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Solution Growth Technique of Silicon Carbide with *In Situ* Observation

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Abstract. In the solution growth method for silicon carbide (SiC) single-crystal fabrication, in-situ observations were performed inside the furnace to monitor the meniscus at the seed–solution interface. A meniscus formed at the contact between the seed crystal and the solution, and variations in the reflections on the solution surface enabled optical monitoring and control of this interface. The observed surface images were also dependent on the frequency of the induction heating. Computational fluid dynamics (CFD) simulations indicated that lowering the heating frequency causes an upward displacement of the solution surface at its central region, producing a locally elevated contact position between the seed crystal and the solution. These findings demonstrate that in-situ observation constitutes an effective approach for precise control of meniscus shape during solution growth of SiC single crystals.

Introduction

Silicon carbide (SiC) is expected to play an increasingly important role in power devices owing to its superior electrical and physical properties compared with silicon (Si). However, various crystal defects in SiC adversely affect device performance and reliability [1]; therefore, reduction of defect density is required. Solution growth is considered to suppress defect formation because it proceeds near thermodynamic equilibrium at the growth interface, and it has thus been applied to SiC crystal growth [2]. Furthermore, incorporation of aluminum (Al) into the solution enables relatively facile p-type heavy doping for SiC single crystals, making solution growth advantageous for producing Al heavy doped p-type SiC substrates required for high-voltage devices such as insulated gate bipolar transistors (IGBTs) [3].

In SiC solution growth, the meniscus formed at the solution-crystal interface significantly influences crystal shape and growth kinetics [4]. In the top-seeded solution growth (TSSG) method, a Si-based alloy solution is contained in a graphite crucible, and a SiC single crystal seed is brought into contact with the solution to grow the SiC single crystal. In this method carbon for SiC growth is supplied by dissolution of the graphite crucible; as the crucible wall dissolves into the solution, carbon is transported to the seed via the solution. Consequently, the inner wall of the graphite crucible is eroded during the growth period. Moreover, because the solution is held at approximately 2000 °C well above the melting point of Si and the other metal elements, an evaporation of the solution is inevitable. For the reasons mentioned above, the solution surface is expected to change continuously throughout the growth period. Because crystal growth proceeds simultaneously, understanding solution behavior is crucial to maintaining precise control of the meniscus over prolonged growth durations.

However, maintaining the solution at around 2000 °C requires enclosing the crucible within an insulating hot zone, which complicates direct observation of the crucible interior. Previously, X-ray transmission imaging inside the furnace was explored to determine the solution shape [5]; however, the required apparatus is too complex for practical manufacturing equipment. Accordingly, this study investigated the feasibility of observing the solution state inside the crucible and conducted preliminary crystal growth experiments with in-situ observations.

Experimental Methods

As shown in Figure 1, an observation port (30 mm × 40 mm) was provided at the top of the hot zone in the induction-heating furnace, and the interior of the hot zone was imaged in real time by a camera positioned outside the chamber. The camera was positioned at an angle of several degrees to the vertical axis of the chamber so that it could observe the area near the contact between the seed crystal and the solution. The camera was equipped with an ND filter to observe high-temperature environments around 2000°C. A common Si-40% Cr alloy was used as the solution and contained in a graphite crucible. The solution was heated to 2000 °C using mid-frequency induction heating, after which it was brought into contact with the seed crystal. The furnace atmosphere was argon at atmospheric pressure. The crystal growth time was set to 20 hours.
A 4H-SiC seed crystal was bonded to a graphite holder. Contact between the seed crystal and the solution was performed manually while monitoring the in-situ images. Note that rotation of the seed crystal and crucible was not performed in order to prioritize the in-situ observation.

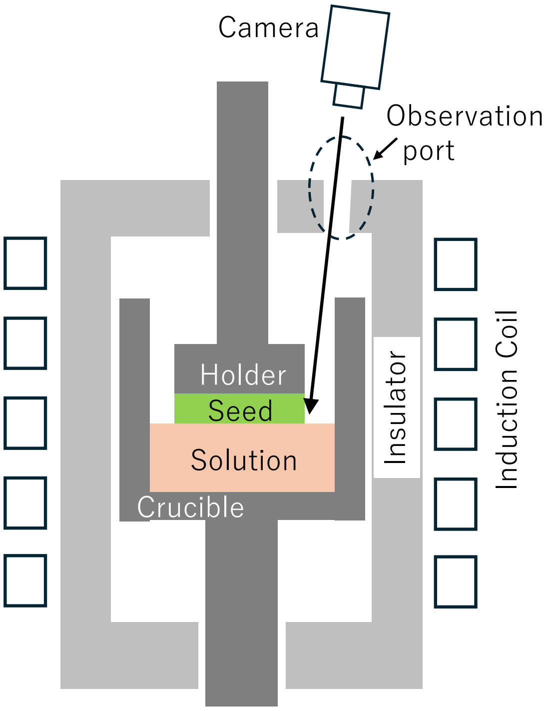


Fig. 1. Solution growth apparatus with an in-situ observation system.

Computational fluid dynamics (CFD) simulations were also performed to elucidate the solution free-surface and the solution flow behaviors. Simulations were carried out using STAR-CCM+ (Siemens Digital Industries Software), and the volume-of-fluid (VOF) method was employed to simulate the free surface of the solution. Because induction heating was used, surface tension and Lorentz forces arising from the electromagnetic field were included in the calculation of the free-surface shape. Regarding the wetting angle between the solution and the graphite crucible wall, a value of 45° was used in the present simulation, corresponding to the wetting angle between Si melt and SiC [6]. This choice is based on the assumption that the crucible wall reacts rapidly, resulting in the formation of SiC on the crucible surface.

Results and Discussion

As shown in Figure 2(a) and (b), the solution surface is presented for a relatively high induction heating frequency and after contact between the seed crystal and the solution, respectively. Owing to the high reflectivity of the Si-Cr alloy solution, the solution surface was clearly visible. In Figure 2(a) the surface remained nearly stationary; however, when the seed was brought into contact with the solution, the reflected image changed markedly (Figure 2(b)), with the observation port aperture mirrored on the solution surface. This change results from meniscus formation upon contact, which substantially alters the surface curvature. Moreover, the inverted appearance of the aperture in Figure 2(b) indicates a concave meniscus with relatively small curvature. As the seed crystal was continuously lowered, this meniscus eventually disappeared, and the solution climbed up onto the graphite holder. Therefore, by observing this meniscus, the contact condition between the seed crystal and the solution can be precisely controlled. We consider that maintaining this contact condition throughout the entire crystal growth process is a critically important technique.

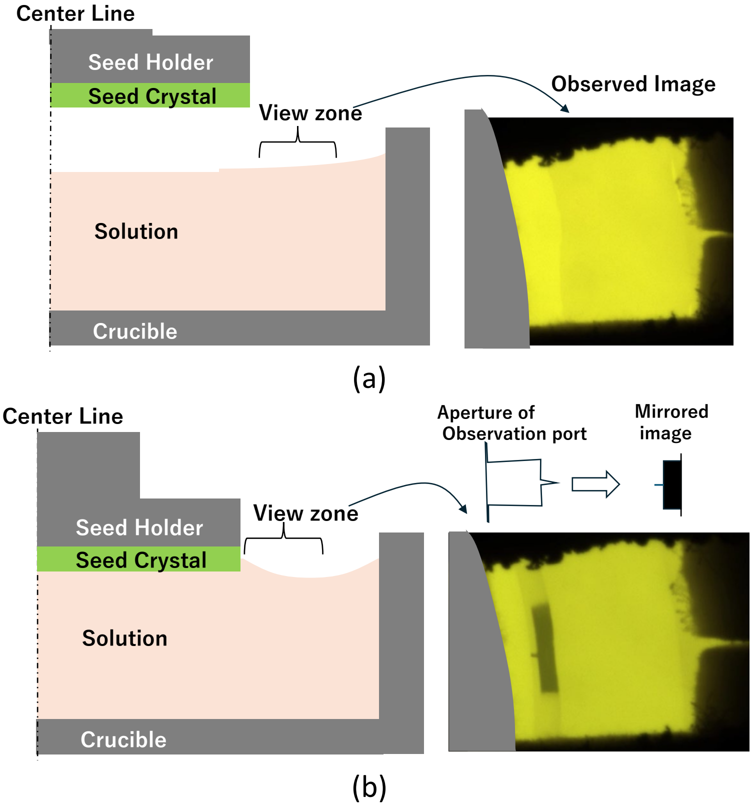
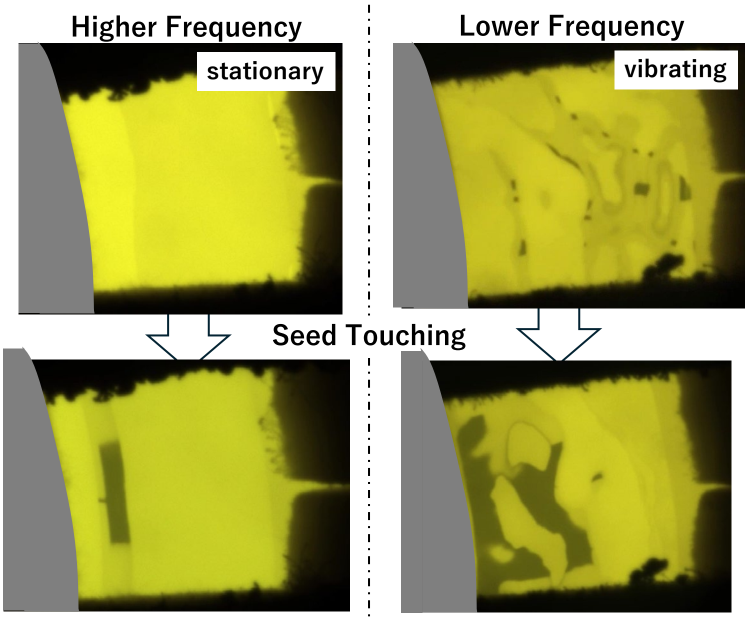


Fig. 2. (a) Image of the solution surface (yellow-shaded region) before contacting the seed. (b) Image after contact; a reflected image of the observation port (dark shape within the yellow region) appears on the solution surface. A schematic diagram of the reflected image is shown in the upper right.

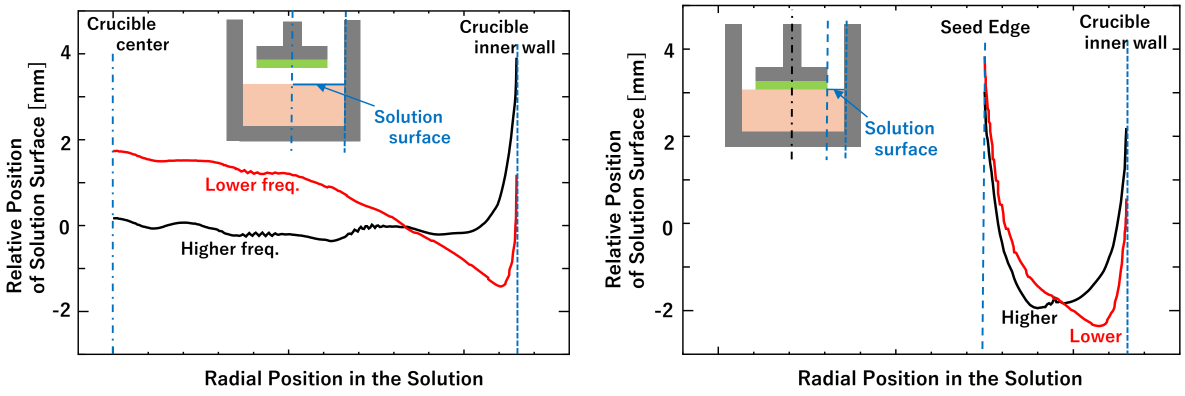


(a) (b)

Fig. 3. Before (upper images) and after (lower images) touching between the seed and the solution. (a) In case of higher frequency, the solution surface is stationary. (b) In case of lower frequency, the solution surface is vibrating.

Next, we investigated the effect of changing the induction heating frequency to one-third of the higher frequency; the results before and after the change of the induction heating frequency are summarized in Figure 3 (a) and (b), respectively. At the lower frequency the solution exhibited clearly oscillations and the observed image became highly vibrating. Nevertheless, contact between the seed and the solution could still be identified from small changes in the image. We confirmed that contact between the seed crystal and the solution can be observed regardless of the induction heating frequency. Lowering the frequency also caused the seed-solution contact to occur at a higher position. In the next paragraph, we will use CFD simulations to explain the vibration of the solution and that difference in contact position.

According to the CFD simulations, in our case, the dominant factor in solution flow was the Lorentz force due to the induction heating. At lower frequency in the induction heating, the velocity of this flow was several times greater than at higher frequency. Therefore, we believe that the magnitude of the solution agitation caused the vibration. Simulation results prior to the contact are shown in Figure 4(a) and those after the contact in Figure 4(b). The vertical axis in Figure 4 denotes relative displacement (initial solution surface set to zero), and the horizontal axis denotes distance from the crucible center. Solution surface profiles at the two frequencies are plotted as black (higher frequency) and red (lower frequency) curves. In Figure 4(a), the lower frequency profile exhibits a central upward rise, indicating a higher contact position of the seed-solution than that of the higher frequency one. Lorentz forces at lower frequency act more uniformly on the solution, raising its central region. This behavior is attributed to increased electromagnetic skin depth at lower frequency, which reduces power dissipation in the crucible and allows the electromagnetic field to be applied to the solution more efficiently. As described above, differences in the solution surface shape occur depending on the induction heating frequency, so it is important to directly observe the contact state between the seed crystal and the solution. In particular, since this method uses a large-diameter wafer as a seed crystal, if the contact between the seed crystal and the solution is only partial, the possibility of voids formation increases.

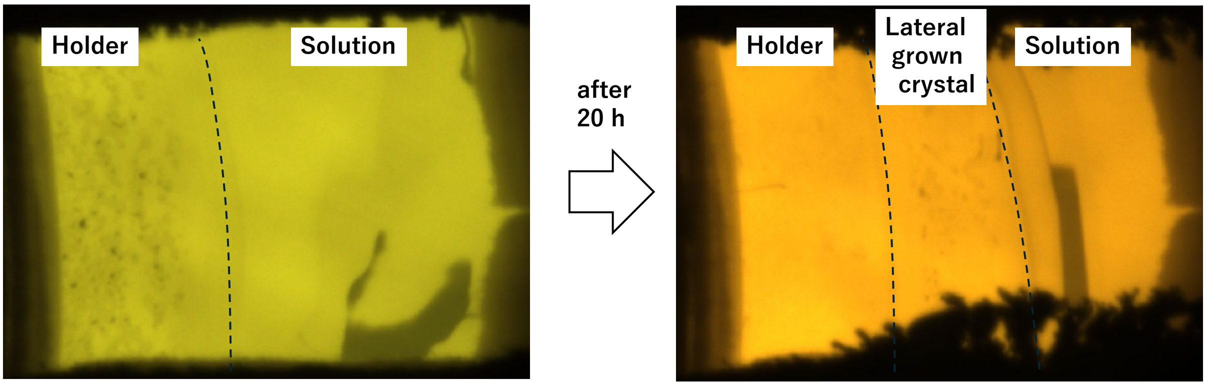


(a) (b)

Fig. 4. Simulation results of solution surface profiles. (a) Before and (b) after the solution contacts the seed.

This paragraph describes the influence of these differences in meniscus shapes on crystal growth. At lower frequency, the meniscus curvature is larger than at higher frequency, which likely affects heat removal from the meniscus. This process operates at temperatures around 2000 °C, and thus heat removal from solid or liquid surfaces is considered to be dominated by radiative heat transfer. As shown the red line in Figure. 4(b), at lower frequency, the meniscus surface is oriented further upward on the crystal side compared with that at higher frequency. Consequently, the radiative energy emitted from the meniscus at lower frequency is directed upward toward the crucible wall. In contrast, as shown the black line in Figure. 4(b), the meniscus surface on the crystal side is oriented more horizontally at higher frequency. As a result, the radiative energy emitted from the meniscus is directed downward compared with the lower frequency case. The crucible exhibits a vertical temperature distribution depending on the relative positions of the crucible and the induction coil. Therefore, changes in the relative angle between the meniscus surface and the crucible wall alter the amount of heat extracted from the meniscus, which is considered to lead to differences in the lateral growth rate of the crystal. We believe that optimal growth conditions may depend on the induction heating frequency.

Figure 5 presents images of crystal growth recorded during the experiment; lateral crystal growth was observed in real time. Figure 5(b) shows the results 20 hours after Figure 5(a). Figure 6 shows a comparison of the crystal with the graphite holder obtained after the experiment and the image during crystal growth. As SiC crystal had grown about 3 mm in the lateral direction, the adjacent solution region shifted laterally. The crystal grows laterally, causing part of the crystal surface to become exposed above the solution. The emissivity of SiC differs from that of the solution surface, with the SiC surface exhibiting a higher emissivity. Consequently, significant heat removal due to thermal radiation occurs at the SiC crystal surface exposed from the solution surface, raising concerns about an increased crystal growth rate in that region. If the crystal growth rate becomes excessive, there is a high likelihood of inducing undesired crystal growth directions or polycrystallization. We believe that such phenomena can be prevented in advance through in situ observation. In other words, maintaining the required crystal shape becomes possible, which is expected to contribute to an improvement in the crystal growth yield. On the other hand, a comparison of the solution surface conditions shown in Figures 5(a) and (b) reveals that fluctuations of the solution were suppressed as the crystal expanded laterally. This behavior is presumed to result from a reduction in the free surface area of the solution. In future work, we plan to investigate changes in the surface condition as a function of solution composition.



(a) (b)

Fig. 5. (a) Initial state of the crystal growth and (b) after 20 hours of the crystal growth.

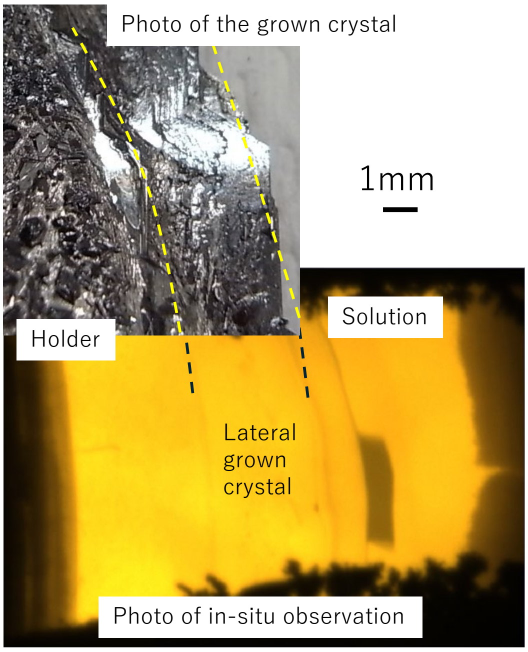


Fig. 6. Photograph of the graphite holder and the grown crystal edge with the in-situ observed image(under).

Summary

As a result of employing in-situ observation for SiC crystal growth by the solution method, changes in the solution surface morphology led to variations in the reflected images, enabling observation of the contact state between the seed crystal and the solution. The solution surface geometry was found to vary with the frequency of induction heating, which was also confirmed by numerical simulations. The contact state between the crystal and the solution not only changes continuously over time due to factors such as crucible erosion and solution evaporation, but also depends strongly on the frequency of induction heating. Furthermore, it was pointed out that changes in the meniscus shape alter the radiative heat transfer between the solution surface and the crucible wall, indicating the potential for a significant impact on crystal growth. From these results, in-situ observation can be regarded as a useful tool for maintaining the meniscus formed between the solution and the crystal over long periods.

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