Defect formation mechanism of 6H-SiC crystals grown by sublimation method

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Abstract

There have two kinds of defects, planar defects and vertical defects which were called micropipes in SiC bulk crystals grown by a sublimation method. We could decrease these defects by adding a little piece of Si in the SiC powder or using Ta cylinder in the crucible. So we report the dependence of these defects in a wafer on silicon/carbon ratio in this paper. The chemical species sublimed from SiC powder is affected by carbon from the graphite wall of the crucible. It is important to control the chemical species on the substrate.

1. Introduction

SiC bulk crystals have been made by the sublimation method[1]. Recently, the development of device applications was also reported. It is important to have high quality bulk crystals and large wafers for development of device. In a wafer, there are a lot of defects. We can see mainly two kinds of defects, vertical defects called micropipes and planar defects. The reliability of device operation on the base of SiC depends on defects in the crystals[2]. It was reported that annealing of SiC single crystals at 2270~2570 K led to a significant diminution of pinholes density[3] and that the bulk crystals grown on faces perpendicular to the (0001) basal plane had no empty tube which caused fatal damage to a SiC wafer[4]. From the viewpoint of growth, effective parameters of the defect formation had not been solved. Therefore it is necessary to trace the defect formation mechanism.

2. Experimental Procedures

SiC growth was carried out in a quartz tube with a water-cooled jacket in Ar-ambient. The source material was a highly purified SiC powder with an average grain size of 2 μm. Before sublimation growth, the purification of the source SiC material was carried out at about 1500 °C source temperature with 100 Torr in order to remove metallic impurities. The substrates were Acheson crystals or 6H-SiC wafers that were cut from the bulk crystals made by sublimation method. The distance between the source and substrate was about 24~25 mm. The inner diameter of graphite crucible was 30 mm. And the crucible was heated by RF induction with a frequency of about 5 kHz. The substrate temperature T_s and the source temperature T_b were measured by optical pyrometers. The growth pressure P was 30~50 Torr and the temperatures were T_b = 2150~2250 °C, T_s = 1950~2050 °C.

3. Results and Discussions

It is important to know the reaction kinetics during sublimation. According to the report, main chemical species are SiC_2, Si_2C and C [5]. However, crucible is made of graphite, so we can not neglect carbon species in the crucible. We often observe carbon inclusion in the grown layer. There is a possibility that this excess carbon is one of the causes of defects. Therefore it is necessary to control this excess carbon.

The first attempt was to compensate the excess carbon by adding small amount of Si together with source SiC powder. The small amount of Si, 0.1 to 2.0 g was added in SiC powder of about 25 g. The optical micrographs of surface morphologies of as-grown layer on

Acheson wafers are shown in Fig. 1. Figures 1(a), 1(b) and 1(c) correspond amount of Si addition 0 g, 0.8 g and 2.0 g, respectively. In Fig. 1(a), there are some large micropipes and planar defects. But the surface morphology of Fig. 1(b) was clean and defects were not observed so much. The surface morphologies of grown layer prepared by adding Si amount in the range of 0.3 to 0.8 g were almost same and resemble to Fig. 1(b). In Fig. 1(c), the flat area was smaller than Fig. 1(b) and the surface became rough. Moreover, there are planar defects under the surface, which looked as a white vague image. Fig. 2(a) and 2(b) show optical micrographs of grown layer on 6H-SiC made by sublimation in advance. Both surfaces were not flat, because these 6H-SiC substrates have some off-angles. In Fig. 2(a), there are a lot of micropipes. These micropipes existed in the substrate before growth and we think the number of micropipes did not increase. In Fig. 2(b) there are a lot of terraces, but these are almost flat like Fig. 1(b).

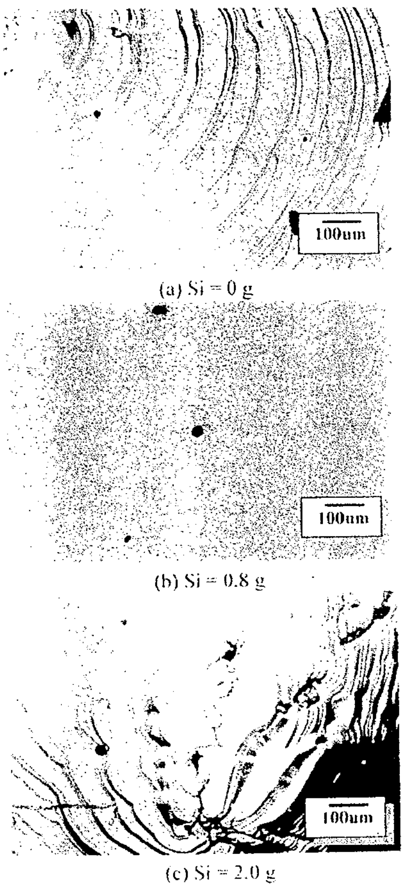

Fig. 1 The surface morphologies of grown layer on Acheson when Si was added in SiC material.

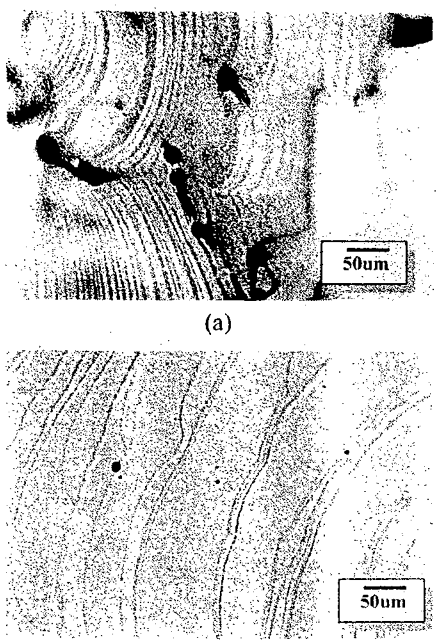

Fig. 2 The surface morphologies of grown layer on home made 6H-SiC wafer made by sublimation method Si was added in SiC material.

The second attempt was to make a bulk crystal using a tantalum container. It was reported that Ta worked as a gettering of carbon[6]. So this is another way to eliminate excess carbon in the crucible. We set Ta cylinder inside of crucible. Before crystal growth, carbonization of Ta was achieved and Ta was transformed to tantalum-carbide (TaC). We call this crucible as Ta container. Fig. 3 shows the surface morphology of grown layer in Ta container by optical microscope. The surface resembles ones in Fig. 1(b). Moreover, we measured the density of defects of grown layer on 6H-SiC wafer substrate made by sublimation with Ta container. But using Ta container did not drastically decrease it. So by using Ta container, defects could be restrained, but could not be decreased.

From these results, we consider the defect formation mechanism. By using Ta and Si, we could control the excess carbon, which arise from graphite crucible, and get clean surface as shown Fig. 1(b). So one of the reasons of the defect formation is Si/C ratio of chemical species in the crucible. In addition, between 0.3 g and 0.8 g added Si, the surface morphologies did not depend on the amount of added Si and they were almost flat surface in all samples. We speculate that the role of added Si in the crucible: First, the chemical species, second, the control of excess carbon from graphite crucible, third, the element for recrystalization of sublimed SiC material. In case of 0.3~0.8 g added Si, Si used almost one of three, so the chemical species was nearly the theory and we got a clear surface. But in case 2.0 g, it was not enough only the three actions and a lot of Si remained by liquid state. Therefore, the surface became rough as Fig. 1(c), because it become the excess Si inside crucible and compensates for this state by etching frown surface. If the chemical species in the crucible was excess carbon or silicon, Si or C atoms will escape from the grown layer to compensate the vapor stoichiometry of the vapor, and consequently holes are created. We suppose these holes were the origin of defects, especially micropipes.

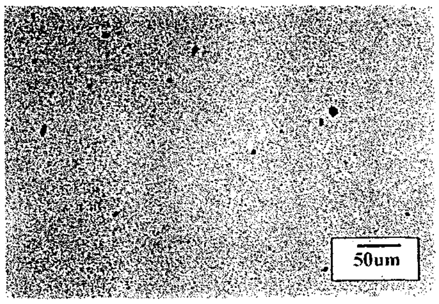

Fig. 3 The surface morphologies of grown layer using Tantalum container.

4. Summary

Added Si and using Ta container decreased the numbers of defects. Therefore, the defect formations are influenced by the Si/C ratio. If the chemical species in the crucible was excess carbon or silicon, Si or C atoms will escape from the grown layer to compensate the vapor stoichiometry of the vapor, and consequently holes are created. These holes may be the origin of defects. The carrier density is independent of the amount of added Si.

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