

# A Novel Approach of Utilizing Mechanically Flexible SiC Substrate to Grow Crack-Free AlN Bulk Crystal by Thermal Strain Relaxation Functionality

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**Abstract.** The fabrication of novel semiconductor seed crystals using hetero-epitaxial growth on substrates such as Si, sapphire, and SiC, which have been successfully grown to large diameter and high quality, is very attractive as a breakthrough technology. However, a critical issue in heteroepitaxial growth is the formation of cracks due to thermal stress caused by the difference in the thermal expansion coefficient between the substrate and the growth layer during the cooling process after growth. In this study, we propose a method to reduce thermal stress by using a "Flexible substrate," which is a substrate with mechanical flexibility enhanced by removing more than 80% of its volume with periodic through holes. Using this method, we obtained an AlN hetero-epitaxial growth layer with absolutely no cracks observed. This method is applicable not only for AlN on SiC but also for the fabrication of various new semiconductor materials.

## Introduction

The recent trend toward lower cost and larger diameter SiC wafers have opened the door for their application as seed substrates for hetero-epitaxy of large diameter new-generation semiconductor bulk crystal. Due to its high band-gap and thermal conductivity, AlN has a high potential for DUV-LED and high-frequency transistors [1]. However, the cost of AlN is very high because the wafer diameter has not expanded over several years due to its extremely slow radial growth rate. Since the lattice mismatch between those materials is as tiny as 1%, high-quality AlN bulk crystals can be obtained using SiC as a substrate. For heteroepitaxial growth of AlN/SiC, the sublimation method at temperatures as high as 1700-2100°C is used. The most severe problem in AlN/SiC hetero-growth is also the formation of the crack in the AlN growth layer due to thermal stress because of the significant difference in the thermal expansion coefficient of 23% (Fig. 1. (a)). Therefore, using Pendeo growth with mesa-structured SiC substrates has been considered to suppress the cracks. In this method, thermal stress destroys the mesa structure and separates the AlN growth layer from SiC substrate to reduce cracking (Fig. 1. (b)). The mesa structure on the SiC surface is fabricated by selective ion etching or the ununiform thermal decomposition of the SiC surface in the initial stage of AlN growth [2]. However, the critical issue of this method is that the lateral wing-like of AlN is deformed due to lattice mismatch and thermal stress, causing dislocation and grain boundary generation [3, 4] (Fig. 2. (a)). Therefore, a method to prevent crack and grain boundary formation in AlN/SiC heteroepitaxial growth is necessary. To solve this issue, we propose the use of the Flexible SiC substrate to reduce the thermal stress (Fig. 1. (c)). The surface of Flexible SiC substrate is patterned to be a single connected surface, resulting in a zippering growth mode of AlN when closing the through-hole. In zippering growth, the grain boundary and the dislocations generation are significantly suppressed (Fig. 2. (b)) [5]. In this study, we examine the crack suppression effect of the AlN hetero-epitaxial growth layer using the Flexible SiC substrate.

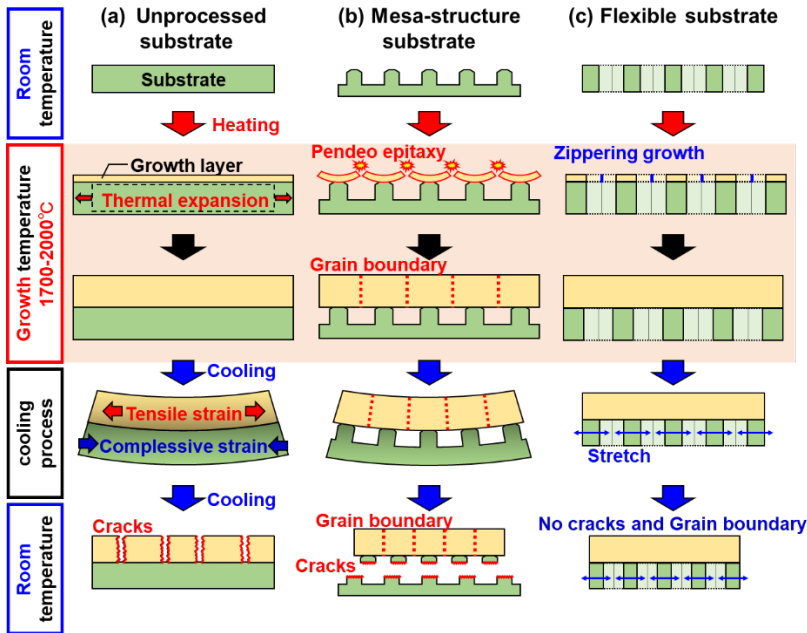


Fig. 1. AlN growth processes on the (a) un-processed, (b) mesa-structured, and (c) Flexible SiC substrate.

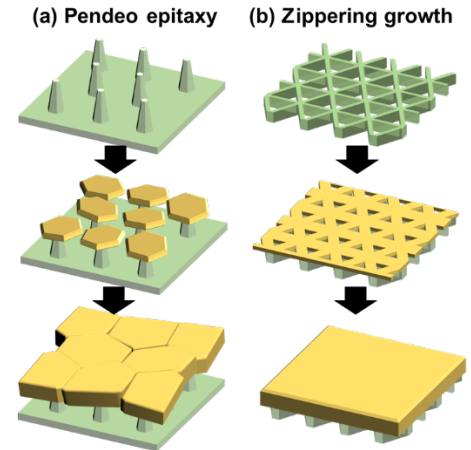


Fig. 2. (a)Pendeo epitaxy on the mesa-structured substrate and (b) Zippering growth on the Flexible substrate.

## Experiment

Flexible SiC substrates were fabricated from 500  $\mu\text{m}$  thick 4° off 4H-SiC (0001) by pulsed laser processing to remove 80% of its volume by periodic through-hole. The patterns were designed to improve the isotropic flexibility, as reported by C. Schumacher et al. [6]. All samples were thermally etched at 1800°C by Si-vapor etching [7] for about 3  $\mu\text{m}$  to remove polishing and laser processing damage on the SiC seed substrate surface. For the AlN sublimation growth, the temperature,  $\Delta T$ , and  $\text{N}_2$  pressure were set at 1870°C, 6.7 K/mm, and 30 kPa. a high-temperature gradient environment of 6.7 K/mm is applied to suppress unintentional SiC surface decomposition at the initial phase of AlN growth [8]. High-purity AlN powder manufactured by Toyo Aluminum K. K. was used as the source material. The SiC substrates and the AlN growth layer were observed using a laser microscope, optical microscope, Raman spectroscopy (excitation wavelength: 532.3nm), and SEM (1kV).

## Results and Discussion

Fig. 3. shows the laser microscopy mapping images of the Unprocessed SiC substrate and the Flexible SiC substrate. It is observed that most of the volume of the Flexible SiC substrate is removed by the through holes made by laser processing. Flexible SiC substrates are treated with periodic through holes by pulsed laser, and about 80% of the volume of the SiC substrate is removed. Since the intense laser processing introduces significant processing damage to the SiC substrate surface, thermal etching was used to remove it. Fig. 4. shows bird's eye view SEM images of (a) after laser processing and (b) thermally etched Flexible SiC substrate. After laser processing, the surface roughness due to laser ablation can be observed on the SiC surface. It can be seen that the thermal etching of 3  $\mu\text{m}$  on the lasered flexible SiC substrate removes the damage and restores the surface flatness. Therefore, in order to eliminate the harmful effects of laser processing, all SiC substrates will be thermally etched before AlN growth.

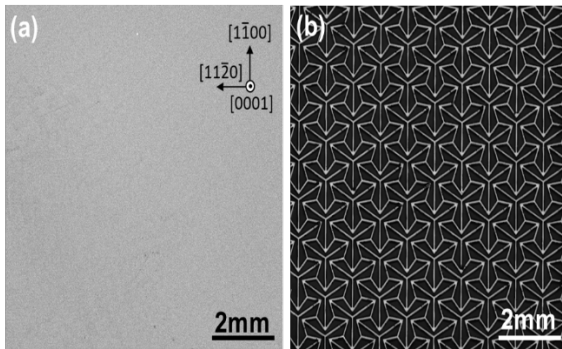


Fig. 3. Laser microscopy images of (a) the unprocessed SiC substrate and (b) the Flexible SiC substrate.

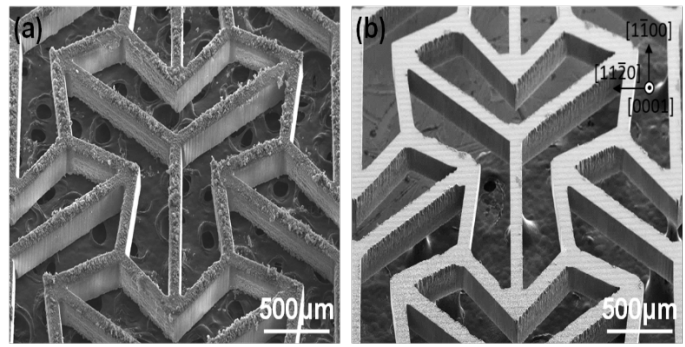


Fig. 4. 1kV-SEM 40° bird-view image of Flexible SiC substrate (a) as laser processing and (b) after thermal etching.

Next, a 500  $\mu\text{m}$  thick AlN growth layer was grown on these SiC substrates using the sublimation method at a growth temperature of 1870°C. Fig. 5. shows the optical microscope and SEM images of AlN grown on unprocessed SiC substrate and Flexible SiC substrate, respectively. The AlN on the unprocessed SiC substrate was observed to have many cracks in both the optical microscope image and SEM, and even the underlying SiC substrate was cracked. In contrast, no cracks were observed in the AlN growth layer on the Flexible SiC substrate in either macroscopic observation by optical microscopy or microscopic observation by SEM. This result suggests that using a Flexible substrate can reduce the thermal strain induced during the cooling process of hetero-epitaxial growth.

The results of the Raman spectroscopy evaluation of these samples are shown in Fig. 6. Fig. 6 (a) and (b) show the measured Raman  $E_2$  peak shift mapping images of AlN on unprocessed SiC substrate and flexible SiC substrate, respectively. The Raman  $E_2$  peak shift is known to vary with the strain of the AlN crystal, and is  $657\text{ cm}^{-1}$  for an unstrained AlN single crystal [9]. The fact that the average Raman shift value shifts to the lower frequency side for unstrained AlN in both samples indicate that tensile strain is introduced. From these results, we can see that both samples have introduced thermal strain. In the AlN growth layer on the Unprocessed SiC substrate, there are linear regions of stress relaxation along the  $\langle 11\bar{2}0 \rangle$  direction. Cracks in the AlN growth layer tend to form along with the  $\langle 11\bar{2}0 \rangle$  direction so that its stable surface,  $\{10\bar{1}0\}$ , is exposed. Therefore, the linear stress relaxation region along the  $\langle 11\bar{2}0 \rangle$  direction is considered to be the relaxation of thermal strain by the cracks. On the other hand, no linear stress relaxation region was observed in the AlN-grown layer on the flexible SiC substrate, which supports the absence of cracks. The fact that the average tensile stress is also slightly higher can be attributed to the fact that stress relaxation has not occurred.

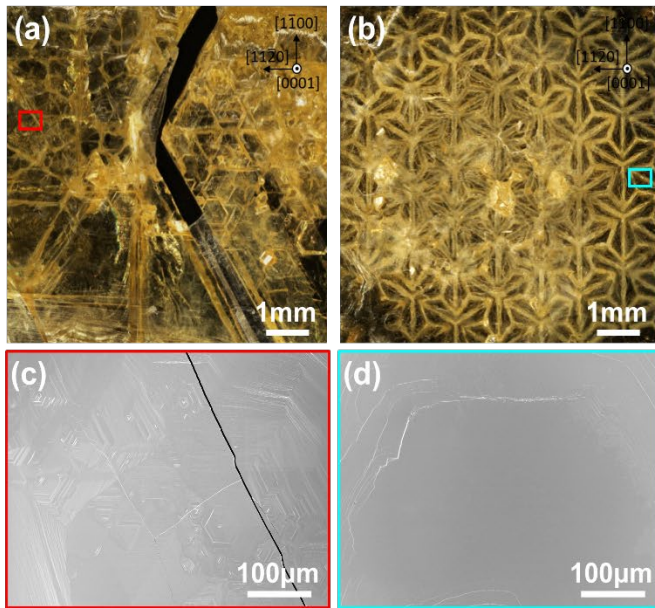


Fig. 5. Optical microscopy images and SEM images of 500μm thick AlN grown layer surface on (a), (c) the unprocessed SiC substrate and (b), (d) the Flexible SiC substrate.

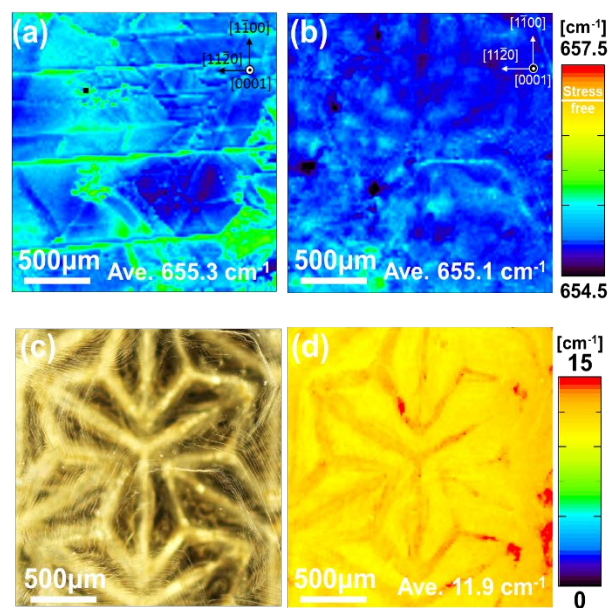


Fig. 6. (a) Raman E<sub>2</sub> peak shift mapping image of AlN on unprocessed SiC. Same area observation images of AlN on Flexible SiC by (b) Raman E<sub>2</sub> peak shift mapping, (c) optical microscopy, and (d) Raman E<sub>2</sub> peak FWHM mapping.

The optical microscope images and Raman E<sub>2</sub> peak FWHM mapping images of the AlN growth layer on the flexible SiC substrate are shown in Fig. 6. (c) and (d), respectively. The flexible SiC substrate is seen through the AlN growth layer in the optical microscope image. It can be seen that the pattern of the SiC substrate and the region with large FWHM observed in the FWHM mapping are in the same location. It is known that a 1% lattice mismatch generates a high density of dislocations of about  $10^{10} \text{ cm}^{-2}$  at the interface between AlN and SiC, and that the dislocation density is significantly smaller when the hetero-grown layer forms a hollow structure [2, 10]. This suggests that the flexible substrate can also be expected to reduce the crystal defect density by reducing the interfacial area.

## Summary

In conclusion, we proposed a method to suppress thermal stress during the cooling process, which is a critical issue in hetero-epitaxial growth, by using Flexible SiC substrate with periodic through holes fabricated by laser processing. As a result of the heteroepitaxial growth of AlN with a thickness of 500 μm on a Flexible SiC substrate with 80% of the volume removed, no cracks were observed by SEM and optical microscopy. In addition, no crack-induced stress relaxation was observed in strain analysis using Raman spectroscopy. Therefore, the use of Flexible substrates is expected to lead to breakthroughs in the development of various new materials using heteroepitaxial growth.

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