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Evaluation of the change in properties caused by axial and radial temperature gradients in silicon carbide crystal growth using the physical vapor transport method

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Abstract

In the process of growing single-crystal silicon carbide (SiC) using the physical vapor transport method, the most sensitive and important factor is temperature. The occurrence of a temperature gradient inside the growth cell acts as a driving force for SiC crystal growth. Temperature gradients are also a significant parameter for determining both the growth rate and the quality of the resultant crystal. In addition to the overall temperature gradient in the growth cell, axial and radial temperature gradients can appear within the SiC crystal ingot. We analyzed changes in thermal stresses caused by these temperature gradients according to their location within the SiC crystal ingot. Using simulation and nanobeam diffraction analysis, we confirmed that thermal stresses, including tensile and compressive stresses, differed in accordance with their location inside of the SiC ingot. In particular, large compressive stresses were observed near the edge of the wafer that was sliced close to the SiC ingot growth interface. We also analyzed the effect of the location within the SiC ingot on crystallinity and the occurrence of defects as a function of thermal stress. We confirmed that lower crystallinity and higher dislocation density not only reduce the SiC bandgap, but also increase the resistivity and diminish the electrical properties of SiC.

Keywords: Temperature gradient; Thermal stress; SiC crystal ingot; Dislocation density; Electrical properties

1. Introduction

Silicon carbide has 200 polytypes and shows different characteristics depending on the crystal structure and the stacking period. Representative stable phases are 3C, 4H and 6H SiC. As each crystal phase has different electrical properties (e.g. energy bandgap and electron mobility), each phase is best suited for different applications. In particular, α phase 4H SiC has excellent bandgap energy and breakdown voltage, relatively high electron and hole mobility, and high thermal conductivity. Therefore, if used in SiC semiconductors, it has the advantages of low cost and high efficiency. 4H SiC has the potential to replace existing Si semiconductor materials for use under high-temperature and high-voltage atmospheres [1–3].

A commonly used method to grow single-crystal 4H SiC is the physical vapor transport (PVT) method which involves sublimation of a powder. However, this method is complicated and requires precise control due to the large number of parameters that can affect the growth of single-crystal SiC. In an attempt to overcome this disadvantage, high-temperature chemical vapor deposition using gas-phase precursors and liquid-phase epitaxy have been studied. Unfortunately, there are disadvantages associated with these processing techniques. Namely, high facility and

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processing costs, lower solubility of C in Si liquid, and the formation of impurities by parasitic phases [4–6]. Despite these aforementioned drawbacks, PVT is the method most frequently used to grow single-crystal 4H SiC. Its high growth rate and ability to grow large-diameter crystals make PVT relatively efficient and easy compared to other processing techniques.

When using the PVT method to grow single-crystal SiC, the most important parameter is temperature. During this process, both sublimation of the source powder and crystal growth from seed occur simultaneously in the crucible; a temperature gradient exists between these two parts. Due to this difference in temperature, the growth rate and the thermal stresses in these two regions are dissimilar. Consequently, the defect density of the crystal being grown increases. However, if the temperature gradient is decreased to reduce dislocations, the growth rate is also diminished. This causes a smaller crystal boule to develop, which leads to a reduction in the SiC yield [7,8]. Therefore, close attention is required when the temperature gradient is set. Additionally, the temperature gradient varies according to the growth time. This is a variable that is difficult to control. Finally, the shape of the crystal ingot growth interface can become either convex or concave according to the temperature gradient [9].

To determine the impact of the temperature gradient on the SiC crystal ingot during growth of the single-crystal SiC using the PVT method, we studied differences in the properties according to the growth stages of the SiC crystal ingot. This was accomplished by examining different locations within the ingot. To research only the impact of the temperature gradient, we examined the middle portion of the crystal ingot and the part of the ingot before deformation of the growth interface. The initial growth region was excluded because this area can be affected by defects and contamination of the seed material. Using simulation modeling and nanobeam electron diffraction (NBD), we verified the types and degrees of thermal stresses according to axial and radial locations within the ingot. We also analyzed the correlation among thermal stresses, crystallinity and dislocation density of the SiC crystal. We determined the impact that these variables had on the electrical properties of SiC including the bandgap and resistivity.

2. Experimental procedure

To grow single-crystal 4H SiC by the PVT method, we used purified β-SiC powder as the source powder; the purity of the β-SiC powder was increased through a purification process that removes impurities within the powder. Due to its low melting temperature the β-SiC powder offers a lower growth temperature. This source material has been used previously [10]. For the seed, we used 4° off-axis 4H SiC. Crystals were grown at 1900 °C under 10 torr of pressure at a growth rate of 250 μm h^{-1}. 4H SiC crystals were sliced approximately perpendicular to the growth direction to form (0 0 0 1) Si wafers. We used a finite-element method simulator program ABAQUS, applying temperature displacement type mode, and the physical parameters were density, Young's modulus, Poisson's ratio and the thermal expansion coefficient to project the changes in thermal stress of the crystal according to the temperature gradient caused during the growth of single-crystal SiC. Using the NBD mode of a high-resolution transmission electron microscope (JEOL JEM 2100F), we analyzed changes in the thermal stress according to the temperature gradient. The crystallinity of the single-crystal SiC was analyzed using high-resolution X-ray diffraction (PANalytical XPert-PRO MED). We measured the ω rocking curves and calculated full width at half maximum (FWHM) values. To confirm the polytype of the crystal, a Raman spectrometer (Jobin-Yvon LabRam HR) with an excitation wavelength of 514.5 nm (Ar laser) was used. To study variations in dislocations as a function of thermal stress, the dislocation density distribution was verified using an automated optical microscopy system. Using UV-Vis-NIR spectrophotometry (JASCO V-570), we measured the transmittance of single-crystal 4H SiC wafers and calculated the bandgap. Additionally, a Hall effect measurement system (Ecopia HMS3000) was utilized to measure the electrical resistivity of single-crystal 4H SiC wafers.

3. Results and discussion

3.1. Thermal stress by location in the SiC crystal ingot

Fig. 1a and c show images taken under transmitted visible light of the middle portion of the SiC crystal ingot and the growth interface region, respectively. These points are marked A, B and C according to their location on the wafer surface. Fig. 1b and d show the results of simulated changes in the thermal stresses of the wafers using ABAQUS. Positive values refer to tensile stresses and negative values indicate compressive stresses. Although both wafers are in tension at their centers and in compression at their edges, there is a significant difference between the stress distributions of the two wafers. Fig. 1b shows that tensile stresses were dominant overall, while compressive stresses were shown locally along the edge of the wafer. On the other hand, Fig. 1d shows compressive stresses in the overall peripheral area of the wafer except at the center. The distinctive difference of the stress distributions in the wafers is caused by differences in the axial and radial temperature distributions. These temperature differences are larger in wafer (c) because it was located near the growth interface where the temperature gradient between the growth interface and the powder increases as the SiC crystal ingot lengthens [11]. Due to this, the growth rate between the center and edge parts of the wafer are not uniform. This causes the wafer to grow in a convex shape, leading to the development of compressive stresses on the peripheral areas of the wafer. Based on the results of the thermal stress distribution derived from simulation, we compared and analyzed the thermal stresses of wafers (a) and (c).