

Green route synthesis of silicon/silicon oxide from bamboo

Vinay Kumar¹, Pranjala Tiwari¹, Lucky Krishnia¹, Reetu Kumari¹, Anshika Singh¹, Arnab Ghosh², Pawan K. Tyagi^{1*}

¹Department of Applied Physics, Delhi Technological University, Delhi 110042, India

²Institute of Physics, Sachivalaya Marg, Bhubaneswar 751005, India

*Corresponding author. Tel: (+91) 11-27852212; Fax: (+91) 11- 27871023; E-mail: pawan.phy@dce.edu

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ABSTRACT

Silica, generally known as silicon dioxide is insulating in nature whereas silicon is semiconducting. These two materials are widely used in sensing and electronic devices. In order to full-fill demand of silicon, sources other than sand, have to explore for silicon extraction. In this report, bamboo culms have been subjected to thermo chemical decomposition at high temperature (1250 °C) in an inert atmosphere. After pyrolysis, contents in residual of bamboo culms have been characterized by using scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray diffraction (XRD). Nanosized, pure crystalline Si has been formed. Si as well as charcoal is found to be crystalline in nature. No silicon carbide formation observed. Copyright © 2016 VBRI Press.

Keywords: Nano-sized Si; bamboo charcoal; pyrolysis process; EDX mapping.

Introduction

Silicon is an extremely versatile material, being semiconducting in nature and has myriad applications ranging from the electronic devices to glass fabrication processes [1]. With continued rapid development in the area of photovoltaics (PV), the overall demand for silicon in both monocrystalline and polycrystalline form has been increased [2]. Availability and well established scalability of silicon makes it one of the most widely used material in microelectronics and photovoltaics. In the recent years, applications of silicon in Li-ion batteries and photonics [3, 4, 5, 6] has emerged as a promising field. During the past decades, in Li-ion batteries silicon has been found to possess high theoretical capacity of 4200 mAh g⁻¹ when the Li₂₂Si₅ phase is formed [3] and emerged as an attractive anode material [3,4,5]. However, crack formation and failure of electrical contact those occurred during the cycling has limited its applications. Although use of silicon nanoparticles seemed to the performance of battery improves significantly [7]. Hence, much research interest is now being shifted in the area of Si nanoparticles. This happened because many applications of Si nanoparticles in various fields such as chemical and biomedical sensors [8, 9], solar cells [10, 11] and secondary batteries [12, 13] have been demonstrated. An extended range of applications of Si in the form of nanowire realized a multi-wavelength light source that can operate both in IR and visible ranges [14]. Furthermore, silica nanoparticles are also being used in photovoltaic systems to reduce the reflectance of protection glass by their coating [15]. Further, SiO₂ was used in photodetector as an interfacial layer. This happened

due to its passivation effect thereby suppressing surface leakage current and reduction in device capacitance resulting in quick photo-response of photo-detector [16]. SiO₂ composites either with Ag or other materials were reported to exhibit unique magnetic, catalytic, thermal, optical and electrical properties and many applications in the field of biomedical, electronic devices, adsorption and separation [17, 18] have been demonstrated. Despite from various applications silica is used in industrial applications for treatment of water and wastewater in form of nanocomposite containing amorphous silica nanoparticles and organic polymer due to low toxicity of silica nanoparticles [19, 20]. Since 2006, demand of mono or poly crystalline silicon as well as nanoparticle exceeded in other areas than that in microelectronics [21]. With aim to produce the cost effective photovoltaics or silicon-based devices, the vital issue is to produce the low-cost Solar Grade Silicon (SGS). Till date mostly, chemical route [21], so-called Siemens process [2, 22] and another route being metallurgical route [22] have been used to produce the SGS. Among all the published reports so far [22, 23, 24], extraction of pure polycrystalline or monocrystalline from bamboo have not been much explored [25], and the research is still in preliminary phase. However, extraction from the rice husks has been reported in recently published reports [26, 27]. Further, importance of bamboo can be understood as it is the fastest growing woody plant in the world, growing with the speed (up to 1 meter/per day) i.e. three times faster than the other rapidly fastest growing trees. This fast growth pattern makes the bamboo extract to be the richest known source of natural silica, containing over 70 % in the total inorganic constituents present in the

extract. In contrast to rice husks, once bamboo pyrolyzed or carbonized at high temperature ($> 600\text{ }^{\circ}\text{C}$) silica profusely formed in crystalline form. Furthermore, bamboo charcoal (BC) has attracted huge attention due to their myriad applications [28]. This BC has shown extraordinary properties such as large surface area, large pore volume, and high hydrophobicity, which was lacking in the activated carbon [29-32]. This endows BC with great potential applications [29]. In addition to the benefits of BC, extracted Si nanoparticle has profuse porosity that has high importance in the sensing applications [32]. Thus, Si nanostructures accompanied with porosity have demonstrated many exciting properties, and can serve as promising material for the development of new era of technology. Generally, porous silicon is made by the electrophoresis process and it has been reported that nanosized porous Si could be directly obtained by the carbonization or pyrolysis [33]. Furthermore, there are several techniques through which Si nanoparticles can be synthesized e.g. vapor-liquid-solid (VLS) mechanism [34, 35, 36], plasma-enhanced chemical vapor deposition (CVD) [37, 38], electrochemical methods using H_2O_2 plus HF solutions [39, 40], laser ablation [41, 42] and hot wire CVD [43]. But these processes are very lengthy, expensive and commercially unenviable. Hence all hurdles discussed above motivate us to develop a method that should be cost effective as well as should produce nanostructures in a controlled manner. The present study was performed with two objectives: first, to synthesize pure crystalline Si nanostructures, and second is to make the crystalline BC of having high porosity.

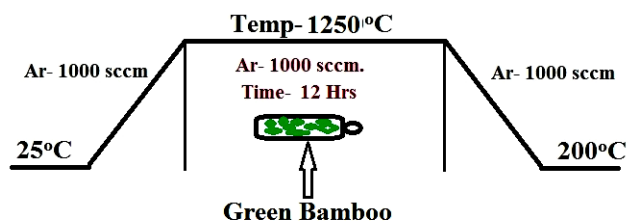


Fig. 1. Schematic diagram of the pyrolysis process for bamboo.

Experimental

Fresh bamboo was collected from a tree grown in Delhi region. The culms of bamboo have been cut in the pieces of 5 mm wide by using a knife. Outer coverings of culms were removed, and only inner part of the bamboo stem was used. These bamboo slices were subjected to pyrolysis process (Fig. 1). In the pyrolysis process, self-thermochemical decomposition of bamboo at high temperature was carried out in the presence of argon gas. In this process, simultaneous irreversible changes in both chemical composition, as well as physical phase, occurred. The pyrolysis was carried out at $1250\text{ }^{\circ}\text{C}$ for 12 hrs in an argon atmosphere to remove water and certain organic groups from fresh bamboo slices as a result of which charcoal bio templates were produced. A heating rate of $5\text{ }^{\circ}\text{C}/\text{min}$ was used to minimize the thermal stress that may occur during the pyrolysis. The produced charcoal biotemplates were

allowed to cool down to $200\text{ }^{\circ}\text{C}$ naturally and then removed after shutting off the argon atmosphere. The produced charcoal was characterized by using SEM (Hitachi S-3700) HRTEM (JEOLTEM 2010) and X-ray Diffractometer (Bruker D8 Advance). Elemental analysis was performed to estimate the fractional quantity of Si/SiO₂ in the produced charcoal.

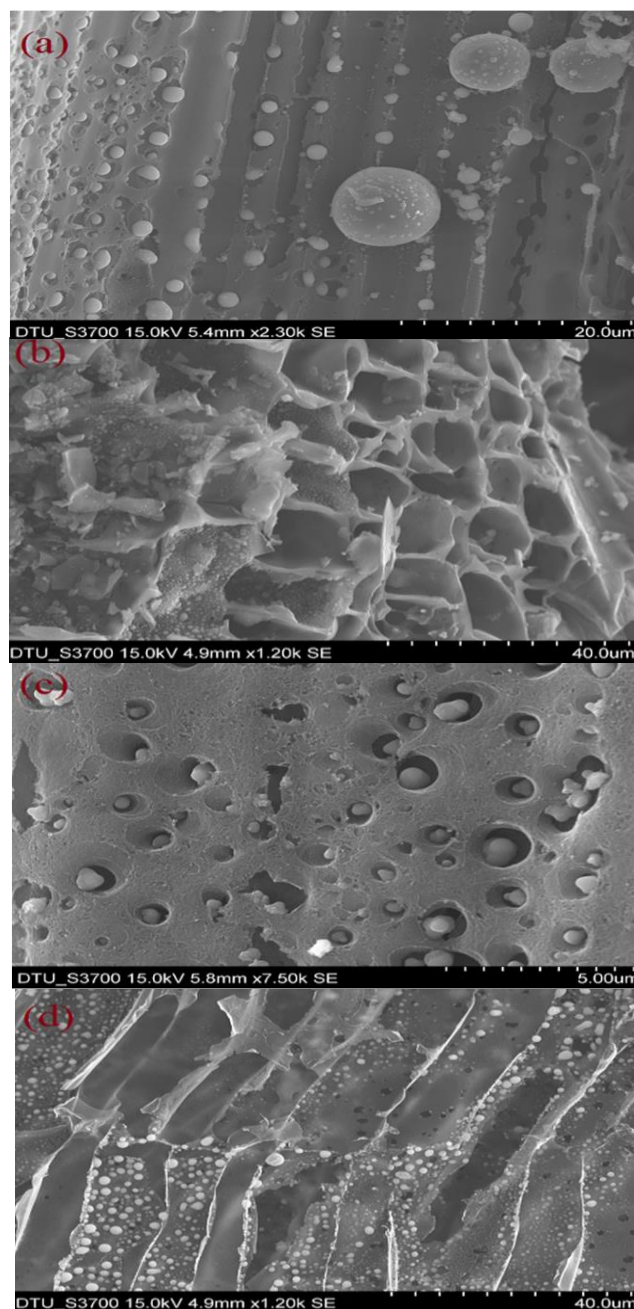


Fig. 2. SEM micrographs of bamboo charcoal (a), (b) and (d) shows lateral view of pyrolyzed bamboo at different magnification scales. (c) Shows the magnified image of lateral view showing the pores and spherical silicon particles.

Results and discussion

The wall of the parenchyma, basic unit inside bamboo, and nanosized particles presented in the soot of BC has been analyzed by using Scanning Electron Microscope (SEM).

SEM images have been shown in **Fig. 2**. Scanning electron microscopy on the BC has been performed at various locations and at different magnification. As shown in **Fig. 2(b)** and **(d)** white spherical particles were observed and further characterized by using XRD and EDX and found to be of pure silicon. As evident in **Fig. 2(c)**, porous nature of BC was confirmed and Si nanoparticles were found to be embedded into the pores of BC. High density of nanoparticles was evident in **Fig. 2(d)**.

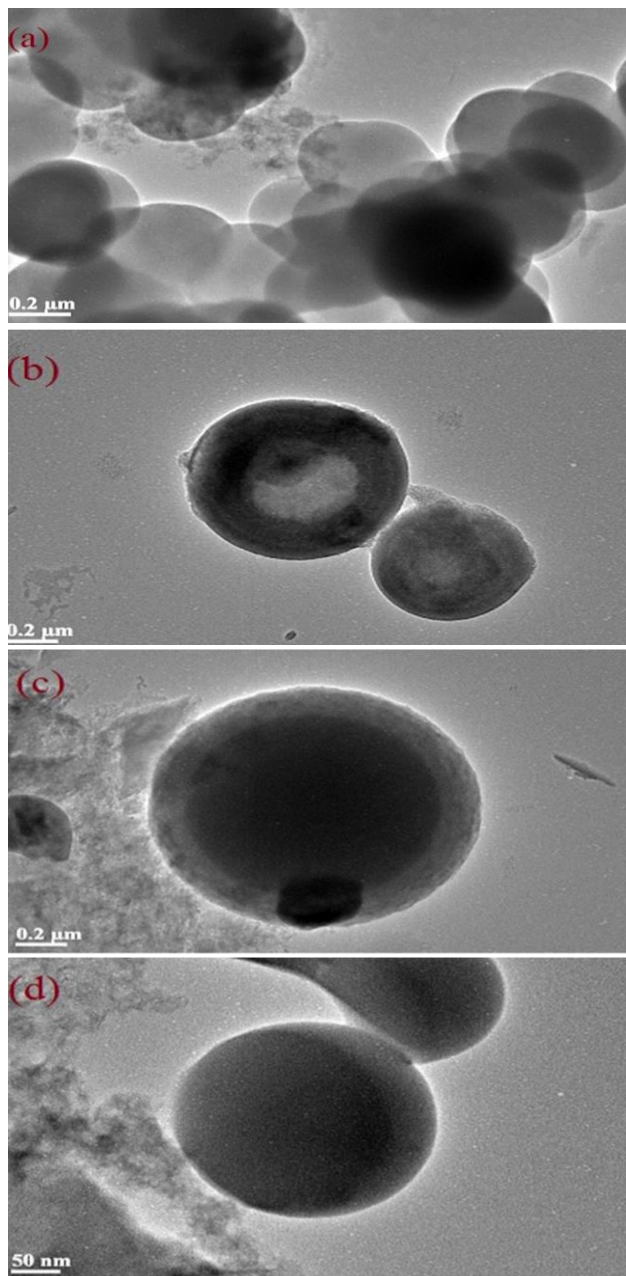


Fig. 3. Bright field TEM micrographs of bamboo charcoal (BC) (a) TEM image shows the presence of silicon spherical particles distributed randomly. (b) and (c) images confirm that particles formed are almost of the same size. (d) The size of silicon nanoparticle seen in figure and found to be approximately 200 nm.

Interestingly, periodic growth pattern of nanosized Si particle was observed as depicting in **Fig. 2(a)**. Periodicity of pattern was nearly uniform and depends on growth location in BC. Nanoparticle size was slightly lower than

pore size of BC. Particles of large size were not readily found as evident from **Fig. 2(a)**. The density of particles was found to be remarkably high in area where BC has convex curvature i.e. inner wall of parenchyma. As pore size of BC increases, cluster of nanoparticle was found to be embedded in pore, as shown in **Fig. 2(c)**. SEM images confirm that the pyrolysed BC has porous structure, and exhibit a wide pore distribution in the range from 100 nm to 1 μm . Pore size was found to play a decisive role in controlling the size as well clustering of nanoparticles. The sample was further characterized by using transmission electron microscope (TEM).

As evident from bright field TEM images in **Fig. 3** random distribution of nanosized spherical particles has been observed. All the particles have almost uniform spherical shape. Core size nanoparticle with shell of carbon has been seen in **Fig. 3(c)**. Variation in contrast of both core and shell reflects that shell is made of material having low atomic mass that may be carbon. Approximate particle size determined and came out to be around 220 nm. As evident from **Fig. 3(b)**, silicon also possesses porous nature.

XRD measurement was performed on the residual soot of BC (**Fig. 4**). XRD results show the presence of mainly carbon and silicon along with other impurities like silica and phosphorous. Carbon is found to exist in two main phases, one which has characteristic peaks at $2\theta = 24.26^\circ$ & 31.733° positions. These peaks correspond to (110) and (111) planes of orthorhombic phase (PCPD file no.89-8493), respectively. Second phase, hexagonal structure (PCPD file no. 26-1083) was evident by the observation of peak at $2\theta = 43.9^\circ$ position which corresponds to (0010) plane of HCP carbon. Apart from carbon, silicon found to have their peaks at $2\theta = 22.9^\circ$, 49.9° , and 80.266° positions and assigned to (120), (412) and (640) planes of silicon of tetragonal structure (PCPD file no. 39-0973), respectively.

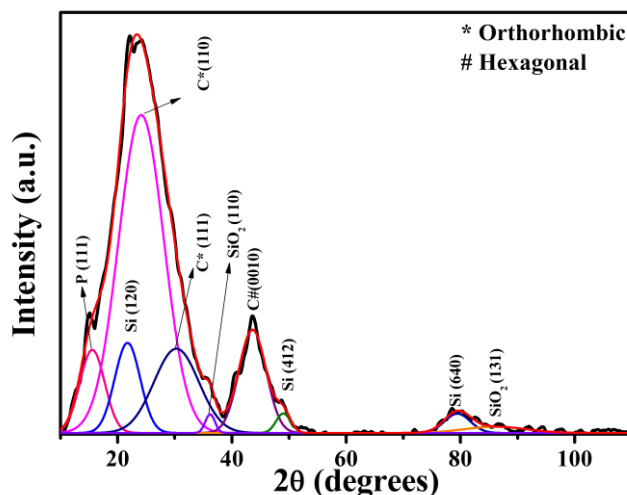


Fig. 4. XRD results for pyrolysed bamboo at 1250 °C.

These multiple peaks confirm that silicon present in BC soot is polycrystalline in nature. In previous published reports, silicon rarely exists alone in elemental state and forms compounds easily with various other elements present [44]. Here, we observed that pure silicon formed. However, silica is also present in very small quantity. Two

peaks observed at $2\theta = 36.54^\circ$, and 83.83° positions (PCPD file no. 89-8934) are assigned to be of silica. In earlier reports, along with main elements and their compounds, a large variety of different minerals have found in the BC depending on the geographical location where bamboo have grown and the processing route/conditions it went through [45]. In the reported pyrolyzed BC, we found the appreciable amounts of phosphorous. One single peak observed at $2\theta = 15.263^\circ$ position is assigned to (111) plane of phosphorous (PCPD file no. 75-0577).

In order to confirm the exclusive extraction of silicon only from the bamboo grown Delhi NCR region, EDX X-ray mapping on the pyrolyzed BC, residual pyrolyzed soot of the bamboo grown outside Delhi region, have been performed as shown in Fig. 5.

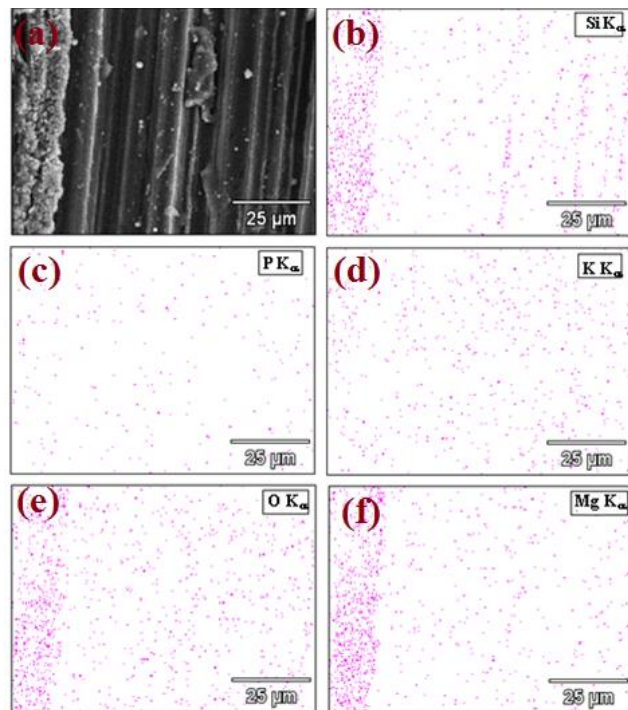


Fig. 5. EDX spectral mapping of elements present in bamboo charcoal in area shown in image (a).

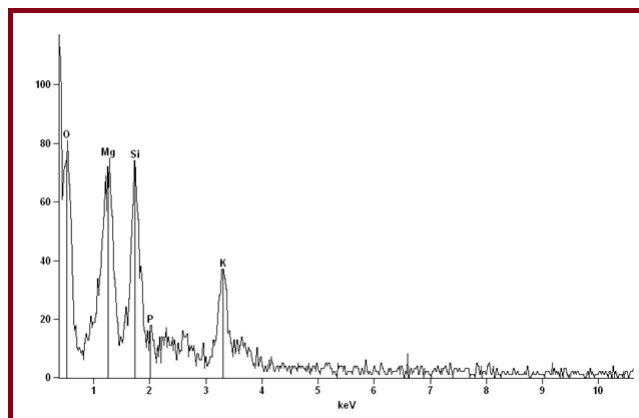


Fig. 6. Resulting EDX spectrum from mapped area revealing that Mg, Si, P, and K are the main elements present with oxygen.

Image in Fig. 5(a) shows an inspection area within where EDX mapping carried out by rastering the incident electron beam to produce the map of elements present. The tabulated (Table 1) results provide a semi-quantitative estimation of the elemental composition in the inspection area in units of both weight percent and atomic percent. The mapping reveals that Si, Mg, K and P are the main elements present within the inspection field. Si is being the third most abundant element. In Fig. 5, the maps of Si, P, K, O and Mg are shown individually and depicts that oxygen is present more in inspection area where Si and Mg are more abundant. P and K have been randomly distributed. This may be due the formation of anhydrous magnesium silicate.

Table 1. Tabulated results of EDX mapping area (Fig. 5a) revealing that Mg, Si, P, and K are the main elements present with oxygen, in pyrolysed bamboo charcoal (BC).

Element Line	Net Counts	Weight %	Weight % Error	Atom %	Atom % Error	Formula
O K _α	1524	54.18	+/- 3.80	68.31	+/- 4.80	O
Mg K _α	964	15.58	+/- 1.31	12.93	+/- 1.09	Mg
Si K _α	882	14.71	+/- 1.37	10.56	+/- 0.98	Si
Si L _{α1}	0	---	---	---	---	
P K _α	79	1.37	+/- 0.61	0.89	+/- 0.40	P
P L _{α1}	0	---	---	---	---	
K K _α	526	14.16	+/- 1.88	7.30	+/- 0.97	K
K L _{α1}	0	---	---	---	---	
Total		100.00		100.00		

Catalyst free synthesis of pure crystalline Si as well as Si nanostructures in BC was made by the pyrolysis of bamboo which was grown in Delhi NCR region. Growth mechanism of these structures can be understood as: first, during the pyrolysis at temperature below 100°C water vapors present in bamboo get released and subsequently large amount of CO_2 and CO gases released at temperature in the range $250^\circ\text{C} - 400^\circ\text{C}$ [46]. As temperature was raised above 700°C , aromatic rings, present in parenchyma, start rearranging and get converted into BC which has long-range graphitic order. However, scaffold of the bamboo remained unchanged but only carbon atoms rearranged their position. This resulted in the crystal structure change as evident from XRD pattern. During the pyrolysis, simultaneous aggregation as well as surface diffusion of atomic Si occurred on the wall/surface of BC and then Si atoms got trapped in the pore of BC. This resulted in the formation of Si nanoparticles. At high temperature migration of silicon particles is also possible. If this happened, then clustering of Si nanoparticles occurred as evident from Fig. 2(c). In present case, synthesis temperature is $\sim 1250^\circ\text{C}$ much lower than that of required for the formation of β -phase SiC [47.] and release of CO_2 & CO gases at this temperature prevents the formation of SiC. This resulted in the formation of pure silicon nanoparticles. Small fractions of Si nanoparticle get oxidized during the cooling which was evident from XRD pattern.

Conclusion

In present report, synthesis of pure polycrystalline silicon nanoparticles embedded in bamboo charcoal has been demonstrated. By adopting the green route for synthesis,

core-shell nanoparticles of almost uniform morphology have been synthesized along with formation of bamboo charcoal. These nanoparticles and bamboo charcoal were found to have porous structure. By estimating the fractional quantity; it was found that purity of silicon also depends on the geographical or soil condition of the region where bamboo grows. We envision that the present route for synthesizing Si/SiO₂ could pave the way for producing the low-cost solar grade silicon for photovoltaics.

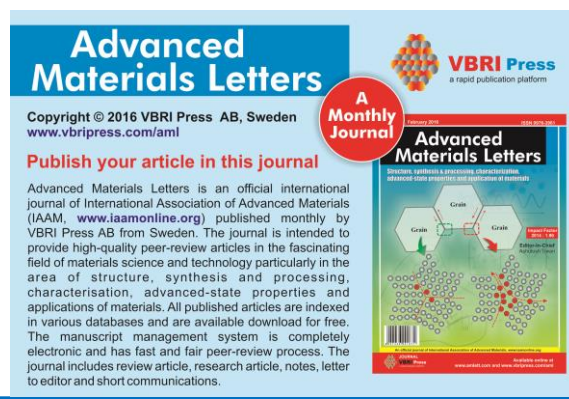
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