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The effect of the amount of activated carbon powder on the synthesis of SiC/SiO₂ nanostructures via a chemical vapor growth method

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In this study, the effects of different amounts of activated carbon powder on the growth of SiC/SiO_2 nanostructures were examined. The nanostructures were formed using a simple chemical vapor growth setup using only oxidized silicon (Si) wafers and activated carbon powder. The addition of carbon powder into the system led to the formation of four main SiC/SiO_2 products namely nanograins, nanowires, nanowebs and nanocables along with other side products of SiO_2 nanowires and nanoparticles, depending on their growth location on the oxidized Si substrates. The possible growth mechanisms for these nanostructures are discussed.

Key words: SiC/SiO₂, Nanowires, Nanocables, Nanowebs, Growth mechanism.

Introduction

SiC is a third generation wide-bandgap semiconductor material [1] that exhibit excellent thermal, mechanical, chemical and electronic properties and thus has been widely applied in various fields especially for applications in harsh environment. Combining the advantage of SiC and the nanosize-effect, SiC nanostructures have become potential candidates in many areas of nanoelectronics, nanooptics and nanosensors [2-4].

In this study, SiC/SiO_2 core-shell nanostructures were synthesized. A SiC/SiO_2 core-shell structure is a structure consisting of a crystalline SiC core, encapsulated by an amorphous SiO_2 shell. The presence of an oxide layer on the SiC nanowires was found to enhance the photoluminescence properties of SiC [5-8] as well as the mechanical strength of a composite by improving the interfacial bonding intensity of SiC in rubber [9], while being able to maintain the intrinsic properties of SiC.

SiC/SiO₂ core-shell nanostructures had been commonly reported to be synthesized using a C-Si-SiO₂ mixture, with [7-14] or without metallic catalysts [5, 15-18]. In the present investigation, a similar route had been used to produce SiC/SiO₂ core-shell nanostructures. A large amount of SiC/SiO₂ core-shell clusters, nanowires, nanowebs and nanocables were synthesized in an Ar atmosphere at high temperature using only activated carbon powder and oxidized silicon (Si) wafers. The route here had the advantage of the basis of direct growth on Si wafers, the lack of metallic catalyst and a simple experimental setup. Direct growth of nanostructures onto the wafers made product collection an easy process as opposed to the growth of nanostructures in the powder mixture that made product separation difficult. It had also been reported that a faster nanostructure growth was possible by separating the Si and C sources, as in this work, because of the controlled supersaturation condition [19].

The same experimental setup had been previously reported in producing SiO_x and Si/SiO_x core-shell nanowires [20] but the oxide layer on Si wafers in the previous work was much thicker due to a longer oxidation time of 12 minutes as compared to 2 minutes in the present work. An increase in the sintering temperature from 1200 to 1300 °C resulted in a morphological change from SiO_x to Si/SiO_x core-shell nanowires and an increment in the holding time from 1 h to 4 h increased the length of the nanowires. A similar experimental setup was also used to produce SiC nanowires by using only activated carbon and pure Si wafers without the oxidation process [21]. Although this setup was able to produce SiC nanowires, it took a longer holding time of 4 h as compared to 2 h in this study. The shorter holding time might be attributed to the addition of SiO₂ that was reported to be a significant help in improving the growth rate of SiC and producing straight nanowires [22, 23]. Here, the effects of the amount of carbon on the synthesis of SiC/ SiO₂ nanostructures by heating 2-minutes-oxidized Si wafers are reported.

Materials and Method

The same experimental setup was reported in a previous study with the only difference that the

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oxidation time of the Si wafers was reduced to 2 minutes as compared to 12 minutes in the previous study [20]. The pre-oxidized n-type Si <100> wafers were placed on top of a graphite crucible filled with different amounts of activated carbon powder (ACP) (0 g, 0.5 g, 1.0 g and 1.5 g) with the ACP having a mean particle size of 149 µm. The Si wafers were held in place by another graphite block on top of the Si wafers. The samples were designated as 0ACP, 0.5ACP, 1.0ACP and 1.5ACP according to the amount of ACP being used. The crucible, with a height of 4.5 cm, was then placed at the center of a vacuum sintering furnace. The furnace was then pre-evacuated to 10⁻³ mTorr (133.322 µPa) using a mechanical pump and later the vacuum was released while Ar gas was introduced at a rate of 15 sccm. Next, the furnace was heated up to 1300 °C and maintained for 2 h.

Finally, the products were collected and characterized by a field-emission scanning electron microscope (FESEM, LEO GEMINI) equipped with an energy dispersive X-ray spectrometer (EDS, ZEISS SUPRATM 35VP), an energy-filtered transmission electron microscope (EFTEM, ZEISS LIBRA 120) equipped with electron spectroscopic imaging (ESI) and X-ray diffraction (XRD, Bruker), which scanned the entire Si surface for each sample. The samples for EFTEM analysis were scrapped from the surface of the Si wafers and dispersed in absolute ethanol, followed by dripping of the suspension onto copper grids covered with holey carbon films.

Results and Discussion

Results and discussion

Different nanostructures were found on the surface of Si wafers, depending on their location, either at the center, boundary or the side of the substrate (Fig. 1). The center is the region where the substrate is exposed



Fig. 1. Location of nanostructures formed on the SiO₂/Si wafers.

directly to activated carbon powder; the boundary is the region in contact with the graphite crucible; and the side is the region outside of the crucible. A summary of the different nanostructures formed depending on the location and amount of ACP is shown in Table 1.

At the center, the surface appeared to be etched, forming voids of various shapes with particles growing in the voids when ACP was added (Fig. 2). EDS showed that these voids contained Si, C and O, with higher amounts of Si and C as compared to the surrounding surface which consisted of only Si and O. This indicated that the voids might be grown with SiC nanograins. When no ACP was added, short nanowires consisting only of Si based on the EDS results were detected. This suggested that the nanowires might be Si or Si/SiO_x nanowires, which are commonly grown by heating a mixture of Si and SiO₂ at temperatures above 1100 °C through an oxide-assisted growth mechanism [24-27].

At the border, the nanostructures changed from randomly oriented nanowires to nanowebs consisting of intersecting nanowires with the presence of a small amount of nanoparticles on the surface of a Si wafer when ACP was added (Fig. 3). The randomly oriented nanowires in 0 ACP showed significantly smaller diameters as compared to the nanowebs. It could also be noticed that the diameters of these nanowebs increased when the amount of ACP was increased from 0.5 g to 1.0 g, with signs of surface roughening of the nanowires, but then the diameter was found to reduce when the



Fig. 2. FESEM images of nanostructures formed at the center under different amounts of ACP for samples (a) 0ACP, (b) 0.5ACP, (c) 1.0ACP and (d) 1.5ACP.

Table. 1. Summary of nanostructures	formed on Si wafe	s depending on the amour	nt of ACP and the location on S	i wafers.
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Sample —	Location on Si Wafer			
	Center	Border	Side	
0ACP	Si or Si/SiO _x nanowires	SiC/SiO ₂ nanowires	Randomly oriented SiC/SiO ₂ nanowires	
0.5ACP	SiC/SiO ₂ nanograins	SiC/SiO ₂ nanowebs	Randomly oriented SiC/SiO2 nanowires	
1.0ACP	SiC/SiO ₂ nanograins	SiC/SiO ₂ nanowebs	Randomly oriented SiC/SiO2 nanowires	
1.5ACP	SiC/SiO ₂ nanograins	SiC/SiO ₂ nanowebs	SiC/SiO ₂ nanowire clusters	



Fig. 3. FESEM images of nanostructures formed at the border under different amounts of ACP for samples (a) 0ACP, (b) 0.5ACP, (c) 1.0ACP and (d) 1.5ACP.



Fig. 4. EFTEM images of nanostructures formed at the border under different amounts of ACP for samples (a) 0ACP, (b) 0.5ACP, (c) 1.0ACP and (d) 1.5ACP.

amount of ACP was further increased to 1.5 g. EDS of these nanowires showed the presence of Si, C and O with higher content of Si and O. In addition, thin nanowires were observed growing from the nanoparticles, suggesting that these nanoparticles might be acting as nuclei for the growth of the nanowires. The EFTEM images of the nanowires formed at the border under different amount of ACP are shown in Fig. 4. From the EFTEM images, the nanowebs consisted of core-shell structures, most with a single core and some could be observed with several thin cores located inside the nanowires. The nanowires had diameters ranging from 15 to 70 nm with the cores having diameters of 4 to 15 nm. Similar nanostructures had been reported by Shim and Huang [17] and were named nanocables due to their resemblance to power transmission cables. The core-shell nanowires were observed with a decreasing diameter of the core when the amount of ACP was increased. Amorphous nanowires were also found growing as branches from the core-shell nanowires [Fig. 4(d)].



Fig. 5. FESEM images of nanostructures formed at the side under different amounts of ACP for samples (a) 0ACP, (b) 0.5ACP, (c) 1.0ACP and (d) 1.5ACP (Inset showing a higher magnification of the nanowire cluster formed in 1.5ACP).



Fig. 6. EFTEM images of nanostructures formed at the side under different amounts of ACP for samples (a) 0ACP, (b) 0.5ACP, (c) 1.0ACP and (d) 1.5ACP (The circle shows the presence of stacking faults).

At the side, randomly oriented nanowires were observed for samples 0 ACP, 0.5 ACP and 1.0 ACP while thick nanowire clusters were observed in 1.5 ACP, as shown in Fig. 5. The randomly oriented nanowires consisted of Si, C and O while the thick nanowires in the sample 1.5 ACP were found growing from the same origin [inset of Fig. 5(d)]. EFTEM images of the samples are shown in Fig. 6. From the EFTEM images, amorphous nanowires and core-shell nanowires were observed as well. However, the diameters of the nanowires were smaller (20 - 50 nm) and with thicker cores (7 - 20 nm). Branching of the thick crystalline core was also observed [Fig. 6(c)]. In addition, some of the crystalline cores could be observed with planar defects of stacking faults, as circled in Fig. 6(d). Stacking faults are common in SiC nanowires due to a lower energy contribution during the growth of nanowires[17, 19, 28].

To determine the composition of the core-shell nanowires, ESI and XRD were performed. XRD was performed on the whole Si wafer surface covering the



Fig. 7. (a) ESI results of the core-shell nanowires at the side of a wafer, (b) ESI results of the nanowebs at the border and (c) XRD spectra of the samples.

three regions. ESI results showed a higher concentration of Si and C at the core and a higher concentration of O at the shell for both nanostructures at the border and at the side [Fig. 7(a) & (b)]. XRD spectra of the entire Si wafer surface for samples 0 ACP, 0.5 ACP, 1.0 ACP and 1.5 ACP are shown in Fig. 7(c). The XRD patterns showed only diffraction peaks of Moissanite 3H-SiC (ICDD 01-073-1708) as well as SiO₂ in the form of quartz (ICDD 01-086-1562) and cristobalite (ICDD 00-039-1425). The detection of SiC peaks even in 0 ACP indicated that the graphite crucible might have acted as a secondary carbon source for SiC formation aside from the ACP. The high SiO₂ peaks might originate from the SiO2 thin film on the pre-oxidized Si wafer. Although thermally-grown SiO₂ is amorphous in nature, devitrification of SiO₂ into a mixture of cristobalite and quartz had been reported to be possible when heated at temperatures above 1200 °C, especially in the presence of hydroxyl (OH) radicals that were bound to the surface of SiO_2 [29]. From the results above, it could be concluded that the cores were SiC and the amorphous shells were SiO₂.

Growth mechanism

The growth of SiC/SiO₂ nanostructures here is quite similar to the Si-C-O system commonly reported and two reactions [reactions (1) and (2) as shown below] have been identified as the most likely reactions that occurred in several intermediate processes that included active SiO and CO species [17]:

$$SiO_2(s) + 2C(s) \rightarrow SiC(s) + CO_2(g)$$
 (1)

$$SiO_2(s) + 3C(s) \rightarrow SiC(s) + 2CO(g)$$
 (2)

Considering the results, the reactions above and their thermodynamics in terms of free energy, the following mechanism for the growth of SiC/SiO₂ nanostructures based on a vapor-solid growth mechanism is proposed. The free energies (G) were calculated at atmospheric pressure and at a temperature (T) of 1300 °C using the equation $\Delta G_T = \Delta H_T - T\Delta S_T$, where H and S represent enthalpy and entropy, respectively. The properties were obtained from NIST-JANAF thermochemical tables [30].

First, SiO and CO need to be generated in order for SiC formation to proceed. One of the possibilities of the formation of SiO is according to reaction (3). At 1300 °C, thermal decomposition of SiO₂/Si at the interface could proceed via reaction (1). Although reaction (1) was not favorable in terms of energy calculations, reports had confirmed that this reaction was still possible kinetically at high temperatures (> 700 °C) when the O₂ partial pressure was low and oxide layer was thin (≤ 70 nm) [31], as proven in the FESEM images showing etched Si surfaces. The etched surface of the wafer indicated the loss of Si species from the wafer. Another possible source of SiO species was the active oxidation of the Si wafer, as shown in reaction (4), which had been reported to occur at a high substrate temperature (> 700 °C) and low O_2 pressure [31]. O_2 could diffuse through the thin oxide layer and react with the Si beneath. The CO species was provided by the reaction between ACP and residual O_2 in the powder according to reaction (5):

 $Si(s) + SiO_2(s) \rightarrow 2SiO(g), \Delta G = 164.53 \text{ kJ/mol}$ (3)

 $2\text{Si}(s) + O_2(g) \rightarrow 2\text{SiO}(g), \Delta G = -466.633 \text{ kJ/mol}$ (4)

$$2C(s) + O_2(g) \rightarrow 2CO(g), \Delta G = -499.97 \text{ kJ/mol}$$
 (5)

A higher partial pressure of CO gas at the bottom resulted in diffusion of CO gas upwards. As the furnace was heated up to 1300 °C, reaction between gaseous SiO and CO was possible for the formation of SiC nuclei through reaction (6). Although the thermo-dynamic calculations that showed the reaction was energetically favorable for temperatures below 927 °C [32], there are few reports that found that this reaction was still possible at higher temperatures if it was under a supersaturated condition of CO vapor in the reaction chamber [33, 34]:

$$SiO(g) + 3CO(g) \rightarrow SiC(s) + 2CO_2(g)$$
(6)

Furthermore, the reaction between the active species of SiO and CO could also proceed near the Si wafer via reaction (7), which had been reported to occur under a supersaturation condition of SiO [34]. From the reaction, SiC and SiO₂ were formed simultaneously.

However, since SiC had a higher melting point than SiO_2 , SiC would solidify first and the amorphous-viscous SiO_2 would encapsulate the crystalline SiC, forming the core-shell structures [10, 15, 19].

$$3SiO(g) + CO(g) \rightarrow SiC(s) + 2SiO_2(s),$$

$$\Delta G = -1273.44 \text{ kJ/mol}$$
(7)

According to the results, SiC nanowires were also found when no ACP was added at the border in 0ACP. This suggested that SiO could also react with graphite directly through reactions (8) and (9) to form SiC:

$$2SiO(g) + 3C(s) \rightarrow 2SiC(s) + CO_2(g),$$

$$\Delta G = -51.7714 \text{ kJ/mol}$$
(8)

 $SiO(g) + 2C(s) \rightarrow SiC(s) + CO(g),$ $\Delta G = -77.7097 \text{ kJ/mol}$ (9)

After the nucleation process, SiC species formed were preferentially absorbed onto the nuclei and further reactions led to the growth of different shapes and sizes of nanoparticles and nanowires depending on the their locations. The nanoparticles formed could also act as nucleation sites for further growth of SiC nanowires. The formation of different nanostructures will be discussed in the following section.

To maintain SiC formation, there must be a sufficient source of CO, which could be replenished through reaction (10):

$$CO_2(g) + C(s) \rightarrow 2CO(g), \Delta G = -103.648 \text{ kJ/mol} (10)$$

When the furnace was cooled down, SiC formation ceased, which in turn resulted in excess SiO species. In the presence of excess SiO species, the reverse reactions took place for reaction (3), thereby producing the Si nanowires, as proposed in the oxide-assisted growth of Si nanowires [24-27]. Furthermore, in the presence of gaseous O_2 , reoxidation occurs through reaction (11), creating the amorphous SiO₂ that would be adsorbed onto the nanowire surfaces due to the large surface energy. Gradual absorption of SiO₂ increased the thickness of the oxide shell. Some SiO droplets on the nanowires would also induce the growth of amorphous SiO₂ branches from the core-shell nanowires since they were energetically favored sites for absorption of SiO vapor[35].

$$SiO(g) + O_2(g) \rightarrow SiO_x, 1 < x < 2$$
(11)

Formation of SiC/SiO₂ core-shell clusters, nanowires, nanowebs and nanocables

The different nanostructures formed at different locations on the Si wafers could be attributed to aerodynamic effects. A small gap existed between the Si wafer and crucible, thereby creating a narrow path



Fig. 8. Growth mechanisms of clusters at the center of silicon wafer.



Fig. 9. Growth mechanisms of nanowires, nanowebs and nanocables at the border of a silicon wafer.

for gas flow towards the outside environment. The existence of this narrow gap resulted in a change of the diffusion path of gases, with more reactants attracted to the side, trying to diffuse through the gap towards the outside environment which had a lower pressure. As a result of this pressure difference, it led to a lower concentration of reactants at the center of substrate. The lower concentration of reactants limited the reactions and thus only a small amount of SiC could be produced. The SiC species were first deposited in the form of nanorods, but as the soaking time was increased to 2 hours, the nanorods agglomerate due to their close proximity and strong Van der Waals, into the final form of clusters. With an increasing amount of ACP, more CO gas was produced. Since CO was the reaction-limiting species in reactions (6) and (7) because SiO gas had a never-ending supply from the Si wafer, this meant that an increase in the amount of ACP might increase the chances of CO gas reaching to the center of the Si wafer and enhanced the reaction rate, which ultimately led to more SiC clusters being formed. The growth mechanism of the clusters at the center of a silicon wafer is shown in Fig. 8.

At the border where the Si wafer was in contact with the crucible, the formation of nanowires and nanowebs could be explained based on Fig. 9(a). From the figure, the nanowires were observed growing from the tip of nanocolumns, as shown in Fig. 9(b), thus suggesting that the nanocolumns induced the growth of nanowires. The growth mechanism of the SiC nanocolumns was similar to that reported by Cheong and co-workers [36-

SiO (CO) A_2, v_2, P_2 $A_2 < A_1$ $v_2 > v_1$ $P_2 < P_1$

Fig. 10. Influence of a Venturi effect on the flow of reactant species as they passed through the gap at the border.

38]. CO gas had to diffuse deeper through the SiC layer into the SiO₂ layer or the Si wafer below and reacted with them. CO gas then reacted with SiO₂, forming more SiO species for a further reaction with CO to form SiC (reaction 6). The difference in molecular size of SiC and atomic size of Si or SiO₂ caused a redistribution of the atoms and molecules in the lattice laterally and/or vertically, so that the induced strain could be relaxed. The spherical and faceted SiC layer structures were then transformed into pyramid-like structures with stacked SiC layers, forming polygonal SiC nanocolumns. Further formation of SiC species would be preferentially adsorbed to the tips of nanocolumns and thus induced the growth of nanowires.

Furthermore, the small gap between the silicon wafer and crucible posed as a constriction to the flow of reactant gas and created a Venturi effect that is based on Bernoulli's principle [39]. When gases tried to pass through the gap, the gases had to flow from a high volume region inside the crucible to a low volume region in the gap. The sudden change in volume caused an increase in the speed of the gas flow and a corresponding reduction in the pressure, as shown in Fig. 10.

The increase in the gas flow rate reduced the reaction rate as a consequence of the high removal rate of reactant species along the gap during the 2-hour holding period. Therefore, the SiC cores had smaller diameters as compared to the nanowires formed at the side of the substrate. Although the SiC cores had smaller diameters, they were observed with thick oxide shells. This could be due to the graphite crucible, which had a higher heat capacity, dissipated heat slower as compared to the surrounding environment and thus a longer cooling period allowed the SiO adsorption to continue to take place and resulted in a thicker oxide shell.

Due to the close proximity of nanowires and the strong van der Waals forces between the nanowires, the nanowires were attracted to each other and formed an aligned bundle or intersection of junctions. Further oxide growth and self-organization through surface



Fig. 11. Formation of SiC/SiO₂nanocable, nanoweb and amorphous SiO₂ branch through self-organization and oxide growth.

diffusion might cause the oxide shells to merge with one another in order to reduce the surface energy, creating the nanocables and nanojunctions in the nanoweb [17, 35]. Amorphous SiO₂ nanowires found as branches might have grown from smaller droplets of SiO adsorbed onto the shell [35]. These amorphous nanowires then grew in length and bridged the separated nanowires and nanocables. The joining of nanowires through "welding" could be proven by the lack of lattice matching of the crystalline cores of the junctions in the EFTEM images and the two nanowires appeared to be overlapped through welding by SiO₂. A schematic diagram showing the growth of the nanocables, nanowebs and amorphous SiO₂ branches from individual SiC/SiO₂ core-shell nanowires is given in Fig. 11.

The different diameters of the samples at the border could be explained by the increase in local pressure in the gap. Since gas is compressible, an increased upstream pressure increases the density of gas through the gap while the gas flow rate remains constant throughout the gap. This meant that an increase in the amount of ACP would increase the vapor pressure of CO in the gap. A slight increase in the pressure had been reported to increase the growth rate and the diameter of nanowires but a large increase would significantly reduce the yield [40-42]. For 0 ACP, the lack of CO species and reduced pressure from the higher gas flow rate promoted the growth of a large amount of randomly oriented nanowires with smaller diameters. When the amount of ACP was increased from 0.5 g to 1.0 g, the increased vapor pressure in that region increased the growth rate due to a higher reactant concentration and thus, larger diameters of nanowires could be observed. However, for 1.5ACP, the high vapor pressure of CO in that region had increased the local pressure and suppressed the evaporation of SiO species. The limited SiO species controlled the growth of the nanowebs and thus only thin nanowebs could be observed.

At the side, the growth of SiC/SiO_2 randomly oriented nanowires could be explained as follows. When reactant species exited the gap, the gas flow rate decreased due to an increase in volume based on a



Fig. 12. (a) Influence of the Venturi effect on the flow of reactant species as then exited the gap at the side of the wafer. (b) Growth mechanisms of nanowires at the side of silicon wafer.

Venturi effect, as shown in Fig. 12(a). The sudden speed reduction caused the reactant species to be accumulated near the crucible and increased the chances for SiC formation to take place at that particular reaction site, as shown in Fig. 12(b). Thus, thicker cores were observed as compared to the border. The flow of Ar gas limited the diameter of the oxide shell because of the constant gas flow that removed excess SiO species during the cooling process. As for the nanowire clusters formed in the sample 1.5 ACP, the nanowires were found originating from a certain spot, which is quite similar to the nanostructures formed according to a vapor-liquid-solid (VLS) mechanism where a single metallic catalyst droplet could induce the growth of several nanowires [43]. Here, no metallic elements were detected by EDS and thus suggesting that the nanowires were self-catalytic, as reported by Zhang et al. [23] but it differed from the normal VLS mechanism since no Si-rich droplets were found at the tips of nanowires.

Conclusions

In summary, SiC/SiO₂ core-shell nanostructures in the form of clusters, nanowires, nanowebs and nanocables were formed along with side products of Si/SiOx or Si nanowires and SiC nanoparticles by chemical vapor growth via a vapor-solid mechanism. The difference in nanostructures could be correlated to the aerodynamic effects created by the pressure difference between the inner crucible chamber and the outside environment. An increase in ACP increased the CO content in the crucible chamber. A slight increment in CO content increased the vapor pressure just enough to promote the growth of nanowires and therefore, thicker nanowires could be observed for 1.0 ACP but when the amount of ACP was increased to 1.5 g, the large amount of CO caused an increase in the total pressure that suppressed the evaporation of SiO for further reaction of SiC and thus only thin network-like nanowires were formed. The addition of ACP also resulted in the formation of nanowebs and nanocables.

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