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Synthesis and Characterization of SiO_2 Nanowires Prepared from Rice Husk Ash

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Abstract

SiO_2 nanowires were synthesized by thermal evaporation of rice husk ash and coconut shell charcoal with the ratio of 1:2 by weight heated at a temperature of 1,350°C in atmosphere of nitrogen for 1 hour. The scanning electron microscope and X-rays diffraction instrument are used to characterize the SiO_2 nanowires. The SiO_2 nanowires synthesized have diameters varying from 40 nm to 200 nm while the lengths were a few micrometers.

Key words: Rice husk ash, SiO_2 nanowires, Coconut shell charcoal

Introduction

Rice husks and coconut shells are abundant residues in agricultural countries. Rice and coconuts are grown in many countries of the world, especially in Asia. There are many rice and coconut farms in Thailand. Rice husks are by-products from mill process of rice. A small amount of rice husks and coconut shells were used in biomass fuel, but the greater part of the rest were treated as waste materials. These agricultural wastes have been studied to produce raw materials such as silicon dioxide, silicon carbide, silicon nitride or new materials and pure silicon for solar cells from rice husks Amick (1982), Hunt, *et al.* (1984) and Real, *et al.* (1996) or activated carbon from coconut shells charcoal.[18] The amount of global rice production is nearly 400 million metric tons per year. Rice husk is a by-product of rice milling, and amounts to proximately 10 % of the grain.[6] Rice husks contain about 13-29 % inorganic constituent, of which 87-97 % is SiO_2 (silica) in an amorphous condition.[15] The major impurities in rice husks are Na, K, Mg, Ca and Fe. Their presence as oxides and silicates can vary from 3 to 13 % in ash. They can be easily removed by acid-leaching, while the other impurities such as Mo, Ti, Ta, Ni, V, Cr are at very low concentrations and can also be eliminated just as effortlessly easily. The high purity silicon of rice husk as a potential source for solar-grade silicon has been demonstrated.[2] Several papers reported that pure polycrystalline silicon can be prepared from rice husk white ash by a metallothermic reduction process.[4, 5] Nano-structured silica has been prepared from rice husks, and uniformly sized ultrafine silica powder can be obtained by nonisothermal decomposition of rice husk in an oxidizing atmosphere.[12] SiO_2 and Si nanowires could be synthesized in many processes from silicon dioxide or silicon oxide source.[11] Recently, amorphous SiO_2 nanowires have been synthesized from a vapor-liquid-solid method using Ga as catalyst, and the Si substrate source very fine SiO_2 nanowires with diameters from 50 to 100 nm and lengths from 10 to 50 micrometers were grown in 5 hours.[9] Silicon powder and graphite were heated at 1200°C under a mixture of Ar/O_2 (99.2/0.8 mol %) for 30 min. Amorphous silicon dioxide nanowires could be synthesized with lengths of up to 500 micrometers and diameters in the range of 10-300 nm. [10] Li, *et al*. (2004) observed that SiOx nanowires were accidentally synthesized on Au-coated Si substrate via solid-liquid-solid (SLS) process of ZnO powder and graphite powder heated at 900 to 1100°C. The production nanowires with diameter vary from 50 to 300 nm and increase with synthesized time.[14] Recently, nanofibers and nanowires materials were prepared from rice husk ash mixed with coconut shell charcoal and GeO_2 as source materials heated at 1200°C under one atmosphere of nitrogen with flow rate of 1 L/min for 3 hours.[17]

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In this work, we used SiO_2 from rice husk ash mixed with coconut shell charcoal as starting materials. The mixtures and Si substrate were heated at 1350°C in nitrogen gas. After the furnace was cooled down to room temperature, the products and the substrates were investigated.

Materials and Experimental Procedures

The rice husks used were taken from rice mills. They were further washed with tap water to remove soils and dirt, and dried in the sun. The dried husks were washed with distilled water and dried again in an oven at 60°C. The dried husks were treated to chemically. The 37% HCl acid used in this work was produced by MERCK, Germany, and was used as received. A proper amount of the HCl was mixed with distilled water to form 1 molar of HCl. The rice husks mixed with 1 molar of HCl in a glass beaker placed on hot plate at 90°C for 1 hour. The solution was filtered and the rice husks were washed with distilled water several times until they were acid-free. The acid-leached rice husks were dried in an oven at 60°C. They were fired at 600°C in air for 1 hour. The coconut shell was cleaned with tap water and distilled water several times and dried in oven at 60°C. The cleaned coconut shell was burned at 400°C in normal atmosphere to form charcoal. The coconut shell charcoal was ground to fine powder and put in a glass beaker. The 1 molar of HCl was poured in the beaker and soaked for 1 hour. The solution was filtered and the charcoal powder was washed with distilled water several times until acid-free. These processes of preparation were explained in a previous report.(17) The rice husk ash consisted of > 99 % amorphous silica. The coconut shell charcoal comprised of > 70% of carbon. The source materials consist of the mixture of cleaned rice husk ask and coconut shell charcoal with ratio of 1:2. They were ground to form powder mixtures. The powder mixtures were put in an alumina boat and then Si substrates were placed on the powder mixtures. The alumina boat and source materials were placed in the middle of quartz tube furnace. They were heated at 1350°C in one atmosphere of nitrogen with a flow rate of 2 L/min for 1 hour. Afterward, the furnace was cooled down to room temperature naturally. The source materials and substrates were studied by scanning electron microscope (SEM) and X-ray diffraction (XRD) instruments.

Results and Discussion

After heat treatment, the furnace was cooled down to room temperature naturally. The substrate and source materials were taken out of the furnace tube. We noticed that some products materialized on substrate and in the powder samples that can be seen with naked eyes. These synthesized products were studied by SEM and XRD instruments. Figure 1 shows a SEM image of source materials before heat treatment. There are some large size particles materials in this figure, consist of rice husk ash and coconut shell charcoal. Figure 2 shows XRD patterns of the source materials before heating, Figure 2(a) indicates the XRD pattern of SiO_2 and Figure 2(b) indicates the XRD pattern of carbon. Figure 3 shows SEM images of the synthesized products on Si substrate. This figure reveals the general morphologies of synthesized nanostructures products, including nanoparticles and nanowires. Figure 3(a-b) illustrates that the low-magnification SEM images of nanostructures materials, Figure 3(c-d) shows high-magnification SEM images.

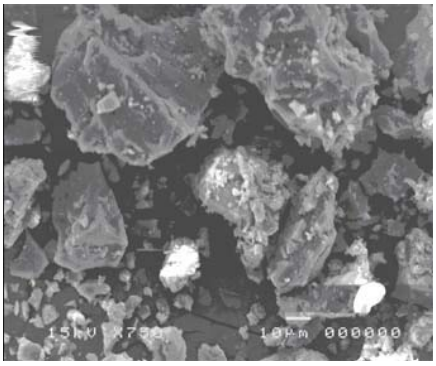
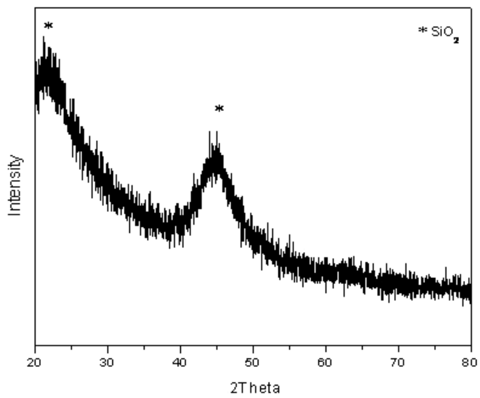
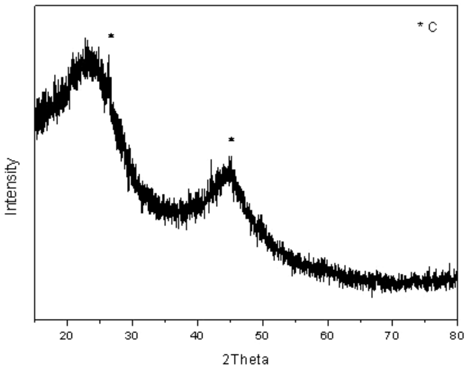


Figure 1. SEM image of the source materials before heat treatment.

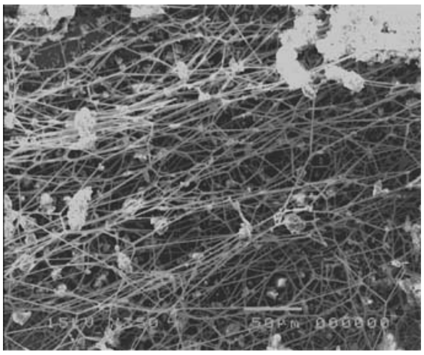


(a)

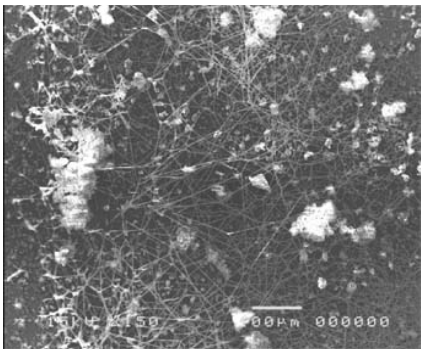


(b)

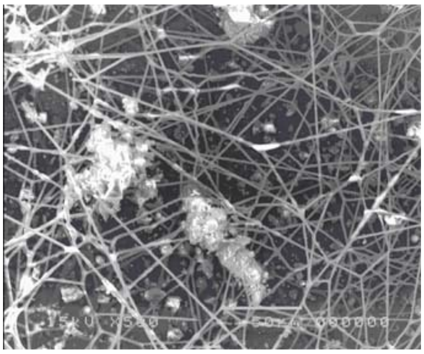
Figure 2. XRD pattern of the source materials (a) curved of SiO_2 and (b) carbon pattern before heat treatment.



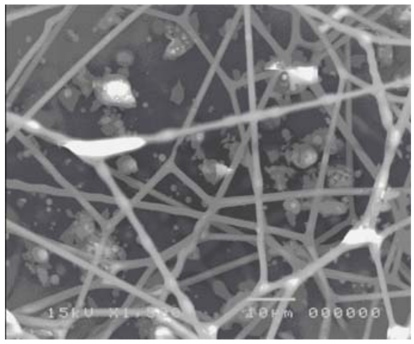
(a)



(b)



(c)



(d)

Figure 3. SEM images of synthesized products on Si substrate after heat treatment.

The diameters of typical nanowires vary from 80-100 nm and the lengths of typical wires are about to micrometers. The crystal structures of these nanowires were investigated by XRD technique. The XRD result in Figure 4 shows SiO_2 and SiC structures. The growth of SiO_2 nanowires was explained by using the reaction $2CO_2(g) + Si(s) \rightarrow 2CO + SiO_2$.[17] The formation of SiC nanowires can be explained by carbothermal reduction, $SiO_2 + 3C \rightarrow SiC + 2CO$ at high temperature in atmosphere of argon gas.[8] The formation of one-dimensional nanostructures can be explained by two similar mechanisms, namely the vapor-liquid-solid (VLS) and the vapor-solid (VS) processes. The VLS growth is a catalyst-assisted process .[13, 16]

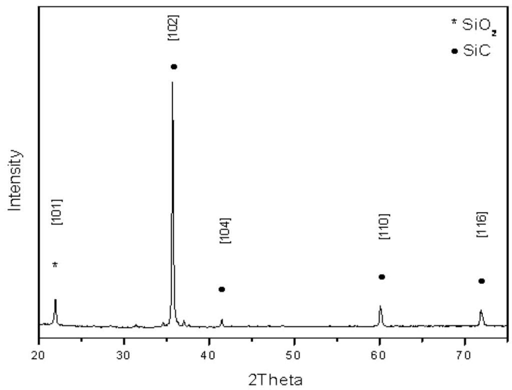
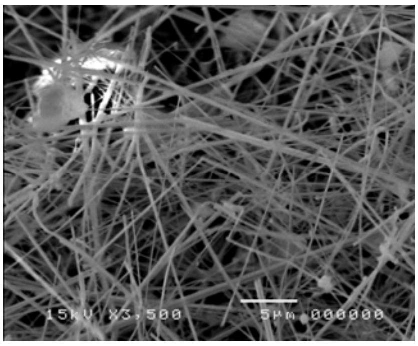


Figure 4. XRD pattern of the prepared products forms on Si substrate.

The source materials were also studied by SEM instrument after heat treatment. Figure 5 shows SEM images of source materials. The SEM images reveal complicated nanowire structure materials. Figure 5(a) indicates the low-magnification SEM images of nanostructures materials. Figure 5(b) shows high-magnification SEM images. The typical nanowires materials vary from 50 to 100 nm and the lengths of typical wires are about to micrometers. These nanowires materials were also investigated by XRD instrument. The XRD pattern in Figure 6 shows the crystal state of SiO_2 structures.

(a)



(b)

Figure 5. SEM images of source materials powder after heat treatment.

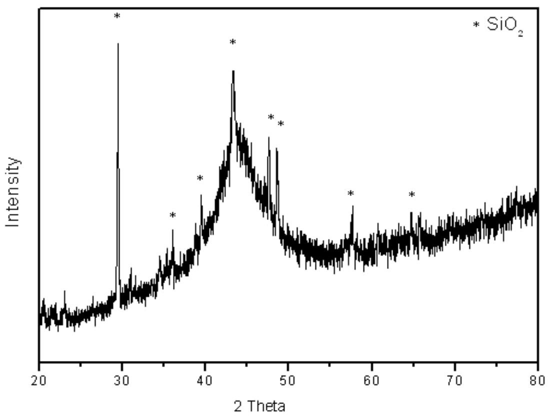


Figure 6. XRD pattern of source materials powder after heat treatment.

Conclusions

In summary, SiO_2 and SiC nanowires were synthesized by thermal evaporation of the mixture of rice husk ash and coconut shell charcoal at a temperature of 1350°C in an atmosphere of nitrogen with a flow rate of 2 L/min for 1 hour. The structures of these products have been characterized by scanning electron microscope and X-rays diffraction instruments with appropriate methods. These SiO_2 and SiC nanowires prepared from rice husk ash may be used in future applications in nanotechnology.

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