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SiC voids, mosaic microstructure and dislocations distribution in Si carbonized layers

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Abstract

The defect structure of SiC/Si layers obtained by carbonization of Si is reported by means of transmission electron microscopy in high-resolution (HREM) and conventional (CTEM) modes. 3C–SiC was obtained after a rapid thermal annealing treatment and good interfacial quality is reported in terms of small void dimensions and densities. Moreover, high misfit dislocation densities are observed close to the Si/SiC interface and inside the SiC layer without observable generation of threading dislocations. The mosaic grain structure is also evidenced, with low misorientation with respect to the substrate. These results are encouraging for further growth of III–N alloy heterostructures.
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Keywords: Silicon carbide (3C–SiC); Si carbonization; High-resolution transmission electron microscopy (HRTEM); Rapid thermal chemical vapor deposition (RTCVD)

1. Introduction

Silicon carbide (SiC) is one of the most versatile wide-bandgap semiconductor materials, as it can either be homoepitaxially grown on commercial substrates or obtained from Si by carbonization. Thus, it is attractive for both mechanical and electronic applications such as micro-electro-mechanical systems (MEMS) [1] and for purely electronic ones as a result of its high thermal conductivity, high electron mobility (for β-SiC, 1000 $cm^2 V^{-1} s^{-1}$), high saturated drift velocity ($>10^7$ cm s^{-1}) and high breakdown field [2]. Moreover, its lattice parameter is very close to that of AlN, making this material a unique substrate for III–V technology [3]. However, the price and dimensions available for SiC substrates are still not tempting for industrial applications. An alternative is Si carbonization [4,5] followed by epitaxial growth. This allows monolithic integration of Si and SiC, making the latter compatible with Si technology.

Here a conventional transmission electron microscopy (CTEM) and high-resolution transmission electron microscopy (HRTEM) study of dislocation distribution, grain size and orientation, and void distribution is reported to show the potential of the Si carbonization treatment. The grain orientation is assessed using high-resolution Moiré contrasts that corroborate selected-area electron diffraction (SAED) results.

2. Experimental technique

Single-side polished (001) and (111) Si wafers were carbonized in an RTCVD system [6] by a rapid thermal process using a mixture of 0.15% propane in H_2 during 60 s with a final temperature of 1280 °C and a ramp rate of 50 °C s^{-1}.

TEM, HREM and SAED were the techniques used to study the inner crystalline structure of the samples. Specimens were prepared by cross-section TEM (XTEM) and plan-view TEM (PVTEM) using mechanical thinning and Ar^+ milling at 4.5 kV in a Gatan dual ion mill system. Conventional TEM was carried out in a JEOL JEM-1200EX electron microscope and a JEOL JEM-2000EX/THR was used for HRTEM. Filtered images were taken using a CCD camera attached to the JEM-2000EX microscope and were processed using DIGITAL MICROGRAPH software.

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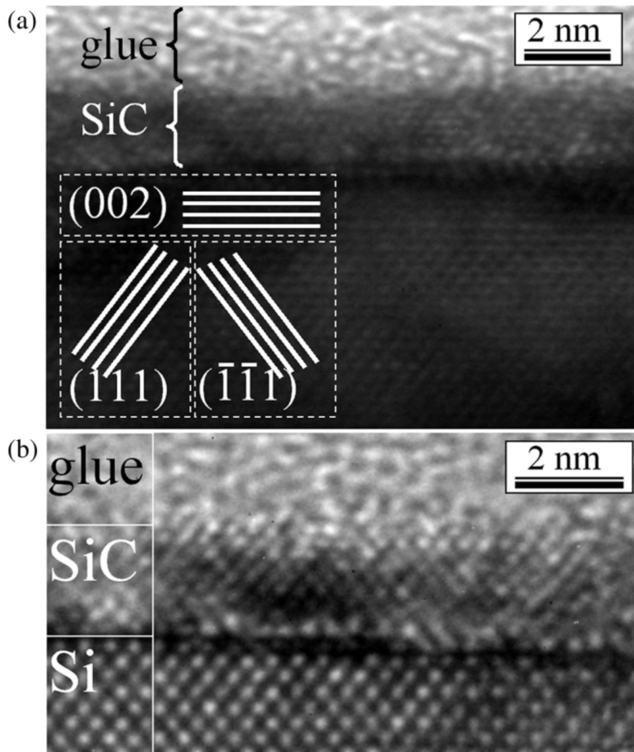


Fig. 1. (a,b) HRTEM micrographs demonstrating the surface and interface quality of epitaxial 3C–SiC formed on silicon substrates.

3. Results

Preliminary studies on these structures showed the formation of ultra-thin β-SiC(001)//Si(001) and β-SiC(111)//Si(111) structures. SiC exhibited well-orientated nano-layers, good crystalline quality and homogeneity in 3-inch. wafers and possessed a parallel continuous and homogeneous thickness all over the specimen surface [7].

Fig. 1 shows HREM micrographs in cross-section orientation for the (001) sample along the $[1\bar{1}0]$ zone axis. The (002), (111) and $(\bar{1}\bar{1}1)$ silicon planes are easily observed. The SiC obtained consists of approximately 10 atomic (002) planes of the cubic polytype (β-SiC). The interface is shown to be very smooth with an epitaxial type of growth. However, contrasts in the SiC layer indicate the presence of some lattice relaxation due to dislocation formation close to the interface.

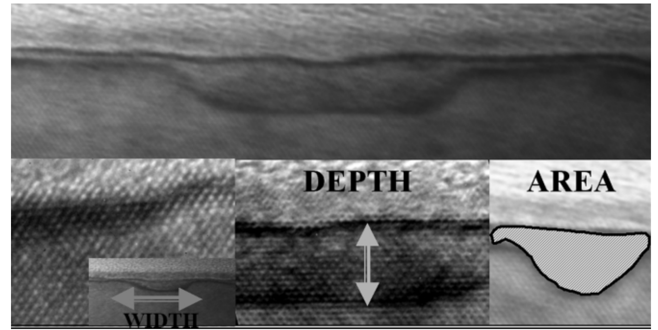


Fig. 3. Void dimensions and HRTEM details of their structure.

To understand such contrasts, a HREM micrograph of a large area is presented in Fig. 2a. The three rectangles correspond to filtered regions to demonstrate the presence of dislocations. An exhaustive study to deduce residual strain in the SiC layer using such filtering and to compare micro-Raman spectroscopy and HREM Moiré data is in progress. Here, only qualitative results are presented. Fig. 2b corresponds to the left-hand-side rectangular filtered region. Misfit dislocations located just at the Si/SiC interface are denoted by white dashed circumferences, while dark dashed circumferences correspond to dislocations located inside the SiC layer. By choosing and filtering the other possible (111) plane family, the dislocation behavior can be deduced. Both 60° and 90° dislocation types are evident, but no threading dislocations induced by misfit dislocation interactions were observed.

In Fig. 3, voids located in the Si are shown. This well-known feature is undesirable and a low density of these defects is present in our samples due to an optimized self-limiting conversion mechanism of the Si surface into SiC. High temperatures are usually required to attain good crystalline quality, but at these temperatures Si out-diffusion generates these Si voids [8]. Reducing the carbonization time at these high temperatures allows a reduction in their dimensions and density under certain conditions. Indeed, no voids were observed

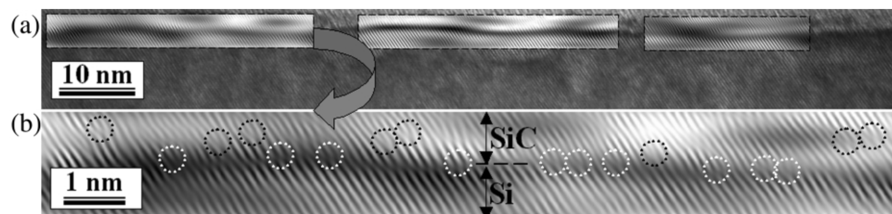
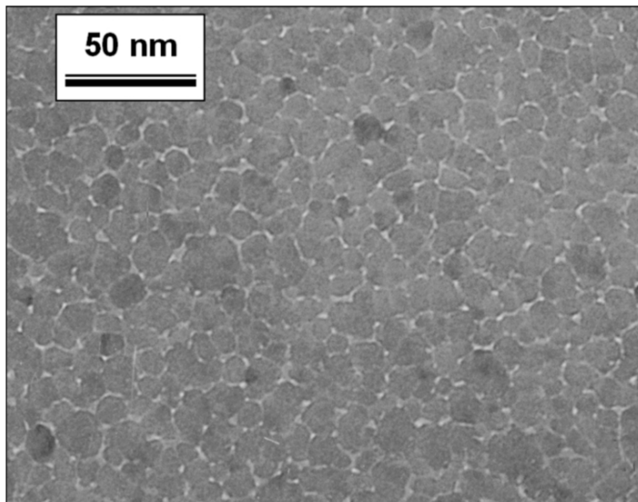
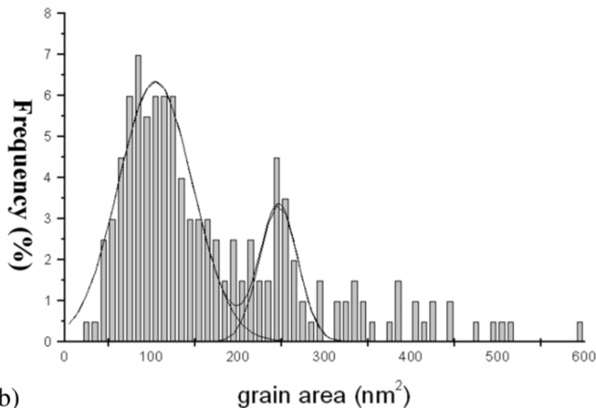


Fig. 2. (a) Insets show filtered regions of the HREM image of a XTEM preparation of the (001) sample where $(\bar{1}\bar{1}1)$ Si and SiC planes are shown. (b) Misfit dislocations are revealed by image filtering and marked with dashed circumferences.



(a)



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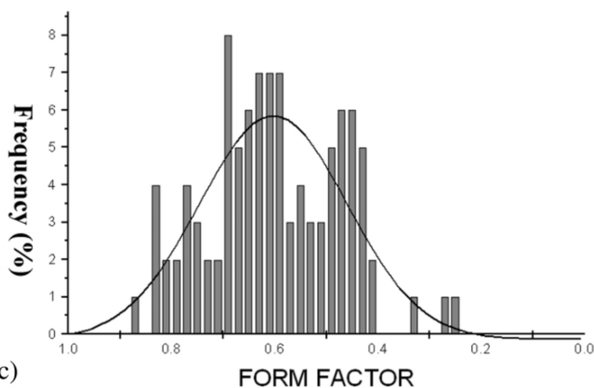

(c)

Fig. 4. (001) mosaic structure. PVTEM of (a) grain boundaries, (b) sizes and (c) form factors.

in PVTEM micrographs due to their small size and low density. Moreover, HREM allowed us to determine the main features of the voids. The average void width and depth observed amount to 18.5 ($\pm$1.0) and 3.2 ($\pm$0.5) nm, respectively. The average separation distance between voids is 15 ($\pm$4) nm. The values in brackets take into account the data range observed for different regions along the (001) wafer studied. These values correspond to an observation normal to the $(1\bar{1}0)$ surface along a $[\bar{1}10]$ line scan.

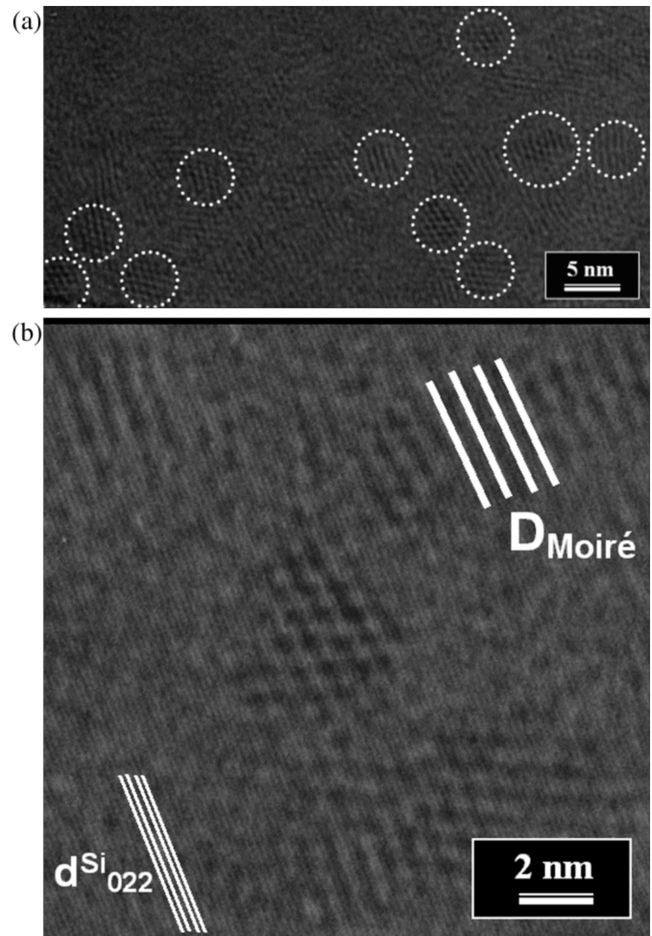

Fig. 5. (a) Plan-view HRTEM image, with Moiré contrasts due to SiC–Si overlap indicated. (b) Magnified detail of HRTEM image (a) with Moiré fringes and {022} Si planes shown.

The mosaic structure induced by the very large lattice mismatch between Si and SiC (approx. 20%) is revealed by the plan-view micrograph of Fig. 4. The grain size is shown to fit a double Gaussian distribution centered at values of approximately 104 and 247 nm^2 (Fig. 4b). The average form factor indicates a quasi-circle shape, as shown in Fig. 4c. In addition to their size, the misorientation of grains with respect to the (111) substrate can be obtained from micrographs, as shown in Fig. 5. The Moiré contrast generated by the SiC grains and the Si substrate lattice in the electron transparent preparation allows determination of the lattice parameter of each grain and its misorientation.

Fig. 5a shows such contrast in plan-view orientation in HREM mode. The ion milling preparation could reduce the grain size in the TEM specimen prepared. However, the white dashed circles indicate some of those Moiré contrasts. Fig. 5b shows the {002} Si lattice planes and their respective Moiré contrasts.

Moiré effects have been extensively used for a long time and equations relating both lattice parameter and misorientation are well known. The formalism introduced by Hirsch et al. [9] and used by other authors for Si/SiC systems [10] is the one used here. The grain misorientation deduced with respect to the substrate is approximately $\pm 2.5°$ and the {022} SiC average interplanar distance measured remains at approximately 0.1551 nm, logically larger than the relaxed parameter (0.1538 nm) due to the expansion induced by the Si substrate. Thus, the average elastic deformation of SiC deduced with respect to the bulk material is -0.0084 (0.8% extension) with an actual misfit between SiC and Si of 0.1909 (19.09%).

4. Conclusions

A high-resolution electron microscopy (HREM) study allowed the characterization of SiC voids, the mosaic microstructure and dislocation distributions in Si carbonized layers. The grain misorientation generally remains below $\pm 2.5°$, while the grain size distribution shows a double Gaussian distribution centered at approximately 100 and 250 nm^2. The voids lying underneath the SiC layer maintain a reasonably reduced size (approx. 42 nm^2) as a result of the rapid thermal process. Finally, misfit dislocations inside the SiC layer and at the SiC/Si interface were evidenced by HREM. Lomer and 60° dislocations were observed in both locations. Large-area misfit dislocation counting allows deduction of the average residual strain of the SiC layer. No threading dislocations were revealed by cross-section observations due to the fact that the large misfit is mainly relieved at the interface by misfit dislocations and low-angle boundaries. Therefore, epitaxial overgrowth on such carbonized structures is expected to lead to high-quality wide-bandgap materials. In conclusion, the good crystalline quality reported motivated further research into SiC and GaN regrowth that will be presented in the near future [11].

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