

# SiC thin films obtained by Si carbonization

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## Abstract

This work reports the fabrication and characterization of SiC thin films obtained by carbonization of Si substrates with a recently designed and fabricated hot-wall reactor by rapid thermal chemical vapor deposition. The reactor design is less complex than other ones previously reported, but it permits to reach a similar SiC material crystalline quality. The composition and structure of the fabricated thin films have been assessed by scanning electron microscopy and transmission electron microscopy. The structural quality of the obtained films has been optimized as a function of the temperature ramp rate, flowing gases, Si surface pre-treatment and sample holder design. SiC obtained layers on Si exhibit a flat free surface, they mainly consist of highly oriented 3C-SiC and some hexagonal SiC inclusions have been detected. © 2001 Elsevier Science B.V. All rights reserved.

## 1. Introduction

Production of silicon carbide layers on silicon wafers is an attractive process because it permits to develop SiC devices on such well-known substrates [1,2]. This sort of development is valuable due to (i) the demonstrated ability of silicon carbide to function under extreme conditions (high-temperature, high-power and/or high-radiation) and (ii) progresses in silicon carbide semiconductor electronics technology [3]. In addition to this advantage, such processes open a way to the production of silicon carbide buffer layers to grow GaN and related materials on silicon [4–6].

Several growth techniques have been used to produce silicon carbide layers on silicon substrates (molecular beam epitaxy [7–9], chemical vapor deposition (CVD) [10–14], carbon implantation and annealing [15] and heat treatment of deposited amorphous films [16]). In this work, we structurally characterize and optimize SiC thin layers on Si substrates by rapid thermal CVD (RTCVD) by using a reactor less complex than those other reactors previously reported.

## 2. Experimental

The design of the carbonization system is based on the system developed by Steckl and Li [11] by simplifying or changing several parts. The main changes have been performed in the reaction chamber. The heating of the Si substrate in the main reactor chamber is carried out by an electrical oven (Carbolite, model CTF 15/75, electrical power 6 kW) controlled by a Eurotherm temperature control system and the reaction chamber is fabricated from an alloy Fe–Cr–Al Incoloy MA 956. The operation maximum temperature is 1400°C and it can be operated at low pressures. The carbonized Si substrates were (0 0 1) n-type wafers. Different temperature ramp rates, flowing gases, Si surface pre-treatments and sample holder designs have been used to improve the crystalline quality of the fabricated SiC layers. This work presents the structural characterization of the optimized obtained layers; a detailed description of the influence of operation conditions and design changes is out of the objective of this paper and it will published elsewhere.

Si substrates samples were treated before their introduction in the reaction chamber by following a mixed procedure based on several pre-treatment procedures [7–9,12,17–19]. Best results are obtained by cleaning the substrate surface with acetone and ethanol in ultrasonic bath for 5 min, and further treatment with HF(48%) for a time between 3 and 15 min. Usual

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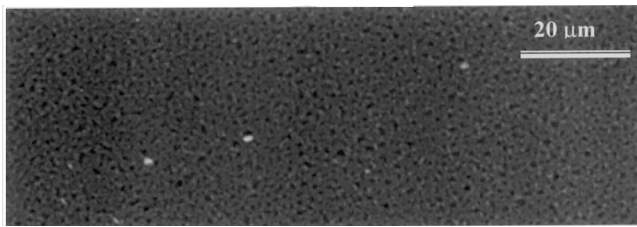


Fig. 1. SEM images of the carbonized Si wafer.

operation conditions for Si carbonization are hydrogen flow of 1 lnm, reaction gas (5% propane and 95% hydrogen) flow of 10.2 ml nm, temperature ramp of  $5\text{--}5.5^\circ\text{C s}^{-1}$ , maximum temperature of  $1250\text{--}1300^\circ\text{C}$  and carbonization time of 90 s.

The structural quality of the fabricated SiC layers on Si was studied by scanning electron microscopy (SEM), conventional transmission electron microscopy (TEM) and selected area electron diffraction (SAED). Preparation of TEM specimens was performed by mechanical thinning and ion milling. These studies were carried out by using cross-sectional (X) and plan view (PV) orientations with the SEM Jeol 820 EM and the TEMs Jeol 1200 EX and 2000 EX.

### 3. Results and discussion

Thin silicon carbide layers exhibiting a flat surface are produced by using, on (0 0 1) Si substrates, carbonization conditions described in Section 2 as usual operation conditions. SEM image of Fig. 1 shows the surface of the carbonized silicon substrate after the surface was chemically pretreated. XTEM image of Fig. 2 is a low magnification image from carbonized Si layer showing the flatness of the produced carbonized Si layer. Voids are also present in this image below the interface between the Si carbonized layer and Si substrate. These voids are typically present on carbonized Si substrates. A detailed analysis of their distribution has previously been published [20]. For optimized operation conditions, thickness of the Si carbonized layer measured by XTEM is about 300 nm. However, by changing such conditions, thickness can be highly increased up to several microns, though the structural quality (SiC polytypes, grain size and crystalline orien-

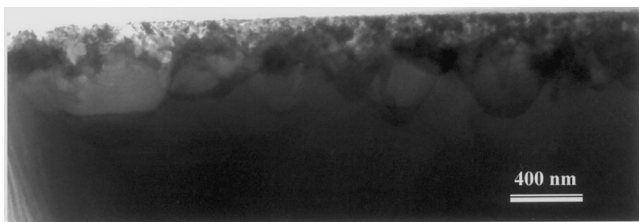


Fig. 2. Low magnification XTEM image of carbonized Si layer.

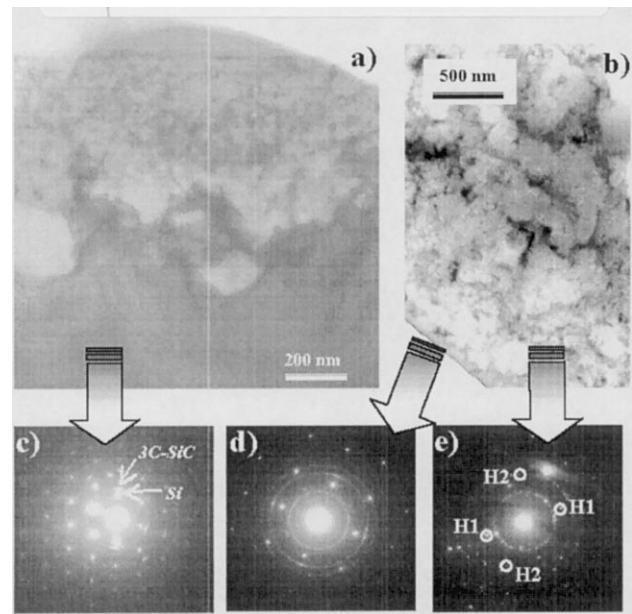


Fig. 3. (a) XTEM and (b) PVTEM images, (c) cross-section and (d) plan view SAED patterns from carbonized Si layer. A very local plan view SAED pattern is showed in (e) where hexagonal spots ( $H_1$  and  $H_2$ ) are labeled.

tation) of the obtained carbonized layers is getting quite poor for such cases.

Fig. 3 shows (a) XTEM and (b) PVTEM images registered from a carbonized Si layer obtained using optimized conditions. Top contrast of the XTEM image is due to glue used for TEM sample preparation. SAED pattern from a selected area of carbonized Si like that showed in Fig. 3(a) is presented in Fig. 3(c). Brighter spots correspond to Si [1 1 0] zone axis and outer spots correspond to cubic 3C-SiC [1 1 0] zone axis. Such patterns can be also explained having into account, high index zone axis of hexagonal structures, but cubic structure is considered as valid because structurally it is the natural option. Cubic structure 3C-SiC also explains the SAED pattern registered from a plan view sample. Fig. 3(d) shows one of these typical SAED plan view patterns. Spots indexed as 3C-SiC do not have perfect circle shape, but they consist of slightly elongated shape along an arc of few degrees. This happens because the 3C-SiC forms a highly oriented mosaic structure instead of a perfect single monocrystal.

However, very local SAED patterns, as that showed in Fig. 3(e), correspond to hexagonal SiC polytypes. Spots labeled as  $H_1$  and  $H_2$  in this figure do not correspond to 3C-SiC. Spot  $H_1$  corresponds to a lattice spacing of 0.143 nm while  $H_2$  corresponds to 0.114 nm. Both spots are explained by assuming hexagonal polytypes (2H, 6H and/or 8H). It is not possible to distinguish among them because their lattice spacings are very similar. Some reflections associated to  $H_1$  are

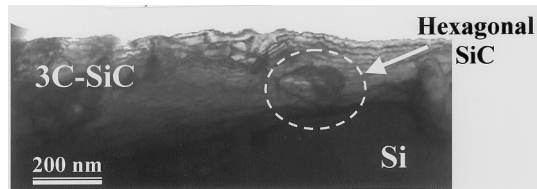


Fig. 4. XTEM image showing a hexagonal crystalline inclusion in the 3C-SiC layer.

(1 0 6) 2H and (0 1 9) 6H, while (1 0 8) 2H, (0 2 7) 6H and (2 0 9) 8H are associated to spot  $H_2$ .

XTEM image of Fig. 4 is an example of a crystalline inclusion in the 3C-SiC layer. The SAED analysis of this particular included crystal concludes that its crystalline structure is a hexagonal (2H and/or 6H) polytype. These hexagonal polytypes have also been detected in and near voids present just below the interface between the Si carbonized layer and the Si substrate.

The Si carbonized layers showing flat surface and highly oriented mosaic structure of the 3C-SiC polytype as the most abundant one have been obtained for optimized carbonization conditions reported in Section 2. However, grain size, crystalline orientation distribution and the relative abundance of polytypes can be controlled by changing the operation conditions. In fact, polytypes 3C, 2H, 4H, 6H and 8H have been detected in carbonized Si samples produced during the optimization of the carbonization process.

#### 4. Conclusions

A system to carbonize (0 0 1) Si wafers designed by following a less complex and cheaper configuration than those ones of previously published systems is presented. Carbonized Si layers obtained in this system show a flat surface and consist of a highly oriented mosaic structure where 3C-SiC is the dominant phase.

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