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300 mm 4H-SiC Crystal and Substrate Development

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Abstract. The development of integrated circuits (IC) is mainly driven by the advanced processes and increased silicon wafer diameter in the past several decades. It is technically believed that the diameter of 300 mm for SiC wafer is too difficult to be achieved since SiC crystal diameter expansion is a long and tough process, the growth process of which is different from that of silicon crystal. Herein, we demonstrate the diameter expansion process of SiC crystal from 200 mm to 300 mm using physical vapor transportation (PVT) method and show the world's first 300 mm 4H-SiC single crystal substrate with 100% 4H polytype. The driving force of crystal diameter expansion and resultant thermal stress are discussed in this paper. Based on the successful preparation of 300 mm SiC seed crystal, 12-inch high-purity, conductive n-type & p-type SiC substrates are subsequently fabricated. Quality characterization of the 300 mm SiC substrate shows very low micropipe and threading screw dislocation density below 0.05 cm^{-2} and 120 cm^{-2}, respectively. Furthermore, both 500 μm and 750 μm thickness substrates are fabricated with bow and warp values lower than 10 μm and 30 μm, indicating high quality 300 mm substrates applicable in power devices and other emerging areas.

Introduction

4H-SiC is one of the most important and promising semiconductor materials due to its excellent electrical, thermal, mechanical and optical properties[1]. Semi-insulating SiC substrates are now used to fabricate radio frequency devices applied in 5G station, while conductive SiC substrates are used in powder devices which plays important roles in electrical vehicles (EVs), photovoltaics (PVs) etc. Meanwhile, new SiC applications such as waveguide in AR glasses and interposer in advanced packaging[2] are emerging which require even larger diameter SiC substrates. Obtaining the first 300-mm SiC substrate requires fabricating a starting 300 mm SiC seed crystal through diameter expansion—a process that is particularly challenging in PVT growth due to the frequent occurrence of polycrystal/defect formation and crystal stress during the multiple dynamic stages of crystal enlargement[3,4]. To suppress crystal breakage and polytype formation during SiC crystal diameter expansion, multiple runs of diameter expansion processes is necessary to obtain enlarged crystal diameter. This requires continuously new designs of crystal growth chamber, thermal field, growth process, wafering tools and processes since each iteration generates new crystals with non-standard diameters (e.g. 260mm).

Herein, we demonstrate successfully prepared 300 mm SiC substrates through diameter expansion process from 200 mm to 300 mm. Lateral growth of SiC crystal driven by the radial temperature gradient (RTG) is elaborated, where maximum growth diameter expansion and minimum residual thermal stress are well balanced[5,6,7]. The quality of 300mm SiC substrates are characterized with Raman microscopy and Synchrotron X-ray Topography. Furthermore, 300 mm high-purity and conductive n & p type SiC substrates are shown in this paper.

Experiment

To simulate SiC crystal growth process and calculate the temperature distribution in the growth chamber, commercial STR_VR software is adopted[8]. The software is also used to analyze the thermal stress in the grown SiC crystal in the models. Based on the thermal field and crystal thermal stress analysis, temperature gradient and chamber structure used for each run of crystal diameter expansion are developed and optimized.

4H-SiC crystals in the diameter expansion process are grown with PVT method. The growth temperature and pressure are set at 2000-2300 ℃ and 5-50 mbar respectively. Nitrogen and argon are introduced into the growth chamber as doping and protective gases[9]. A self-produced 200 mm 4H-SiC seed crystal with 4° off-cut is used at the beginning of crystal diameter expansion process. After each run of crystal growth, newly grown SiC crystal with expanded diameter is fabricated into seed crystal used in the next generation. Meanwhile, the internal structure of crystal growth chamber and the external insulation structure have been modified at each generation[10,11]. Simultaneously, corresponding wafering tools are also customized for this process.

Results and Discussion

It's crucial to set up proper temperature gradients to drive the crystal grow in both radial and axial direction in SiC crystal diameter expansion process. Theoretically, larger RTG is preferred for lateral growth, however, a large RTG will inevitably introduce thermal stress in crystal which leads to the formation of defects or even crystal breakage. On the other hand, a small RTG will not drive monocrystal grow efficiently and polycrystal may appear in the periphery instead. Therefore, it is of great importance to balance temperature gradient and thermal stress during the continuous diameter processes.

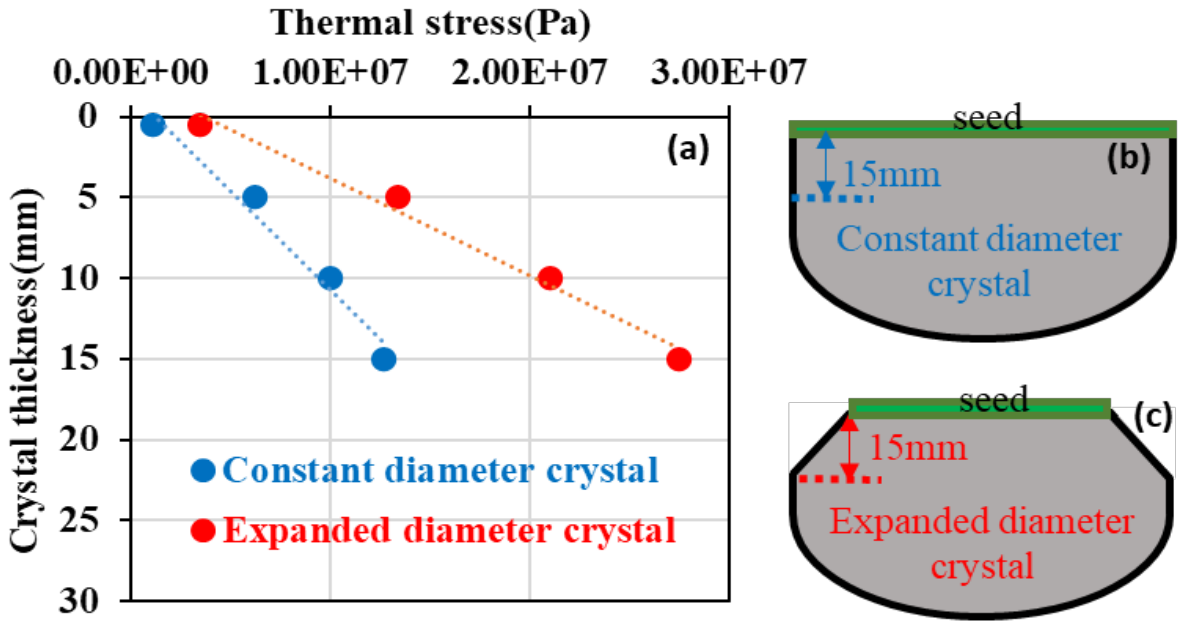

Fig. 1. (a) Calculation of SiC crystal thermal stress under different conditions, (b) Constant diameter crystal growth, (c) Diameter expansion crystal growth.

Based on the discussion above, the design of the crystal diameter expansion process is schematically shown in Fig.1. Different from the constant diameter crystal growth (CDCG) process, a tilt angle in the crystal growth periphery and corresponding RTG is designed in expanded diameter crystal growth (EDCG), which allows crystal diameter to increase gradually at the start of crystal growth. Fig.1 (a) shows the calculation of the crystal thermal stress of CDCG and EDCG process (Fig. 1 (b) and (c)), respectively. Calculation results indicate that crystal thermal stress increases with thickness due to

the stress accumulation. Even with the same temperature gradient, SiC crystal diameter expansion process has larger thermal stress in the crystal from the start of the growth, and its stress increasing rate is almost twice of that in constant-diameter grown crystal. The calculation result in Fig.1 indicates that the maximum RTG should be carefully designed according to the thermal stress in the diameter expanded crystal.

In order to further illustrate the correlation between lateral driving force and crystal thermal stress, different RTG curve is set on the seed surface to perform diameter expansion growth by modifying the thermal field structure (Fig. 2 (a)). Thermal stress distribution in grown crystal and corresponding RTG curve along the radial direction on the seed crystal surface are shown in Fig.2(b) and (c) respectively. As shown in Fig. 2 (c), RTG value is larger at the edge of crystal compared to that in the center, which facilitates the lateral growth of the crystals while maintaining relatively small stress in the entire crystal lattice. Calculated thermal stress in the diameter expansion area of the crystal is marked by the red dotted box in Fig. 2 (b), which is in good correlation with the plot of the RTG.

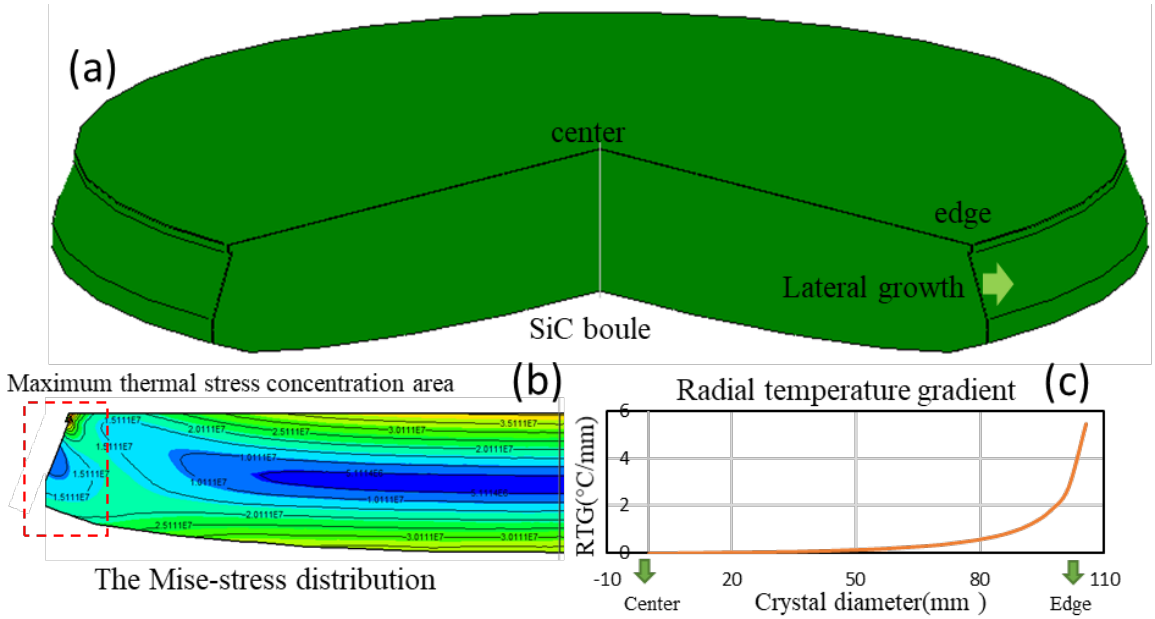

Fig. 2. (a) Schematic illustration of diameter expansion crystal. (b) The distribution of the thermal stress in crystal. (c) The RTG curve at seed crystal surface.

A suitable temperature gradient range should be derived for various diameter of crystals. A tougher balance between temperature gradient and crystal stress is speculated with diameter increasing in the EDCG process, as shown in Fig.3 (a). The best scheme for a successful crystal diameter expansion process should be the optimized combination of enlarged diameter, temperature gradient and thermal stress. Theoretically, free-style crystal growth without constraints in diameter is beneficial for both diameter enlargement and quality control. However, the actual situation is that hexagonality of 4H-SiC and the distribution of vapor species lead to much larger convexity, causing larger thermal stress with less effective diameter enlargement. To resolve this problem, a tilt graphite ring which guides crystal growth with expansion angle (θ) is applied. The correlation between θ and thermal gradient can be expressed as,

$$\theta = \arctan\frac{G_r}{G_a}. \tag{1}$$

where G_r is radial temperature gradient and G_a is axial temperature gradient. During crystal growth, the expansion angle θ depends on the other two parameters and a desirable diameter increase can be realized by adjusting G_r and G_a. Furthermore, curve fitting between the increase of crystal diameter (d) and temperature gradient (G_r/G_a) in Fig.3 (b) is derived as below,

$$d=115.3\frac{G_r}{G_a}-13.16. \quad (2)$$

When the G_r/G_a value exceeds 0.25, an effective monocrystal diameter expansion larger than 15mm can be achieved. which comes with a high ratio of crystal breakage (Fig. 3 (c)) caused by thermal stress. If G_r/G_a value is set to be smaller than 0.15, insufficient driving force will lead to extremely slow monocrystal growth in radial direction, resulting in polycrystal deposition (Fig.3(d)). Then, RTG value in the range of 0.7~2.4 °C/cm and diameter expansion angle in the range of 8°~14° is set up. In order to achieve desirable crystal diameter expansion at different process nodes, it is important to design corresponding crucibles, insulation structures and RTG values based on the achieved diameter of the seed crystal. Therefore, various thermal field structures and temperature gradient are designed for each iteration of EDCG process.

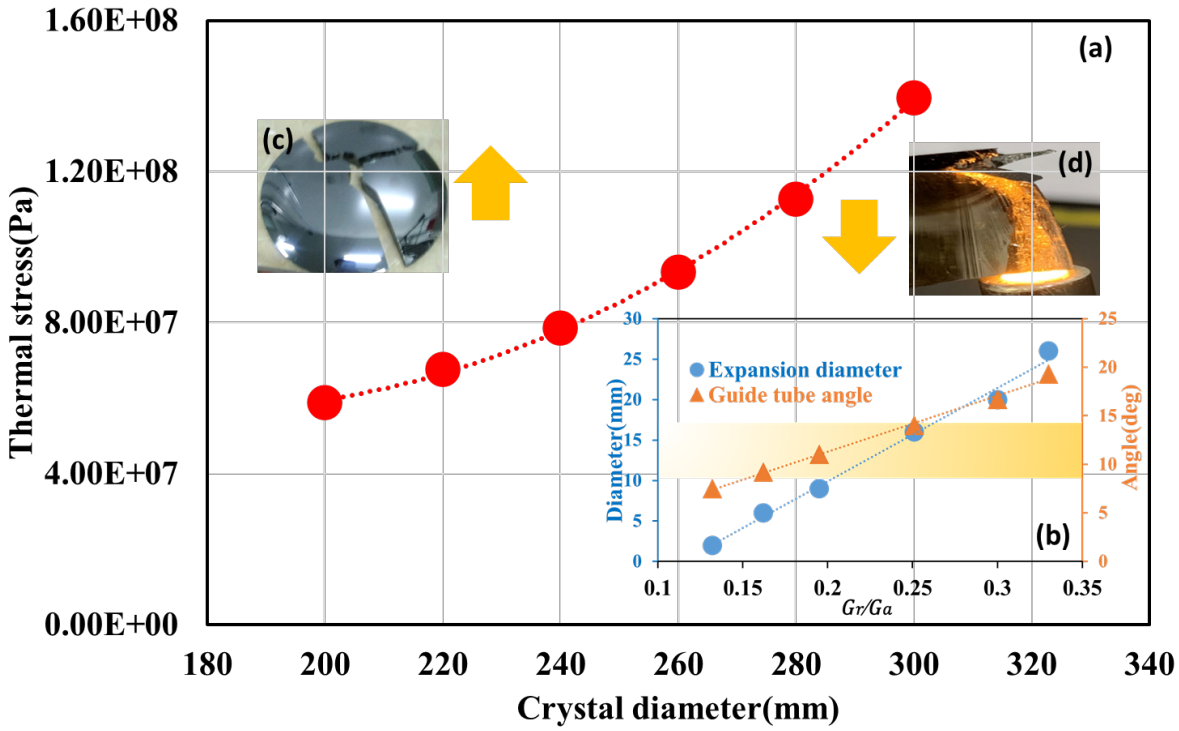

Fig. 3. (a) Calculation of the thermal stress during crystal expansion process. (b) The correlation between the effective expansion diameter of the crystal, the edge G_r/G_a value and guide tube angle. (c) Cracked crystals. (d) Crystal peripheral region with failed expansion.

Based on theoretical analysis and crystal growth experiments, SiC crystal diameter is successfully enlarged from 200 mm to 300 mm. Each generation of expanded SiC crystal, fabricated SiC substrates and the eventually obtained 300 mm SiC crystal and substrate are shown in Fig.4. As the 300 mm SiC seed substrates are ready, CDCG process is carried out for 300 mm SiC crystal growth. As shown in Fig. 5, high-purity and conductive n-type & p-type SiC substrates are successfully fabricated, which covers all series of SiC substrates and all areas in which SiC material can be adopted.

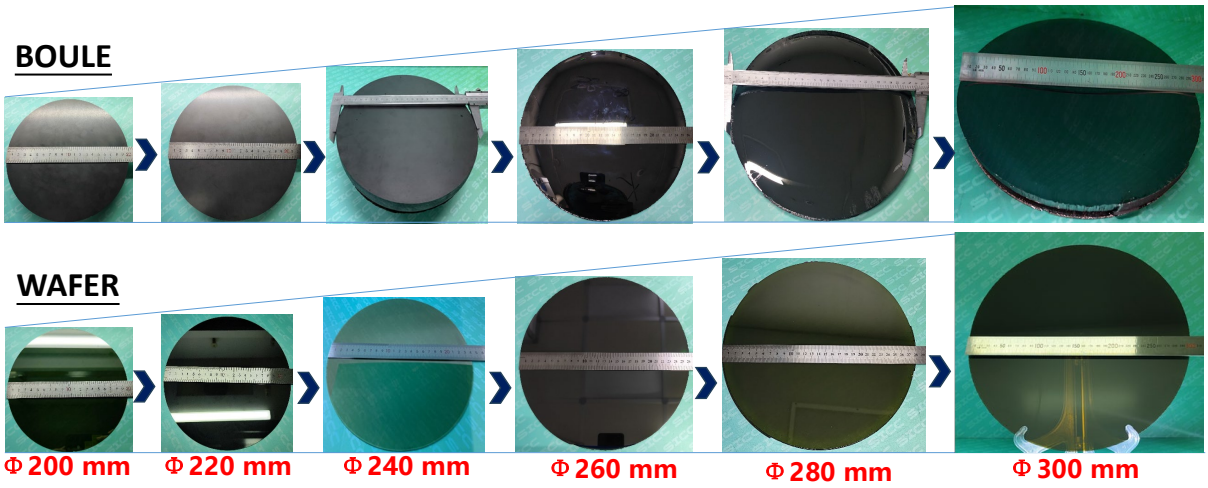

Fig. 4. SiC crystals and corresponding substrates with various diameter during expansion process.

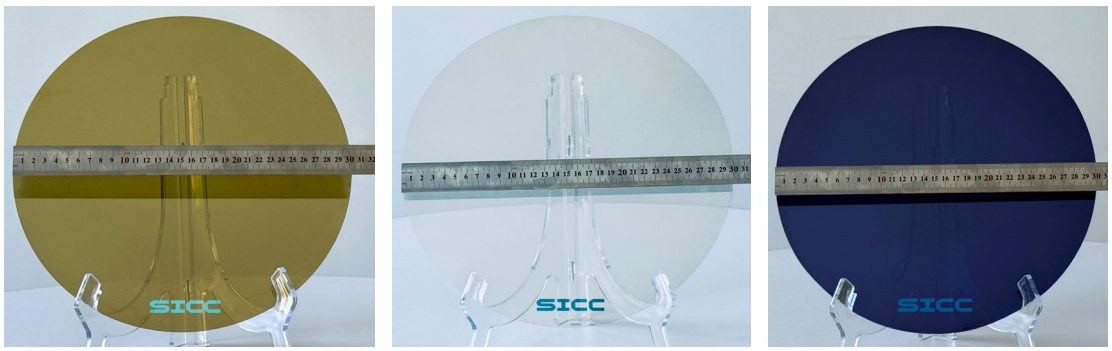

Fig. 5. Images of fabricated 12 inch n-type SiC substrate, 12 inch high-purity SiC substrate and 12 inch p-type SiC substrate.

The polytype and thermal stress of the 300 mm SiC substrate are characterized using micro-Raman. Fig. 6 (a) shows a FTO (2/4) E2 peak locates at 776-777 cm^{-1}, which is the typical 4H-SiC crystal. Mapping data (Fig. 6 (b)) of the whole 300 mm substrate shows consistent 776.5 cm^{-1} Raman spectrum peak, indicating that the 300mm SiC substrate is 100% 4H polytype. Additionally, negilible peak shift is observed at 776.5 cm^{-1} in the Raman spectrum, indicating that the thermal stress is small in 300 mm substrate since Raman spectrum is sensitive to the crystal lattice strain[12]. Calculation from the spectrum shift results in a typical stress value of 3.5 MPa, which is comparable to the current 200 mm SiC substrates. Fig. 6 (c) further shows the threading screw dislocation (TSD) density characterization by XRT. Average micropipe and TSD density in the 300 mm n type 4H-SiC substrate is measured to be less than 0.05 cm^{-2} and 120 cm^{-2}, this almost replicates the TSD density of initial 200 mm 4H-SiC seed substrate when diameter expansion process started, which further shows the diameter expansion process is well designed without deteriorating the initial crystal quality.

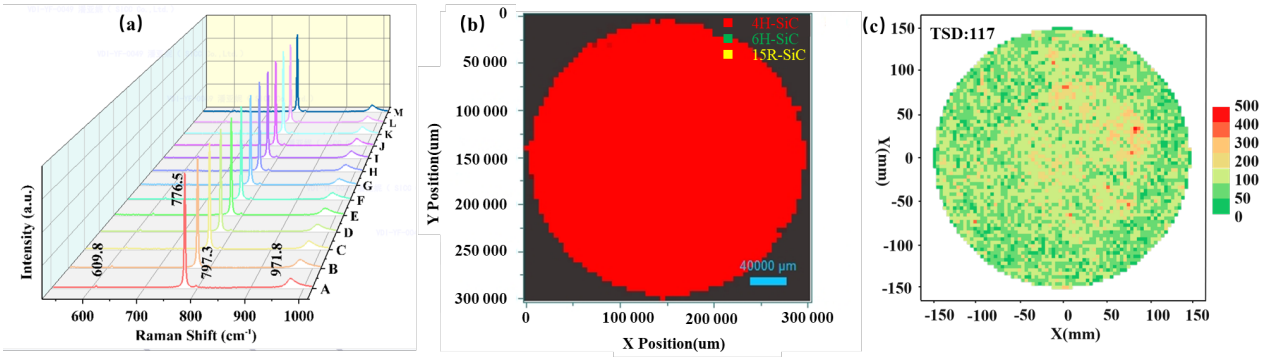

Fig. 6. Raman and XRT characterization of the 300 mm 4H-SiC substrates.

One concern of 300 mm SiC substrates is the warpage increase. Fig. 7 shows the typical values of fabricated 750 μm SiC substrates, in which the bow and warp values are controlled well below 10 μm and 30 μm, respectively. Besides, the total thickness variation (TTV) value is controlled below 6 μm, which indicates applicable substrates for device fabrication process.

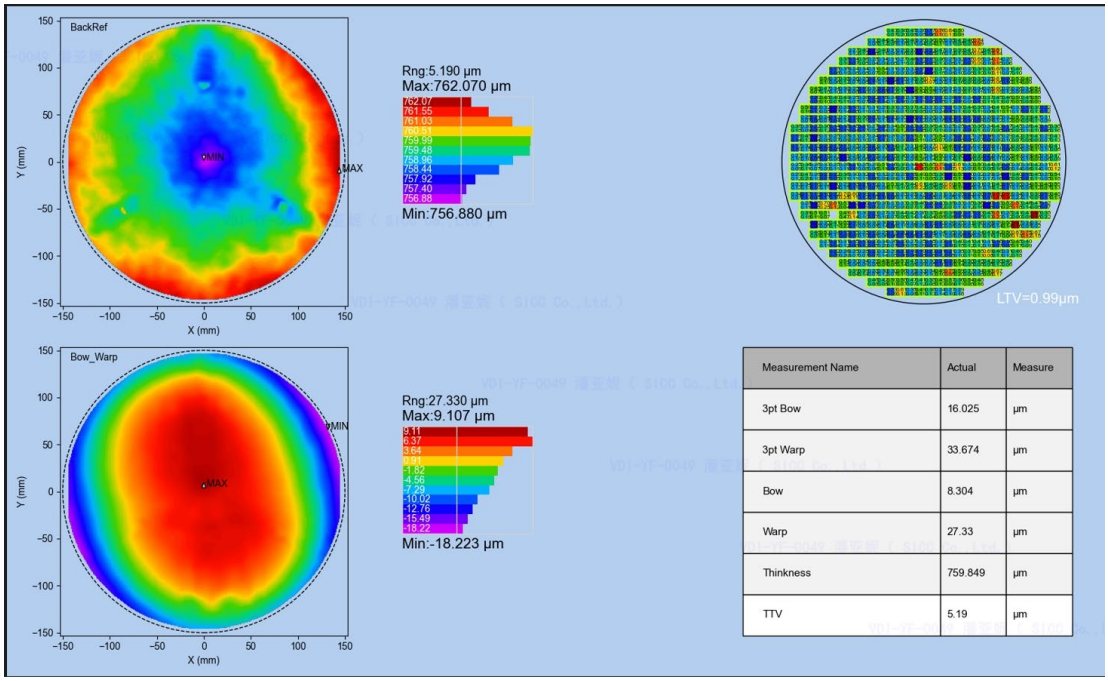

Fig. 7. Wafering quality characterization of 750 μm thickness 300 mm 4H-SiC substrates

Summary

The first 300 mm 4H-SiC substrates is demonstrated in November 2024 by SICC. Detailed diameter expansion process of 4H-SiC crystals from 200 mm to 300 mm is shown in this paper. Correlation between the temperature gradient and thermal stress as well as the process window for successful diameter expansion process is elaborated based on simulation and experiments. Based on the expansion obtained 300 mm 4H-SiC seed, high quality 300 mm high-purity and conductive n-type & p-type SiC substrates are subsequently fabricated. The 300 mm SiC substrate is 100% 4H polytype with TSD density below 120 cm^{-2}. Substrates with both thickness 500 μm and 750 μm are fabricated with bow and warp values smaller than 10 μm and 30 μm, respectively. High quality of 300 mm SiC substrates will support future development of power devices, waveguide in AR glass as well as thermal management in advanced packaging.

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