Lateral Growth Expansion of 4H/6H-SiC m-plane Pseudo Fiber Crystals by Hot Wall CVD Epitaxy

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Abstract. Lateral expansion of small mixed polytype 4H/6H-SiC slivers were realized by hot wall chemical vapor deposition (HWCVD). Small slivers cut from m-oriented $(1\bar{1}00)$ SiC boule slices containing regions of 4H and 6H SiC were exposed to HWCVD conditions using standard silane/propane chemstry for a period of up to eight hours. The slivers exhibited approximately 1500 μm (1.5 mm) of total lateral expansion. Initial analysis by synchrotron white beam x-ray topography (SWBXT) confirms, that the lateral growth was homoepitaxial, matching the polytype of the respective underlying region of the seed sliver.

Introduction

Reducing the number of dislocations in SiC substrates is essential for further commercialization of SiC power elctronics. Unfortunately, due to the polytypic nature of SiC, dislocations in the seed crystal are essential for maintaining polytype and commercially viable growth rates [1]. There have been some studies of growing SiC in the non-polar $[1\bar{1}00]$ and $[11\bar{2}0]$ directions as a method to reduce dislocations [1,2]. Toyota explored the possiblility of reducing screw dislocations (SD) by developing the physical vapor transport repeated a-face (RAF) growth process [1]. However, these processes are either very difficult or resulted in increased stacking fault formation [2]. The NASA Glenn Research Center has recently proposed a new bulk growth process for producing SiC boules that has the potential for dramatically reducing the number of SD's per wafer, ideally as few as one [3]. Unlike the RAF process, this approach relies on chemical vapor deposition for the lateral homoepitaxial expansion of a SiC fiber without introducing a significant number of additional defects [2, 3]. This paper reports on the lateral growth enlargement of 4H/6H-SiC m-plane slivers up to millimeter diameter crystals via lateral growth in a HWCVD system.

Experimental

Currently, a source of single crystal SiC fibers does not exist, therefore slivers (0.8 mm x 0.5 mm x 15 mm), with a crystal orientation as diagrammed in Fig. 1, were diced from polished m-oriented $(1\bar{1}00)$ SiC boule slices obtained from a purchased source. The m-oriented SiC boule slice contained a thick region of 6H-SiC sandwiched between two 4H-SiC regions containing numerous micropipes. The slivers act as pseudo fibers, herein referred to as fibers. This work reports on the results from a five (Fiber 1) and eight (Fiber 2) hour long growth runs. The dicing results in a fiber with two polished m-plane faces and two saw cut a-plane faces. Prior to growth, fibers were immersed in a 1:1 solution of H_2SO_4 and H_2O_2 for 15 minutes then blown dry with N_2. Fibers were immediately transferred to an argon filled glove box. All growth experiments

proceeded within fours of cleaning. Growth runs were accomplished in a custom designed, inductively heated, horizontal flow hot wall reactor. The tantalum carbide coated tubular susceptor has a 40mm inside diameter and a length of 140mm. A carbon foam insulation package was used to reduce heat loss and improve temperature uniformity. The fibers were supported in the reactor by an uncoated graphite carrier, which also facilitated in the loading and unloading of the fibers, Fig. 2. Approximately, two millimeters of the fibers overall length were used for mounting and do not participate in growth. With the exception of the mounted portion of the fiber, the rest of the fiber is uniformly exposed to growth conditions. The carrier gas was hydrogen (H_2) with standard carbon (C_3H_8) and silicon (SiH_4) precursors used for growth. HCl was added to the gas stream to improve pregrowth etching performance and reduce gas phase nucleation during growth. A twelve minute *in-situ* pregrowth etch was performed on the fibers before transitioning directly to growth. Aggressive etching conditions were employed to remove crystalline damage caused by the saw cutting operation. Growth parameters for both fibers are given in Table 1.

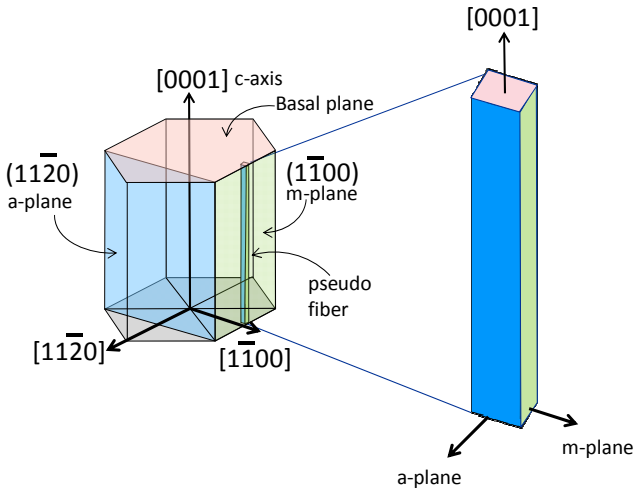

Fig. 1: Schematic diagram showing the SiC unit cell with associated crystallographic planes and the orientation of the seed slivers (pseudo fibers) as cut from m-plane polished boule slices.

Growth Parameters for five (Fiber 1) and eight (Fiber 2) hour growth runs

Growth	*In-situ* etch [min]	Etch Pressure [mb]	Growth [min]	Growth Pressure [mb]	Hydrogen [sccm]	Silane[1] [sccm]	Propane[1] [sccm]	HCl[1] [sccm]	Estimated Temperature[2] [°C]
Fiber 1 (five hour)	12	40	300	325	4260	0/4	1.5/1.5	15/20	1600
Fiber 2 (eight hour)	12	40	480	325	4260	0/8	1.5/2.25	15/40	1590

1. First number is for etching conditions, second number for growth conditions.
2. No direct observation of temperature by pyrometry was possible. An inferred temperature was calculated based upon the melting points of Si and Pd.

Table 1. Growth parameters for two seperate growth runs. Fiber 1 was five hours in length. Fiber 2 was eight hours in length. Note that the intentional Si/C is 2.7 and 3.5 respectively. There is a substantial and unkown quantity of carbon contribution coming from the uncoated graphite sample carrier and carbon foam insulation.

Results and Discussion

In the first growth, a fiber from a m-oriented SiC boule slice was exposed to five hours of growth. Since the fiber is mounted approximately vertically in the gas stream, all surfaces (excluding the 2mm portion used for mounting) are exposed to growth conditions, Fig. 2. The post-growth fiber (Fig. 3) has begun to evolve towards hexagonal crystal symmetry [4]. It is interesting to note that, growth progressed on the a-plane side of the pseudo fiber, lead by nucleation at the intersection of the m and a–planes. A trough is formed along the a-plane side of the fiber as the growth in the middle trails the corners by a significant amount. Examination of the m-plane face shows that a very small depression exits but is much less than on the a-plane surface. Even though

the m-plane surface is ~30% wider than the a-plane surface, the slower growth rate in the $< 1\bar{1}00 >$ direction allows the facet time to fill in. The overall growth rate is ~80 μm/hr in the $< 11\bar{2}0 >$ directions. As expected, the growth rate in the $< 1\bar{1}00 >$ directions is ~60 μm/hr or 75% of the growth rate in the $< 11\bar{2}0 >$ directions. Synchrotron white beam x-ray topography (SWBXT) confirms the growth is homoepitaxial, matching the polytype of the respective underlying region of the fiber.

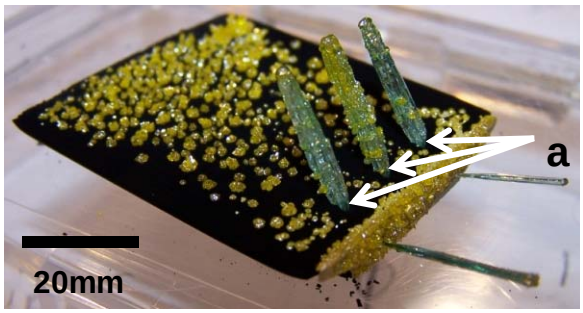

Fig. 2: Post growth results for the eight hour growth showing three m-plane fibers mounted on top of an uncoated graphite carrier (a). Two other control fibers protude from the front of the carrier. Yellow 3C-SiC decorates the surface of the graphite.

A second 6H-SiC m-plane polished fiber (Fiber 2 in Table 1) was exposed to eight hours of growth at higher super-saturation conditions. Similar to fiber 1, this fiber also has begun to evolve towards hexagonal crystal symmetry, Fig. 4. However, the greatest lateral expansion occurred in the $< 1\bar{1}00 >$ directions with approximately 1500 μm (1.5 mm) of total lateral expansion. Similar to the five hour growth fiber, a deep trough has developed along the a-plane side of the fiber. A much shallower depression is present on the m-plane face of the fiber. The crystal has several locations where 3C-SiC has nucleated and negatively impacted the growth morphology. It is proposed that carbon particulates coming from the uncoated graphite carrier contributed to the formation of the 3C-SiC. The total overall growth rate is ~180 μm/hr in the $< 1\bar{1}00 >$ directions. However, the approximate overall growth rate is 30 μm/hr in the $< 11\bar{2}0 >$ directions. SWBXT confirms the growth was homoepitaxial, matching the polytype of the respective underlying region of the seed sliver. The facets are of high crystalline quality and are mostly free of strain.

Under the higher super-saturation conditions used for fiber 2, the crystal is expanding at the highest rate along the $< 1\bar{1}00 >$ direction. The fast growth direction is normally understood to be along the $< 11\bar{2}0 >$ direction. It appears that the evolving crystal shape for these higher super-saturation conditions indicates that the highest nucleation probability is occurring at the edges/corners formed by intersections of the m-planes, Fig. 4. This is not unexpected; as the NASA Glenn Research Center reported similar growth behavior obtained on hexagonal shaped mesas whose sidewalls share the same crystallographic orientation as the sides of the fibers [5].

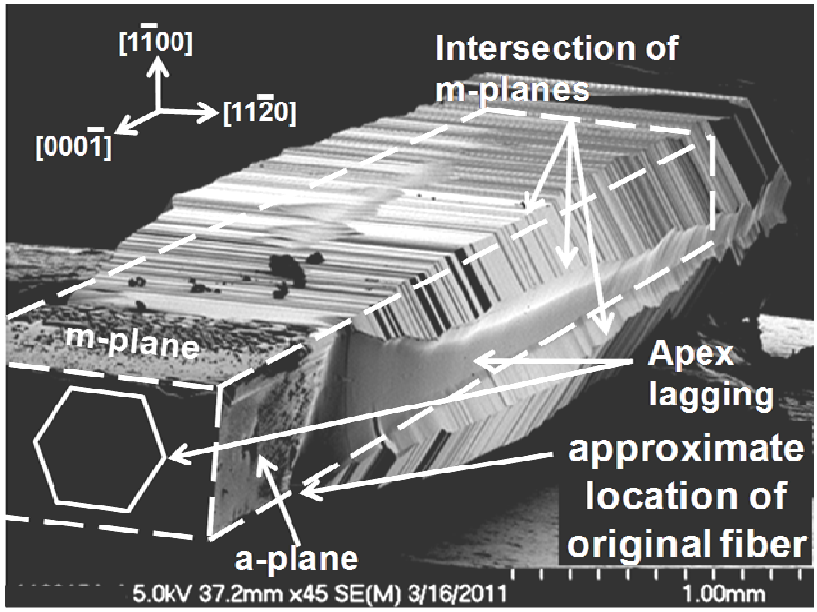

Fig. 3: SEM of m-plane fiber 1 after five hours of growth. The white dashed lines indicate the aproximate shape and location of the original fiber. The hexagonal unit cell orientation has been superimposed on the end of the fiber. Growth at the intersection of the m -planes dominates. The left side of the fiber was used for mounting and exhibits almost no growth. The fiber has increased in width by ~440 μm along the $< 11\bar{2}0 >$ direction and is evolving towards a hexagonal shape.

Both fibers exibit 3C-SiC growth near the tip. A large number of steps are present on both fibers, particularly on the five hour growth fiber. This could be due to the imperfect nature of the starting sliver. However, similar morphology is displayed by the

RAF process and a model for such behavior has been proposed by Takahashi et al [1, 2].

Conclusion:

By exposing all surfaces of small slivers diced from m-plane boule slices to uniform growth conditions, homoepitaxial lateral expansion of m-plane oriented fibers has been demonstrated. Growing perpendicular to the (0001) axis without the benefit of SD's to maintain polytype has been demonstrated. SWBXT confirms that the underlying substrate polytype was successfully replicated and is of a high crystalline quality with minimal or no strain, Fig. 5.

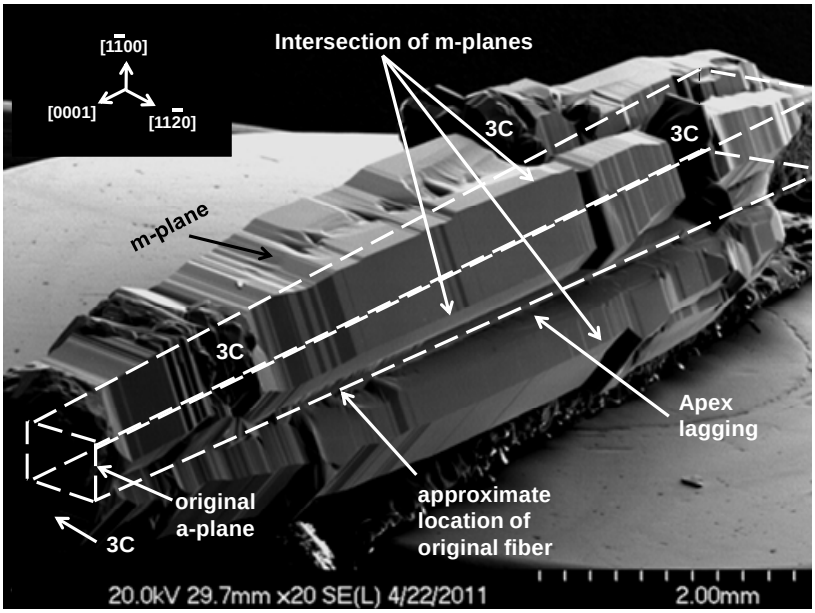

Fig. 4: SEM of 6H-SiC m-plane polished fiber 2 after eight hours of growth at higher growth rate conditions. The white dashed lines indicate the aproximate shape and location of the original fiber. Note the deep trough (apex lagging) that has evolved as growth at the intersection of the m-planes has outpaced the growth rate at the center of the fiber. Some regions of 3C-SiC have nucleated near either end of the fiber. The fiber has increased in width by ~1500 μm in the $< 1\bar{1}00 >$ direction.

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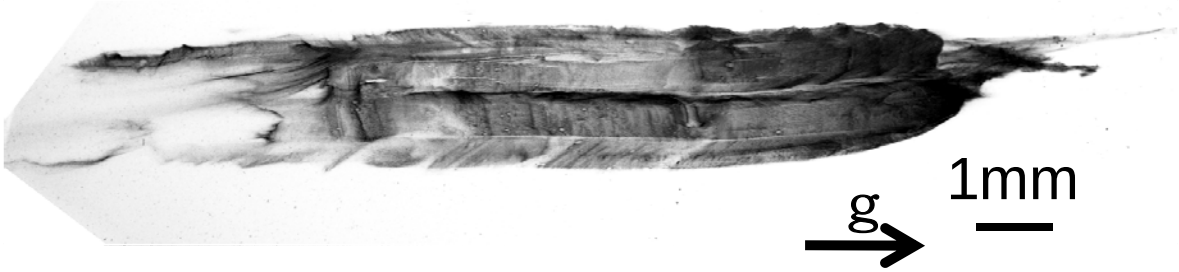

Fig. 5: SWXBT topograph of fiber 2 showing the high crystaline quality of the epitaxial growth.

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