Acquisition and Research of the p-3C-SiC Epitaxial Layers Based on the 6H-SiC Semi-Insulating Substrates

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Abstract: By now we know almost nothing about works on production of p-3C-SiC. Probably, it is due to the fact that the basic acceptor impurity (aluminium) accumulates on the interfaces of the twins and other structural defects in the 3C film and it is electrically neutral. We managed to produce the highly doped layers p-3C based on the conducting substrates of hexagonal SiC using the method of Sublimation Epitaxy (SE). Probably, it is due to the growth high temperature used in this method. First, it results in production of the more structural-perfect epifilm and, second, in increasing the Al atomic mobility. This works purpose is further optimization of the production technology for epilayers p-3C-SiC with usage of semi-insulating substrates 6H-SiC.

Keywords: p-3C-SiC, Sublimation Epitaxy, Epifilm, Substrates

Introduction

Silicon carbide has a possibility, if the chemical composition is the same, to crystallize in both a cubical polytype and in more than two hundred hexagonal and rhombohedral ones. In the hexagonal polytype the lattice constant does not differ from the lattice constant of the cubical polytype with the accuracy to three decimal places in the direction perpendicular to axis C. Therefore, it is a rather perspective material for creation of different types of heterostructures (Lebedev, 2006). It was theoretically proved that the heteropolytypic structures based on SiC are more perspective, for example, for HEMT transistors manufacturing than compound III-N (Polyakov and Schweirz, 2005). There is a number of experimental works where heteropolytypic structures were produced using both the method of sublimation epitaxy (Lebedev et al., 2001a) and the CVD method (Chandrashekhar, 2007; Li et al., 2009).

Experiment

The 3C-SiC epilayers were grown using the method of Sublimation Epitaxy in vacuum (SE) (Lebedev et al., 2001b). The 3C-SiC epilayers were grown at temperatures 1700-2100°C, the time of growth is 15 min. The 6H-SiC semi-insulating substrates made by Svetlana-Elektronpribor CJSC (Lebedev et al., 2008; 2014) were used, the angle of misorientation of the substrate surface did not exceed 0.5°. The growth was held both on silicon Si (0001) and on carbon C(001) faces. The silicon carbide charge of high purity with grain size 20-40 microns was applied as the source of growth. For production of r-type conductivity, we added the Al impurity into the source of growth. Immediately before the beginning of growth, the substrate sublimation etching “in situ” was realized to remove the layer destroyed by mechanical polishing. The polytype were transformed during homoepitaxial growth due to the following factors:

- The growth temperature drop relatively to the growth of polytype 6H. At the same time, the structure of a vapor phase shifts toward the silicon enrichment
- Usage of the silicon carbide substrates with the no disorientation surface

The area of the grown epitaxial layers was ~ 1 cm², thickness was ~3-5 µm. The specimen structural perfection was studied using x-ray topography and the method of X-ray back reflection. The topograms were made in reflections (113) in the CuKα radiation. An optical microscopy and method of photoluminescence also were used. The method of photoluminescence (FL) was applied to identify the polytype of the grown epilayer. For the FL excitation the nitrogen laser with wave length 337.1 nm and with the following parameters was used: Pulse power -2kw, pulse duration -10 ns, pulse repetition frequency -100 Hz. The
pump power density was \( \sim 50 \text{kWs/cm}^2 \). The FL spectra were studied at the temperature of liquid nitrogen (77K).

The concentrations of uncompensated acceptors (N\(_a\)–N\(_d\)) in the samples were specified following the capacitor-voltage characteristics (C-U) on the standard stand with a parallel equivalent circuit and sinusoidal frequency 10 kHz at room temperature.

**Results and Discussion**

It is known, when a cubical epilayer arises at once in several different points of the hexagonal substrate, the orientation of the 3C-SiC nucleuses along the growth surface can be of two types different with a 60° turn relative to each other (twinning structure). On the x-ray topograms derived in some Bragg reflexes, the twinning structure appears in the form of areas of black or white contrast (Fig. 1) which changes for an opposite one if the crystal turns 60° around the axis (111) (Andreev et al., 2002). We produced in our work (Lebedev et al., 2007) 3C p-type layers consisting practically of one twin. As you can see in Fig. 1, the structural perfection of the p-type layers appeared worse than for the p-type layers grown in the same technological conditions. However, the twins density was anyway lower than in the p-3C-SiC layers grown based on the silicon substrates (Reyes et al., 2006).

Figure 2 shows the photoluminescence spectra of the grown 3C-SiC epifilm (curve 1) and spectrum of the 6H substrate (curve 2). The 3C-polytype is characterized by red radiation, the spectrum demonstrates a broad band with the maximal radiation 2.1 eV. The nature of this band is related to the radiation of the Al-N donor-acceptor pairs in 3C-SiC. This radiation energy is less than the width of the forbidden area 3C (the width of the 3C-SiC forbidden area is 2.36 eV). The 6H polytype is characterized by blue luminescence, this radiation band nature is also related to the Al-N donor-acceptor recombination, but for the 6H polytype \( E_g \) is equal to 3.02 eV. It should be noted that the spectrum received from the epilayer (curve 1) has a radiation band coincident with the radiation band from the 6H polytype. It can be explained by the fact that the depth of penetration of the laser ultraviolet radiation is more that the thickness of the epilayer, therefore, the epilayer radiation is fixed, as well as the radiation from a part of the substrate under the layer.

Therefore, the cubical and hexagonal polytypes are easily recognized according to the radiation excited in the epilayer by ultraviolet radiation. Using photo fluorescence microscope “Lumam I2” with ultraviolet radiation, it is possible, according to the color of the luminescence, to characterize the sample homogeneity, to identify the grown layer’s polytype and also the polytypes of impurities, which sizes do not exceed ten microns.

![Fig. 1. X-ray topogram of the typical epitaxial film p-3C-SiC. Reflection [311], CuKα-radiation, magnification 14X](image1)

![Fig. 2. 6H/3C heterostructure photoluminescence spectra at 77 K](image2)

With the help of the optical microscope we determined the sizes of the cubical polytype individual twins and also the density and thickness of intertwining interfaces.

The studies have shown that the most stable growth of the 3C polytype was reached at temperatures 1900-2000°C.

At these temperatures the sizes of the twinning areas reach the maximum sizes (>500 microns), at the same time, the share of the cubical polytype in the epilayer can reach 99%, i.e., the 6H polytype is present only in the form of small impurities 10-20 microns (Fig. 3B). At lower temperatures of the growth (<1800°C), the 3C and 6H polytypes mixing was observed in the epilayer (Fig.
3A). At high temperatures (> 2000°C) the 6H polytype with impurities 3C (Fig. 3C) grew predominantly, it is explained by the silicon-carbon ratio decrease in the vapor phase (Fig. 3), that, in turn, is more favourable for the growth of hexagonal polytypes.

Also, the quality of the grown layer was significantly influenced by the substrate edge. The sizes of the twins at growth on the Si(0001) edge were almost one order less than at growth on the C(0001) edge (Fig. 4). Besides, the thickness of the intertwinning interfaces on the Si edge was much more, which testifies to the greater density of defects on the interface of the twinning areas. Apart from the twins sizes, the difference between the edges consisted of an amount of impurities of the 6H polytype in the cubical epilayer (Fig. 5). If for the C(0001) edge the share of the 6H impurities did not exceed 1-2% of the total area of the epilayer and the sizes of impurities were 10-20 microns, for the Si edge the share of the 6H impurities reached 10% and the sizes of individual impurities reached up to 40-50 microns.

Fig. 3. (A) Picture of the FL epilayer grown at different temperatures (B and C) Picture of the FL epilayer grown at different temperatures

Fig. 4. Optical picture (0.5×0.5 microns) of the 3C-SiC epilayer of the Si edge (A), C edge (B)

Fig. 5. Picture of the FL epilayer grown on (A) the Si edge, (B) the C edge
The given capacitor-voltage measurement with the use of a mercury probe has confirmed, the r-type conductivity of the grown 3C SiC epilayer with the $N_A - N_D$ concentration $\sim 1 - 3 \times 10^{18}$ cm$^{-3}$ (Fig. 6).

**Conclusion**

In the course of the study, we showed the possibility of heteroepitaxy of the p-3C-SiC layers based on the 6H-SiC semi-insulating substrates. Also, it was specified that the optimal temperature range for heteroepitaxy is 1900-2000°C and the C edge of the substrate is more preferential for production of the 3C-SiC epilayer without formation of impurities of other SiC polytypes.

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**Author's Contributions**

**Lebedev Alexander Alexandrovich:** Coordinated data analysis and prepared manuscript for publication.

**S.P. Lebedev:** Designed the research plan and organized the epitaxial growth experiments.

**E.V. Bogdanova:** Organized the capacitance-voltage measurements.

**N.V. Seredova:** Organized the photoluminescence measurements.

**L.V. Shakhov:** Organized the epitaxial growth experiments.

**Ethics**

This article is original and contains unpublished material. The corresponding author confirms that all of the other authors have read and approved the manuscript and no ethical issues involved.

**References**


