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# Nucleation of Dislocations during Physical Vapor Transport Growth of Silicon Carbide

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**Abstract**: Two possible nucleation mechanisms for threading edge and screw dislocations during the physical vapor transport growth of SiC have been investigated. First, growth over intentionally deposited carbon inclusions led to an edge and screw dislocation density orders of magnitude higher than the surrounding crystal. Second, seeds with mechanical polishing damage have been shown to lead to a dislocation density nearly three orders of magnitude higher than seeds that were hydrogen etched. A new linear step source has been observed and correlated with an increase in the dislocation density.

## Introduction

It has recently been shown that single Burgers vector screw dislocations aligned along the [0001] direction limit silicon carbide (SiC) device performance [1]. Even though they may not be as detrimental to a device as super screw dislocations (micropipes) [2], their densities are much higher, on the order of $10^4/\mathrm{cm}^2$ [3,4]. Their formation mechanism is still not fully understood. Considering the magnitude of the Burgers vector (1.5 nm in 6H-SiC), it is unlikely that they form by plastic deformation [5]. It is more probable that screw dislocations are a grown-in defect. Several authors have suggested that the seed surface quality can be a factor in the generation of defects [6,7]. Super screw dislocations have been observed to start at second phase inclusions (carbon or silicon) [3,8]. Dudley *et al.* [6] have taken this observation further and proposed a mechanism in which second phase inclusions act as the nucleation sites for screw dislocations. A second factor is surface polishing; Powell *et al.* [7] have shown that surface polishing in chemical vapor deposition of SiC can lead to growth pits. This paper will focus on the nucleation of screw dislocations on second phase inclusions and mechanically polished surfaces during physical vapor transport (PVT) growth of SiC.

## Experimental

All experiments used basal plane (0001) 6H SiC Lely platelets as seeds. A wet oxidation treatment (1100 ± 50° C, for a half hour in $H_2O$ saturated $O_2$) was used to determine the surface polarity. Two sets of experiments were carried out. In the first set, only seeds with virgin surfaces were used. However, the virgin surfaces contained carbon deposits ranging from 10 to 500 μm in size. In addition, carbon deposits were intentionally produced by placing small spots of melted sucrose on the seed surface and annealing at high temperature. The second set of experiments used seeds with different surface finishes. All seeds were first polished with 0.5 μm diamond paste. One was left as polished and used as a control; a second was polished using 30 μm diamond paste to introduce severe mechanical damage. The last two seeds underwent a hydrogen etch for 15 minutes under a flow of hydrogen and nitrogen at 1600 °C [9]. In each run, two seeds (a hydrogen etched seed and a mechanically polished seed) were attached to the same lid and grown on simultaneously.

Seed surfaces were cleaned using a standard RCA cleaning and mounted to the crucible lid using the carbonized sucrose method [10]. Growth experiments were carried out in a physical vapor transport system similar to those described in literature [11]. The source material used was grade B-hp from H.C. Starck. Prior to growth, the system was evacuated to a pressure below $3x10^{-7}$ torr. It was then heated in stages to about 1200 °C at a pressure below $1 \times 10^{-4}$ torr. The system was then backfilled with ultra high purity argon to a pressure of 650 torr and the temperature was gradually increased to the growth temperature of 2300 °C, at a rate of 24°C/min. The seeds with carbon deposits were grown on at 650 torr for time periods ranging from 5 to 30 minutes. For the polished seeds, the system pressure was slowly decreased to 10 torr and held for 1 hour. Contact mode atomic force microscopy (AFM) was carried out using standard $Si_3N_4$ probes and contact forces in the 0.1 to 5 nN range. Scanning electron microscopy (SEM) and energy dispersive x-ray (EDX) images were obtained on a Philips XL-30 microscope under an accelerating voltage of 25 kV. To reveal dislocation etch pits, the crystals were etched in molten KOH at 500°C for 3-8 minutes.

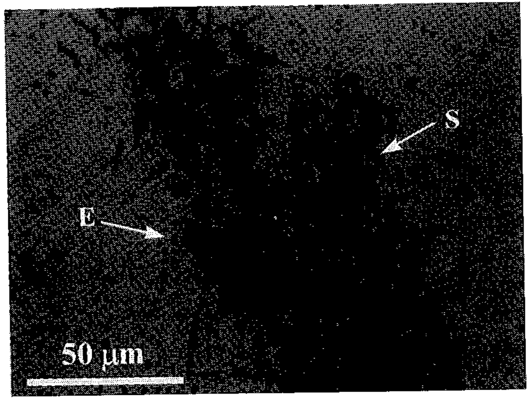

Fig 1. Optical image of a KOH etched surface. Dislocations are revealed on top of carbon precipitates. E is an edge dislocation, S is a screw dislocation.

**Results**

The first set of experiments was conducted to test the effect of surface deposits on the nucleation of dislocations. After growth, AFM was carried out on the seeds followed by etching in molten KOH. AFM scans revealed a screw dislocation density of $2x10^5/cm^2$ in areas above carbon inclusions. Fig. 1 shows an optical image, after etching, of the areas directly above a carbon deposit. Two types of etch pits are distinguishable: the small round etch pits labeled with E, and the lager faceted pits labeled with S. Smaller pits are most likely due to threading edge dislocations and the larger pits are most likely screw dislocations lined along the [0001] direction [12]. The density of larger pits above the carbon inclusions is $8x10^5/cm^2$; this agrees well with screw dislocation density observed by AFM. In contrast, the density of screw dislocations in the rest of the crystal is only $10^4/cm^2$, nearly two orders of magnitude less then above the precipitate. The threading edge dislocations had a density of $10^7/cm^2$ above the precipitate and a density of $5x10^5/cm^2$ in the rest of the crystal. This correlation indicates that surface deposits play a role in the generation of dislocations in SiC. One possible mechanism to explain the formation of dislocations above a precipitate has been suggested by Dudley *et al* [6]. When surface carbon deposits are overgrown, misorientations are created as growth fronts from opposite sides of the inclusion meet. Another possible mechanism involves

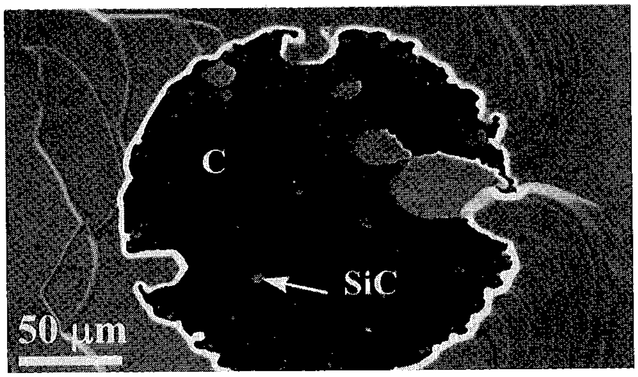

Fig 2. SEM micrograph of a carbon precipitate (C). SiC marks the location of one of the many SiC growth islands.

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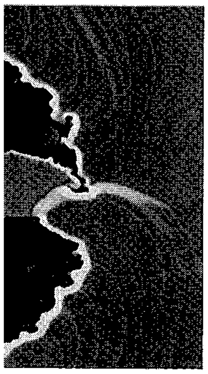

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the nucleation of SiC islands on a second phase deposit. Individual islands are expected to be misoriented with respect to one another and, when they coalesce, this misorientation can be accommodated by dislocations. A key step in the process is the nucleation of SiC on carbon precipitates. The initial stages of the overgrowth of carbon deposits were investigated with SEM. Fig. 2 is an SEM image of a carbon deposit on a SiC seed after a 5 minute growth at 2300 °C, 10 °C/cm, and 650 torr. EDX indicates that the small islands (labeled SiC) have the same silicon and carbon concentration as the rest of the crystal. AFM on the surface of an island revealed that the surfaces are atomically flat. These observations suggest that nucleation of SiC islands has occurred on top of a carbon deposit.

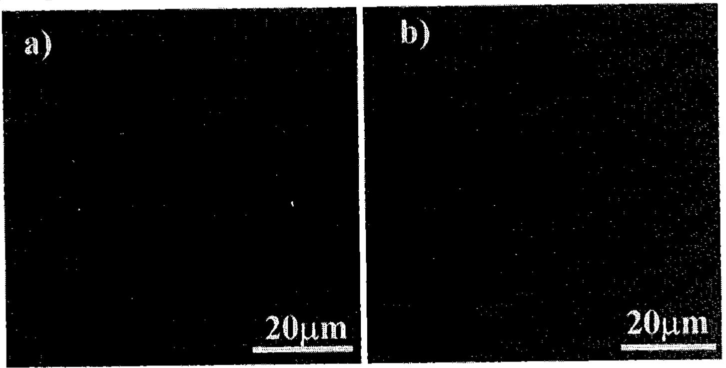

Fig 3. AFM images of a KOH etched (a) crystal grown on a hydrogen etched seed, and (b) a crystal grown on an industry polished seed.

The second set of experiments was conducted to test the effect of surface finish. Following growth, the seeds were characterized by AFM to determine the screw dislocation density and were then KOH etched to reveal both screws and threading edge dislocations. SiC was grown simultaneously on a seed polished with 30 μm diamond grit and on a hydrogen etched seed, mounted side by side. The polished seed had a screw dislocation density of $10^6/cm^2$ while the hydrogen etched sample had a screw dislocation density of less then $10^3/cm^2$. The results for the hydrogen etched and fine grit polished samples are shown in Fig. 3a and 3b, respectively. In Fig. 3a, two etch pits can be seen at the top of the image. The total etch pit density on this crystal was about $10^4/cm^2$. Fig. 3b shows a line of etch pits, along the left side of the image. This is interpreted as dislocations arranged in a greater density along a scratch left behind by polishing. The density of total etch pits on this crystal is 3.5 x $10^7/cm^2$, three orders of magnitude higher then the hydrogen etched sample. This provides evidence that polishing leaves behind damage in the crystal that leads to dislocation nucleation during PVT growth. It further suggests that hydrogen etching helps to remove this damage.

An AFM image of a SiC growth surface is shown in Fig. 4. This image was taken after etching in KOH for 5 minutes at 500 °C. The image reveals a linear step source, 30 μm long, aligned along the $<11\bar{2}0>$ direction. Several screw dislocations (etch pits where steps end) and several threading edge dislocations (isolated etch pits) are revealed in close proximity to the linear step source. These linear step sources were observed on all surfaces formed by short growth treatments. All of the observed lines have a Burgers vector in the [0001] direction, making them step sources. Taking a Burgers circuit around the one in Fig. 4, reveals a height difference of 3 nm or two 6H lattice cells. These linear step sources are 3 to 30 μm in length and have been seen to rise above the surface as linear hills to heights of 7 nm or fall below the surface as narrow trenches to

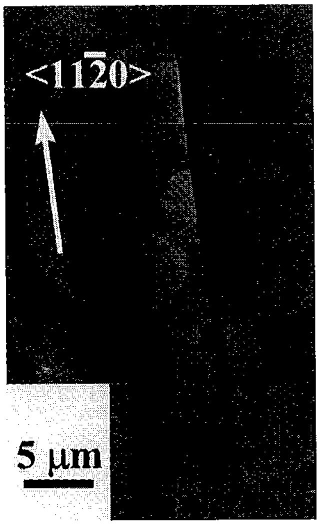

Fig 4. AFM image of a KOH etched linear step source.

depths of 3 nm. Laue x-ray revealed that they are all orientated along $<11\bar{2}0>$ directions. It is interesting that there is a direct correlation between these lines and both threading edge and screw dislocations. Examining several of these sources reveals that both edge dislocations and screw dislocations decrease in density as you move radially away from the linear step source. Edge dislocations decrease from $10^6/cm^2$, within 10 μm of the line sources, to $10^4/cm^2$ at 80 μm from the line. Screw dislocations decrease from $5x10^5/cm^2$, near the lines, to less than $10^3/cm^2$ at 40 μm away from the linear sources. This suggests that there is a mechanistic link between the formation of these linear step sources and dislocations. Further work is in progress to determine the exact nature of this step source. However, we speculate that this could be a stacking fault along one of the pyramidal planes, similar to those described by Heindl *et al.* [13]. Since they all align along $<11\bar{2}0>$ directions, it is most likely a stacking fault on either the $\{10\bar{1}1\}$ or $\{10\bar{1}2\}$ pyramidal planes.

## Conclusions

The seed surface quality before growth has a direct relation to the dislocation density in the PVT grown material. Second phase precipitates on the seed growth surface lead to the nucleation of both screw dislocations and threading edge dislocations. This formation appears to tie into the nucleation of SiC islands on top of second phase material. There is also a direct relationship between the surface polish and dislocation density. Hydrogen etching has been shown to remove this damage and in turn lower the dislocation density. When the surface of a seed is cleaned and the damage removed, the major step source is related to a newly discovered defect. Long narrow defects aligned along the $<11\bar{2}0>$ directions act as step sources and are correlated to elevated populations of both threading edge and screw dislocations.

## Acknowledgments

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