



Vanadium oxide assisted synthesis of networked silicon oxide nanowires and their growth dependence

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ARTICLE INFO

Article history:

Received 15 December 2010

Received in revised form 14 February 2011

Accepted 21 February 2011

Keywords:

Networked SiO₂ nanowire

CVD

Vanadium dioxide

Gold

ABSTRACT

Networked silicon oxide nanowires have been synthesized by VO₂-assisted chemical vapor deposition at 1000 °C on silicon substrate without supplying any gaseous or liquid Si source. Systematic study on the nanowire growth has indicated that morphology and composition of the final products are sensitive to the catalyst components, reaction atmosphere and temperature. Compared to Au and VO₂ as catalysts individually, co-catalysts of Au and VO₂ play a critical role in the formation of networked SiO₂ nanowires. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) observations indicate that the silicon oxide nanowires have smooth surfaces with uniform diameters of 30–100 nm, and their lengths reach several hundred micrometers. X-ray photoelectron spectroscopy (XPS) results reveal the atomic ratio of silicon to oxygen is about 1:2. Growth dependence of the networked nanowires on hydrogen and temperature is also discussed. Vapor-liquid-solid (VLS) process is proposed for the growth mechanism of the networked nanowires. It is also found that the growth mechanism of SiO₂ nanowires by increasing the temperature up to 1200 °C changes to vapor–solid (VS) processes since wire-like structures can be formed without any catalyst or H₂ gas introduced into the system.

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1. Introduction

In the past two decades, research on one-dimensional (1D) nanomaterials such as nanowires and nanotubes has increased dramatically due to their anisotropic shape and variable stoichiometry, which offer interesting physical and chemical properties and make them suitable for various applications.

Recently, 1D silicon oxide (SiO_x) nanostructures have attracted substantial interest because of their various morphology such as nanowires (Yang, Tay, Sun, Fan, & Shen, 2006), nanotubes (Zhang, Bando, Wada, & Kurashima, 1999), nanoflowers (Chen, Li, Han, & Dai, 2002), helical and nanosprings (Zhang et al., 2008). These diverse silicon oxide nanostructures show unique properties which make them potential candidates for various technologies such as optics (Hu, Jiang, Meng, Lee, & Lee, 2003; Kim, Shim, & Lee, 2007; Yang et al., 2006; Zhu et al., 1999), semiconductors (Park et al., 2007; Zhang et al., 1999) and catalysis (Zhang et al., 1999).

Up to now, different synthesis methods such as vapor transport (Yang et al., 2006), bio-mimetic strategies (Davis, Burkett,

Mendelson, & Mann, 1997), laser ablation (Zhu et al., 1999), physical and thermal chemical evaporation (Ni, Ying, Luo, & Li, 2007), thermal oxidation (Hu et al., 2003) and solution methods (Zhang et al., 1999) have been used to prepare silicon based nanostructures with different morphologies. However, synthesis of silicon oxide nanostructures with controlled and predictable morphology for commercial application remains a challenge (Huang, Ostrikov, & Xu, 2008).

Among the synthesis methods, vapor transport methods are often used to grow silicon based nanostructures owing to their simple systems and fast growth rates as compared to solution based methods. In addition, various mechanisms have been proposed for the growth of silicon oxide nanowires using vapor transport methods such as VLS and VS mechanism. In these methods, a source material, e.g. Si powder, together with a catalyst like Pb (VLS process), is used to grow Si based nanowires (Zhang, Jiang, Yang, & Li, 2007). Recently, studies of growing silicon oxide nanowires directly from silicon substrate without any external silicon source have been reported. Park et al. (2007) synthesized amorphous SiO_x nanowires using Au and Pd–Au alloy as catalysts. Kim et al. (2007) found that SiO_x nanowires can be grown from Cu coated silicon substrates. Lee et al. (2004) reported the growth of silica nanowires from silicon substrates covered with thin films of TiN/Ni. Some

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reports of other catalysts such as Fe, Ga, Ga–In and Co have also been presented (Kim et al., 2007). Generally for these processes, metal particles have been commonly employed as the catalyst and the solid–liquid–solid (SLS) growth mechanism has been proposed to obtain silicon oxide nanowires (Park et al., 2007; Wang, Chan, Xiao, Lin, & Shih, 2006).

In this paper, we report the synthesis of uniform silicon oxide nanowires with distinct network features with the aid of VO₂ powder directly on a silicon substrate. We found that the network characteristic of silicon oxide nanowires can be significantly enhanced by combining VO₂ and gold as a co-catalyst. We have also investigated the effect of hydrogen and temperature on the nanostructures deposited on the substrate and discussed the possible growth mechanism with respect to the role of catalysts and hydrogen in the system.

2. Materials and methods

The synthesis of silicon oxide nanowires was carried out in a horizontal tube furnace. In this study different combinations of catalysts were investigated including gold (Au), VO₂ powder and Au–VO₂ systems. Depending on the catalyst employed, bare or Au sputtered silicon (100) wafers were used as the substrate and silicon source. The Au layer with a thickness of 5 nm was sputtered on the silicon wafers using an Emitech K550X sputter coater. To synthesize silicon oxide nanowires, a silicon substrate with a catalyst layer was placed in an alumina boat which was transferred into the furnace. The furnace was heated to 1000 °C at a rate of 20 °C/min and kept at this temperature for 5 h. During the experiment a constant flow of high purity argon (200 sccm) and hydrogen (20 sccm) was maintained in the chamber. At the end of the experiment, white wool-like products were found on the silicon substrate.

The synthesized nanostructures were characterized using Hitachi S-4500 field-emission scanning electron microscope (SEM) operated at 5.0 kV, Oxford INCA energy dispersive X-ray spectrometer (EDX) operated at 20 kV, JEOL 2010F transmission electron microscope (TEM) operated at 200 kV, and Kratos Axis Ultra Al(alpha) X-ray photoelectron spectrometer (XPS) operated at 14 kV.

3. Results and discussion

3.1. Effect of catalyst

Fig. 1(a) and (b) shows SEM images of silicon oxide nanowires synthesized by heating the silicon substrates in the vicinity of only VO₂ powder at 1000 °C in an Ar/H₂ atmosphere. These images show randomly oriented nanowires and bundles formed in some areas completely covering the substrate (Fig. 1(a)). High magnification image of these bundles seen in Fig. 1(b) reveals highly tangled network structure of these nanowires. These nanostructures are similar to the nanostructures synthesized by Huang et al. (2008) and Kim et al. (2007), reporting the synthesis of helical silicon oxide nanostructures on silicon substrates.

SEM images of networked silicon oxide nanowires synthesized using co-catalysts of VO₂ and gold are shown in Fig. 1(c) and (d). As seen in the low magnification SEM image of these nanowires (Fig. 1(c)), application of VO₂ powder and a gold thin layer on silicon substrate together, results in the uniform synthesis of high-density networked silicon oxide nanowires (similar to morphologies synthesized using VO₂ powder) covering the substrate. SEM observations indicate, the diameter of these nanowires is between 30–70 nm and their length

reaches up to tens of micrometers. High magnification image of these networked structures (Fig. 1(d)) reveals the silicon oxide nanowires have branched structures, suggesting simultaneous nucleation and growth of these nanowires. In some cases, these branched nanowires bundle together to form rope-like structures. Previously similar silicon oxide nanostructures have been reported by other researchers using metallic catalysts such as Ga (Pan, Dai, Beach, & Lowndes, 2003), Sn (Sun et al., 2003) and Fe (Liang, Zhang, Meng, Wang, & Chu, 2000; Xu et al., 2004).

SEM images of silicon oxide nanowires synthesized using gold as catalyst are demonstrated in Fig. 1(e) and (f). Fig. 1(e) shows dense formation of nanowires on the substrate. Close-up examination at these nanowires (Fig. 1(f)) reveals that the nanostructures are composed of thick nanowires and thin nanowire-bundles, possibly due to excess of gold on the surface of the silicon substrate. Compared to well dispersed network of nanowires grown by co-catalysts, these nanowires have shorter lengths and wider diameter distribution from 20 nm to 200 nm as seen in Fig. 1(e). The synthesis of silicon oxide nanowires using gold directly from silicon substrate was previously reported by Park et al. (2007), and Kim, Shalav, and Elliman (2010) without the use of hydrogen and at higher temperatures.

Based on the SEM observations it is seen that gold and VO₂ powder each plays an important role in shaping the morphology of the nanostructures. Park et al. (2007) reported a similar conclusion, when studying Pd and Au as catalysts. According to their reports, silicon oxide nanowires could be grown at a lower temperature of around 1000 °C using the alloy catalyst as compared to nanowires grown with the use of gold only at 1150 °C. For a detailed study of the effect of VO₂ powder on the synthesis of silicon oxide nanowires, TEM observations and XPS measurements were carried out. Fig. 2(a) is a typical TEM image of the silicon oxide nanowires, showing the branching of the silicon oxide nanowires which can result in the formation of network nanostructures. Furthermore these observations reveal that no catalyst particles were found at the tips of the nanowires and the nanowires were clean and with smooth and curved morphologies. As shown in the inset of Fig. 2(a), the highly dispersed selected area electron diffraction pattern indicates the amorphous nature of these structures. This is similar to reports using other catalysts at these temperatures (Kim et al., 2007; Lee et al., 2004; Park et al., 2007).

The composition of these nanostructures was determined using XPS (Fig. 2(b)), illustrating that the nanowires are mainly composed of silicon and oxygen with an atomic ratio close to 1:2. Further study of the valence state of the Si element (shown in inset of Fig. 2(b)), indicates the Si 2p_{3/2} peak is located at 103.4 eV, which is consistent with the Si⁴⁺ state of silicon. This confirms the nanowires synthesized by thermal heating of silicon substrates with VO₂ powder are mainly SiO₂ nanowires. The carbon peak seen in the XPS spectrum could be from impurities such as carbon oxides absorbed on the silicon oxide nanowires.

No catalyst peaks can be observed in the XPS spectrum, which is consistent with the TEM and SEM observations. This is different from the result reported by Yang et al. (2006), who synthesized amorphous silica nanowires by heating the silicon with a mixture of hydrous zinc chloride and vanadium dioxide powder. In their case, vanadium particles were detected on the tip of the silicon oxide nanowires.

3.2. Effect of hydrogen

A number of groups have reported the synthesis of silicon based nanowires under H₂ atmosphere from silicon substrate with various catalysts. Lee et al. (2004) used TiN/Ni coated

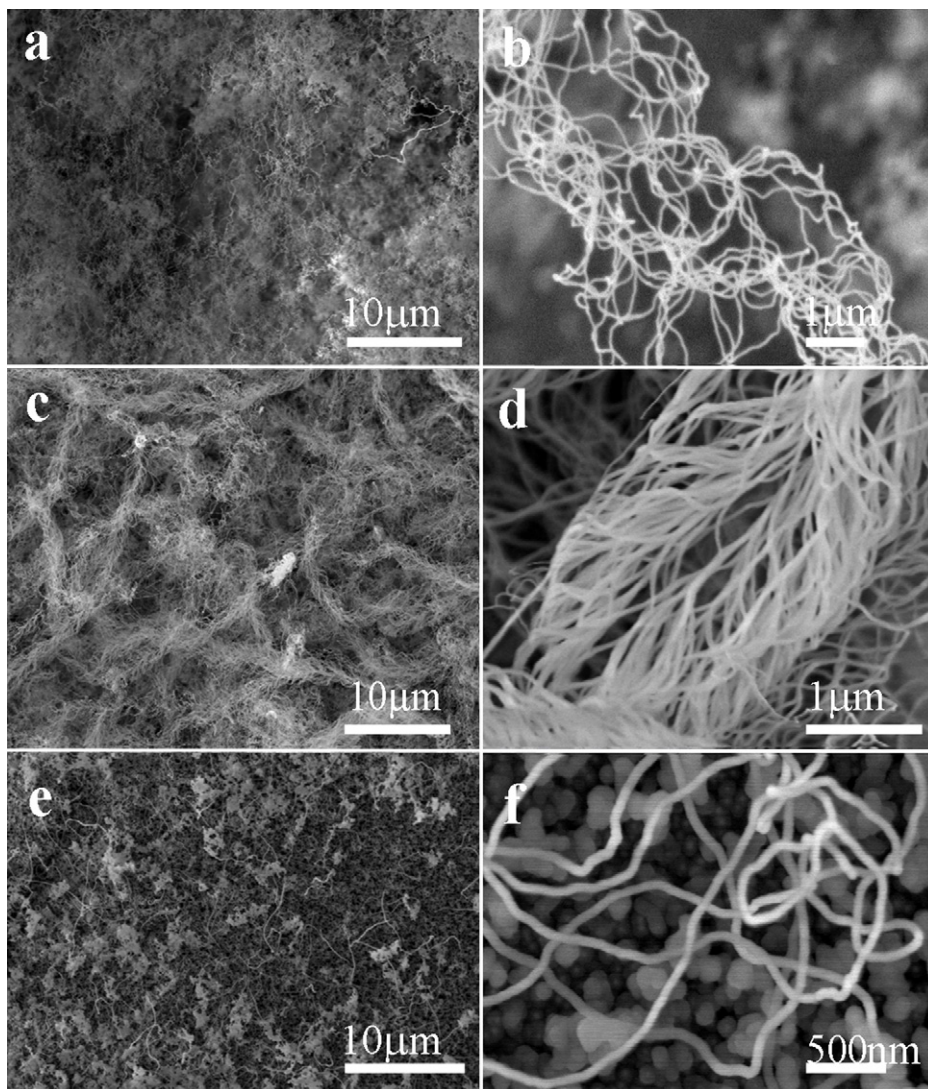


Fig. 1. Typical SEM images of silicon oxide nanostructures synthesized using (a) and (b) VO_2 powder, (c) and (d) VO_2 -Au co-catalyst, (e) and (f) Au catalyst by simple thermal annealing of silicon substrate.

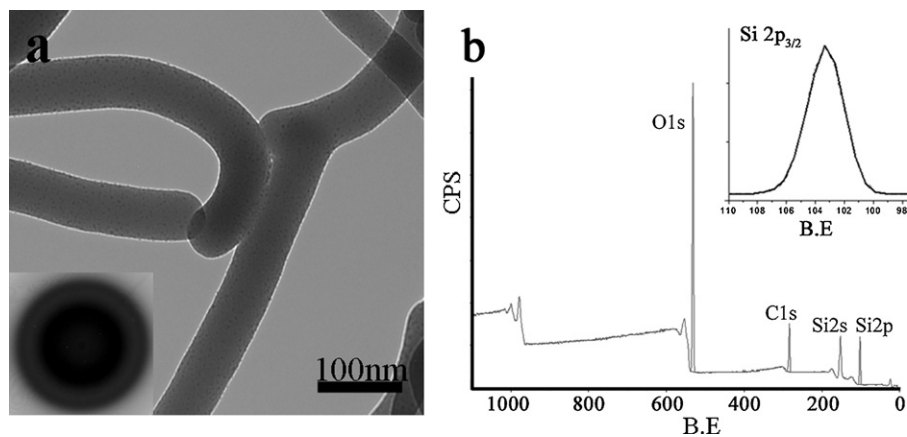


Fig. 2. (a) TEM image of silicon oxide nanowires (synthesized using VO_2 powder under H_2 and Ar flow at 1000°C) showing smooth morphology and a diameter of 50–70 nm. The SAED pattern shown in the inset reveals a characteristic diffusive ring pattern indicating that silicon oxide nanowires are completely amorphous. (b) XPS spectrum of silicon oxide nanowires with silicon to oxygen peak ratio of approximately 0.5. There are no peaks for VO_2 , suggesting that the nanowires are pure silicon oxide nanowires. The inset shows a detailed study of valence state of silicon indicating the presence of only Si^{4+} state.

silicon substrate in H₂ atmosphere and synthesized amorphous silica nanowires. Xing, Yu, Xi, and Xue (2003) reported the growth of silicon nanowires from silicon coated with gold in H₂ atmosphere, a condition similar to our experiment. Recently, Shao, Hu, Ban, Li, and Gao (2007) reported the synthesis of low oxygen content silicon nanowires from a single quartz crystal in H₂ atmosphere at relatively high temperatures.

In this study, it was found the presence of H₂ is essential for the growth of high-density nanowires at temperatures around 1000 °C. Fig. 3(a) shows an SEM image of silicon substrate heated with VO₂ powder in only Ar atmosphere at 1000 °C. Unlike previous results using H₂ (Fig. 1(a) and (b)), the substrate is covered by rod like structures with different diameters. EDX results (inset of Fig. 3(a)) on these structures reveal that these structures comprise vanadium and oxygen, the silicon peak being mainly due to the substrate itself. According to these observations, H₂ inhibits the deposition of vanadium oxide on the silicon and results in the growth of silicon oxide nanowires. In addition, controlled experiments were conducted with only Ar/H₂ gas and silicon substrate in the chamber and no products were found deposited on the substrate. It can therefore be concluded that both H₂ and VO₂ powder are crucial for the growth of high-density networked silicon oxide nanowires.

SEM image of the silicon substrate coated with gold in Ar atmosphere at 1000 °C (Fig. 3(b)) shows that very small nanowires are synthesized on the substrate and there are a few bundles of silicon oxide nanowires non-uniformly distributed on the substrate. EDX result shown in inset of Fig. 3(b) confirms that these nanostructures are composed of silicon and oxygen. Therefore, the results point out that H₂ in the atmosphere can contribute a positive effect to the density of silicon oxide nanowires synthesized from the substrate.

It should be noted that in the above two cases, a wide range of H₂ flow rate has been applied (60–180 sccm) and the results show the growth of the silicon oxide nanowires is not sensitive to the amount of hydrogen.

3.3. Effect of temperature

Temperature also plays an important role in the synthesis and growth mechanism of silicon oxide nanowires. Studies of synthesizing silicon oxide nanowires directly from the substrate in a temperatures range from 950 °C to 1400 °C were reported earlier (Davis et al., 1997; Hu et al., 2003; Shao et al., 2007). In the present study, a series of experiments were conducted to study the effect of temperature on the synthesis of silicon oxide nanowires. It was found that silicon oxide nanowires were synthesized above 950 °C using gold and VO₂–gold co-catalyst in an Ar/H₂ atmosphere. The main difference between the nanowires grown at different temperatures lies in their density and diameter. With increasing temperature, both the density and average diameter of nanowires increase dramatically. However, at temperatures higher than 1200 °C, silicon oxide wire-like structures with large diameters can be synthesized in the absence of catalysts or H₂ gas (Fig. 4). It is seen in Fig. 4(a) that the products on the substrate are a mixture of microfibers and nanowires and have a wide range of diameters from 100 nm to over 1 μm. Closer look at these structures, as shown in Fig. 4(b), indicates helical morphologies of the products, similar to the nanostructures reported by Zhang et al. (2008).

Based on these results, it can be concluded that the presence of catalyst and H₂ is necessary in generating silicon oxide nanowires at lower temperatures (~950 °C). At higher temperatures (~1200 °C), neither catalyst nor H₂ is necessary for growing silicon oxide micro- and nanowires.

3.4. Growth mechanism

Table 1 summarizes the effect of different growth parameters on the morphology of nanostructures synthesized on silicon substrate. Based on these results a growth mechanism can be proposed for the synthesis of silicon oxide nanostructures.

Different mechanisms have been proposed for the growth of silicon oxide nanowires such as VLS (Kwak, Cho, & Yang, 2007; Sekhar, Ramgir, Joshi, & Bhansali, 2008; Xiao et al., 2006), VS (Lee, Lee, Vanfleet, & Sigmund, 2003) and SLS (Park et al., 2007). In all these growth mechanisms, the most important parameter is the silicon source for the growth of nanowires. Since in the present study no external silicon source is employed, silicon substrate can be considered as the sole source for the growth of nanowires.

Based on SEM observation of silicon substrate at the initial growth stage of the synthesis procedure, etched pits were seen at random areas of the substrate when the three types of catalysts were used, shown in Fig. 5. As reported by Kim et al. (2010), these etched pits are related to the oxidation and evaporation of silicon monoxide species. Based on previous studies on the oxidation of silicon (Engstrom, Bonser, Nelson, & Engel, 1991), such oxidation occurs through the formation of a passive SiO₂ layer under high oxygen partial pressures. However, volatile SiO_(g) instead of SiO₂ is formed under low oxygen pressure. Kim et al. (2010) related the etched pits to the formation of these species. In this study the oxygen content in the chamber is decreased by the introduction of H₂ into the growth chamber. Hence the condition is favorable for the formation of SiO_(g) and etched pits.

Furthermore there have been a number of studies on the synthesis of silicon based nanowires in H₂ atmosphere using catalyst at relatively low temperatures or without the use of catalysts at higher temperatures (Shao et al., 2007; Xing et al., 2002; Yan et al., 2000). Previous reports proposed that the role of H₂ is to enhance the supersaturation of liquid droplet thus building up the driving force for the precipitation of silicon as silicon nanowires (Xing et al., 2003; Yan et al., 2000). In addition, Choi, Johnson, and Ural (2009) reported the increase in the catalyst nanoparticle size and density when H₂ is introduced during the growth of SiO₂ nanowires. They attributed this to the enhancement of diffusion of impurities in SiO₂ in the presence of H₂ on the basis of previous studies (Grove, Leistikko, & Sah, 1964). In this study, H₂ gas plays a crucial role in the formation of SiO_(g) species from silicon substrate. In addition, H₂ prevents the large scale deposition of VO_x nanostructures (as seen in Fig. 3(a)) by reducing the oxygen partial pressure and inhibiting the oxidation and evaporation of VO₂ (melting point ~1970 °C) to other oxides, such as V₂O₅ (melting point ~690 °C).

In the presence of a catalyst such as gold on the surface of the silicon substrate, silicon oxide nanowires can be grown following the VLS growth mechanism proposed in several studies (Yan et al., 2000). In this process, Si atoms in the vapor phase are dissolved into the mediating alloy liquid droplets formed at high temperature, and then SiO_x nanowires precipitate from the supersaturated alloy droplets (Kwak et al., 2007). Usually droplets of catalyst are observed on the top of the nanostructures in a VLS based growth system, although the position of catalyst, at the tip or root of the nanostructure, depends on the interaction of catalyst nanoparticles and the substrate as reported by Sohn, Ok, Seong, and Lee (2007).

In the absence of obvious catalysis of Au in the growth procedure, such as in VO₂ assisted synthesis of silicon oxide nanostructures, it is possible for the nanostructures to be deposited on silicon substrate using VO_x as the catalyst. To further confirm this assumption, SEM observations on the vanadium oxide powder in the vicinity of the silicon substrates showed the growth of silicon oxide network structures directly from the VO₂ powder, indicating the catalytic effect of these powders (Fig. 6). This

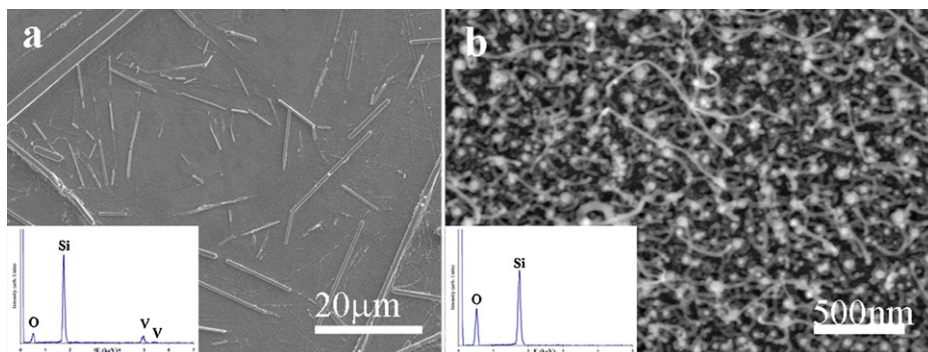


Fig. 3. SEM images showing the effect of hydrogen in the synthesis of silicon oxide nanowires. (a) Vanadium oxide structures synthesized by thermal annealing of silicon substrates in vicinity of VO₂ powder without the presence of H₂ in atmosphere. Inset shows EDX spectrum of nanowires deposited on silicon substrate. (b) SEM image of low density of silicon oxide nanowires synthesized on silicon substrate coated with gold without the use of H₂ gas. Inset shows EDX spectrum of nanostructures on the substrate.

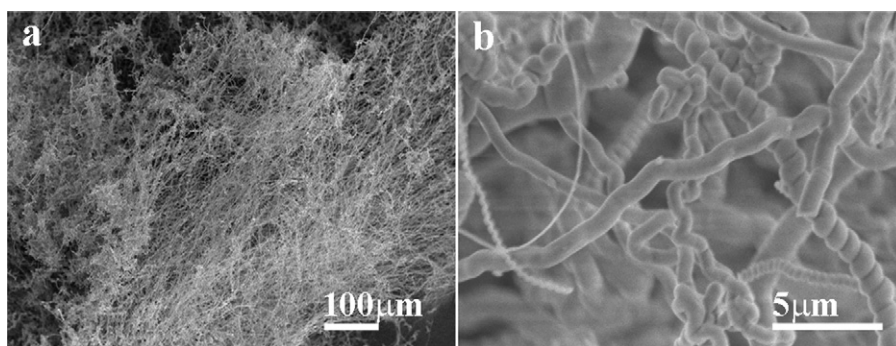


Fig. 4. SEM images of nanostructures synthesized on silicon substrate in the absence of any catalysts and H₂ at high temperatures (1200 °C).

Table 1
Effect of different parameters on the synthesis of SiO₂ nanostructures.

Temperature (°C)	Gas	Additives	Final morphology
950–1000	Ar/H ₂	VO ₂ powder	SiO ₂ networked nanowires
950–1000	Ar/H ₂	Au layer (3–5 nm)	SiO ₂ nanowires
950–1000	Ar/H ₂	VO ₂ powder/Au	High density SiO ₂ networked nanowires
950–1000	Ar	VO ₂ powder	VO _x microrods
950–1000	Ar	Au layer (3–5 nm)	Non-uniform short SiO ₂ nanowires
950–1000	Ar/H ₂	–	None
1200	Ar/H ₂	VO ₂ powder	SiO ₂ microfibers
1200	Ar/H ₂	Au layer (3–5 nm)	SiO ₂ microfibers
1200	Ar/H ₂	–	SiO ₂ microfibers

is similar to the results Yang et al. (2006) reported, in which VO₂ nanoparticle acted as the catalyst for the growth of silicon oxide nanowires. They proposed a VLS mechanism for the growth of silicon oxide nanowires which can be applied to the results in this

study. Under the current experimental conditions, small fraction of VO₂ powder can be oxidized and evaporated and reduced to VO_x depositing as nanoparticles on the silicon substrate. These nanoparticles can then act as catalyst for the growth of silicon

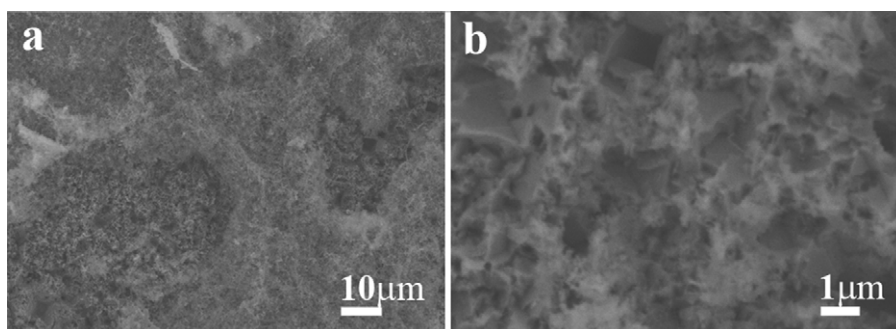


Fig. 5. SEM images of etched pits on the surface of silicon substrate formed via VO₂ powder under H₂ and Ar gas flow at 1000 °C for 2 h. These pits are formed due to evaporation of SiO_(g) species under low oxygen partial pressure using VO₂ as catalyst for growth of SiO_x nanostructures.

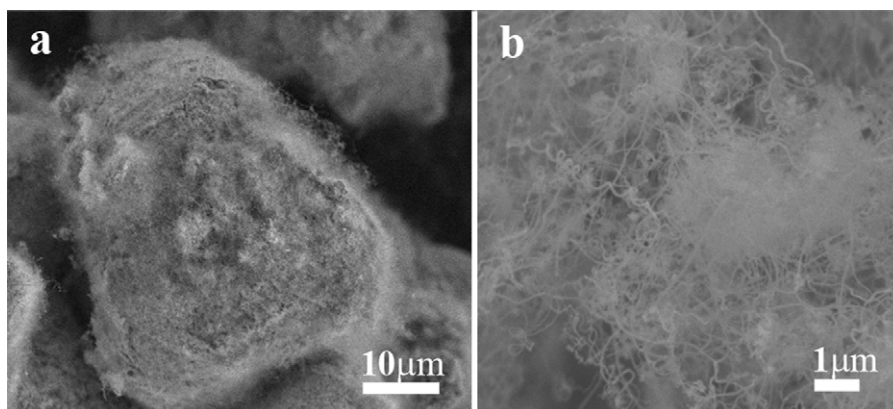


Fig. 6. SEM images of silicon oxide network nanostructures synthesized directly on VO_2 powder under H_2 and Ar gas flow at 1000°C for 5 h.

oxide network structures. However, unlike the findings of Yang et al. (2006), no catalyst particles were found at the tip of silicon oxide network structures (Fig. 2). This can be explained by the base growth VLS mechanism similar to nanostructures synthesized by Au.

This growth mechanism can be further extended to the Au– VO_2 powder experimental conditions to explain the synthesis of enhanced networked silicon oxide nanostructures. Under these conditions, it is possible for VO_x to be deposited on silicon substrate with Au semisolid droplets. Au–Si alloy can be formed as in the previously reported growth of SiO_2 following the VLS mechanism. Each Au droplet can absorb several VO_x nanoparticles on its surface, consequently increasing the possibility of multiple silicon oxide nanowires growing from a single Au droplet. In addition, as reported in many studies, gold can absorb the $\text{SiO}_{(g)}$ in the atmosphere, thus further increasing the source of silicon oxide species for silicon oxide network nanostructure growth.

At higher temperatures ($\sim 1000^\circ\text{C}$), the production of $\text{SiO}_{(g)}$ from the silicon substrate is easier, and similar to what was reported by Zhang et al. (2001), a VS growth mechanism can be proposed for the growth of silicon oxide nanostructures when no catalyst is introduced into the system. In this growth mechanism, silicon oxide nanostructures are synthesized by oxidation of the volatile silicon monoxide vapor. The difference between the diameter and density of nanostructures synthesized under the two different conditions can be related to the partial pressure of $\text{SiO}_{(g)}$ produced during synthesis.

To explain the helical structure of the network nanostructures (synthesized at low temperature) and nanowires (at high temperatures), three growth models, viz., contact angle anisotropy (CAA) (Kim et al., 2007; Zhang et al., 2008), screw dislocation driven (SCD) (Zhang et al., 2008) and polar surface driven (PSD) (Kong, Ding, Yang, & Wang, 2004; Zhang et al., 2008), have been proposed. The CAA model is based on the angle anisotropy between the catalyst nanoparticles and nanowires interface. In the present study, however, the catalyst nanoparticles (Au) were not seen at the top of the nanostructures. In the SCD model, the helical formation of nanostructures is proposed to originate from screw dislocations in the crystal lattice. Since the nanostructures synthesized here are amorphous, this model cannot be used to explain the growth mechanism. In this study, the PSD model seems to be the most logical model which is based on the presence of polar SiO_2 surfaces as reported by Zhang et al. (2008). Based on this model, the asymmetrical charge distribution on the nanowires surface can lead to the formation of amorphous SiO_2 helical nanostructures.

4. Conclusions

In summary, we have successfully synthesized networked silicon oxide nanowire with diameters of 30–100 nm by heating the silicon substrate with VO_2 powder at temperatures around 1000°C . In addition, it was found that the mixture of VO_2 and gold as co-catalysts enhances the achievement of silicon oxide nanowires with network characteristics. Microscopic characterization indicates that VLS mechanism governs the synthesis of these nanowires at lower temperatures ($<1000^\circ\text{C}$), while VS mechanism dominates at a higher temperature ($\sim 1200^\circ\text{C}$). Further, the presence of H_2 gas is important in elevating nanowire density at temperatures below 1000°C , but at higher temperatures ($\sim 1200^\circ\text{C}$), mixtures of microfibers and nanowires are synthesized without the use of any metal catalyst or H_2 .

Acknowledgments

This research was supported by Natural Sciences and Engineering Research Council of Canada (NSERC), Canadian Space Agency, NSERC Canada Research Chair (CRC) Program, Canada Foundation for Innovation (CFI), Ontario Research Fund (ORF), Ontario Early Researcher Award (ERA) and the University of Western Ontario.

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