Solid State Phenomena
ISSN: 1662-9779, Vol. 362, pp 59-64
doi:10.4028/p-uOTL9e

Submitted: 2023-09-30
Revised: 2024-05-09
Accepted: 2024-05-09
Online: 2024-08-27

The Role of Air-Pocket in Crucible Structure for High Quality SiC Crystal Growth

Seung Jun Lee[1,a], Su Ho Kim[1,b], Jung Woo Choi[1,c], Jong Hwi Park[1,d], Jung Gyu Kim[1,e], Kap Ryeol Ku[1,f], Ha Lin Lee[2,g], Gi Uk Lee[2,h] and Won Jae Lee[2,i*]

[1]Senic, 17-15, 4sandan 7-ro, Jiksan-eup, Seobuk-gu, Cheonan-si, Chungcheongnam-do, Korea

[2]Department of Advanced Materials Engineering, Dong-Eui University, 176, Eomgwang-ro, Busanjin-gu, Busan, Korea

[a]sjlee@senic.co.kr, [b]shkim@senic.co.kr, [c]jwchoi@senic.co.kr, [d]jhpark@senic.co.kr, [e]jgkim@senic.co.kr, [f]krku@senic.co.kr, [g]halinlee99@naver.com, [h]dlrldnr7774@naver.com, [i]leewj@deu.ac.kr

Keywords: 4H-SiC Growth, PVT, Air pocket, C/Si ratio, Graphite structure

Abstract. The modified design using an air-pocket existing on inside of crucible has been proposed for the growth of 6-inch SiC single crystal. The actual growth has been performed for conventional, focus ring design and modified hot-zone designs under the same growth conditions and then three SiC crystals were systematically compared in terms of crystal quality. Since stacking faults and polytype inclusions, which could cause dislocation formation, were suppressed by the suitable C/Si ratio control, it was possible to grow SiC ingots with reduced defect density and excellent crystal quality with crucible structures proposed in this study.

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]. For high-quality epitaxial growth, a high-quality SiC substrate with a low density of crystal defects and suppressed polytype inclusion must be required. Therefore, the control of temperature gradient and C/Si ratio of vapor source in front of growing crystal is important to obtain high quality SiC crystal growth.

In this study, the modified design using an air-pocket existing on inside of crucible has been proposed for the growth of 6-inch SiC single crystal. With adjusting the shape of graphite structure with air-pocket, the suppression of double temperature gradient formation in the crucible and the C/Si ratio control were successfully performed [4-6]. The actual growth has been performed for conventional design, focus ring design and modified hot-zone design under the same growth conditions and then three SiC grown crystals were systematically compared in terms of crystal quality.

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 2000~2400℃ and with argon inert gas of 1~40 torr including 5~10% nitrogen. The axial thermal gradient of the SiC crystal during the growth is estimated at the range of 15~20℃/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. To minimize the influence of seed crystals, three consecutive wafers were applied as seed crystals in one ingot, and the EPD was 6,000ea/cm^2. Fig. 2 exhibited schematic diagram of the conventional crucible structure, the crucible structure with focus ring and the modified graphite structure with air pocket used for 6-inch SiC crystal growth. The air-pocket in graphite structure was adopted for designing temperature gradient and C/Si ratio control in the SiC crystal growth. All three methods have the same MSD (Material – Seed distance) condition. Also, the distance between the structure and the powder surface was kept the same to reduce the difference in the movement of the gas source depending on the distance. It was observed that the formation of a double temperature gradient in the growth zone was suppressed depending on the shape of the graphite structure. The C/Si ratio was improved by the reaction between a graphite structure with a large specific surface area and a vapor source, which is important factor to control polytype inclusion [7]. All graphite structures are made of porous graphite, and the focus ring has a thickness of 3 mm and an inner diameter of 40 mm. The air pocket graphite structure consisted of two bodies and had an inner diameter of 100 mm. After the growth experiment, the growth rate and crystallinity of crystals were evaluated after wafering each of the three 150 mm ingots obtained using the three designs. X-ray rocking curve measurement and an etch pit density (EPD) analysis were performed to investigate the effect of the C/Si ratio control due to the arrangement of graphite structures on crystal quality of SiC crystal.

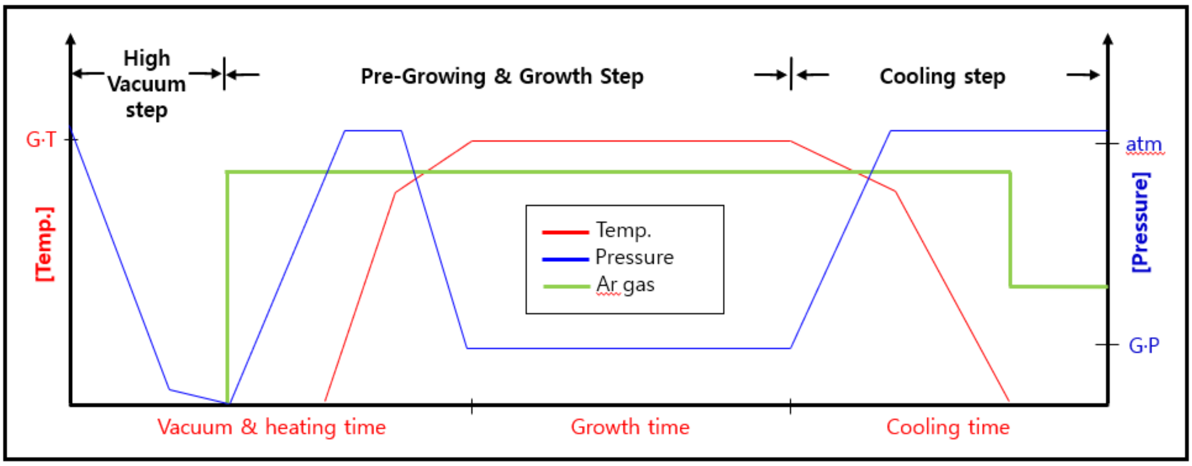


Fig. 1. The growth condition for SiC single crystal. Temperature and pressure profile for growth step (G.T., G.P.) were indicated on the graph.

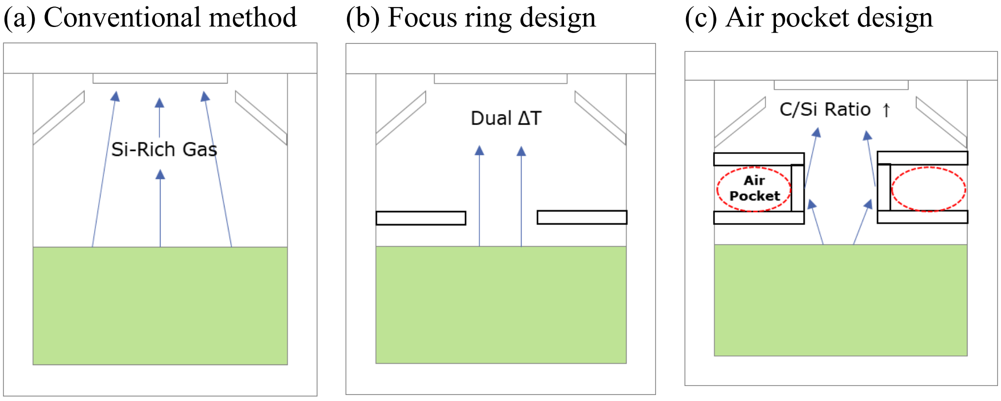


Fig. 2. Schematic diagrams of each graphite structure. (a) Conventional method, (b) Focus-ring method, (c) Air pocket design.

Result and Discussions

Table 1 summarized growth results concerning with the polytype inclusion, the growth thickness, and the front shape of ingot observed from SiC crystals grown with three different crucible structures. While polytype inclusion was observed in conventional crucible design, none of other polytype was formed in crucible structures with focus ring and air pocket designs. Inserting new graphite structures into the conventional crucible could change a temperature gradient inside crucible resulting in different growth rate and front shape in grown crystals. Fig. 3 is Ultra-Violet Fluorescence (UVF) photographs of crystal ingots grown with three different crucible structures. The polytype inclusions were formed in the conventional structure because the C/Si ratio could not be adequately controlled. In case of growth processes using the focus ring and air pocket graphite structure, the C/Si ratio was controlled and the polytype inclusion was successfully suppressed. However, the growth rate of SiC crystal grown with the focus ring was lower than that in conventional structure due to the blocking effect of the focus ring structure.

Table 1. Growth result of SiC ingots grown with different crucible structure.

	Conventional method	Focus ring	Air pocket
Polytype inclusion	Existed	None	None
Growth thickness (mm)	18	12	20
Front shape	Concave	Flat	Convex

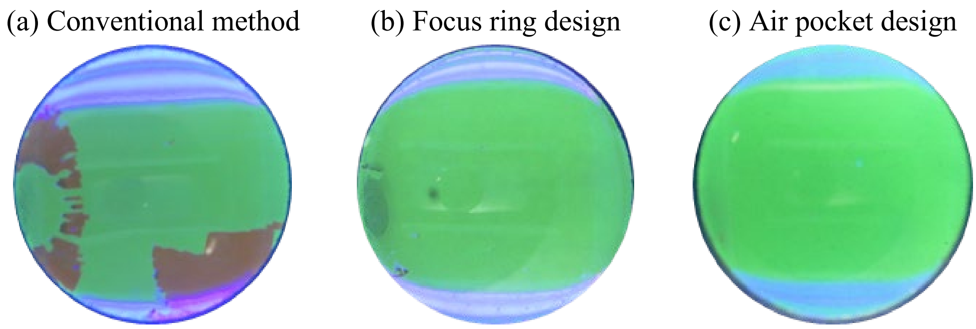


Fig. 3. UVF images of 150mm SiC ingot grown by applying each method.

As shown in Fig. 4, when the focus ring structure was applied, a relatively lower temperature was formed due to little heat generation near the ring, and a double temperature gradient was formed. Therefore, actual growth was blocked by vapor source deposition in focus ring structure and were prevented spreading of the vapor sources to the seed area, resulting in a decrease in growth rate. To overcome this phenomenon, a graphite structure with air pockets was designed. Since the temperature of the air pocket structure could rise due to radiant heat as the temperature in the crucible increases, the vapor sources could diffuse to the seed area, not the graphite structure, due to the temperature gradient. In addition, since the graphite structure with air pockets has a larger surface area than the focus ring structure, the reactivity with Si-rich vapor gas increased and the C/Si ratio could be controlled more uniformly [7]. Fig. 5 exhibited photograph images of graphite structures using focus ring and air pocket graphite structure after the growth process.

The FWHM value and shift degree of SiC crystals grown with three different crucible structures was investigated by a high resolution XRD system to observe the crystallinity and crystal quality of crystals. As can be seen in Table 2, the FWHM value of SiC crystals grown with the focus ring and the air pocket structures was lower than that of crystal grown with the conventional structure. In addition, the air pocket graphite structure improved the quality of SiC crystal, showing 15.9 arcsec of the FWHM value and the shift degree of Δ0.2°. These improved crystallinity and crystal quality could be related with the suppressed polytype inclusion through C/Si ratio control.

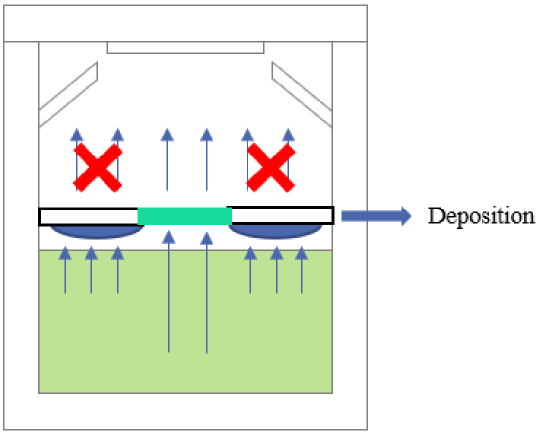
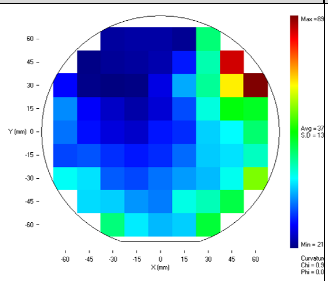
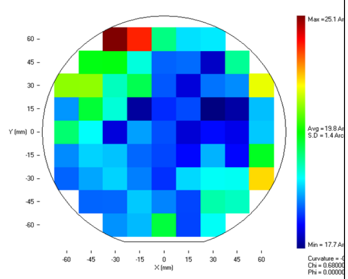
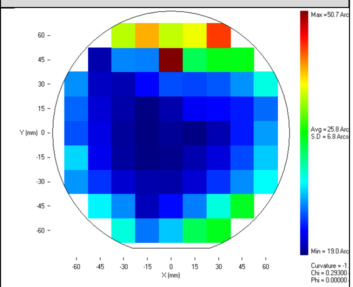
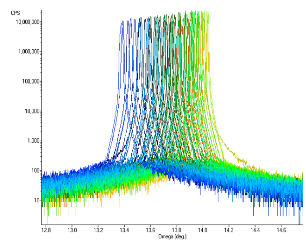
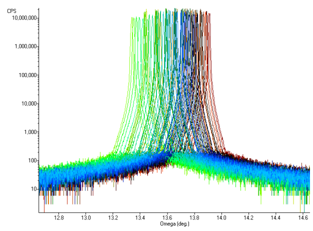
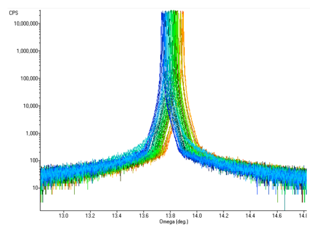


Fig. 4. Schematic diagrams of dual temperature gradient and deposition to Focus Ring structure.

(a) Focus Ring graphite structure (b) Air-Pocket graphite structure

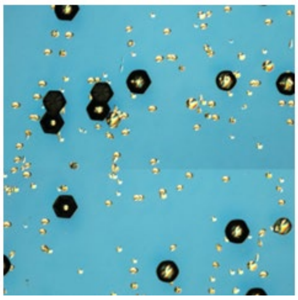
Fig. 5. Photograph images of graphite structures after the growth process. (a) Focus ring graphite structure, (b) Air pocket graphite structure.

Table 2. FWHM and shift degree results of SiC crystals grown with different crucible structures.

		Conventional method	Focus ring	Air pocket
Rocking-curve mapping Image				
FWHM (arcsec)	Avg.	37.4	19.8	15.9
	Max.	89.2	25.1	16.8
	Min.	21.7	17.7	15.2
Shift degree (°)				
		0.6	0.6	0.2

Finally, an etch pit density (EPD) result of SiC crystals grown with different crucible structures was investigated on etched surface (Table 3). The EPD value was improved when focus ring structure was applied in the conventional crucible. TSD and TED values were greatly reduced. TSD value of SiC crystal grown with the air pocket graphite structure was completely suppressed, and the total EPD value was further improved compared to that of the focus ring structure. In conclusion, since stacking faults and polytype inclusions, which could cause dislocation formation, were suppressed by polytype stability and convex ingot shape, it was possible to grow SiC ingots with reduced defect density and excellent crystal quality with crucible structures proposed in this study.

Table 3. EPD result of SiC crystals grown with different crucible structures.

	Conventional method	Focus ring	Air pocket
OM Image			
TSD (ea/cm^2)	4,500	200	0
TED (ea/cm^2)	19,300	400	1,300
BPD (ea/cm^2)	50,300	4,200	2,400

Summary

A study on obtaining high-quality SiC ingots by suppressing polytype inclusions through C/Si ratio control of vapor source was proposed. The C/Si ratio was increased by applying various shapes of graphite structures to control the C/Si ratio of the Si-rich gas dominant in the crucible. In addition, the graphite structure was relatively heated by a radiant heat so that the vapor source was directed to the seed area, and an optimal structure to prevent the vapor source from being deposited on the graphite structure was also studied. When the graphite structure with the air pocket was used, the growth rate was the highest and the polytype inclusion was suppressed. In addition, it was confirmed that the FWHM value and the shift value were improved, and the suppression of the dislocation defect was also confirmed.

Acknowledgements

This work was supported by Technology Innovation Program (RS-2022-00154720) and the Technology Innovation Program (Project Number: 20022843, Project Name: Development of automated inspection equipment capable of high speed high precision analysis of large diameter SiC bare and Epi wafers) funded by the Ministry of Trade, Industry(MOTIE, Korea), 2023 and by the Energy and Korea Institute for Advancement of Technology(KIAT) grant funded by the Korea Government(MOTIE, P0012451, The Competency Development Program for Industry Specialist).

References

[1] A. R. Powell *et al*., Mater. Sci. Forum, Vol. 858 (016), p.5

[2] E. Y. Tupitsyn *et al*., Mater. Sci. Forum, Vol. 483-485 (2005), p. 21

[3] A. Kumar, M. S. Aspalli: Int. J. Res. Eng. Tech., Vol. 3 (2014), p. 248

[4] A. Gupta *et al*., U.S. Patent No. 159, 803 (24 October 2013)

[5] A. Gupta *et al*., U.S. Patent No. 447,742 (24 September 2021)

[6] K. M. Dovbnya, N. D. Er'omina: Materials Science Vol. 22, No. 2. (2016), p. 287

[7] R. R. Ciechonski: J. Cryst. Growth Vol. 275 (2005), p.e461