3C-SiC growth by sublimation in vacuum technology optimization

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Abstract. Results of an epitaxial growth of 3C-SiC epilayers on hexagonal 6H-SiC, 4H-SiC substrates are described. The study of the obtained epitaxial layers grown on 6H substrates was made by photoluminescence and optical microscopy. Also, an image analysis of the interface of 3C-SiC epitaxial layers with 6H, 4H, and 15R substrates obtained by Transmission Electron Microscopy (TEM) are presented. Difference between the layers on the Si and C faces are discussed.

1. Introduction
The attractiveness of silicon carbide for semiconductor electronics is well known and theoretically justified. Significant advances in technology over the past 20 years have made it possible to create almost all the main types of semiconductor devices based on SiC.

Due to the wide band gap, high thermal conductivity, high thermal, chemical and radiation resistance, silicon carbide (SiC) is a very promising material for power electronics. At the same time, SiC has the ability to crystallize in various modifications (polypotypes) having the same chemical composition, but different electrical parameters. Due to the high mobility (1200 cm²/(V·s)) [1], drift velocity of electron saturation (2.7 * 10⁷ cm/s) and the high value of the critical breakdown field (3 * 10⁶ V/cm) in combination with a wide forbidden zone (2.39 eV) explain the advisability of using 3C-SiC for the production of semiconductor devices for various purposes (power, microwave electronics, devices designed to operate in extreme conditions). To create devices, the cubic polytype is most interesting.

Currently, two technologies are used to grow 3C-SiC epitaxial layers: growth on silicon substrates [2] and growth on hexagonal polytypes on substrates. But with growth in both ways, structural defects are formed. In the first case, due to low growth temperatures, limited by the melting temperature of the Si substrate, a large number of dislocations are formed in the layer (3-4). These structural defects do not allow semiconductor devices to be manufactured on the basis of epitaxial layer data. With the growth of hexagonal polytypes (5-6) on substrates, few dislocations form, but twin boundaries arise as a result of epitaxy of the cubic material on the hexagonal. These twin boundaries, as well as dislocations in the first case, do not allow the use of these films for the manufacture of devices.

2. Experimental
The aim of this work is to optimize the growth technology of epitaxial films of cubic silicon carbide (3C-SiC) and to obtain layers of high structural quality. In our growth experiments an sublimation epitaxy in vacuum unit [7] was used. The growth of 3C-SiC epitaxial layers based on substrates of
hexagonal polytypes was made at temperatures of $1800 \div 2000 \, ^\circ \text{C}$. The growth time was 1-2 hours. As a pre-growth preparation of the samples an oxidation operation was performed. The oxidation was made in a separate reactor in an atmosphere of moist oxygen for 3 hours at a temperature of $1100 \, ^\circ \text{C}$. The oxide was etched in hydrofluoric acid, and then a standard washing operation was made in deionized water and organic solvents.

As a source for growth, commercial SiC with a grain size of $\sim 10–20 \, \mu \text{m}$ was used. In a number of experiments silicon (2% of the total mass of the source) with the same grain size was added to the composition of the source. In a number of experiments, we used the same growth source. As the source is used, it becomes depleted with silicon and density of double position boundary defects in subsequent experiments was increasing. To avoid this, we enriched the source with pure silicon.

The growth was made on industrially produced 6H-SiC substrates which were manufactured by «Nitride Crystals» [8] and 4H-SiC substrates which were manufactured by «Svetlana». Immediately before the start of growth sublimation etching of the «in situ» substrate was also made to remove the layer impaired by mechanical polishing and to clean the surface of the substrate. Using an optical microscope, we determined the sizes of individual twins of the cubic polytype, as well as the density and thickness of the twin boundaries.

3. Results
The quality of the grown layer was also strongly affected by the face of the substrate. The sizes of the twins during growth on the Si (0001) -face were almost an order of magnitude smaller than during growth on the C-face (Figure 1).

![Figure 1. Optical image (0.5x0.5 µm) of 3C-SiC epitaxial layer of Si-face (A), C-face (B)](image)

In addition, the thickness of the twin boundaries on the Si-face was much larger. It indicates a higher density defects at the boundary of twinning regions. In addition to the sizes of the twins, the difference between the faces was the number of inclusions of the 6H polytype in the cubic epitaxial layer (Figure...
Figure 2. Photoluminescence of (A) Si-face, (B) C-face

Whereas for the C face, the fraction of 6H inclusions did not exceed 1-2% of the total area of the epitaxial layer, and the sizes of inclusions were 10-20 µm for the Si face. The share of 6H inclusions reached 10%, and the sizes of individual inclusions reached 40-50 µm.

It was experimentally established that for covering more than 85% of the surface with a 3C-SiC the growth conditions for Si and C faces will differ. For growth on the Si face, growth temperatures should be closer to 2000°C. Growth speed of more than 0.7 µm / min are required with the C face for which these temperatures are in the range from 1950 to 2000 °C and 0.4-0.5 µm / min, respectively. With growth on the C-face, the twins reach bigger size during growth process and the concentration of DPB defects due to this decreases. At lower growth temperatures (<1800 °C), a combination of 3C and 6H polytypes in the epitaxial layer was observed. The 6H polytype with 3C inclusions mainly grew at high temperatures (> 2000 °C). This is explained by a decrease in the ratio of silicon to carbon in the vapor phase. It is a more favorable factor for the growth of hexagonal polytypes.

After determining the technological parameters for the growth of 3-SiC polytype on 6H SiC, we began to refine the technology on other polytypes as well. In our experiments, we performed growth on 4H-SiC polytype. Here are the results of some experiments.

In addition to experiments with hetero-polytype growth, a new approach to 3C SiC growth is being investigated, trying to combine the advantages of two previously developed growth technologies without inheriting their shortcomings [9]. As part of this approach we preliminarily used 3C-SiC film grown by CVD on a silicon substrate. Such a epitaxial layer grew on the basis of the cubic substrate. Then the grown 3C-SiC / Si structure from the side of the 3C SiC film was glued to the hexagonal SiC substrate and the silicon substrate was removed chemically. (Figure 3)
Table 1. Optical Images and photoluminescence of grown 3C-SiC layers on 6H-SiC and 4H-SiC substrates.

| Polytype | Face | Temperature, °C | Result, % 3C | Microscope Image | Photoluminescence |
|----------|------|----------------|--------------|-----------------|------------------|
| 6H       | C    | 1850           | 95 %         | ![Image](image1) | ![Image](image2) |
| 6H       | Si   | 1900           | 88 %         | ![Image](image3) | ![Image](image4) |
| 4H       | C    | 1900           | 90%          | ![Image](image5) | ![Image](image6) |
| 4H       | Si   | 1900           | 35%          | ![Image](image7) | ![Image](image8) |

Figure 3. Diagram of the process of transferring from CVD grown 3C-SiC on Si to the final stack of 3C-SiC / carbon glue / SiC (a) and (b) The schematic 2D view of the PVT reactor used for sublimation epitaxy, showing the Crucible and the basic property of high temperature gradient.
The resulting structure was used for further growth of the 3C-SiC layer by sublimation in vacuum, but already at significantly higher temperatures (~ 1850 C). The obtained samples were studied by photoluminescence, Raman spectroscopy, optical microscopy and x-ray diffractometry.

**Figure 4.** Surface photo using a Differential interference contrast microscope.

**Figure 5.** Photoluminescence of grown 3C-SiC layer.
Figure 6. X-ray diffractometry of 3C-SiC layer grown on 3C-SiC glued on 6H-SiC

Figure 7. Comparing Raman spectrums of 3C-SiC layer grown on Si and 3C-SiC layer grown on 3C-SiC glued on 6H-SiC.
4. Conclusion
The technological parameters for the growth of 3-SiC polytype on 6H and 4H-SiC were determined. For covering more than 85% of the surface with a 3C-SiC the growth conditions for Si and C faces will differ. The new approach for 3C SiC growth needs further investigation.

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