Technical Report

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Development of High Quality 4H-SiC Single Crystal Wafers Grown by Solution Growth Technique

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Abstract

As silicon carbide (SiC) has potential for application to power devices with significantly lower power losses compared to conventional Si devices, it is garnering increased attention as a next-generation semiconductor for realizing considerable energy-saving and CO_2 reduction. However, it is difficult to grow a perfect single crystal; this has long been one of the main reasons hindering real SiC power device application. This paper introduces our newly developed SiC solution growth technology. In particular, we have succeeded in suppressing solvent inclusions, which have been a serious technological problem unique to the solution growth method. We have fabricated 2-inch diameter 4° off-axis 4H-SiC wafers and evaluated their crystal defects. As a result, the superior quality of our solution-grown crystal was confirmed.

1. Preface

Silicon carbide (SiC) is positioned as a front-runner semiconductor for the next-generation of power devices. In both areas of material development and device development, R&D in pursuit of the practical use of the compound is progressing favorably. Contrary to silicon (Si), which is the prevalent material of current power devices, SiC has advantageous properties to realize a power device with ultralow power loss. Such a device will significantly contribute to the reduction of CO₂ and other greenhouse gas emissions considered as a cause of global warming. In order to elicit the intrinsic capabilities of SiC power devices, as well as to produce them at low cost for a wide range of uses in society, high quality and large diameter SiC single crystal wafers are required. Today's SiC single crystal production method mainly involves the sublimation and recrystallization in order to obtain large diameter wafers, the control of the electrical properties, the crystal defect control, and the optimization of productivity.¹⁾ Using the method, Nippon Steel & Sumitomo Metal Corporation has developed a wafer 150 mm (6 inch) in diameter.²⁾ In recent years, a manufacturer in the United States has announced that it produced a 200 mm (8-inch) in diameter wafer experimentally.3)

The improvement of the sublimation growth technique has long been carried out to suppress the occurrence of crystalline defects by improving and optimizing the crystal growth process. Dislocation defects exist in the order of 103/cm2 in a grown crystal.4,5) However, since the crystalline quality generally tends to deteriorate when a single crystal has been grown to have a larger diameter, the large diameters of single crystal wafers have not been obtained together with high quality. Against this backdrop, the development of methods of crystal growth other than that involving the sublimation and recrystallization is underway for the purpose of obtaining an ultrahigh-quality SiC single crystal.^{6,7)} One of such methods is the solution growth method. The solution growth method involves growing a single crystal under the condition close to a thermodynamic equilibrium and is expected to grow single crystals with a significantly low defect density. Due to this, the method presents the prospect of itself evolving into a method of crystal growth playing a role of complementing the sublimating and recrystallizing method and vice versa. R&D of the solution growth method is under way mainly in Japan. This paper outlines Nippon Steel & Sumitomo Metal's SiC single crystal growing technology based on the solution growth method together with the recent development.

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2. Growing SiC Single Crystals Using the Solution Growth Method

This chapter explains the basic technologies of the SiC single crystal growth implemented in Nippon Steel & Sumitomo Metal using the solution growth method. First, the selection of a solution suitable for growing SiC single crystal is described, and then the current technologies for bulk crystal growth and the conductivity control are outlined.

2.1 Selection of the solution

No SiC melt with the same chemical composition as that of solid state SiC can be obtained at normal pressures. In principle, SiC melt growth by solidification is not possible, contrary to Si and GaAs (gallium arsenide) semiconductors or oxide single crystal materials. However, solution growth of SiC is possible using the supersaturation as the driving force by dissolving SiC in a metal solvent. For the crystal growth under the solution growth method, liquidus lines (solubility curves) on phase diagrams are used. The most popular solvent in SiC solution growth is Si. Figure 1 shows a Si-C binary phase diagram.⁸⁾ However, in general, the growth rate of SiC is extremely slow such as 10 μ m/h or less, since the amount of carbon that dissolves to Si melt is as small as 1 at% or even less at 1800°C. For this reason, while several basic researches on the SiC crystal growth from a Si solvent were conducted as a method for growing thin film crystals from 1960 to 1970, this method never developed into a technique to obtain bulk single crystals.

In recent years, in order to increase carbon solubility in a solution, various alloy solvents using Si and transition metal have been proposed for use in growing bulk crystals.^{9–11} In order to select a multi-component solvent suitable for SiC crystal growth, elements and their relative proportions were carefully examined to find a liquid phase that reaches equilibrium with the SiC compound. We selected Si-Ti and Si-Cr solvents from among many candidates, considering the ease of handling at high temperatures, availability of high purity materials, abundance of natural resources, low probability of inclusion in the SiC crystal, etc. Using these solvents, the growth rate reaches from several hundred μ m/h up to 2 mm/h.¹² At present, we are working on the technology for growing 4H-SiC bulk single crystals suitable for power devices using the top-seeded solution growth (TSSG) method described later.



Fig. 1 Calculated Si-C binary phase diagram together with experimental liquidus compositions⁸⁾

2.2 Growing a bulk single crystal

2.2.1 Structure of the solution crystal growth system

Figure 2 shows a schematic view of the system used for growing a SiC crystal from a solution using the TSSG method. A Si-Ti or Si-Cr alloy solvent material is put in a graphite crucible, and is melted by high frequency induction heating under an atmospheric pressure of helium. Si is supplied from the solvent to the solution. while carbon is supplied from the inner wall of the graphite crucible that dissolves into the solution. The 4H-SiC seed crystal is attached to the end of a graphite shaft (crystal holding shaft). This shaft is made to descend to be in contact with the solution surface. The crucible and crystal holding shaft are rotated as necessary. A temperature difference is created in the vertical direction of the solution so that the temperature in the vicinity of the seed crystal is lower than the other parts of the solution. When carbon contained in the hightemperature part of the solution at the bottom of the crucible is carried by the flow of the solution to the vicinity of the seed crystal, supersaturation occurs around the seed crystal to initiate the growth of SiC. The crystal growth temperature is approx. 1800°C to 2100°C.

In general, heating is performed by resistance heating or highfrequency induction heating. Nippon Steel & Sumitomo Metal has adopted high-frequency induction heating, since it can make the solution flow by electromagnetic force. Such forced flow efficiently transports carbon dissolved from the inner wall of the crucible to the growth interface, playing an important role to ensure a high growth rate.¹³⁾ Our research group has worked on growing SiC ingots 2 to 4-inch in diameter to date. **Figure 3** schematically shows the major technical challenges regarding the solution growth that constitute the obstacles to growing bulk crystals of high quality. The challenges include the suppression of the spontaneous nucleation that causes the growing crystal to be multicrystalline (undesirable crystals) and the macroscopic solvent inclusion defects caused by solvent molecules included in the crystal structure.

2.2.2 Suppression of undesirable crystal formation

Contrary to the melt growth method involving growing a crystal from a liquid phase with a constant melting point, the solution growth method allows crystal growth to make progress in a wide range of temperatures, depending on the temperature distribution in the solution. When a SiC crystal is grown using the TSSG method, the presence of a low temperature part other than the vicinity of the seed crystal causes spontaneous nucleation, which will bring about the growth of undesirable crystals. Undesirable crystals hinder the growth of a single crystal. As shown in Fig. 3, undesirable crystals grow mainly in the vicinity of the crystal holding shaft, in the solution, and on the inner wall of the graphite crucible. An example of



Fig. 2 Schematic representation of the TSSG set-up Graphite crucible which acts both as a container for the solvent and as a carbon source is directly heated by induction.



Fig. 3 Schematic illustration of the major technicalogical problems for the SiC solution growth

the study we conducted to suppress undesirable crystal growth is described below.

Conventionally, when crystal growth is started from a solution, the seed crystal is immersed in the solution as shown in Fig. 4(a). The part of the graphite crystal holding shaft end contacts the solution, causing SiC nucleation to occur on the graphite surface to form undesirable crystals. The undesirable crystals reach the vicinity of the seed crystal in due course and make it difficult for the single crystal to continue growing. Our research group proposed a new crystal growth method involving formation of the meniscus in order to suppress the undesirable crystal growth around the crystal holding shaft.¹⁴⁾ As shown in Fig. 4(b), in this technique, the seed crystal is first brought into contact with the solution surface, and then is pulled up by approx. 0.2 mm to 1.0 mm above the solution surface to form a meniscus; after such a process, crystal growth is started. The height of the pulled-up seed crystal is called meniscus height. The meniscus formation method avoids the crystal holding shaft touching the solution, thereby completely suppressing the growth of undesirable crystals in the vicinity of the shaft.

On the other hand, undesirable crystals that appear on the graphite crucible inner wall and in the solution will float in the solution to attach to the growth interface and hinder the growth of the single crystal. In order to suppress the formation of such undesirable crystals, it is effective to optimize the heat insulation material structure used for the hot zone in the crystal growth furnace to make the temperature in the solution uniform. **Figure 5** shows the simulation results (inside the solution only) on the temperature distribution and flow rate vector distribution (vertical component) in the solution after optimizing the structure in the hot zone. If heat is homogenized in the vicinity of the seed crystal as well, the supersaturation, which is the driving force of crystal growth, will decline to slow the growth rate. For this reason, heat is released from the back of the seed crystal via the crystal holding shaft to facilitate the growth of the single crystal.

The temperature difference in the solution except the part in which the single crystal grows is kept within around 4°C. Such heat homogenization in the solution has significantly prevented undesirable crystals from appearing in the solution and on the inner wall of the graphite crucible. As a result, a long-time growth of nearly 100 hours has now been enabled.¹⁵⁾ It is important to accurately control the temperature distribution in the furnace for growing a SiC crystal in a solution. Today, simulation technology plays an important role in optimizing the temperature distribution.

2.2.3 Suppression of solvent inclusion

When growing a SiC crystal in a solution, as the thickness of the growing crystal increases, the crystal surface tends to be rough.

(a) Conventional growth configuration



Fig. 4 Schematic illustrations of the configurations of the seed in the case of (a) Conventional growth method, and (b) Meniscus formation technique

0.2 - 1.0mm

Seed crystal is dipped into the solution in the conventional growth method. On the other hand, the seed is contacted with solution surface and then pulled instantly upward from the surface.

(Single crstal)



Fig. 5 Simulation of the temperature and fluid flow distribution in the solution after optimization of the thermal insulation structure Length and direction of arrow indicate the flow rate and direction, respectively.

Such a phenomenon is called surface morphology instability.¹⁶ If the surface becomes rough and concavities and convexities in the order of several hundred microns to several millimeters are formed, the crystal growth advances while leaving solvent microdrops in the fine concavities, thereby forming macroscopic defects called solvent inclusion. A SiC crystal with solvent inclusion is not suitable for power device use. In order to prevent solvent inclusion, it is important to keep the growing surface smooth over a long period of time



Photo 1 Photograph of the typical surface mophrology of SiC grown by conventional solution growth method Step-bunching and wandering are frequently observed in the long-term growth.

during the crystal growth.

As shown in **Photo 1**, roughness of the crystal surface is produced by macroscopic bunching or meandering of steps on the crystal surface during the crystal growth. When a bulk crystal is grown using the solution growth method, a substrate at an angle of 0° off the (000-1) plane is used for the seed crystal for the reason that the step density is reduced in order to hinder the progress of the stepbunching. However, since a complete prevention method of macroscopic step-bunching has not yet been devised, a long crystal without solvent inclusion has been difficult to obtain. Recently, out of numerous process factors, we have revealed that the control of the growing interface outline and the suppression of temporal supersaturation change are extremely effective for stabilizing the surface morphology, leading to success in growing a bulk crystal without solvent inclusion. The study is described below.

Our research group has proposed a new crystal growth method involving making the growing interface of a crystal grown in a solution curve inward.¹⁷⁾ For crystal growth using the solution method, step-bunching can be prevented more effectively by making the solution flow counter to the direction of the forward movement of steps than by orienting the solution flow to the direction of the forward movement of steps.^{18, 19)} This is considered due to the fact that with the solution flow counter to the forward direction of steps, the solute concentration gradient on the front side of the steps becomes large, making the forward movement of steps smooth. During the crystal growth performed by Nippon Steel & Sumitomo Metal using high frequency induction heating, Lorentz force acts to agitate the solution and form an upward flow in the center of the solution as shown in Fig. 5.²⁰⁾ Given this, when the growing interface is made to be concave, the solution flow can be set counter to the direction of the forward movement of steps.

This concept allowed for a significant reduction of surface roughness compared with the crystal growth under the condition of the convex growing interface. In addition, under a method combining the uses of the concave growing interface and the solution surface meniscus, keeping the meniscus height within an appropriate range during the crystal growth for many hours would enable a bulk crystal excellent in surface flatness to be obtained. **Figure 6** shows the simulation results of supersaturation distribution in the vicinity of the growing interface with changed meniscus height.¹⁵

As the figure shows, as the meniscus height increases, the supersaturation at the growth interface increases. During actual crystal growth, as the growth progresses, the solution level is lowered due



Fig. 6 Calculated 3 dimensional distribution of supersaturated carbon concentration near the seed/solution interface
(a) Meniscus height is 0 mm, (b) Meniscus height is 2 mm,
(c) Meniscus height is 3 mm



Photo 2 (a) Photograph of the 4H-SiC ingot grown by solution growth technique. (b) Transmission X-ray image of the ingot. No dark spot ascribed to solidified metal solvent is observed for the whole volume, indicating the solvent inclusion free.

to the dissolution of the crucible inner wall and the evaporation of the solution. As a result, if the crystal holding shaft is not moved at all during the growth that takes a long time, the meniscus height will vary from the initial conditions. In particular, when the meniscus height temporally changes to be higher during the crystal growth and the supersaturation increases as well, the SiC solid phase stable regions are extended from the growing interface to the solution side, facilitating the transition to three-dimensional growth mode. The variation in supersaturation is considered responsible for the unstable surface morphology.

Given that, we moved the crystal holding shaft accurately in the vertical direction during crystal growth for many hours while keeping the meniscus height within an appropriate range. Today, this approach has enabled us to obtain a 4H-SiC bulk crystal with a 2-inch diameter and 5 mm or more thickness without solvent inclusion under the growing condition of a temperature at 2080°C using a Si-Cr solvent as shown in **Photo 2**. The solvent inclusion suppression technique described in this subsection is applicable to growing a bulk crystal with a diameter larger than 2 inches using the solution growth method.

2.3 Electrical property control for a SiC single crystal grown by the solution growth method

For a SiC single crystal for the power device use, it is necessary to control electrical conduction properties to make the crystal have low resistance in order to prevent such properties from constituting the device resistance component. For n-type SiC crystals, there is an ongoing study on doping using the nitrogen element as the donor. We have found that adding a small amount of nitrogen in the atmosphere gas (atmospheric pressure helium) allows nitrogen doping of a SiC crystal to be performed using either a Si-Ti solvent or Si-Cr solvent. It is considered that nitrogen is dissolved from the atmosphere to the solution in proportion to the square root of the nitrogen partial pressure according to Sieverts' law, and then is transported to the growing interface and incorporated into the crystal.²¹⁾

Figure 7 shows the relationship between the nitrogen amount in a crystal developed using the solution growth method and the specific resistance, in comparison with the values provided in previous

studies^{22, 23)} involving the sublimation growth method. The relationship between the nitrogen doping amount of the crystal grown using the solution growth method and the specific resistance is almost the same as that using the sublimation growth method. This result indicates that both methods involve almost the same level of charge compensation by impurities in a crystal. Until now, we have succeeded in growing n-type crystals with the resistivity at 15 to 20 m $\Omega \cdot$ cm, comparable to commercially-available wafers produced using the sublimation growth method.

2.4 Dislocation defect evaluation of the SiC single crystal wafer grown by the solution growth method

A commercially-available wafer produced using the sublimation growth method contains various dislocation defects. Dislocation defects fall into two large categories based on the developing direction: basal plane dislocations present on the basal plane, which is perpendicular to the c-axis direction, i.e., the growing direction; and threading dislocations, which extend in the c-axis direction. Threading dislocations can be further grouped into screw dislocations and edge dislocations. In order to improve the performance and longterm reliability of a power device, the reduction of these dislocation defects is strongly desired.



Fig. 7 Relationship between nitrogen concentration and resistivity in 4H-SiC grown from Si-Ti and Si-Cr based solvent

Since the production of 4H-SiC single crystal ingots without solvent inclusion has been made possible using the solution growth method, Nippon Steel & Sumitomo Metal has proceeded with the production of wafers from the ingots. **Photo 3** shows examples of 4H-SiC wafers with a 2-inch diameter and at an off angle of 4° produced using the solution growth method. When the specific resistance of these wafers was measured by the eddy current method, the entire surface of each wafer indicated specific resistance at 16 to 18 m $\Omega \cdot$ cm. From an SIMS (Secondary Ion Mass Spectroscopy) analysis, it was confirmed that the crystal contained 1.2 to 1.4×10^{19} nitrogen atoms per cubic centimeter, and that they were nitrogen-doped low resistance wafers.²⁴⁾ In this section, the quality of the wafers produced using the solution growth method is described.

First, in order to clarify the propagation behavior of dislocation defects from a seed crystal to a crystal grown using the solution growth method (hereinafter referred to as the "solution grown crystal"), the dislocation defect density of the solution grown crystal was compared with that of the seed crystal. For the seed crystal, a crystal produced using the sublimation growth method was used. As shown in **Fig. 8**, the evaluation wafer sample was prepared first by growing a crystal in a solution on a plane of the seed crystal at an angle of 0° off the (000-1) plane, and then was tilted at 4°. After that, the grown crystal was sliced, polished, and adjusted so that the interface between the seed crystal and solution grown crystal was included in a wafer. The sample as prepared underwent molten KOH etching (at 500°C for 2 minutes) and the types and density of dislocations were determined based on the shapes and number of etch pits that appeared.

2.4.1 Basal plane dislocations

Figure 9 shows the change in basal plane dislocation density from the seed crystal to the solution grown crystal. While the seed crystal had several hundred basal plane dislocations per square centimeter, in the solution grown crystal, no basal plane dislocation was found at all. This result indicates that since the crystal was grown in the solution on a plane of the seed crystal at an off angle of 0° from the (000-1) plane, the basal plane dislocations contained in the seed crystal were not propagated to the solution grown crystal. The result also indicates that no basal plane dislocation newly generates during solution growth. It has been confirmed that no basal plane dislocation



Photo 3 Photograh of 2 inch diameter 4° off-axis 4H-SiC wafers grown by solution growth tequnique



Fig. 8 Procedure of molten KOH etch pit observation on the tilt angle polished surface exposing both the seed and solution grown crystal



Fig. 9 Relationship bwtween the distance from the interface and basal plane dislocation density Upper x-axis denotes the conversion value of the grown thickness.

tion exists in a solution grown crystal from synchrotron radiation topography observation as well.²⁵⁾ This is due to the processes of the solution growth method conducted at lower temperatures than the temperatures at which the processes of the sublimation growth method are carried out. In view of thermal stresses, the possibility of dislocation occurrence and multiplication is limited in low temperature processes because of the temperature gradient of the crystal growing interface, which is smaller in low temperature processes than in high temperature processes. Furthermore, in view of the change in energy (latent heat) along with the growth, the microscopic form of the growing interface is considered stable when a crystal is grown in a solution, making dislocation occurrence unlikely, since the latent heat is smaller in the case of crystal growth in a solution than that in the gas phase.

In the SiC power device production, in order to grow an epitaxial layer of high quality suitable for device production by CVD, the wafer is provided with an off angle of 4° to 8°. The majority of basal plane dislocations that exist in a wafer that is undergoing epitaxial layer deposition are known to alter their structures to become threading edge dislocations with the same Burgers vectors to exert no adverse effect causing the deterioration of device properties.²⁶⁾ However, when part of the wafer basal plane dislocations is propagated to the epitaxial layer as-is, stacking faults are produced in a device with a p-n junction during forward direction operations, resulting in deterioration of device properties.²⁷⁾ While such property deterioration was previously regarded as a problem specific to bipolar devices, it has become conspicuous in unipolar devices along with the recent progress in the device structure. As seen from the above, a basal plane dislocation is a killer defect that deteriorates device properties. Since a SiC single crystal grown using the solution growth method has almost no basal plane dislocation, the crystal is suitable for producing a high- performance power device.

2.4.2 Threading dislocations (screw dislocations and edge dislocations)

An examination of the change in the threading screw dislocation density from the seed crystal to the solution grown crystal revealed that while the seed crystal had several hundred threading screw dislocations per square centimeter, the number was reduced to 100 or less in the solution grown crystal. Screw dislocations are reported to have altered the structures with high efficiency to become stacking faults on the basal plane during the growth of the seed crystal with an off angle when growing a SiC single crystal in a solution.²⁸⁾ This

structural conversion is considered to advance caused by the interaction between macroscopic steps (step height >100 nm) developing in the lateral direction during crystal growth in a solution and threading screw dislocations. Nippon Steel & Sumitomo Metal has a solution method crystal growing technology capable of suppressing the formation of macroscopic steps by stabilizing the surface morphology. Nevertheless, the efficiency of screw dislocation conversion remains relatively high.

According to a recent report, the step edge shape as well as the step height have an influence on the change in the screw dislocation structure.²⁹⁾ Taking into account the finding as well, we would like to clarify the screw dislocation reduction mechanism for the bulk crystal growth using the solution method performed at Nippon Steel & Sumitomo Metal going forward. Incidentally, threading screw dislocations are said to cause leak current in a vertical power device to which high voltage is applied to significantly deteriorate the voltage resistance.³⁰⁾ The use of the solution growth method allows for reducing threading screw dislocations, which are killer defects.

Lastly, the threading edge dislocation density was obtained for each of the seed crystal and solution grown crystal. As a result, in both the seed crystal and solution grown crystal, there were several thousand threading edge dislocations per square centimeter. As seen from this, threading edge dislocations do not significantly increase or decrease in number during the crystal growth in a solution. While we consider that threading edge dislocations have only a small impact on power device properties at present, it will be necessary to reduce the number of edge dislocations in the future for SiC single crystals to eliminate dislocations as in Si to be used as a power device material with high reliability.

3. Conclusion

We have described the latest development of Nippon Steel & Sumitomo Metal's SiC single crystal growth technology that uses the solution growth method. With the surmounting of various technical problems specific to the solution method, the possible production of power device wafers is finally being initiated. In the industry, the solution growth method has not yet reached the wafer diameter level that can be obtained using methods involving the gas phase crystal growing including the sublimation growth method, and thus has not yet been practically used as the gas phase methods have done. However, SiC crystals grown using the solution growth method are those of high quality with much less killer defects such as basal plane dislocations and threading screw dislocations. In addition, using the solution growth method, crystals can be grown at lower temperatures than other methods, and at normal pressures as well, a cost reduction can be expected for the crystal growing process. While forming a mutually complementary relationship with the sublimation growth method, the solution growth method is expected to develop as a low-cost method capable of producing crystals with less dislocation defects, which has long been desired for realizing a high-performance and high-reliability SiC power device. Nothing would give us greater pleasure than to have the SiC R&D of Nippon Steel & Sumitomo Metal using the solution growth method contribute to the market formation of high-quality SiC single crystal wafers.

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