

# Interaction of silicon carbide with silicon melt formed under conditions of direct bonding of epitaxial structures of 3C-SiC/Si and 6H-SiC wafers

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The article presents the results of a study of the processes occurring at the interphase boundaries under conditions of the transfer of 3C-SiC epitaxial layers from Si substrates to 6H-SiC wafers by the direct bonding method. It is shown that the bonding is ensured by the formation of an intermediate layer of silicon melt. The source of the melt is the silicon substrate of the starting 3C-SiC/Si structures. The effects of the interaction of SiC with the Si melt are studied. The requirements for the properties of the initial materials are determined with the ultimate goal of obtaining of 3C-SiC/6H-SiC templates for homo polytype growth of 3C-SiC by sublimation method.

**Keywords:** Silicon carbide, polytypism, 3C-SiC, Si melt, direct bonding.

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## 1. Introduction

The cubic silicon carbide polytype 3C-SiC is promising for creating efficient semiconductor devices with high electron mobility based on it [1]. However, high-quality epitaxy of 3C-SiC is a technological problem due to the lack of industrial technologies for producing monocrystalline homopolytype substrates. Single crystal silicon substrates are used for epitaxial growth of 3C-SiC. The existing mismatches of the crystal lattice constants of 3C-SiC and Si (20%) and the coefficients of thermal expansion (8%) are the cause of the formation of structural defects, bending and cracking of epitaxial structures due to the thermal stress [2]. This problem can be solved by creation of a composite substrate (template) by the method of coordinated substitution of atoms [3] or, as we showed earlier, the transfer of heteroepitaxial layers of 3C-SiC from a silicon substrate to single crystal plates of 6H-SiC by direct bonding method at the temperature higher than silicon melting point [4]. In the last cited work, it was suggested that the key moment of the bonding process is the formation of a liquid phase of molten silicon, which is formed during the melting of the substrates of the used CVD 3C-SiC/Si structures. As is known, despite the high chemical resistance of SiC in aggressive environments, its interaction with melts of various compositions is observed. During this interaction, the processes of dissolution of SiC and solid-phase processes on its surface, as well as the simultaneously occurring processes of surface and volume diffusion lead to formation of new phases [5,6]. Therefore, the purpose of the presented work is to identify processes involving silicon melt under conditions used for direct bonding of epitaxial layers of 3C-SiC with 6H-SiC wafers.

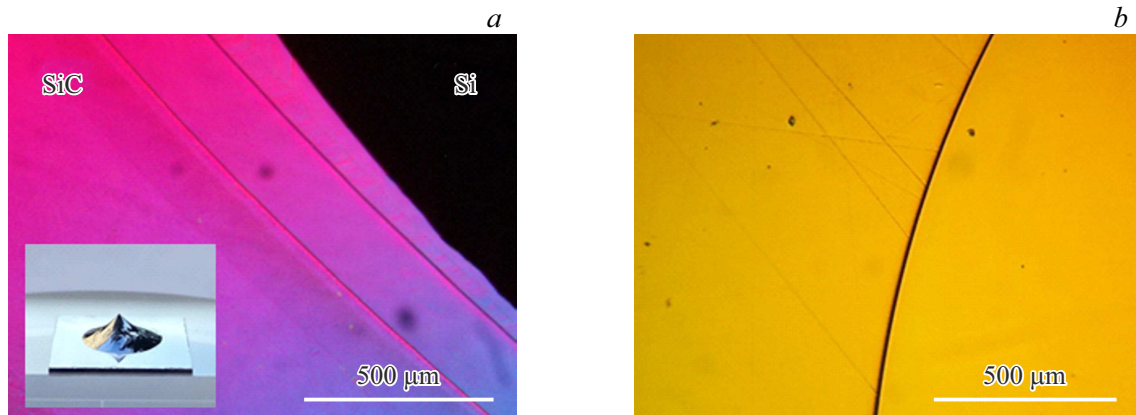
## 2. Samples and experimental procedure

6H-SiC wafers, samples of Si of electronic quality and structure 3C-SiC/Si with 10 μm thick epitaxial layers grown by chemical vapor deposition (CVD) were used in the study. The experiments were carried out on a lab scale plant equipped with high-frequency heating system under vacuum conditions 10<sup>-3</sup> Torr at temperatures above the melting point of silicon (1414°C). The microstructure of the surface of the experimental samples was studied using optical and atomic force microscopy (AFM) methods. The structure and chemical composition of the samples were studied using transmission electron microscopy (TEM) and Auger electron spectroscopy (AES).

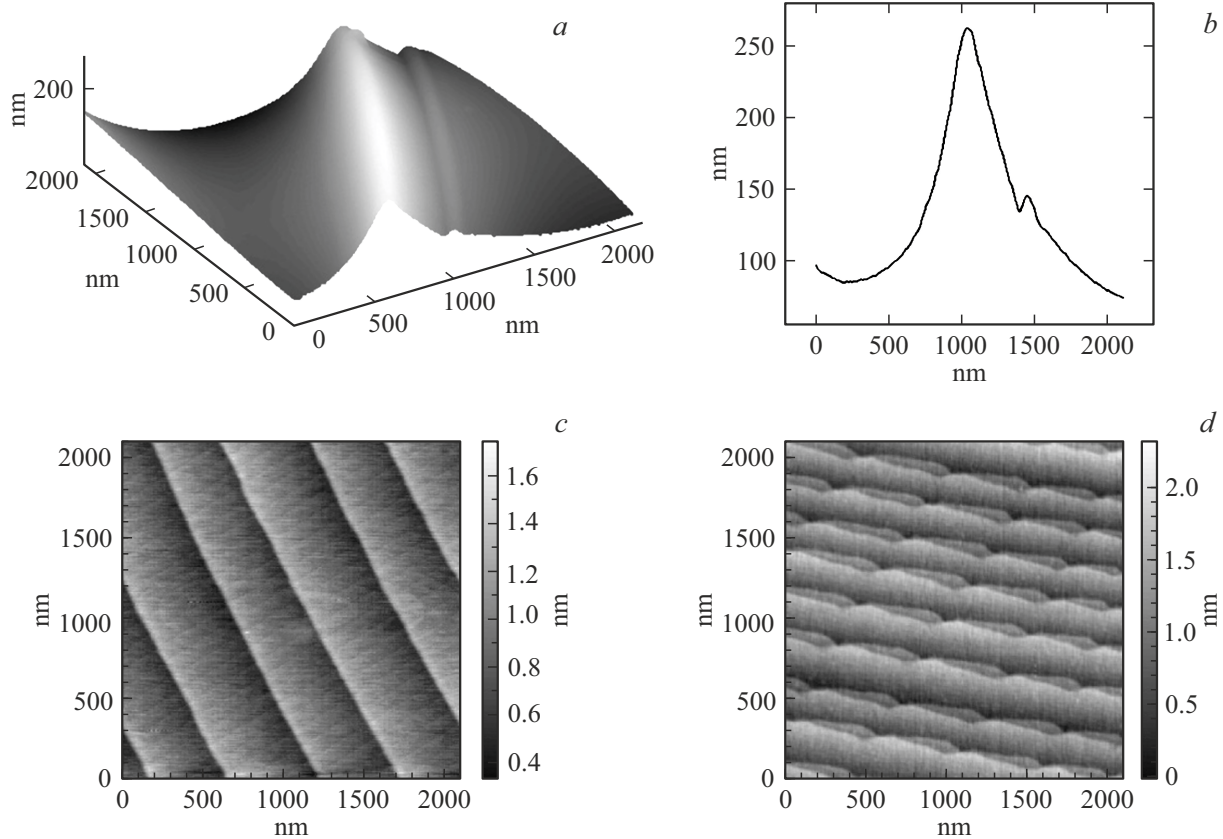
## 3. Results and discussion

The results of studies of the interaction of SiC with Si melt are provided below, in which silicon samples without an epitaxial layer 3C-SiC were used to obtain a melt on the surface of SiC wafers. After standard cleaning Si samples (with dimensions ~ 5 × 5 mm) were placed on the surface of 6H-SiC wafers (with dimensions 11 × 11 mm). Next, the samples were placed in a vacuum chamber and gradually heated up to a temperature of 1200–1500°C for 1.5–2 h with exposure at maximum temperature for half an hour. After cooling down to room temperature, the structures were taken out from the chamber. The silicon melt, which is solidified in the form of a droplet, was etched off in a mixture of nitric (HNO<sub>3</sub>) and hydrofluoric (HF) acids. Figure 1, *a, b* shows micrographs of the surface of the SiC substrate before and after removal of a droplet of molten silicon, respectively

Figure 2, *a–d* shows AFM images of the substrate surface after removal of the melt. Figure 2, *a* shows that a ridge-like



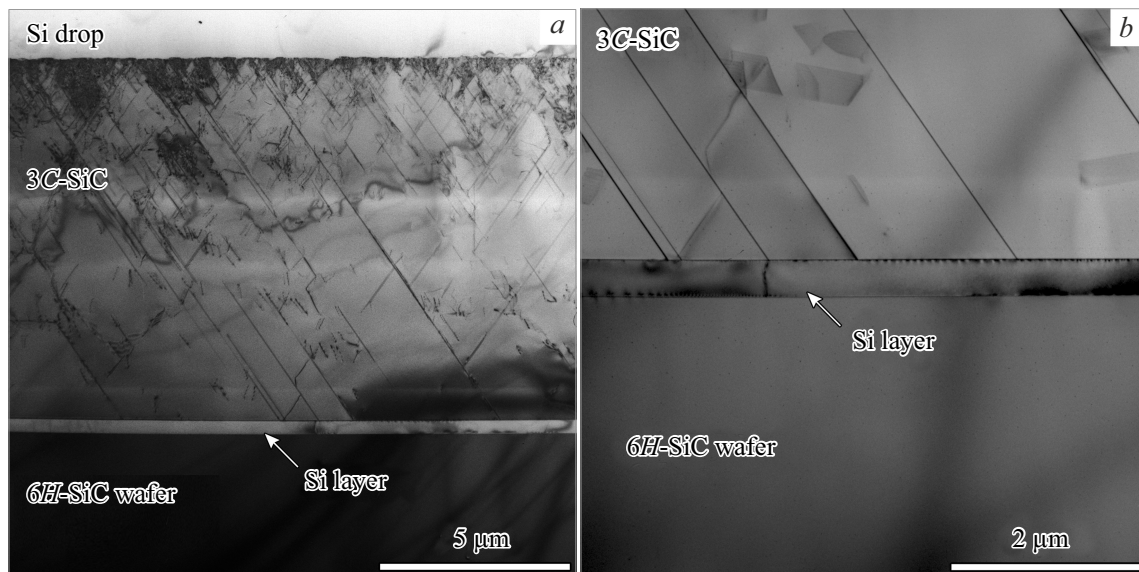
**Figure 1.** Micrographs of the 6H-SiC substrate surface: *a* — near a droplet of solidified melt obtained by DIC microscopy (droplet view on the insert); *b* — after Si etching. (A color version of the figure is provided in the online version of the paper).



**Figure 2.** AFM images of the 6H-SiC substrate surface: *a* — the area of the melt boundary and *b* — the corresponding AFM profile; *c* — steps on the surface, outside the melt zone; *d* — steps formed in the contact zone of the melt–SiC.

structure with a height of  $\sim 250$  nm is formed on the SiC surface on the periphery of the droplet. In addition, the effect of changing the position of Si droplet front, which is accompanied by the formation of concentric „traces“ on the substrate (Figure 1, *a*) was detected. The effects of ridge formation (ridging effect) in the area of the triple junction „solid-liquid-vapor“ were well studied. It is shown that they appear under conditions of high-temperature melt spreading as a result of surface diffusion of atoms of contacting

materials under action of surface tension. The movement of the melt front is a consequence of the reactive interaction, which results in the dissolution of the substrate surface [7,8]. The composition of various regions of the substrate-droplet structure was analyzed using AES. It was found that the upper volume of the droplet is formed by silicon with a carbon content at the level of units of percent (Si = 94%, C = 6%). The lower volume of the droplet is enriched with carbon.



**Figure 3.** TEM images: *a* — structures 3C-SiC/6H-SiC, *b* — interface image.

It was also found that in the area of SiC contact with the melt, a layer with a thickness of  $\sim 600$  nm is formed, where the content of the main components was: Si = 36%, C = 54%. The droplets of the solidified Si melt have a conical shape with a wide base and a narrow tip as shown in Figure 1, *a*. Taking into account the AES results, it can be assumed that the upper volume of droplets, which is pure silicon, actively evaporates during the annealing process. The lower carbon-enriched volume exhibits resistance to evaporation and corresponds to the „diffusion zone“ formed during the transition of carbon from the substrate to the melt. Thus, the shape of the solidified droplet reflects the presence of a reactive interaction of SiC with the liquid phase of Si and is due to the vertical gradient of carbon concentration.

Figure 2, *c, d* shows AFM images of the Si face of the SiC wafer in the area of contact with the melt and beyond. It can be seen that atomically smooth ordered steps with a terrace width of  $\sim 500$  nm, have formed on the surface outside the droplet, which is a typical consequence of high-temperature annealing of SiC in vacuum [8]. At the same time, the structure of the surface that was in contact with the melt has significant differences. The effects of reconstruction of the SiC surface at the boundary with melts have been described in a number of theoretical and experimental papers. It is shown that the solid-phase microstructuring process of the substrate surface accompanies the process of its dissolution in the liquid phase [9]. Under the conditions of our experiments, the dissolution of SiC occurs to a depth at least equal to the thickness of the disturbed layer. This follows from Figure 1, *b*, where it can be seen that traces of mechanochemical polishing remain on the SiC surface outside the melt area, and no traces of processing are observed on the surface in contact with the melt. Experiments were also conducted using 6H-SiC wafers with different misorientation. The „macroscopic“ effect of change

of the position of the Si droplet was observed within the framework of these experiments, which is more pronounced in the case of substrates with a higher misorientation.

The following are the results of direct bonding of 3C-SiC/Si CVD structures with single-crystal SiC substrates (the experimental scheme see in Ref. [4]). TEM studies have shown that in cases where strong mechanical contact between the 3C-SiC layers and the 6H-SiC substrates is formed, an intermediate silicon layers are present at their boundaries, which survive after the melt droplet is removed from the surface of the bonded structures (see Figure 3). The figure shows a view of cross-sections of the resulting structure 3C-SiC/6H-SiC: an overview image where you can see the defective structure of the transferred CVD layer with multiple stacking faults (Figure 3, *a*), and a snapshot of the interface area where a layer of solidified silicon melt is clearly visible (Figure 3, *b*). It was also found by the TEM method that the reactive interaction with the melt is more pronounced in the case of 3C-SiC. As a result, the thickness of the transferred layers decreased from  $10\ \mu\text{m}$  to  $7\text{--}8\ \mu\text{m}$ .

## 4. Conclusion

The following main conclusions can be drawn based on the results obtained in this study. Direct bonding of 3C-SiC epitaxial layers to 6H-SiC wafers occurs with the formation of intermediate layer of molten silicon. Si substrates on which the transfer layers were grown serve as the melt source. The interaction of silicon carbide with silicon melt is accompanied by solid-phase surface microstructuring processes and the formation of a carbon-rich layer in the area of melt-SiC surface contact. Considering the observed effect of liquid phase movement of the melt along the vicinal surfaces, it was concluded that the use of SiC wafers with minimal surface misorientation angles is preferable. Taking

into account the established reactivity of SiC with the melt, it is preferable to use transferred layers of 3C-SiC with a thicknesses exceeding  $3\ \mu\text{m}$ .

Thus, experimental confirmations of the assumption previously expressed in Ref. [4] were obtained, namely, that the direct bonding of epitaxial layers of 3C-SiC with 6H-SiC wafers under conditions of high-temperature induction heating in vacuum occurs with the participation of the liquid phase of the silicon melt. The effects of SiC interaction with the melt, which can affect the properties of the composite substrates of 3C-SiC/6H-SiC have been determined. The basic requirements for the properties of the initial layers of 3C-SiC and 6H-SiC wafers were formulated.

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### Conflict of interest

The authors declare that they have no conflict of interest.

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