Reviews on Advanced Materials and Technologies, 2025, vol. 7, no. 1, pp. 18–23
DOI: 10.17586/2687-0568-2025-7-1-18-23

Formation of Interfaces in Direct Bonded Heteropolytype SiC Structures Mediated with Liquid and Vapor Phase of Silicon

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Article history

Received March 10, 2025
Accepted March 24, 2025
Available online March 31, 2025

Abstract

It is shown that heteropolytype silicon carbide structures can be obtained by direct bonding of wafers of different SiC polytypes by high-temperature treatment in vacuum. Heteroepitaxial 3*C*-SiC layers grown by the CVD method on a Si substrate were successfully transferred to 6*H*-SiC wafers. It was found that nanometer-thick bonding layers formed at the 3*C*-SiC/6*H*-SiC interface were the layers of recrystallized melt originating in a meltdown of the Si substrate of starting 3*C*-SiC/Si specimens. This example of transferring is a promising way for producing 3*C*-SiC/6*H*-SiC template for growing homoepitaxial 3*C*-SiC films of device quality. Feasibility of direct bonding of SiC single-crystal wafers in a silicon vapor environment also demonstrated. The motivation for these studies is development of prospective power devices on the base of 4*H*-SiC/6*H*-SiC heteropolytype junctions. It is shown that a necessary condition for bonding is a gap capable of providing vapor transport at the interface between the wafers. The gap was obtained by preliminary self-structuring of the surface of bonded SiC wafers with their annealing in vacuum.

Keywords: Silicon carbide; Direct wafer bonding; Heteropolytype structures; Silicon; Liquid and vapor phase

1. INTRODUCTION

Silicon carbide is considered as strong competitor to silicon in high-temperature and high-voltage device application due to its unique physical properties such as wider band gap, higher thermal and corrosion resistance, as well as higher blocking voltage, lower losses and faster switching speed [1]. Compared to hexagonal polytypes (4*H*- and 6*H*-SiC), which are widely used for the development of advanced power devices, cubic polytype 3*C*-SiC is a promising material for high-frequency transistors as well as for a number of new applications in photonics, micro-electromechanical devices, and biomedical technologies [2,3].

A serious challenge in 3*C*-SiC technology is the lack of methods for the "bulk" growth of single-crystal material required for the manufacture of substrates for 3*C*-SiC homoepitaxy. Seed sublimation, also called physical vapor transport (PVT), remains to be the most common method for growing 6*H*- and 4*H*-SiC ingots. In contrast, up to now, bulk 3*C*-SiC single crystals not yet fully realized, mainly due to metastable nature of 3*C*-SiC. This is due to solid phase transition of cubic to hexagonal phase of SiC under the operating conditions of PVT growth processes [4].

The lack of high quality 3*C*-SiC substrates forces 3*C*-SiC to be grown heteroepitaxially by CVD (Chemical Vapor Deposition) on monocrystalline Si substrates [5]. Alternative methods of formation of 3*C*-SiC layers on Si substrates are also known, such as coordinated substitution of atoms [6], or rapid vacuum thermal processing of Si wafers [7]. However, the use of such 3*C*-SiC/Si templates for subsequent epitaxy by methods requiring growth

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temperatures above the melting point of Si (1414 °C) appears to be difficult. In particular, this concerns homoepitaxy of 3*C*-SiC by vacuum sublimation method with typical growth temperatures above 1550 °C.

A solution to this problem can be a proper template fabricated through transferring CVD-grown 3*C*-SiC layers from a Si substrate to single crystalline SiC wafers with a wafer bonding method. Nowadays wafer bonding methods are widely used for integration of dissimilar materials in order to prepare new type of heterogeneous structures for wide variety of applications [8]. In this work, we show implementation of wafer bonding technique for fabrication of 3*C*-SiC/6*H*-SiC templates suitable for subsequent homoepitaxial growth of 3*C*-SiC by sublimation [9] and formation of 4*H*-SiC/6*H*-SiC structure promising for the development of power devices based on heteropolytype junction [10–12]. We used a direct bonding approach without applying external mechanical pressure and without preliminary application of any intermediate adhesive or sputtered layers. Peculiarities of interfaces of the bonded structures formed at different conditions of the experiments are considered.

2. MATERIALS AND METHODS

Commercially available (CREE, Inc.) monocrystalline (0001) 6*H*-SiC wafers and 3*C*-SiC CVD-grown (001) 3*C*-SiC/Si structures (NOVASIC) with a 10 µm-thick epitaxial layer were used for experiments of transferring of cubic epitaxial layers from Si substrates to SiC wafers. The sample sizes were 1.0×1.0 cm and 0.6×0.6 cm, respectively. Heteropolytype 4*H*-SiC/6*H*-SiC structures were obtained by direct bonding of specimens of the two polytypes with a size of 1.0×1.0 cm. All experiments were carried out at temperature T = 1300–1550 °C in a vacuum chamber with induction heating system.

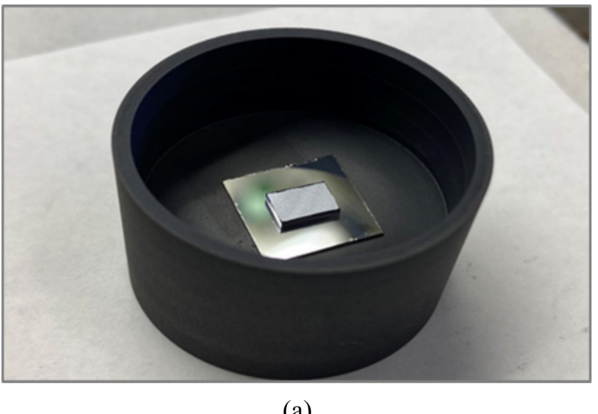
Structural characterization of the specimens was done with optical and atomic force microscopy (AFM) and transmission electron microscopy (TEM). TEM investigation was carried out with a Philips EM420 microscope operating at accelerating voltage of 100 kV. Elemental composition of the samples was investigated with Auger electron spectroscopy (AES).

3. RESULTS AND DISCUSSION

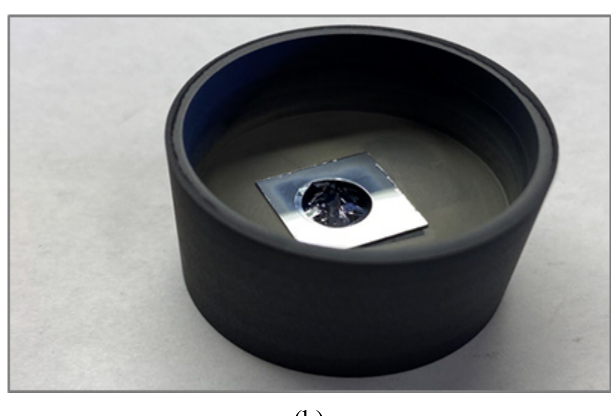
To transfer CVD epitaxial layers from Si substrates to SiC wafers, 3*C*-SiC/Si and 6*H*-SiC samples were placed together in a graphite crucible with the 3*C*-SiC layers facing down, towards the surface of the 6*H*-SiC sample (Fig. 1a), and then heated up to 1500 °C in a vacuum chamber. Melting of the silicon substrate of 3*C*-SiC/Si structure was achieved under these conditions. This resulted in formation of a droplet of Si melt upon the sandwiched samples as shown in Fig. 1b. Full fusion of the samples was observed when the Si droplet extended beyond the edges of the specimen with the 3*C*-SiC layer and partially covered the surface of the SiC substrate.

After the solidified droplet was etched away in a mixture of hydrofluoric (HF) and nitric (HNO_3) acids, we discovered that the *3*C-SiC layers were successfully transferred to SiC wafers, demonstrating a mechanically strong contact with them. Series of TEM examination of the interface region of the newly formed 3*C*-SiC/6*H*-SiC structures showed that the contact was achieved through formation of 2 to 30 nm-thick Si interlayers between the transferred film and the substrate (Fig. 2). Visible linear features in 3*C*-SiC layer are stacking fault defects. It is obvious that formation of Si interlayers is a result of melt flowing into the interface space of the samples. This phenomenon could be tentatively attributed to the thermocapillary flow effect [13].

For more details on the transmission electron microscopy study of the bonding interface formation during the transfer of 3*C*-SiC layers onto 6*H*-SiC wafers, see the recent publication [14].



(a)



(b)

Fig. 1. View of the samples in the crucible: (a) before and (b) after annealing.

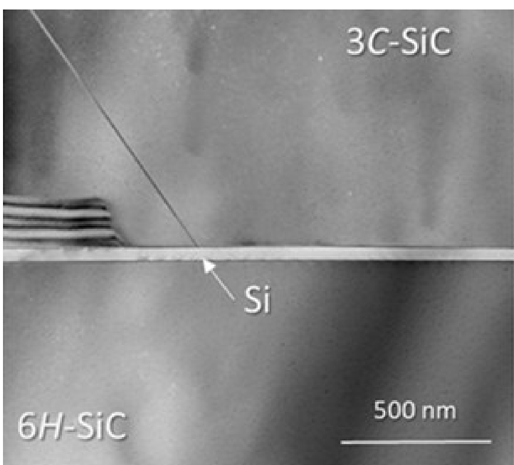


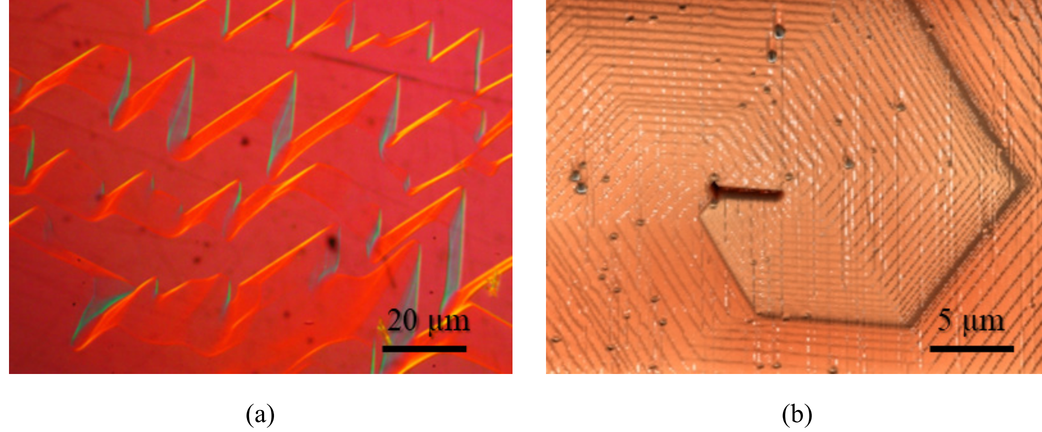
Fig. 2. Cross-sectional TEM-image of a 6*H*-SiC/3*C*-SiC boundary with a Si interlayer.

The structure of the upper interface between the bonded 3*C*-SiC layers and the Si melt was also investigated. An optical microscope image of the back surface of the transferred CVD-grown layer taken in differential interference contrast (DIC) after removing of solidified droplet of Si melt by wet etching is shown in Fig. 3a. As can be seen, the surface has a stepped microstructure with atomically flat (according to AFM analysis) triangle-shaped terraces with height of sidewall facets ~ 100 nm. Orientation of the terrace planes has some deviations relative to the basal plane (0001) of the 6*H*-SiC substrate. At present, the exact reason for the formation of such surface pattern is unclear. We consider two possible options. The first one relates to the effect of the interaction of 3*C*-SiC with the Si melt at high temperature [15]. In particular, results of sessile drop approach implemented for the study of morphology evolution of 4*H*-SiC wafers in contact with Si melt showed that the interaction results in formation of steps on the wafer surface [16]. The second possibility is the realization of self-organized growth process from the Si melt enriched with carbon. This assumption is supported by the observation of spiral pattern at the same surface of 3*C*-SiC layer (Fig. 3b). The pattern could be related to 6*H*-SiC domain formation by of dislocation-controlled spiral growth mode [17–19].

Besides this, AES elemental analysis of a solidified Si droplet near the boundary with the 3*C*-SiC layer revealed a carbon-rich region with a thickness of ~ 600 nm, where the content of the main components was Si = 36% and C = 58%. This indicated that under the annealing conditions, diffusion mass transfer of carbon from the substrate to the melt occurred. By saturating Si melt with carbon, one may provide epitaxial growth from the liquid phase upon the backside surface of transferred CVD-grown 3*C*-SiC layer.

Bonding of 4*H*-SiC (0001) and 6*H*-SiC (0001) wafers was carried out in an atmosphere of silicon vapor. The source of the vapor was pieces of monocrystalline Si wafers. Before bonding experiments, SiC samples were pretreated in vacuum at 1300 °C to form a crystallographic step-terrace relief on their surface. This should have provided a gap for better vapor penetration [20]. AFM image of the stepped surface of the processed 4*H*-SiC wafer is shown in Fig. 4a. While the height of the steps varies in the range 1.8 to 3.6 nm, the width of (0001) plane terraces is 4–5 µm. Formation of such straight-ordered terraces on the surface of SiC wafers subjected to high-temperature annealing is a well-studied phenomenon and is associated with sublimation of atoms and subsequent surface reconstruction [21].

At the next step of the experiment, the processed SiC wafers were placed face-to-face in a graphite crucible. As Si evaporates from its liquid phase at the temperatures higher than its melting point [22], to obtain a vapor, pieces of Si wafers were placed in a separate cell within the main graphite crucible together with the specimens to be bonded. Bonding proceeded at 1550 °C after pumping out



(a) (b)

Fig. 3. (a) DIC microscopy image of back side surface of transferred 3*C*-SiC layer taken after Si droplet removal; (b) AFM image of the area with spiral hillocks growth.

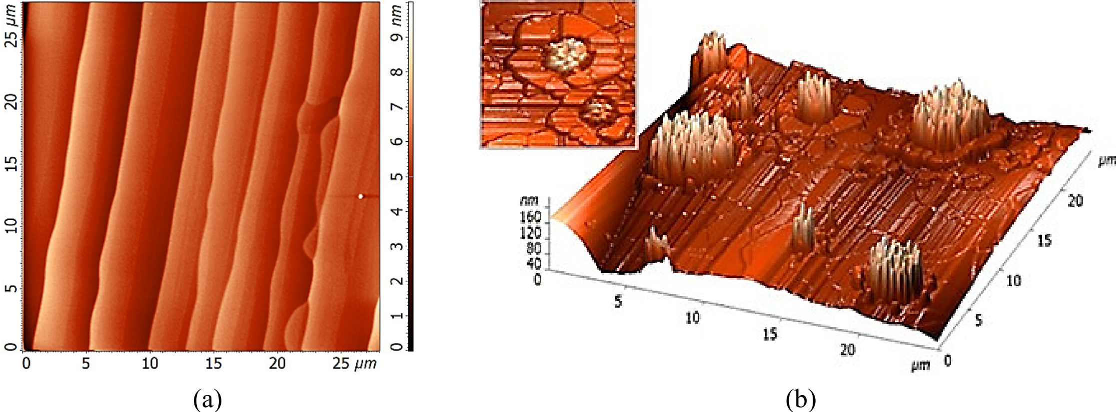


Fig. 4. AFM images of stepped surface of 4*H*-SiC wafer after preliminary annealing (a) and after annealing in Si vapor ambient (b).

the chamber to 10^{-5}–10^{-6} Torr pressure. When cooled down to the room temperature, the samples were found to be stacked together. To study the interface structure, the bonded wafers were mechanically separated. Optical microscopy and AFM studies showed that both separated surfaces have a similar structure, which was radically different from the stepped structure obtained by the preliminary annealing in vacuum. AFM image of the surface of the 4*H*-SiC wafer is shown in Fig. 4b as an example.

The most intriguing fact is the observation of structured "hillocks" on the surfaces of debonded specimens (Fig. 4b). The hillocks look as an agglomeration of microcrystallites elongated in the [0001] direction, which is perpendicular to the plane of the 4*H*-SiC substrate surface. Diameter of the base of the hillocks is 2–10 µm, the height of the microcrystallites is up to 40 nm. It is also seen that the "ring"-like structures have formed at the periphery of the base of the hillocks (see inset in Fig. 4b).

At this stage, we assume that both types of structures were formed by the "Vapor-Liquid-Solid" mechanism according to the following scenario. Si atoms from the vapor phase enter the gap between two SiC plates, where they segregate and form a liquid phase in the form of microdroplets. Newly formed liquid phase serves as a pathway for the diffusion mass transfer of carbon atoms from the solid SiC as is shown above. Agglomeration of SiC microcrystallites forms inside the Si microdroplets. Formation of SiC "rings" structures occur at triple vapor–liquid–crystal contact (i.e., along droplet base). At the same time, it cannot be ruled out that the microstructures represent recrystallized silicon. The exact mechanism of the formation of the microcrystalline phase in the narrow gap between fused SiC wafers will be determined after completing investigation of its properties with micro-Raman spectroscopy and microcathodoluminescence.

Still, the above results showed that we succeeded in forming fused SiC air-gap structure filled with vapor species. It should be noted that development of "air-gap technology" is considered to be a promising way for a variety of other important applications in semiconductor science, for example, for the stress management in heteroepitaxial structures [23,24], for the development of microelectromechanical sensors [25,26], for power generation devices based on tunneling-thermionic emission [27,28].

4. CONCLUSION

We demonstrate that heteropolytype structures can be obtained by direct bonding of SiC wafers with contribution by the Si melt or Si vapor at high temperature. Observed effects, related to the flow of liquid and vapor phases of silicon in a narrow confined space, is an interesting subject for fundamental research to develop physical foundations for applied materials science in the field of prospective technologies of semiconductors. Further research will be aimed at achieving optimal Si bonding interlayers and the air gap parameters and its filling process as well as testing the impact of the of properties of intermediate layers on the electrical characteristics of the heteropolytype SiC structures.

ACKNOWLEDGEMENTS

This study supported by the Russian Science Foundation, project no. 24-22-00232. TEM characterizations were performed using equipment of the Federal Joint Research Center "Material science and characterization in advanced technology" supported by the Ministry of Education and Science of the Russian Federation (id RFMEFI62117X0018).

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УДК 538.91

Формирование интерфейсов в гетерополитипных структурах SiC, полученных методом прямого сращивания с участием жидкой и паровой фаз кремния

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Аннотация. Методом прямого сращивания в условиях высокотемпературного отжига в вакууме получены гетерополитипные структуры 3*C*-SiC/6*H*-SiC и 4*H*-SiC/6*H*-SiC. Мотивацией данных исследований является создание технологии получения комбинированной подложки 3*C*-SiC/6H-SiC для гомоэпитаксиального роста слоёв 3*C*-SiC приборного качества и перспективных силовых приборов на основе гетерополитипных переходов 4*H*-SiC/6*H*-SiC. Исходными материалами служили гетероэпитаксиальные структуры 3*C*-SiC/Si, полученные химическим осаждением из паровой фазы, и пластины монокристаллического карбида кремния политипов 6*H*-SiC и 4*H*-SiC. Контакт между сращиваемыми материалами в первом случае был получен за счёт формирования промежуточного слоя рекристаллизованного расплава кремния, возникающего при расплавлении Si подложки исходных образцов 3*C*-SiC/Si, а во втором, — за счёт формирования связующих микроструктур в зазоре между пластинами SiC, который обеспечивал транспорт паров кремния по границе раздела. Зазор был получен посредством реализации процесса самоструктурирования поверхности сращиваемых пластин в ходе их предварительного отжига.

Ключевые слова: карбид кремния; прямое сращивание; гетерополитипные структуры; кремний; жидкая и паровая фаза