# Temperature Gradient Control with an Air-Pocket Design for Growth of High Quality SiC Crystal

Submitted: 2022-09-04

Revised: 2022-11-25

Online: 2023-05-30

Accepted: 2022-11-25

Seung-June Lee<sup>1,a</sup>, Su-Ho Kim<sup>1,b</sup>, Jung-Woo Choi<sup>1,c</sup>, Jong-Hwi Park<sup>1,d</sup>, Jung-Doo Seo<sup>1,e</sup>, Myung-Ok Kyun<sup>1,f</sup>, Jung-Gyu Kim<sup>1,g</sup>, Kap-Ryeol Ku<sup>1,h</sup>, Yeon-Suk Jang<sup>2,i</sup>, Won-Jae Lee<sup>2,j</sup>\*

<sup>1</sup>Senic, 17-15, 4sandan 7-ro, Jiksan-eup, Seobuk-gu, Cheonan-si, Chungcheongnam-do, Korea <sup>2</sup>Department of Advanced Materials Engineering, Dong-Eui University, 176, Eomgwang-ro, Busanjin-gu, Korea

<sup>a</sup>sjlee@senic.co.kr, <sup>b</sup>shkim@senic.co.kr, <sup>c</sup>jwchoi@senic.co.kr, <sup>d</sup>jhpark@senic.co.kr, <sup>e</sup>jdseo@senic.co.kr, <sup>f</sup>mokyun@senic.co.kr, <sup>g</sup>jgkim@senic.co.kr, <sup>h</sup>krku@senic.co.kr, <sup>i</sup>d91052@nate.com, <sup>j</sup>leewj@deu.ac.kr\*

Keywords: Air pocket, PVT, 6-inch, Temperature gradient, defect density, hot zone

**Abstract.** The hot-zone design using an air-pocket was adopted to produce uniform temperature gradient in horizontal direction. In order to investigate the change of temperature gradient toward horizontal direction with growth time, the front shape of SiC growing crystal was measured with different growth stages such as initial, growing and finished stage. While SiC ingot grown in conventional hot-zone design exhibited inhomogeneous growth front in the initial stage of growth and multi facet formation in final stage, which could result in increased defect density, a homogeneous temperature gradient and improved crystal quality was obtained in the modified hot-zone design. Based on the mapping measurement of FWHM (Full width at half maximum) value in X-ray rocking curve, the crystal quality of SiC crystals grown with the modified hot-zone design was observed to be definitely better than conventional design.

# Introduction

Commercially available SiC-power-devices of MOSFETs and SBDs are recently fabricated on n-type 4H-SiC substrates with 6-inch in diameter. For improving device performance and device yield, the quality of large diameter SiC wafer is important. [1-3] In particular, it was well known that high quality SiC epitaxial layer with low defect density can be grown mainly on high quality SiC substrate having low crystal defect and low warpage value. Therefore, the careful control of temperature gradient in the front of growing crystal is crucial on large-sized SiC crystal growth.

In this study, the modified hot-zone design using an air-pocket existing on backside of seed crystal has been proposed for the growth of 6-inch SiC single crystal. With adjusting the shape of air-pocket, the temperature gradient of seed crystal in the initial stage of crystal growth could be successfully controlled. The homogeneous temperature gradient could be obtained on initial stage of growth process, which is important factor to improve crystal quality.

The actual growth has been performed for the conventional and the modified hot-zone designs and then systematically compared in terms of crystal quality. In order to improve SiC ingot quality through Physical Vapor Transport(PVT) method, it is essential to secure a lightly convex ingot shape. When temperature non-uniformity caused by the material deviation is formed, the quality deterioration such as polytype inclusion occurs due to local nucleation and abnormal growth. Therefore, a structural management method to apply the air-pocket is presented as a solution to improve non-uniform temperature gradient.

## **Experiments**

Fig. 1 showed the growth condition including temperature and pressure profile of each growth step for SiC single crystal. The SiC crystal were grown at the temperature of 2400°C and with argon inert gas of 20 torr including 5% nitrogen.[4] The axial thermal gradient of the SiC crystal during the growth is estimated at the range of 15~20°C/cm. The seeds and the source materials of high purity SiC are placed on opposite side in a sealed graphite crucible which is surrounded by graphite insulator. The conventional hot-zone design with no air-pocket and the modified hot-zone design with the air-pocket were used for SiC crystal growth. Fig. 2 exhibited schematic diagram of conventional hot-zone design and modified hot-zone design. The modified hot-zone design consisted of an air-pocket in the backside of SiC seed holder to control the temperature gradient in the horizontal direction. X-ray rocking curve measurement and etch pit density (EPD) analysis were performed to investigate the effect of modified hot-zone design on crystal quality of SiC crystal.

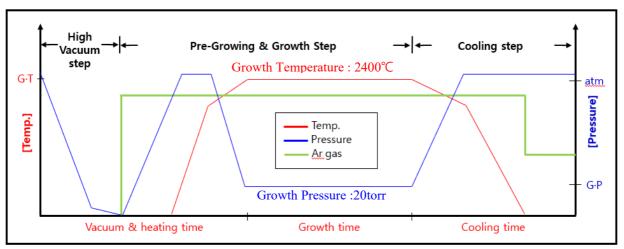


Fig. 1: The growth condition for SiC single crystal. Temperature and pressure profile for growth step (G.T., G.P.) were indicated on the graph.

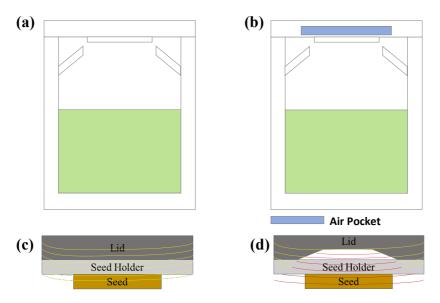


Fig. 2: Schematic diagram of (a, c) conventional hot-zone design and (b, d) modified hot-zone design. The modified hot-zone design consisted of an air-pocket in the backside of SiC seed holder to control the temperature gradient in the horizontal direction.

#### **Result and Discussions**

Fig. 3 exhibited the front shape of SiC crystal ingot measured at different growth stages (initial, growing, finished stages) in the conventional hot-zone design. To obtain the front shape of SiC crystal ingot with the growth stage, three different SiC ingots were prepared at identical growth condition with different growth time. As shown in ingot shape of growing and finished stages in Fig.3, the front shape of SiC crystal ingot were definitely convex indicating the existence of convex temperature gradient in growth cell during the growth process. However, the front shape of SiC crystal ingot at initial stage in the conventional hot-zone design presented bumpy line, which could result from inhomogeneous temperature gradient in horizontal direction. The formation of not uniform  $\Delta T$  at the growth front in the initial stage of growth could generate multi facet structure at the growth front and increase the probability of polytype inclusion by the stacking fault generation and defect formation. Fig. 4 exhibited schematic diagram forming polytype inclusion and defect generation with the growth process in the conventional hot-zone design.

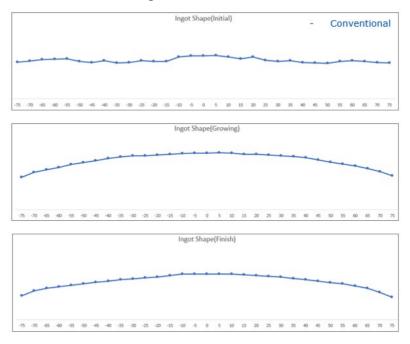


Fig. 3: The front shape of SiC crystal ingot measured at different growth stages (initial, growing, finished) in conventional hot-zone design.

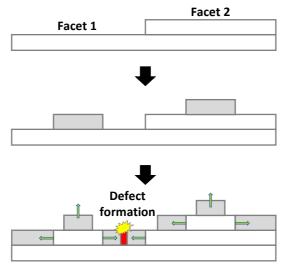


Fig. 4: Schematic diagram forming polytype inclusion and defect generation with growth process in conventional hot-zone design.

The front shape of SiC crystal ingot measured at different growth stages (initial, growing, finished stages) in the modified hot-zone design was shown in Fig.5. With adjusting the shape of air-pocket, the temperature gradient of seed crystal in the initial stage of crystal growth was successfully controlled. Temperature gradient could be definitely changed because the heat radiation could add to the heat conduction on air-pocket structure. During the entire growth process, the front shape of crystal ingot was almost same implying that ΔT was uniformly maintained. Fig. 6 exhibited optical photographs and ultra-volet fluorescence (UVF) images of SiC crystal ingots prepared with the conventional hot-zone design (a) and the modified hot-zone design (b). While SiC ingot image obtained by UV lamp (*Vilber, CN-15CC*) consisted of regions having different colors, which indicating different polytype in the coventional design, SiC crystal grown in the modified design showed uniform color.

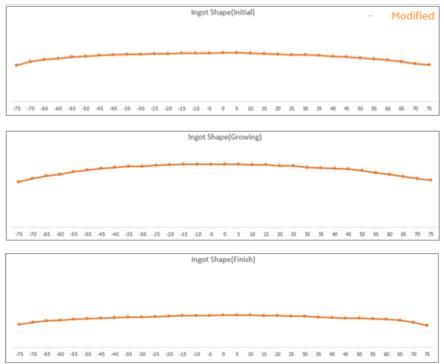


Fig. 5: The front shape of SiC crystal ingot measured at different growth stages (initial, growing, finished) in the modified hot-zone design.

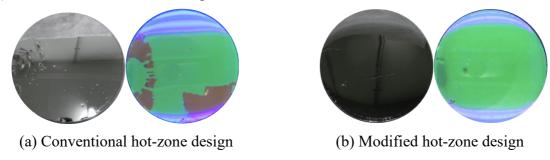


Fig. 6: Optical photographs and UVF images of SiC crystal ingots prepared with (a) the conventional hot-zone design and (b) the modified hot-zone design.

The crystal quality of SiC crystals grown with the conventional hot-zone design and the modified hot-zone design was compared using X-ray rocking curve measurement (*Rigaku*, *Smart-lab*). The mapping measurement of FWHM (Full width at half maximum) value in X-ray rocking curve and a shift of X-ray peak position for two different SiC wafers from SiC grown ingots are presented in Fig.7 and Table.1. From the average FWHM values in the Table.1, 37.4 arcsec in SiC crystal grown by the conventional hot-zone design was definitely improved to 19.8 arcsec in SiC crystal grown by the modified hot-zone design. The shift degrees of X-ray peak position of conventional and modified design were 0.6° and 0.2°, respectively. The shift degree of X-ray peak position may relate with the

bow value of crystal plane and the lower value of shift degree in SiC crystal grown by the modified hot-zone design is more advantageous for next process step in a device fabrication.

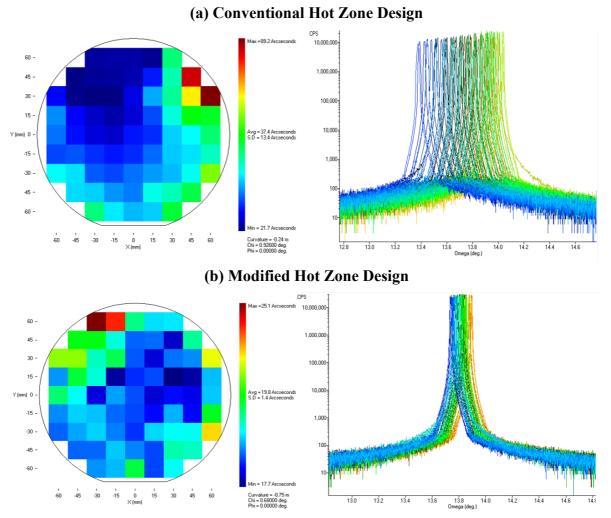


Fig. 7: The mapping measurement of FWHM (Full width at half maximum) value in X-ray rocking curve and a shift of X-ray peak position for two different SiC wafers prepared by (a) Conventional hot-zone design and (b) modified hot-zone design.

Table. 1. The FWHM values(max, min and average) in X-ray rocking curve and the shift degree of X-ray peak position for two different SiC wafers prepared by (a) conventional hot-zone design and (b) modified hot-zone design.

	(a) Conventional hot-zone design	(b) modified hot-zone design
AVG.[ arcsec]	37.4	19.8
Max.[arcsec]	89.2	25.1
MIN. [arcsec]	21.7	17.7
Shift degree	Δ0.6°(13.4~14.0°)	Δ0.2°(13.7~13.9°)

In order to investigate the defect density of SiC crystal, the chemical etch in molten KOH at 480°C for 5 min was performed. Fig. 8 exhibited the photograph (*Nikon, Eclipse LV150*) of etched surface in SiC crystal grown with the conventional hot-zone design and the modified hot-zone design. Fig.8(a) showed an etched image of specific region around a facet and higher density of basal plane dislocation (BPD) was observed. Defect densities of etched surface in SiC crystals grown with the conventional hot-zone design and the modified hot-zone design are presented in Table. 2. The defect density of SiC crystal grown by the modified design is definitely lower than the conventional design.

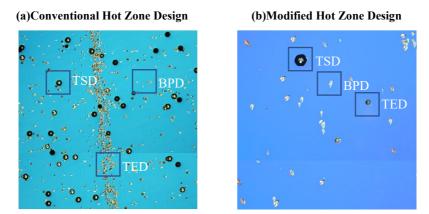


Fig. 8: The photograph of etched surface. (a) Conventional hot-zone design (b) Modified hot-zone design.

Table. 2. Defect densities of etched surface in SiC crystals grown with (a) conventional hot-zone design (b) modified hot-zone design.

	(a) conventional hot-zone design	(b) modified hot-zone design
TSD[ea/cm <sup>2</sup> ]	4,500	200
TED[ea/cm <sup>2</sup> ]	19,300	400
BPD[ea/cm <sup>2</sup> ]	50,300	4,200

\*TSD: Threading Screw Dislocation, TED: Threading Edge Dislocation

# **Summary**

The hot-zone design using the air-pocket was proposed for improving crystal quality of SiC crystal. While SiC ingot grown in the conventional hot-zone design exhibited inhomogeneous growth front in the initial growth stage and multi facet formation in final stage, resulting in increased defect density, the modified hot-zone design produced homogeneous temperature gradient and improved crystal quality. Based on FWHM value in X-ray rocking curve and etch pit density analysis, the crystal quality of SiC crystals grown with the modified hot-zone design was observed to be definitely better than the conventional hot-zone design.

## References

- [1] A. R. Powell et al., Mater. Sci. Forum, Vol. 858 (2016) 5
- [2] E. Y. Tupitsyn et al., Mater. Sci. Forum, Vol. 483-485 (2005) 21
- [3] A. Kumar, M. S. Aspalli, Int. J. Res. Eng. Tech., Vol. 3 (2014) 248
- [4] B. K. Jang et al., Mater. Sci. Forum, Vol. 1004 (2020) 32