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Formation of silicon oxide nanowires directly from Au/Si and Pd–Au/Si substrates

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Abstract

Amorphous silicon oxide (SiO_x) nanowires were directly grown by thermal processing of Si substrates. Au and Pd–Au thin films with thicknesses of 3 nm deposited on Si (0 0 1) substrates were used as catalysts for the growth of nanowires. High-yield synthesis of SiO_x nanowires was achieved by a simple heating process (1000–1150 °C) in an Ar ambient atmosphere without introducing any additional Si source materials. The as-synthesized products were characterized by field-emission scanning electron microscopy, energy-dispersive X-ray spectroscopy, and transmission electron microscopy measurements. The SiO_x nanowires with lengths of a few and tens of micrometers had an amorphous crystal structure. The solid–liquid–solid model of nanowire formation was shown to be valid.

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1. Introduction

Studies on the fabrication and characterization of one-dimensional (1D) nanostructures such as nanowires and nanotubes have flourished in recent years because of their fundamental importance to nanotechnology [1,2]. A number of applications using 1D nanostructures have been investigated for nanoscale devices, devices using flexible substrates, and sensor applications [3–5]. Recently, silicon oxide (SiO_x) nanowires have attracted intensive interest due to their novel physical properties and potential applications in realizing multi-functional nanosized devices, such as blue light emitters and optical sensors with high sensitivity [6,7]. Various fabrication methods, including laser ablation, chemical vapor deposition, and evaporation, have been used to produce SiO_x nanowires via a vapor–liquid–solid (VLS) process requiring Si source materials [8–10]. In this work, we report simple synthesis of amorphous SiO_x nanowires achieved by thermal heating of Au and Pd–Au coated Si substrates in an Ar ambient atmosphere without any additional silicon source supply.

2. Experiments

Si (0 0 1) substrates were used in our experiments. The substrates were alternately ultrasonically cleaned in acetone and in methanol for 5 min, and then dried by nitrogen blowing. Each 3 nm thin layer of Au and Pd–Au was deposited on the substrates in a simple sputtering system. For deposition of the Pd–Au catalyst layer, Pd–Au alloy (Pd:Au = 1:1) was introduced as the target material. The substrate was placed in an alumina tube, which was then heated in a tube furnace at 1000–1150 °C. While heating the substrates for 90 min, Ar gas with the flow rate of 50 sccm was introduced in the alumina tube at an atmospheric pressure of 760 Torr. After cooling down to room temperature, a thin layer of white-colored deposit was

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found on the substrate surface, indirectly indicating the formation of SiO_x nanowires. Measurements utilizing a field-emission scanning electron microscope (FE-SEM), equipped with an energy-dispersive X-ray spectroscope (EDX) as well as a transmission electron microscope (TEM), were carried out for analysis of the morphology and atomic composition of the grown nanowires.

3. Results and discussion

Fig. 1 shows FE-SEM images revealing the general morphologies of the thermally heated Au deposited Si (0 0 1) [Au/Si] and Pd–Au deposited Si (0 0 1) [Pd–Au/Si] substrates in the tube furnace. As shown in Fig. 1(a), Au nanoislands with diameters of 10–80 nm were formed on the surface of the substrate by heating the Au/Si substrate at 1000 °C. On the other hand, the formation of Pd–Au nanoislands with a uniform size distribution (a diameter of around 100 nm) by heating the Pd–Au/Si substrate at 1000 °C was observed in the FE-SEM measurement (Fig. 1(b)). From the EDX analysis, it was found that the formed Pd–Au nanoislands have a dual structure consisting of Pd surrounded by Au. In addition, the EDX spectrum collected from the nanoislands showed the presence of Au, Pd, Si, and O elements. Interestingly, no nanowire growth was seen on the Au/Si substrate, while SiO_x nanowires were locally formed on the Pd–Au/Si substrates, as shown in Fig. 1(b), in spite of the same heating temperature of 1000 °C. This fact indicates that the dual structure consisting of Pd surrounded by Au facilitates the formation of nanowires.

Fig. 2 shows FE-SEM images revealing the general morphologies of SiO_x nanowires grown on Au/Si and Pd–Au/Si substrates at 1050 °C. As shown in the FE-SEM images, a large number of nanowires with lengths of tens of micrometers and diameters around 100 nm were formed on the Pd–Au/Si substrate surface, while a small number of nanowires with lengths of a few micrometers were locally formed on the Au/Si substrate. EDX results in Fig. 3 show that our SiO_x nanowires grown on the Au/Si substrate have an atomic ratio of Si/O higher than the $\frac{1}{2}$ of SiO_2. On the other hand, the atomic ratio of Si/O in the SiO_x nanowires formed on the Pd–Au/Si substrate is nearly consistent with the $\frac{1}{2}$ of SiO_2, indicating the formation of SiO_2 nanowires on the Pd–Au/Si substrate at this growth temperature.

Since no source of Si vapor existed, and the process temperature was not high enough to evaporate Si atoms into the Si substrate, the formation mechanism is different from the well-known VLS process requiring source materials and an evaporation technique. In addition, Si substrates were covered by a thin layer of Pd–Au and Au. Thus, the possible source for formation of SiO_x nanowires comes from the Si substrate, indicating that the formation mechanism of the SiO_x nanowires relies on a solid–liquid–solid (SLS) process. The melting point (MP) of pure Au is 1063 °C. As shown in Fig. 1, no nanowires were observed on the Au/Si substrate heated at 1000 °C, while a small number of nanowires with lengths of a few micrometers were locally formed on the Au/Si substrate at 1050 °C. These results suggest that the formation of SiO_x nanowires on Si via a SLS process by introducing the Au catalyst is somewhat limited below the MP of pure Au, even though the Au–Si eutectic temperature of around 370 °C is much lower than the process temperature of 1000 °C. On the other hand, a large number of nanowires were uniformly formed on the Pd–Au/Si substrate surface in spite of using a heating temperature lower than the MP of both Au and Pd (1554 °C). This might be due to enhanced catalytic activities by Pd–Au alloying compared to pure Au.

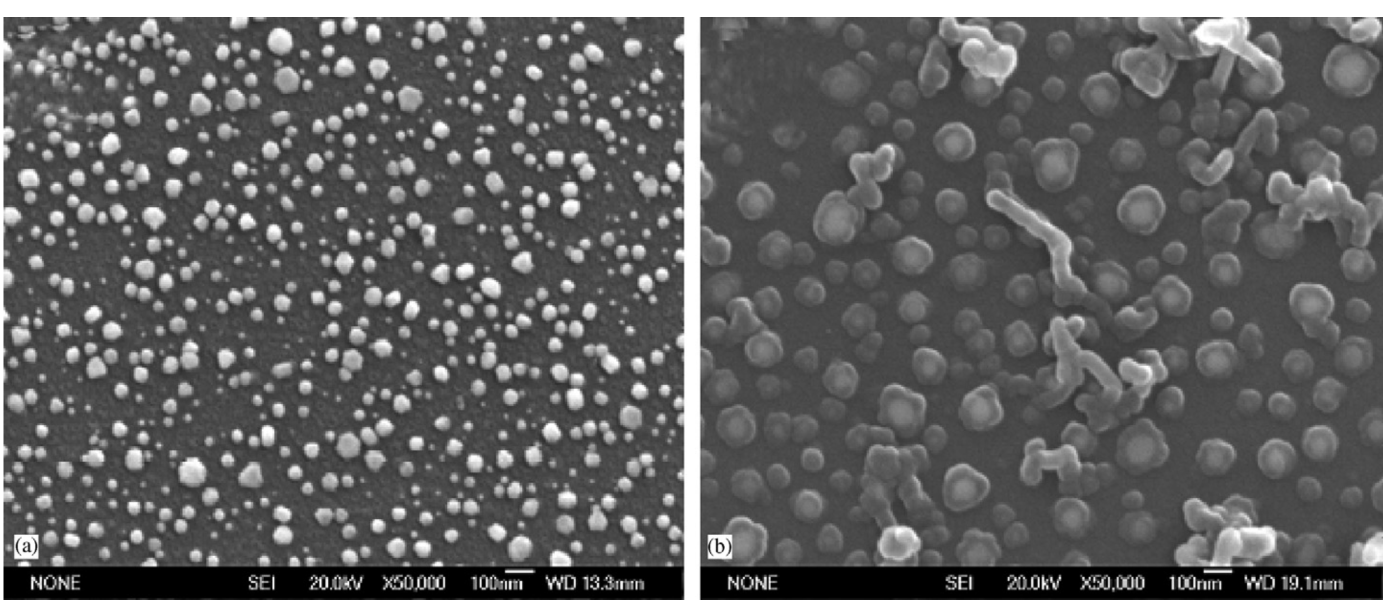

Fig. 1. FE-SEM images revealing general surface morphologies of (a) Au/Si and (b) Pd–Au/Si substrates treated by thermal processing at 1000 °C in an Ar ambient atmosphere in the tube furnace. The scale bar indicates 100 nm.