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# Facile catalyst-free straightforward thermal evaporation of ultra-long antimony oxide microwires: Synthesis and characterization

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ABSTRACT

Antimony oxide microwires (MWs) were grown on $Si/SiO_2$ substrate by proficient non-catalytic economically promising method based on ambient heating of metallic source materials in crucible in a facile conventional muffle furnace. The produced antimony oxide microwires were 200–300 nm in diameter and tens micron in length. These ultra-long microwires were characterized by FESEM, TEM, XRD and Raman analysis. This approach is useful to develop grams quantities of microwires on $Si/SiO_2$ substrate.



## 1. Introduction

One-dimensional (1D) metal oxide semiconducting nanowires, such as $SnO_2$, $In_2O_3$ and ZnO, are key building blocks for the next generation optoelectronics and have attracted considerable attention for scientific research due to their remarkable optical, electrical, catalytic, and photovoltaic properties, which are different from those of the their bulk counterpart.

Among metal oxides antimony oxide is an important V–VI main group compound which has been widely used in industry as fire retardant, polymer fillers, flocculent in titania production catalysis for organic reactions, anode materials in lithium battery and so on [1–5]. It is anticipated that antimony oxide micro/nanowires will shows unique optoelectronic properties. However less attention has been paid to the optoelectronic properties of antimony oxide.

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Various low temperature methods have been used for the synthesis of antimony oxides micro/nano nanostructures, such as microemulsion, hydrolysis-precipitation approach, solvothermal, hydrothermal aqueous strategy electrochemical and biosynthesis methods, respectively [6–17]. Also high temperature vapor condensation and carbothermal reduction have been reported [18,19]. These methods need relatively operates at high temperature, demand expensive chemical vapor deposition (CVD), needs carrier and reactive gases for vapor transport of product on the substrate and mostly uses catalyst to initiate the growth.

In this work, we used catalyst-free facile evaporation method using simple muffle furnace to grow high-yield of antimony oxide microwires without using any catalyst or carrier gas. This method can be extended for synthesis of other metal oxide micro/nanostructures.

## 2. Experimental details

The ultra-long antimony oxide micro-wires were fabricated by novel ambient thermal evaporation method. In this experiment, small quantity of high-purity antimony metal (99.999%) was placed into an alumina crucible of 40 mm in height with increasing diameter from the bottom (cone shape). A well-defined $Si/SiO_2$ wafer was hanged horizontally in the crucible at 5 mm above the antimony metal for the deposition of product, as shown in Fig. 1. Finally, the crucible was shielded from the top with adjustable alumina cover and muffle furnace heated from 500 to 650 °C for 40–60 min.

A thin layer of whitish product was deposited under overall surface of the substrate. The surface morphology of the product analyzed by using a field emission scanning electron microscope (FESEM; JEOL JSM-6700F). Detailed structural and compositional analysis of microwires were carried out by transmission electron microscopy ((TEM) TOPCON EM-002B) equipped by energy dispersive X-ray spectroscopy (EDX). The structural phase was analyzed by the X-ray diffraction (XRD) Shimadzu XRD-6100, Cu K (0.15406 nm). Raman spectra of microwires were recorded with a LabRAM HR high-resolution Raman spectrometer (Horiba-Jobin Yvon) using a He–Ne laser ($\lambda$ = 632.8 nm) with D1 filter.

## 3. Results and discussion

The surface morphology of whitish gray product deposited on the $Si/SiO_2$ substrate is examined by field emission scanning electron microscopy. Fig. 2(a)–(d) shows low and high magnification micrographs of antimony oxide microwires. These microwires are grown on the complete surface of the substrate. The microwires are 200–300 nm in diameter and tens of micron in length. These microwires are ultra-long with high aspect-ratio. The product also unveiled high density of microwires and noticeably showed the homogeneity straightness along longitudinal axis. Fig. 3(a) shows TEM image of single

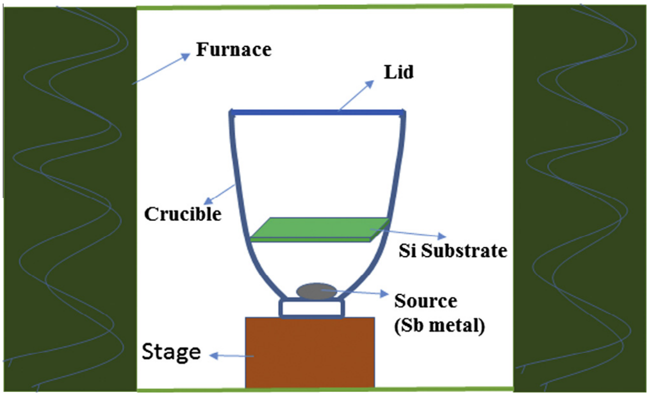


**Fig. 1.** Schematic diagram of furnace used for the growth of antimony oxide microwires.