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Decoration of hierarchical Au/Pd nanostructures on 3C–SiC nanorods

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ABSTRACT

Fabrication of hierarchical nanostructures offers many advantages and can be exploited for creating superhydrophobic surfaces as well as heterojunctions and branched nanostructures, etc. In this work, catalytic growth of cubic-silicon carbide (3C–SiC) nanorods was carried out in an inductively heated horizontal cold-wall atmospheric pressure chemical vapor deposition (APCVD) reactor operated at the growth temperature i.e., 1200 °C for 1 h. Following this, hierarchical nanostructures of Au/Pd metal alloy were deposited on the surface of micron size long nanorods by DC sputtering. These hierarchical nanostructures were physically adsorbed on the surface of nanorods covering the exposed area. The average diameter of the nanorods as well as the average size of the adsorbed Au/Pd nanostructures was found to be in the range of 10–80 nm and 2–10 nm, respectively.

1. Introduction

The decoration of hierarchical nanostructures on the surface of micro/nano structures can be achieved either by physical or chemical adsorption and the multiscale hierarchical micro/nano structures thus formed can be exploited for various applications like, fabrication of superhydrophobic surfaces, as demonstrated by others [1–6]. These nanostructures can be further used for fabricating heterojunctions as well as branched structures [7–11]. For these applications, one dimensional nanostructures of silicon carbide (SiC) are a good choice of material due to their high mechanical strength and chemical inertness [12]. The growth of 1D nanostructures of SiC, particularly the cubic (3C) polytype, via chemical vapor deposition (CVD) is one of the most common methods that has been reported [13–19]. The ability to control the size of the grown nanostructures during CVD growth opens up the potential to control the band structures [20]. Further, the decoration of 3C–SiC nanowires with Pt nanoparticles [21] and polycrystalline SiC nanoparticles [22] has been used for high temperature H_2 sensing [21]. There are very few reports on the decoration of 1D SiC nanostructures. Most of the methods are costly and tedious. Hence, in this letter, a quick, inexpensive and simple method for the decoration of hierarchical Au/Pd nanostructures on the surface of CVD grown 3C–SiC nanorods via DC sputtering is reported. These hierarchical Au/Pd nanostructures cover the surface of the 3C–SiC nanorods and are physically adsorbed on it.

2. Experimental

A 100 nm nickel (Ni) catalyst film on a Si (1 1 1) substrate was used for the growth of 3C–SiC nanorods by CVD [19]. A DC sputter coater (POLARON-SC7620, UK) was used to decorate the surface of the grown nanorods by using an Au/Pd (99.99%, 60/40) sputtering target and a sputtering time of 2 min. Scanning electron microscopy (SUPRA 40 FESEM, ZEISS, Germany, operated at 5 kV) was used to see the surface morphology. To confirm the crystalline nature and cubic symmetry, X-ray diffraction analysis and micro Raman analysis were carried out by an X'pert PANalytical (PW 3040/60) system, using CuK_α radiation (λ = 1.54 Å) in the 2θ range of 20–80° and a HORIBA Jobin Yvon T64000 Raman spectrometer using an Ar–Kr laser fixed at a wavelength of 514.5 nm with 0.5 cm^{-1} spectral resolution, respectively. A transmission electron microscope (TEM) (FEI-TECNAI G2 20S-TWIN, USA, operated at 200 kV) was used to see the shape, size and crystallinity of the grown nanorods. Elemental analysis of clean and decorated SiC nanorods was carried out using an energy dispersive X-ray spectrometer (EDS), (Oxford instruments, UK) in the TEM.

3. Results and discussion

The FESEM photographs in Fig. 1(a) and (b) confirm the dense growth of nanorods all over the substrate. These nanorods are

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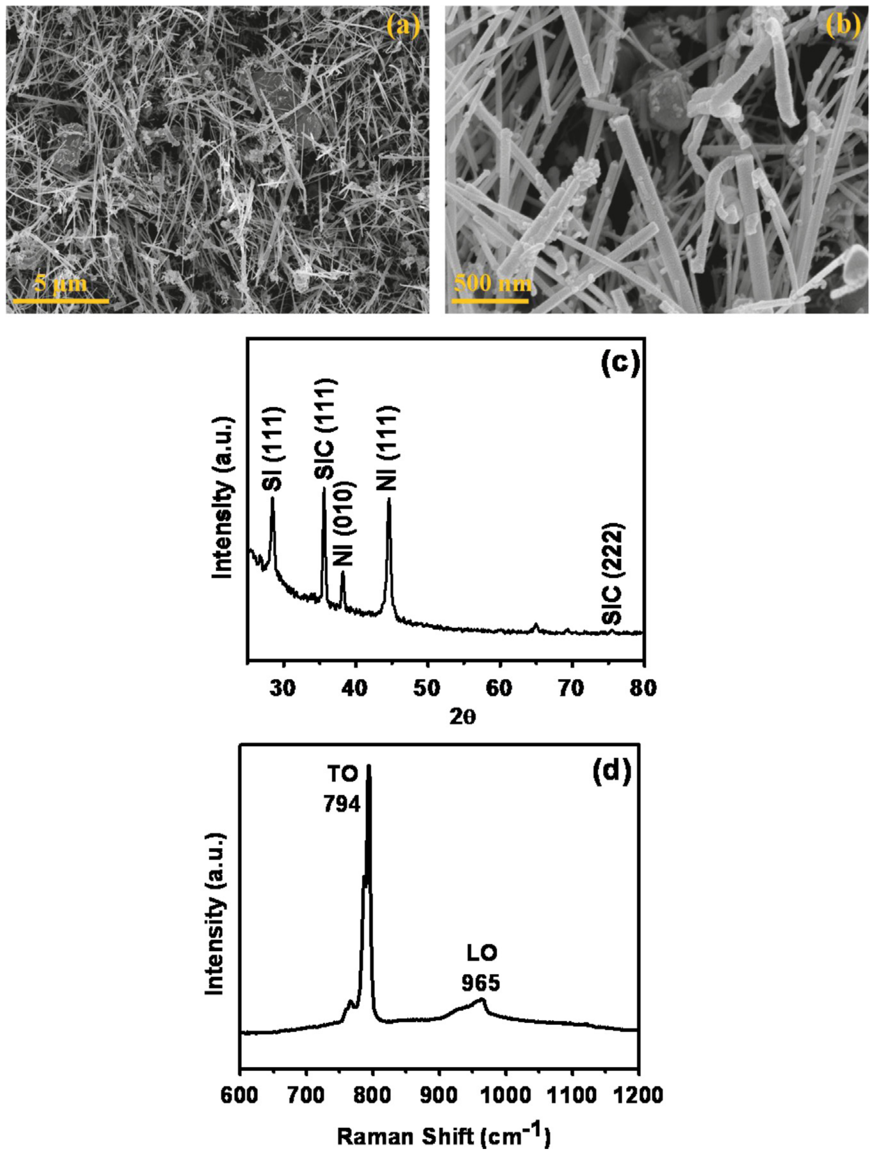

Fig. 1. (a and b) FESEM micrographs of grown 3C–SiC nanorods on Si (1 1 1) substrate taken at different magnifications, (c) XRD pattern and (d) Raman spectrum of grown 3C–SiC nanorods on Si (1 1 1).

randomly oriented and entangled. They have different diameters as shown in Fig. 1(b). The XRD pattern of as-grown nanorods is shown in Fig. 1(c). The main peak at 35.62° and a very weak intensity peak at 75.48° belong to (1 1 1) and (2 2 2) planes of 3C–SiC, respectively (JCPDS-Card No. 29-1129). Fig. 1(d) shows the Raman spectrum of the grown nanorods having transverse optical (TO) and longitudinal optical (LO) phonon modes at 794 cm^{-1} and 965 cm^{-1}, respectively. These TO and LO modes belong to 3C–SiC and are red shifted by about 2 cm^{-1} and 7 cm^{-1} as compared to modes of bulk 3C–SiC [23] and are often attributed to the presence of stacking faults in grown 3C–SiC nanorods [24].

Fig. 2(a) shows the TEM micrograph of undecorated clean 3C–SiC nanorods having diameters in the range of 10–50 nm and lengths in micrometers. The contrast bands along the length of the nanorods are due to stacking faults (SFs). Similarly, Fig. 2(b) shows the TEM micrograph of decorated 3C–SiC nanorods in an agglomerated form having diameters in the range of 10–80 nm and lengths up to several microns. Fig. 2(c) shows the high magnification TEM micrograph of a few other undecorated 3C–SiC nanorods containing low density of SFs. Similarly, Fig. 2(d) shows the TEM micrograph of decorated 3C–SiC nanorods confirming the formation of 2–10 nm hierarchical nanostructures on the surface. These decorated hierarchical nanostructures of Au/Pd are very small in size ranging between 2–10 nm and very easily deposited as compared to other materials [1–6].

Fig. 3(a) shows the TEM micrograph of an individual undecorated 3C–SiC nanorod. The EDS spectrum, taken from the region indicated by the arrow is shown in Fig. 3(d), confirms the presence of C, Si, O (due to thin native oxide layer) and Cu (due to TEM Cu grid). Similarly, Fig. 3(b and c) show two beautifully decorated 3C–SiC nanorods. The EDS spectrum, taken from the region indicated by the arrow (shown in Fig. 3b) is shown in Fig. 3(e), confirms the presence of Au and Pd along with C, Si, O and Cu. Also, Fig. 4(a) shows the selected area electron diffraction (SAED) pattern from a heavily faulted region (indicated by an arrow in Fig. 3a) of undecorated 3C–SiC nanorods showing streaks in the pattern due to presence of a large number of SFs. On the other hand, Fig. 4(b) shows the SAED pattern taken from a faulted region (middle nanorod in Fig. 2d) of a decorated nanorod showing the superimposed streaks and rings in the pattern, due to the faulted 3C–SiC