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## 主 論 文 の 要 旨

論文題目            High quality 4H-SiC crystal growth and  
                              dislocations behavior during solution method  
                              (溶液法における高品質 4H-SiC 成長および欠陥挙動)

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## 論 文 内 容 の 要 旨

Silicon carbide (SiC) has been proved to be a choice of material for the next-generation power devices because of its excellent physical properties such as high break down field, wide band gap and high thermal conductivity. Nowadays, the rapid development of semiconductor power devices requires higher quality SiC bulk crystals. Various defects such as threading screw dislocations (TSDs), threading edge dislocations (TEDs) and basal plane dislocations (BPDs) are contained in commercial SiC wafers grown by physical vapor transport (PVT) method. Meanwhile, several types of defects like carrot defects and comet defects formed during the chemical vapor deposition (CVD) growth of SiC have been reported. It is well known that defects in SiC have various deleterious effects on power device performance. For example, the existence of TSDs in power devices results in the degradation of blocking voltage performance and epitaxial defects. BPDs cause the degradation of on-state conduction performance for PiN diode and so on. In order to realize the full potential of SiC power devices, the reduction of defects density becomes extremely crucial.

Solution growth is a potential method to produce high quality crystals because the growth proceeds under the condition close to thermal equilibrium. In the previous research, it has been reported that TSDs can be dramatically reduced by the conversion of TSDs to defects on basal plane, which was caused by the advance of macrosteps on the growth surface. As the growth proceeds, the defects on the basal planes are excluded due to extending along in-plane direction and consequently the ultrahigh-quality SiC

grown crystals can be obtained. Therefore defects in seed crystals could be reduced by conversion phenomenon. However, little research has been done for defects generated during growth. Moreover, no TSD conversion is observed on C-face solution growth due to the absence of macrosteps. Recently, 4H-SiC C-face bulk growth has attracted many attentions due to the difference to Si face in many aspects. C-face seed crystals are often used for stabilizing polytype during bulk growth. However, the TSD density cannot be decreased on C face by utilizing TSD conversion phenomenon like on Si face. Thus the reduction of TSD density on C-face growth is still a challenge.

The aim of this study is to reduce defects and obtain high quality 4H-SiC grown crystals by solution growth. These background and aims are described in chapter 1.

The chapter 2 introduces the growth force and model. In addition, experiment detail is described.

The chapter 3 reveals the existence of unidentified defects corresponding to V-shaped contrasts in X-ray topographic images of the crystals grown by 4H-SiC solution growth. The detailed analysis combining transmit TEM results revealed that the newly generated defects are identified as a pair of dislocations on basal plane with opposite burgers vectors parallel to the [11-20] direction. It is found that no defects in the substrate are directly associated with the formation of the V-shaped defects. Geometric analysis of the size and shape of the V-shaped defects indicated that they nucleate in pairs intermittently during growth process. Based on the observed morphology of these defect configurations a mechanism of two dimensional (2D) nucleation during solution growth is postulated for the generation of the V-shaped defects.

The chapter 4 provides a method to realize TSD conversion on 4H-SiC C-face solution growth. Several solvents were investigated in order to induce the dislocation conversion caused by the formation of macrosteps on C face. Macrosteps were observed on C-face crystals grown from Si-5at%Ti solvent. Topography images revealed that TSD conversion was induced and the conversion ratio reached to 10% with 5at%Ti addition into solvent. We investigated steps structure on Si face and C face of crystals grown with pure Si and C face of crystals grown with Si-5at%Ti solvent by cross-sectional TEM observation respectively. We proposed that the influence factor of TSD conversion ratio was not only step height but also the angle between facet and basal plane. The mechanism of angle influence was discussed from dislocations energy aspect.

The chapter 5 describes the increase of TSD conversion ratio on C face of grown crystals. It was assumed that TSD conversion ratio increased with growth thickness. However, topography image became fussy with elongating growth time. TEM

observation revealed that high stacking fault (SF) density caused by N<sub>2</sub> doping from background probably was responsible for unclear topography images. Thus, pre-baking, high growth temperature and Zr and Hf as N<sub>2</sub> absorber were used to decrease N<sub>2</sub> doping concentration from background. Consequently, the TSDs conversion ratio increased from 10% to 70% by increasing growth thickness to 65 μm. The result is beneficial for the reduction of TSD density in C face of grown crystals.

The chapter 6 proposes two-step growth method to grow high quality SiC C-face bulk crystals. In order to solve low growth rate and rough morphology during growth with Si-5at%Ti solvent, two-step growth by controlling crystals morphology was proposed. At first, TSD conversion phenomenon on C face was realized by using Si-5at%Ti solvent. The high-quality seed crystal for second step growth was prepared using the crystal grown on C face, where about 70% TSD conversion ratio occurred. Si-39at%Cr-2at%Al solvent was used in second step growth to increase growth rate and keep grown surface smooth. As a consequence, the TSD density of C-face grown crystal decreased from 2000 cm<sup>-2</sup> to below 8 cm<sup>-2</sup> which was three orders of magnitude lower than that of seed crystal. The thickness of grown crystals was about 460 μm.

In chapter 7, conclusions from each chapter were summarized.

In this thesis, the author obtained high-quality 4H-SiC C face grown crystals by solution growth. The TSD density was decreased from 2000 cm<sup>-2</sup> to below 8 cm<sup>-2</sup> which was three orders of magnitude lower than that of seed crystal. This study proposed a novel and effective method (by controlling crystal morphology during growth) to decrease defects during 4H-SiC C-face bulk growth. Moreover, this study revealed V-shaped defects generated during Si-face growth due to 2D islands and provided that C-face growth could restrain their generation.