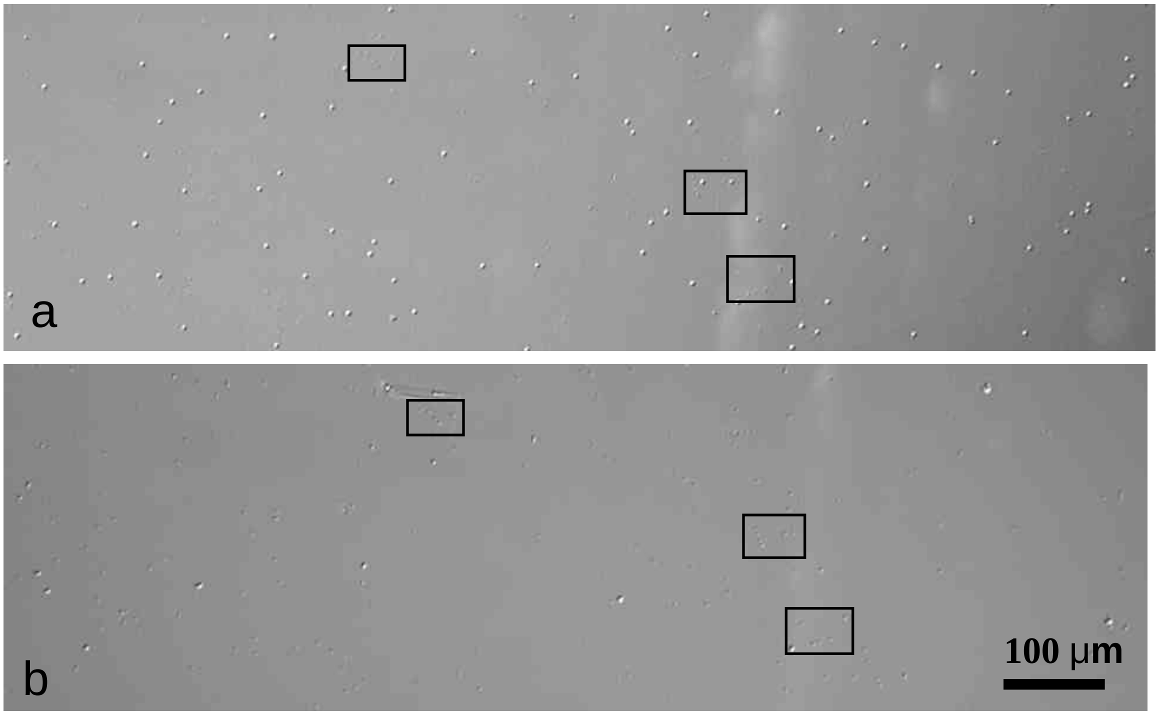

Fig. 2. Nomarski photographs of the same area of a 6H-SiC sample with two different epilayers. (a) first epilayer, (b) second epilayer, after first was removed. Boxes indicate growth pits common to both epilayers.

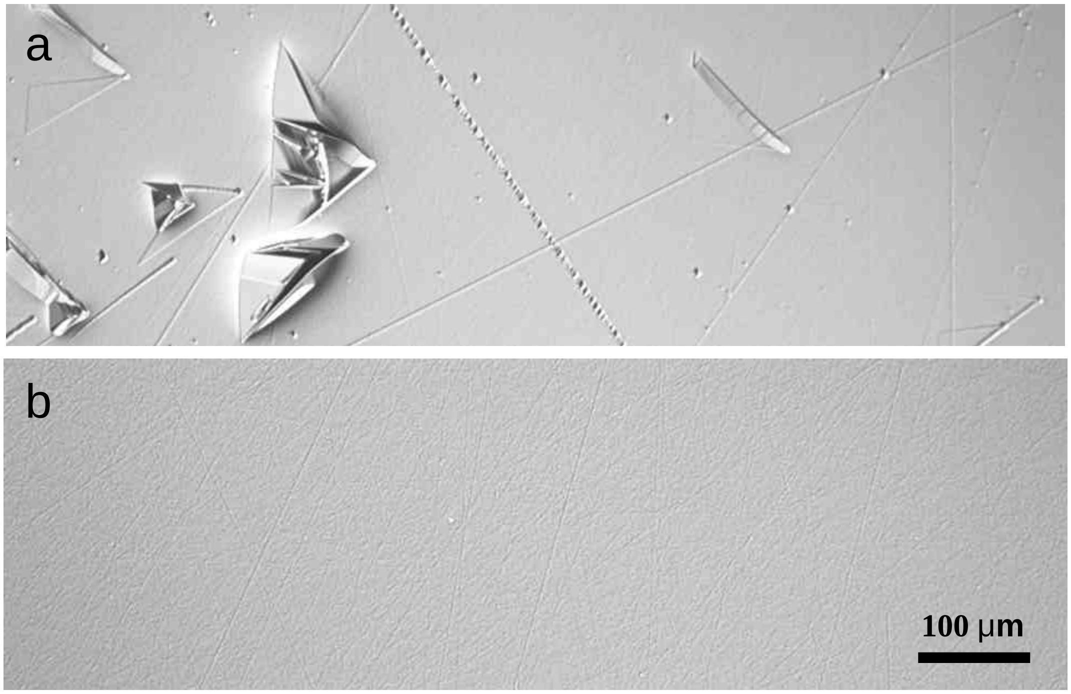

Fig. 3. Nomarski photographs of 12-mm-thick 4H-SiC epilayers grown on 4H-SiC substrates with tilt angles of (a) 3.5° and (b) 8°.

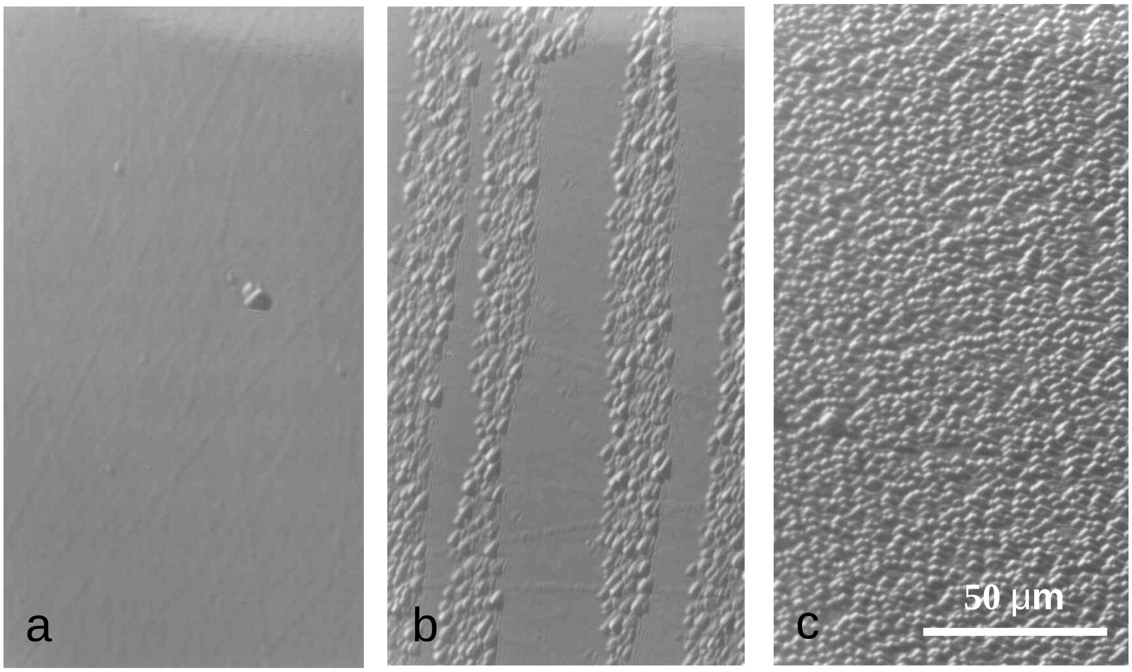

Fig. 4. Nomarski photographs of 6H-SiC epilayers grown with Si/C ratio = 0.19, but with different silane and propane concentrations. (a) [Si] = 170 ppm, (b) [Si] = 200 ppm, (c) [Si] = 220 ppm.