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Electron microscopy study of SiC obtained by the carbonization of Si(111)

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Abstract

A SiC thin film grown by propane carbonization of a Si(111) substrate has been characterized by transmission electron microscopy and scanning electron microscopy techniques. This study reveals the presence of planar defects in the SiC layer and voids in the Si(111) substrate as well as misfit dislocations at the SiC/Si interface. The resulting SiC layer consists of a mosaic structure and is shown to have low stress.
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1. Introduction

Recently, SiC have attracted a lot of interest as a basic material for a multitude of applications. Indeed, in addition to some direct opto- and micro-electronic devices as an emitting diode for visible spectra and for high power and temperature microelectronic applications, SiC is a suitable candidate as a substrate for III-N growth. The deposition of SiC on Si makes it cheaper and compatible with Si technology.

In this work a SiC film obtained by propane carbonization of Si(111) is investigated by scanning electron microscopy (SEM), cross-sectional transmission electron microscopy (XTEM), including high resolution electron microscopy (HREM), transmission electron diffraction (TED) and plan-view transmission electron microscopy (PVTEM).

2. Experimental technique

The studied sample was produced by the rapid thermal chemical vapour deposition (RTCVD) technique. A SiC thin film was obtained by propane carbonization of a Si(111) substrate at 1250°C with a flow rate of 15 sccm of C_3H_8 and 1 slpm of H_2 at atmospheric pressure. The temperature ramp rate was 25°C/s. More details about the growth technique and the RTCVD system can be found in Ref. [1]. These growth conditions resulted in a SiC film with thickness of about 40 nm (determined by XTEM).

SEM specimens were observed in a Jeol 820 JSM. The conventional TEM studies were performed on a Jeol 1200-EX transmission electron microscope operating at 120 kV and the HREM analysis was carried out in a Jeol 2000-EX working at 200 kV. The TEM specimens preparation was achieved by Ar^+ ion milling after mechanical thinning.

3. Experimental results and discussion

To investigate the crystal structure of the SiC film, SEM, PVTEM XTEM, TED and HREM techniques were used. The selected area TED (SAED) pattern shown in Fig. 1, taken from a cross-section prepared specimen, shows a good SiC film crystallinity. The distance between the main diffracted spots associated to an epitaxial cubic SiC in SAED patterns (Fig. 1) are constant in the whole layer. The existence of cubic SiC was confirmed by the analysis of intensity profiles in HREM images acquired with a Gatan slow scan CCD camera set in the Jeol 2000-EX microscope. (Fig. 2) The analysis method used here has been previously utilized with success [2].

The lattice parameter, even for monolayers close to the SiC/Si interface, corresponds with the relaxed bulk lattice constant of cubic SiC. These results provide evidence that the SiC layer immediately takes the unstrained SiC bulk structure which is slightly disorientated to the Si substrate, as we have observed in ⟨110⟩ HREM images of the SiC/Si interface. Therefore, the relaxation of strain between the

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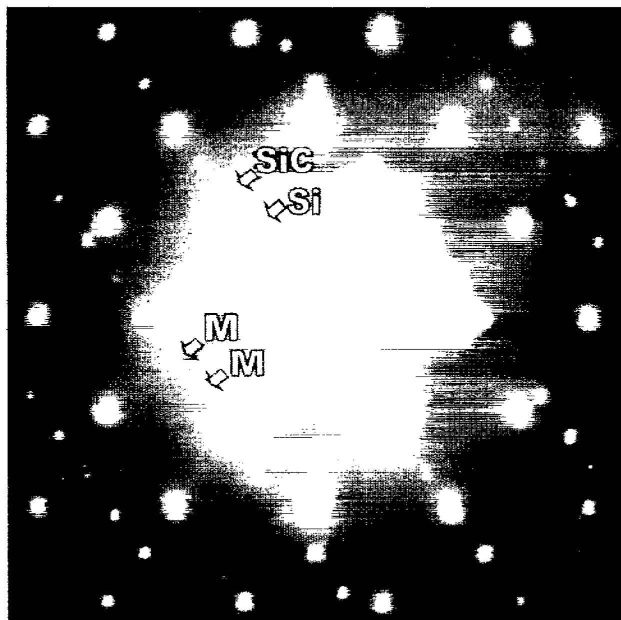

Fig. 1. The ⟨110⟩ selected area transmission electron diffraction pattern of the SiC/Si system showing the zinc-blende structure of both materials. Extra spots are associated to planar defects in the SiC layer.

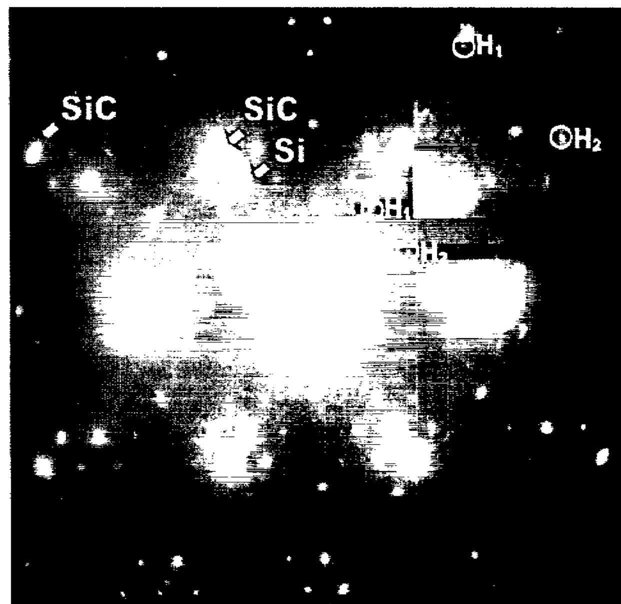

Fig. 3. The ⟨111⟩ selected area transmission electron diffraction pattern of the SiC/Si system showing the zinc-blende structure of both materials.

two materials seems to be carried out through a high defect density at the SiC/Si interface.

In the SAED pattern of Fig. 1 some extra spots are also visible. Those spots labeled as M can be associated with planar defects. Such defects have been also observed in conventional XTEM and HREM images.

From the SAED pattern of Fig. 1 the SiC layer seems to be monocrystalline. Nevertheless, SAED patterns taken

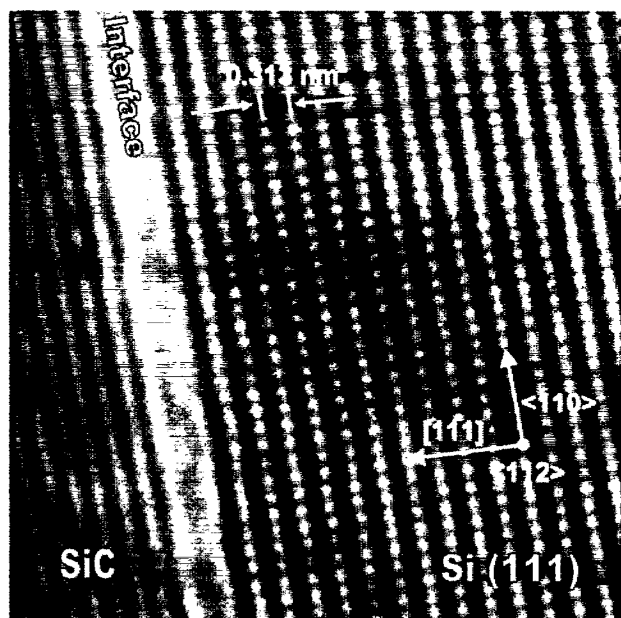

Fig. 2. High-resolution electron microscopy micrograph, viewed along the ⟨112⟩ zone axis, of the SiC/Si(111) interface region.

from areas of about 65 μm^2 of a PVTEM specimen (Fig. 3) revealed the presence of a mosaic structure. In the diffraction patterns extra spots due to double diffraction can be clearly observed around the transmitted beam and all the diffracted spots corresponding to Si[111]. The SiC spots appear at a distance that corresponds with its lattice parameter considering a cubic structure. The existence of SiC crystals with a very small angle of disorientation is shown by the curved shape elongated spots in the SAED pattern. This result can be expected because epitaxial films with a large lattice mismatch with respect to the substrate invariably form a mosaic structure of slightly misoriented subgrains [3]. The lattice mismatch between the β-SiC ($a_{SiC} = 0.4359$ nm) and Si ($a_{Si} = 0.5430$ nm) is extremely high, 19.7%, in tension. In our case, the grain diameter was estimated by SEM to be 170 nm. In PVTEM images a granular structure is observed.

Spots with the same distance from the transmitted beam but rotated $\pm 30°$ are also clearly observed in the pattern of Fig. 3. By taking dark field images with only one of these diffracted spots a number of the grains corresponding to such spots are easily observed in PVTEM images. Their average diameter was estimated to be 150 nm and with a density of 9.3 grains per μm^2 of growth plane area. Spots labeled as H_1 and H_2 can only be indexed in the hexagonal system [0001] zone axis rotated $\pm$ 30° and without rotation with respect to the main SiC cubic crystal [111] growth direction. In SiC it has been observed the existence of the two analogous HCP and FCC polymorphs [4].

In agreement with TEM observations, SEM study revealed the presence of voids and a surface roughness. In SEM micrographs as shown in Fig. 4a, equilateral triangular

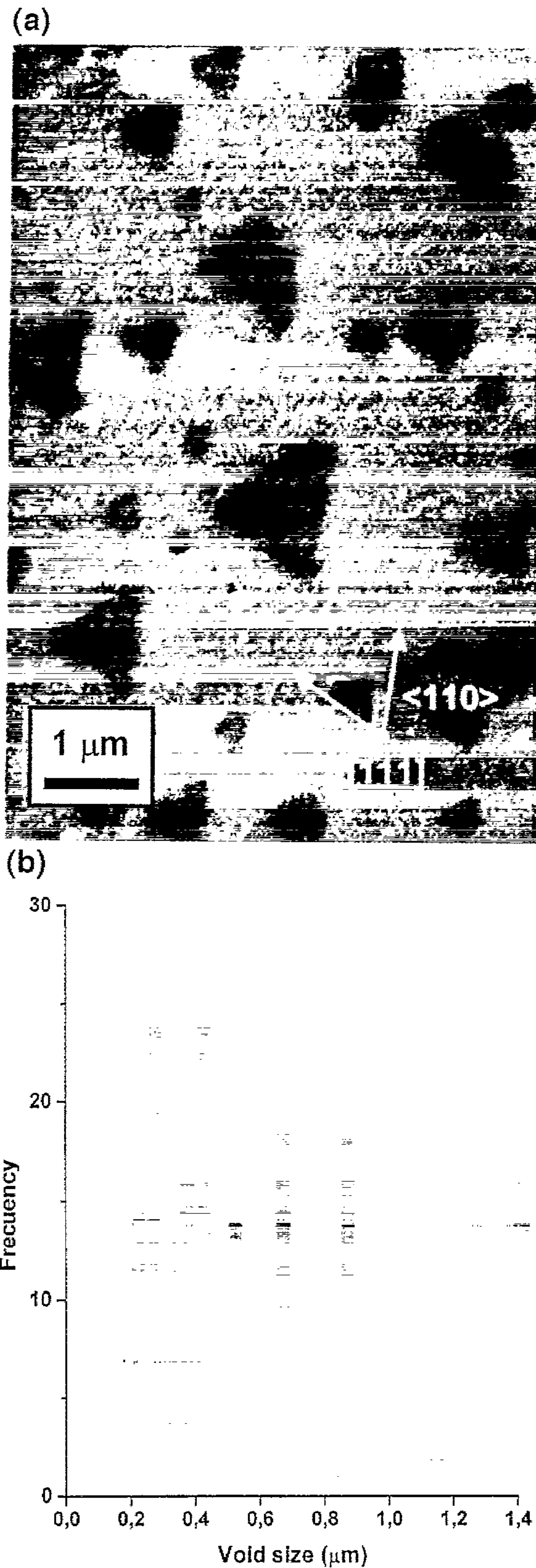

Fig. 4. (a) Scanning electron microscopy image revealing the presence of equilateral triangular and non-regular shape voids and a rough surface. (b) Void side size histogram showing the existence of at least two different distributions.

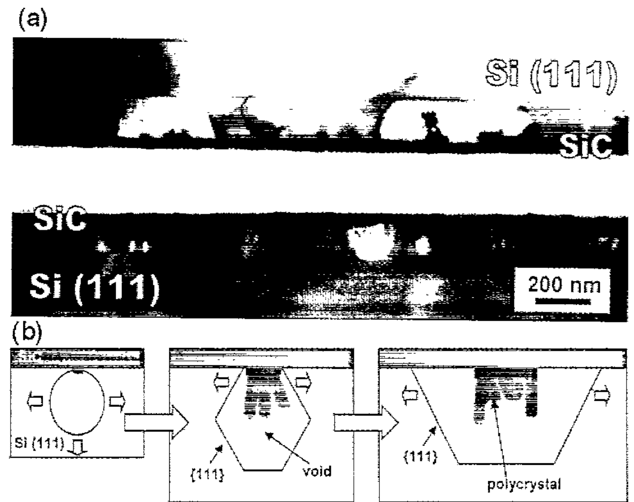

Fig. 5. (a) Low magnification bright field cross-sectional transmission electron microscopy image on a {110} projection showing a general view of the SiC layer and facetted and non-facetted voids in the (111) Si substrate. (b) Scheme of the void growth.

shape voids with their sides along {110} directions are evidenced as well as non-regular shape voids with smaller sizes. By studying SEM images of the sample surface corresponding to a total area of 280 μm^2, the void size distribution resulted to be as shown in Fig. 4.b. The average void size was 0.52 ± 0.27 nm and the area fraction in the growth plane occupied by voids resulted to be 9.6% approximately.

XTEM micrographs as in Fig. 5a show (i) voids faceted by {111} planes and (ii) quasi-spherical voids. The void depth was estimated from low magnification XTEM images of a about 15 µm of SiC/Si interface and resulted to be 0.22 ± 0.07 µm.

From TEM observations it can be deduced that the void growth front follows the scheme shown in Fig. 5b. The voids present a quasi-spherical shape at the first stages of their formation. As the carbonization progresses, they continue growing without faceting until they reach a given depth. Then, faceting in {111} planes takes place. Next, the voids only increase their lateral dimensions following ⟨110⟩ directions.

The voids are not completely empty but there are some crystals inside as can be observed in Fig. 5a. The presence of such crystals has been confirmed by TED. In SAED patterns including only a void a considerable number of extra points can be observed. This fact must be related to the Si out-diffusion mechanism during the SiC layer growth.

The SiC/Si interface is well defined and the roughness is small. In HREM images (Fig. 2) a white contrast always appears at the SiC/Si interface. This is associated with different focus conditions for Si in this line due to a different thickness of the XTEM specimen at the SiC/Si interface. This fact is a consequence of the uneven SiC and Si thinning rates during the Ar^+ ion milling [5]. The presence of a high density of misfit dislocations at the interface necessary to accommodate the high lattice mismatch between the two

materials is evidenced in ⟨110⟩ HREM images. Every four Si {110} interplanar spacings, an extra SiC plane appears as a consequence of misfit dislocations.

4. Conclusions

A thin SiC film was obtained by Si carbonization. The crystalline quality of the SiC has been investigated by TEM and SEM. The observations reveal a mosaic structure resulting from the high mismatch between Si and SiC. However, the lattice is shown to be well relaxed. This results from the high density of misfit dislocations located at the SiC/Si interface. The only threading defects observed were planar defects. The mosaic structure includes, in addition to slightly disoriented grains, a considerable density of in-plane 30° disoriented crystals. Finally, voids located in the Si substrate at the SiC/Si interface result from the C and Si interdiffusion.

Acknowledgements

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