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Growth of SiC NWs by vapor phase technique using Fe as catalyst

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ABSTRACT

In this paper we report on the growth of silicon carbide nanowires deposited on silicon substrate with vapor phase technique at atmospheric pressure, using propane and silane as precursors and hydrogen as carrier gas.

A thin layer of iron deposited on the silicon surface was used as catalyst.

The morphology, crystal structure and details such as the growth direction of the as-prepared SiC NWs were characterized by Scanning Electron Microscopy (SEM), X-ray diffraction (XRD) and Transmission Electron Microscopy (TEM).

The SiC NWs have a diameter ranging from 30 to 100 nm and length of tens of micrometers.

XRD and High resolution TEM confirm the cubic structure of the nanowires and evidence a growth habit along the ⟨111⟩ direction.

1. Introduction

Cubic silicon carbide (β-SiC or 3C–SiC) is a wide-bandgap semiconductor with high hardness, high electron mobility, high thermal conductivity and high resistance to chemical attacks.

3C–SiC nanowires (SiC-NWs) are gathering a lot of interest because the exceptional physical and chemical properties of the material can be coupled to the possibilities offered by the nanoscale design of devices, for example smaller sample volumes, decreased power consumption and improved performance [1]. Moreover, nanostructured materials show unique characteristics such as increased surface and enhanced electrical/optical properties, not traditionally observed in bulk materials. These peculiarities make them suitable for numerous applications such as nanoelectronics or chemical/biological sensing.

Functionalized β-silicon carbide nanowires have also the potential to act as highly sensitive detector elements in bio-chemical field. Furthermore, β-SiC nanowires with amorphous silicon dioxide wrapping layer are interesting for fundamental research and technological applications.

Many methods have been reported for the preparation of SiC NWs, using different metals as catalyst: chemical vapor deposition (CVD), physical vapor deposition (PVD), direct synthesis at high-temperature by reaction of different powders such as SiO, graphite [2–5] and oxides with carbothermal reduction [6], using carbon tetrachloride [7]; a more complete list of references is reported in [8].

Silicon carbide nanowires have been grown with metalorganic chemical vapor deposition with dichloromethylvynilsilane using nickel as catalyst [9] and using methilchlorosilane [10–12].

Recently, we successfully prepared SiC–SiO_2 core–shell nanowires using carbon monoxide on silicon substrates using nickel [13] and iron [14] as catalysts.

In order to explore the potentiality of this system for different nano-applications it is important to study and understand how different catalysts affects the NW growth. The metal catalysts are usually incorporated in the NW, may have different properties and could be useful or detrimental for some applications. For this reason, in this paper we report on the growth of SiC nanowires in a Vapor Phase Epitaxy (VPE) reactor, using a thin layer of iron as catalyst deposited on silicon substrate.

Biocompatibility studies show that silicon carbide is a good promising material for in vitro and in vivo bio-applications. Silicon cytotoxicity has been reported by several studies [15], while SiC is indeed a good candidate for biocompatible electronic and sensing devices: it thus may replace silicon for some specific applications.

Fe has previously been used as catalyst for the synthesis of silicon carbide nanowires, for example mixed as metal in a solid phase source [5,16], finely mixed using sol–gel methods [17], or mixed with silicon powder to get a silicon-iron alloy deposited on a graphite plate and used in a solution method [18]. Silicon monoxide together with propane [19] or methane [20] were also used to synthesize Fe-catalyzed nanowires with iron nitrate as catalyst from gas phase sources.

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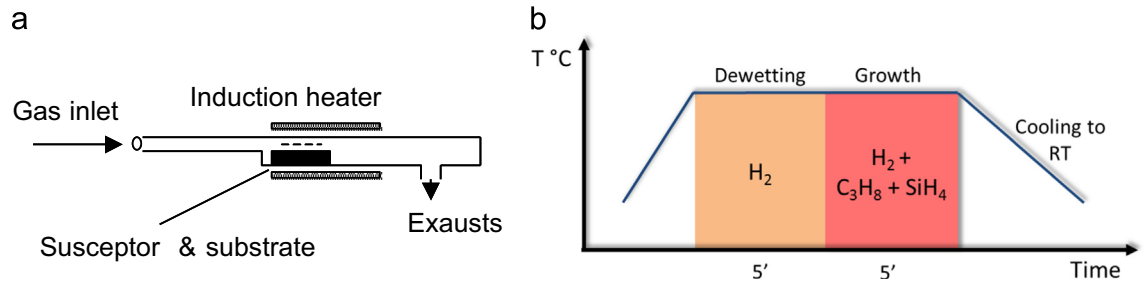

Fig. 1. (a) Apparatus set-up and (b) scheme of the procedure.

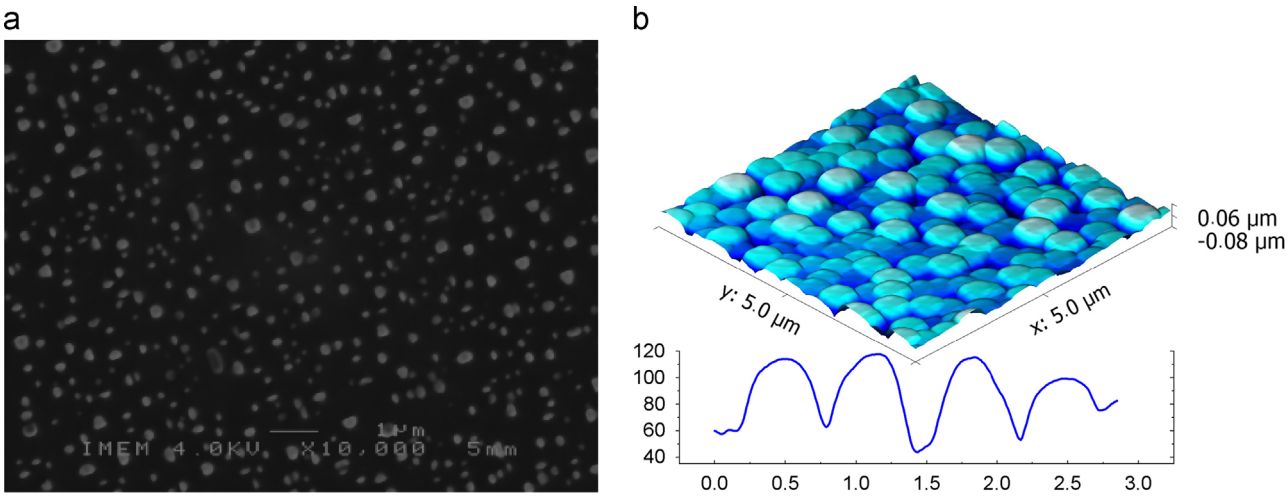

Fig. 2. Dewetting of the iron thin layer on the silicon substrate: (a) SEM image and (b) AFM morphological characterization (image in false colors). The overlapping of the islands is an artefact due to the finite size of the AFM probe. In the lower part a one-dimensional profile is shown.

Iron catalyst is used as component of metalorganic compounds too, such as iron pentacarbonyl [21] or ferrocene [22].

To the best of our knowledge, it was impossible to find in literature a description of the epitaxial growth of SiC NWs using silane and propane as precursors and metallic iron as catalyst and the correspondent morphological and structural characterization. Since one of the long term goals is to realize SiC devices to be used in nanomedicine or for in-vivo biological devices, we developed the NW growth with iron as catalyst instead of nickel, because Fe is reported to be less cytotoxic with respect to Ni [23,24].

The morphological aspects of the as grown NW were studied with Scanning Electron Microscopy (SEM), while structural properties were investigated by Transmission Electron Microscopy (TEM) and by X-ray diffraction (XRD), while Atomic Force Microscopy (AFM) was used to investigate the dewetting features.

2. Experimental

The SiC NWs samples were prepared on (100) Si substrate in a VPE system with propane and silane diluted in hydrogen (3%) as precursors and palladium purified hydrogen as carrier gas.

The experimental apparatus consists of a quartz tube with a graphite susceptor inside, on which the substrate is supported; the heating system is of the inductive type, as sketched in Fig. 1a.

The substrates preparation involved a cleaning with organic solvent in ultrasonic bath, an etching with standard RCA solution and a dip in hydrofluoric acid to remove the native oxide. After this treatments they were immediately coated with a thin (2 nm) layer of iron using a radio frequency sputtering system. Finally, the substrates were stored in a vacuum chamber before loading in the reactor. A typical growth experiment was performed at atmospheric pressure and included two steps: first, the Si substrate was heated at 1250 °C for 5 min to obtain a patterned distribution of iron (dewetting). Then, the NW were grown at the same temperature by injecting a mixture of SiH_4 and C_3H_8 with C/Si=1.6 for 5 min (Fig. 1b). After the growth the furnace was switched off and the sample cooled down at room temperature.

The morphology and crystal habit of the nanowires were further investigated by Field Emission Gun Scanning Electron Microscopy (FEG-SEM) while the Transmission Electron Microscopy (TEM) (Jeol – JEM 2200 FS) was used for detailed nanowires structure investigation using High-Resolution (HR-TEM) and High Angle Dark Field imaging in Scanning mode (HAADF–STEM) (not reported here).

The morphology of the dewetting was analyzed by Atomic Force Microscopy (AFM) in contact mode by using a Digital Instruments Nanoscope IIIa.

X-ray diffraction (XRD) characterization was used to identify the nanowires structure.

3. Results and discussion

Silane and propane mixed with hydrogen are common precursors used to grow silicon carbide layers at both atmospheric and low pressure [25,26]. In this work, a similar procedure developed for the film growth was used to prepare the nanowires. When the precursors flow over the heated substrate they decompose giving manly methane, which is favoured by the presence of