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GROWTH MECHANISM OF SILICON OXIDE NANOWIRES SYNTHESIZED IN A CARBOTHERMAL CHEMICAL VAPOR DEPOSITION REACTOR

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ABSTRACT

Silicon oxide nanowires (SiO_2NWs) have been synthesized using the carbothermal method on silicon at 1000°C utilizing gold as the catalyst. Field Emission Scanning Electron Microscopy (FESEM) analysis revealed that thin gold layer agglomerated into nanosized particles which formed the spots for SiO_2NWs growth. A growth mechanism model based on the dissolution and condensation of Silicon on the gold nanoparticles is proposed. Energy Dispersive X-ray Spectroscopy (EDX) and X-ray Diffraction Analysis (XRD) were performed on the SiO_2 nanowires samples.

Keywords: SiO_2 Nanowires; Carbothermal; Growth Mechanism

INTRODUCTION

SiO_2 fibers have many applications in photonics and there has been active researchers in the synthesis and application of SiO_2NWs[1-4]. In this paper, we describe the synthesis of SiO_2nanowires using the carbothermal synthesis method. Here, a mixture of carbon and ZnO powder were heated inside a tube furnace to produce CO_2 gas and Zn metal. By adjusting the position of the silicon substrate used, Zn metal was prevented to form on the substrate. The CO_2 gas provided the oxygen source for the formation of SiO_2.

MATERIAL AND METHOD

SiO_2nanowires were synthesized using carbo-thermal chemical vapor deposition route. The N-type Si (100) substrates were cut into 1cm x 1cm and cleaned ultrasonically with acetone for 15 minutes. Then, it was dipped into hydrofluoric (HF) acid for 15 minutes to remove the SiO_2layer. The substrates were rinsed with running distilled water and dried in air. The cleaned substrates were coated with thin layer of gold in a dc sputtering for 30s. Horizontal tube furnace with maximum of 1200 ^{0}C was used to accomplish the

synthesis process. In the experiment to synthesis silicon oxide nanowires, SiO and graphite powder with 1:1 mass ratio were mixed together. Then, mixture was kept in a porcelain boat (2cm diameter, 8 cm long). The prepared source and substrate were loaded (downstream) in the middle of horizontal tube furnace as shown in Figure1**.** Other parameters were set as follows; the furnace temperature was set at 900^0C, 950^0C and 1000^0C, the dwelling time at 90 minutes (1.5 hours) and in each experiment trial, the substrates were placed 2 cm apart from the source. The argon gas was flowed into the tube throughout the heating, synthesis and cooling times. After cooling down at room temperature, a thin layer of gray color was found on the surface of substrate. The surface morphology of growth nanowires were characterized using field emission scanning electron microscope (FESEM, FEI Quanta 200F) equipped with an energy dispersive X-ray spectrometer (EDX, Quanta 200F). The crystalline characteristics of formed nanostructures were studied by X-ray diffraction (XRD, SiemensD-5000).

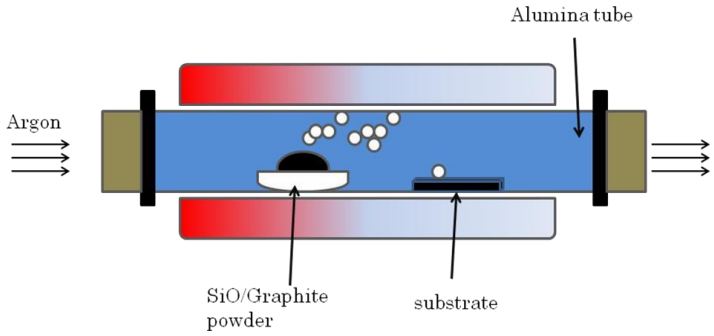
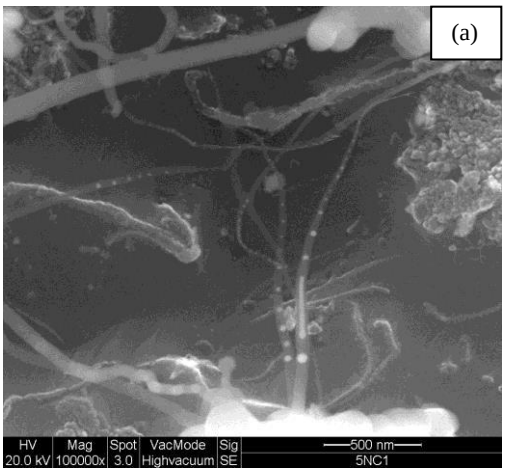
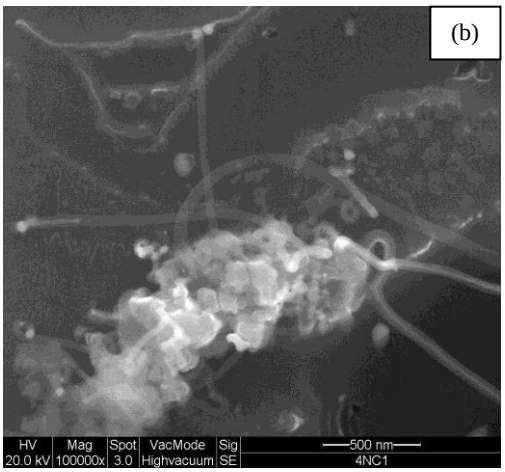


Figure 1: Experimental set up diagram of the Carbo-thermal CVD method





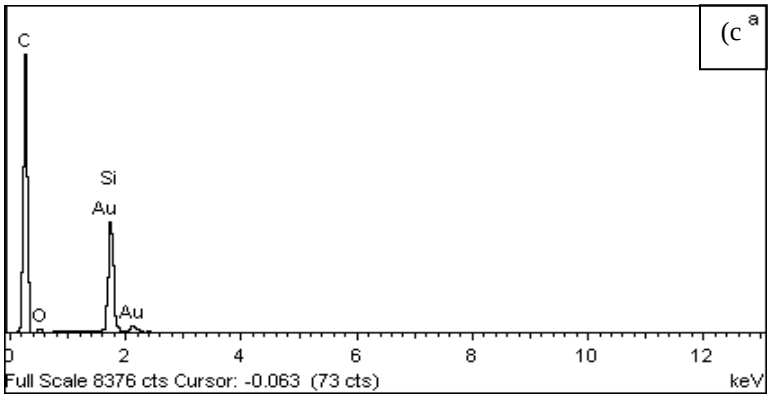


Figure 2: FESEM SiO_x nanowires grown using 30s-Au coated silicon substrate at (a) 900ºC (b) 950^0C while (c) is the typical EDX spectrum for the samples

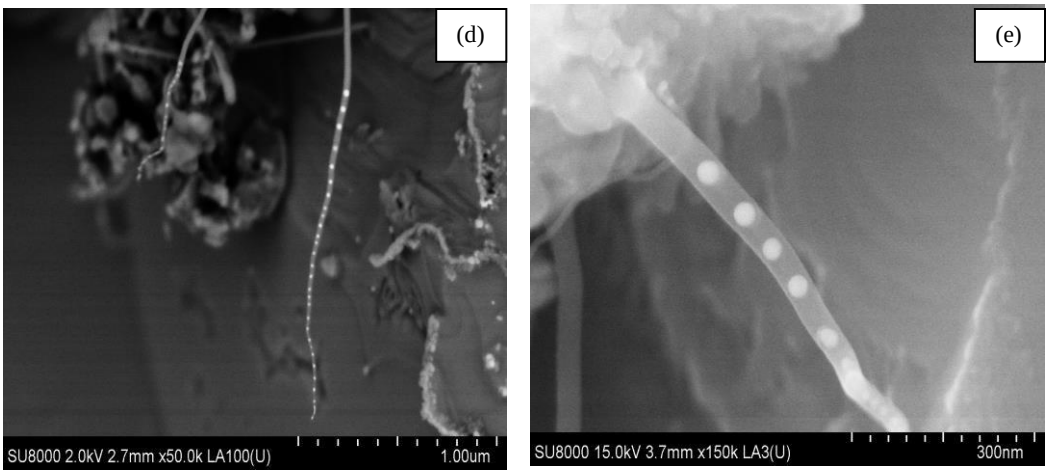


Figure 3: FESEM micrograph of SiO_2 nanowires on Au-Si coated at 1000^0C at (d) low and (e) higher magnification

RESULT AND DISCUSSIONS

In this study, the Au layer thickness was prepared by constantly set the sputtering time at 30s only. But the deposition temperature was varying three times which is from 900^0C to 1000^0C. Figure 2 shows the FESEM images for SiO_2nanowires grown on Au-Si coated substrates at (a) 900^0C and (b) 950^0C source temperature, while (c) is the typical EDX spectrum for the nanostructures. The EDX reveals that the nanostructures consist of Si, O, Au and the Carbon peak is believed comes from the graphite powder used in the source. Further on doing the experiment, the source temperature was then

enlarged at 1000^0C to observe the formation of SiO_2NWs. Figure 3 reveals the FESEM images of that particular experiment at low and high magnifications. It can be seen some of the wires were embedded with gold which can be seen clearly in Figure 3 (e). The possible mechanism of the formation of SiO2 nanostructures can be explained here. Upon heating the coated Si substrate at appropriate temperature, the Au thin film formed to be nanoparticles (like spherical ball). The Au catalyst dot served as a nucleation seed for the subsequent nanowire growth. The vapors are transported towards the Si by using Ar as the carrier gas. Even we are not yet study the effect of Ar flow gas, but according to [1], the flowing may cause the substrate surface temperature to change or fluctuated. Thus, it is believed, if the temperature suddenly drops, the growing nanostructures can be solidify with wider diameter before it's completed.

The XRD were carried out for all samples. In figure 4, we can observe amorphous structure of SiO2 nanowires. The associated marked peaks reveal the attendance of gold and silicon at different facets.

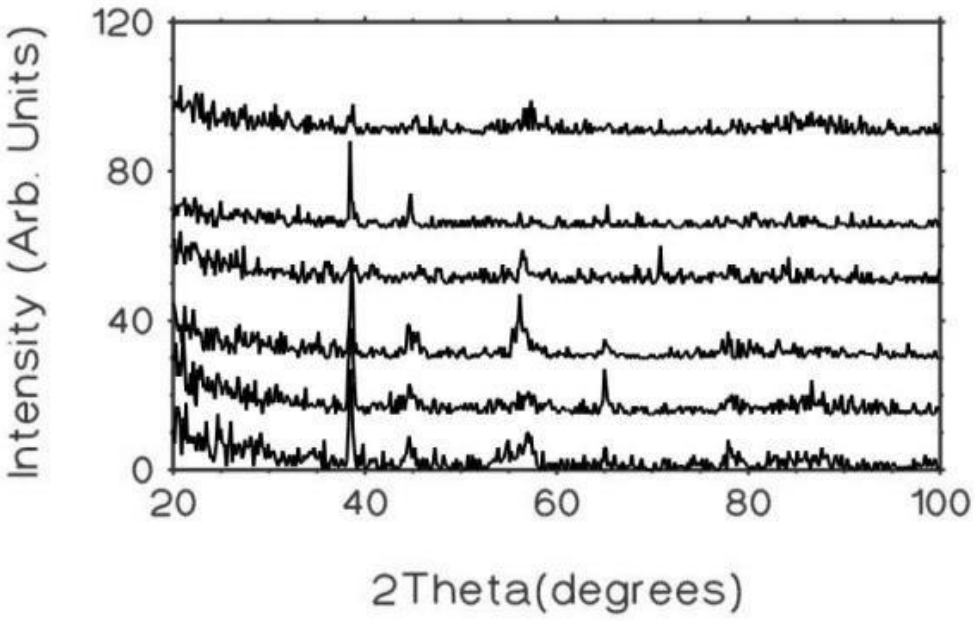


Figure 4: XRD spectrum of A) 900^0C, B) 950^0C and C) 1000^0C nanowires for 90 minutes

The possible mechanism for the SiO_2 formation is depicted in Figure 5. Figure 5(a) the Si chip was coated with thin gold layer. Once heating, the gold layer agglomerated and formed nanosized particles at the surface of the substrate. This particles were formed due to the melting of the Au nanolayer whereas the surface tension was the result of the Si surface. Si atoms dissolved in the gold particles at the elevated temperature used until saturation as in Figure 5(b). The Ar gas flow at small temperature fluctuation caused the Si atom to condense on the Au particle surface, and subsequently being oxidized by the

CO_2 gas in the deposition atmosphere (Figure 5(b)). Many cycles of dissolution and condensation, eventually will resulted in the growth of long SiO2 nanowires. Gold nanoparticle catalysts, may be embedded in the SiO_2nanowires due to the interaction of melted Au nanoparticles and the surface tension of the SiO2 subtrate.

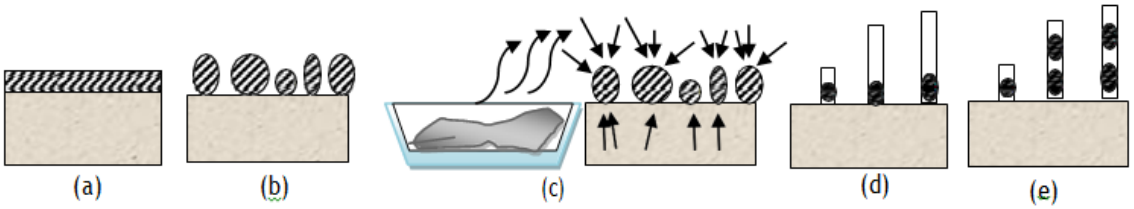


Figure 5: Possible mechanism of the SiO_2NWs. (a)Silicon chip coated with Au at room temperature. (b)Spherical ball form at the surface of silicon chip. (c) SiO_2 dissolved in Au. (d-e) Nanowires were form

CONCLUSION

SiO_2NWS have been synthesized on Si using Au as catalyst verified by FESEM, EDX, and XRD analyses. It was observed that the gold catalyst served as medium for dissolving Si which recondensed under slight fluctuating temperature conditions to forms nanowires. This seems to depend on the size of the gold particles. A model based on this phenomenon was proposed. Besides, gold nanoparticles were observed to be incorporated into the SiO_2NWs. This creates the possibility the creation of stable nanosized Au nanoparticles.

The characterized by the few methods has proved that nanowires exist at the surface of the materials. The characteristics using FESEM, show the structure of the nanowires. To get the best result in term of the surface, refer to the powder mixture used, the temperature used in heating process, and other factors. From the results, the diameters of nanowires can be determined, which range from 10nm to 200nm. The characterizations using EDX spectrum shows that the substrate contain Si, O, Au and carbon. While in XRD, the XRD spectrum shows that it is cover with Au and Si.

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