Synthesis of FeO nanorods on silicon substrate using annealing technique

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We have synthesized nanorods of FeO by annealing $FeCl_3$ on silicon substrate at 950 ^oC in presence of H_2 gas diluted in argon (Ar). Field emission electron microscopy (FESEM), Energy dispersive x-ray (EDX) and High resolution transmission electron microscopy (HRTEM) techniques have been used to characterize the nanorods. HRTEM study shows crystalline formation of nanorods. The electron diffraction pattern along viewing direction (111) and HRTEM show the interplaner distance equal to 2.17 Å which is nearly equal to the slandered value 2.3 Å of the FeO, EDX data also confirmed nanorods of FeO.

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1. Introduction

In the recent years the report of spontaneous nanwires formation in rare-earth silicides on Si (100) has attracted much attention (1–4). These nanorods on the Si substrate structures offer the possibility of electrical interconnects on a scale that cannot be attained with conventional lithographic methods. Also they can be used to display novel electronic properties that could be exploited in functional circuit elements similar to the carbon nanotube and semiconducting nanowire systems as recently demonstrated (5–7). Epitaxial silicides comprise an important materials system because they are eminently silicon compatible and can form atomically perfect structures with a natural Schottky barrier electrical isolation from the silicon substrate (8–10). Also this provides motivation to search for NW formation in other silicide systems. Also, a large interest has been generated in the fabrication of nanoscale wires (11). When wire width and height become comparable to characteristic lengths such as the electronic wavelength or magnetic domain width, new physical properties are expected that may lead to a new generation of electronic, optoelectronic, and magnetic devices (12, 13). To realize these new properties, the width and height of the wires usually need to be in the single-digit-nanometer scale or even less. Lithography methods fail to get the thin nanowire or nanorod. In these case natural self-assembly methods become the method of choice, and various techniques have been used to grow quasi-one-dimensional structures on Si substrate.

In this study, the successful preparation of FeO nanorods on Si substrates using the annealing method has been demonstrated. In addition, the structural properties of these nanorods were examined.

2. Experimental

Synthesis of FeO nanorods has been carried out with a simple annealing method. A Si (100) substrate was cleaned by hydrofluoric acid solution (5%) in order to remove the pre-existing native oxide layer. This substrate after a heavy rinsed under running deionized water was dried in a stream of N_2 gas. Then a drop of 5M solution of $FeCl_3$ in ethanol and water (1:1 by volume) was placed on cleaned Si substrate. Si substrate sampled by $FeCl_3$ solution drop was then transferred to the quartz tube (reaction chamber) inside a furnace, and Ar gas was allowed to flow at a rate of 100 ml/min into the reaction chamber immediately after loading the sample. The Ar gas was continued throughout the annealing process. Next the temperature was raised to 950 ^oC (at a heating rate of 15.83 /min) and it was maintained at 950 ^oC for 2 hours. During the increase of temperature H_2 gas was introduced into the quartz tube at a rate of 100 ml/min in order to cause the reduction of $FeCl_3$ on the Si substrate. After annealing the sample for 2 hours at 950^{o}C the furnace was cooled to the room temperature at its normal cooling rate. The nanostructures formed for above two different temperatures and gas flow rates were characterized by field emission scanning electron microscopy (FESEM), energy dispersive X-rays (EDX) installed in FESEM, high resolution transmission electron microscopy (HRTEM) installed with EDX and mapping facilities and atomic force microscopy (AFM).

3. Results and discussion

Fig. 1(a) indicates the top view of FESEM image of nanorods synthesized on Si substrate at annealing temperature 950 ^oC, and Ar = 100 ml/min and H_2 = 100 ml/min for 2 hours annealing.

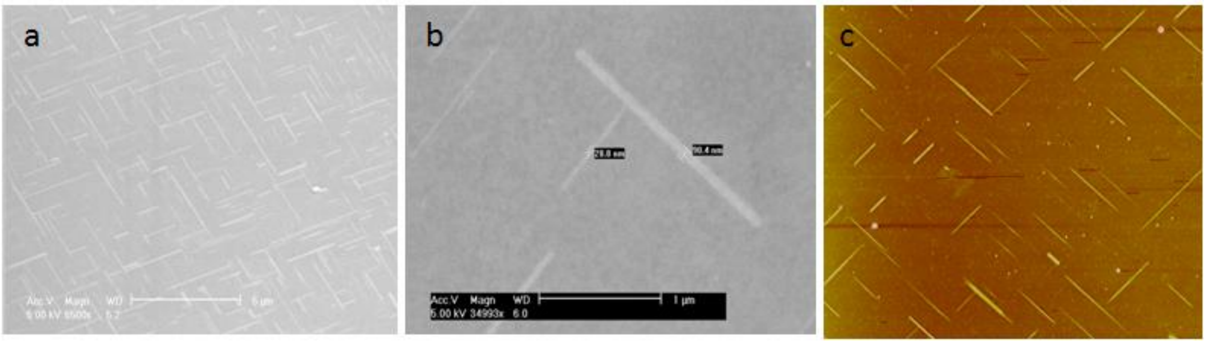

Fig. 1. (a) is SEM image of the FeO nanorods grown at Si (100), (b) is high resolution FESEM image and (c) AFM flatten image of FeO nanorods on Si surface.

In the FESEM image, a large number of the nanorods are observed on Si substrate, which varies in their diameter as measured in SEM characterization. For the thickest nanorod the diameter was measured 90.4 nm and for the thinnest nanorod it was 28.8 nm. Fig. 1(b) is high magnification FESEM image of these nanorods. From these SEM images it is clear that the nanorods on the surface of Si are arranged normally to each other. It has been observed in case of Si (100) substrate the nanorods were arranged normally to each other; while in case of Si (111) substrate the nanorods were arranged at 60^o with each other. It is believed that a particular arrangement of the nanorods on the Si substrate is in accordance to the atomic arrangement inside the Si crystal. During FESEM characterization we observed that the nanorods were having the height above the surface of the Si. Since from FESEM measurements we could not measure the height of the nanorods above the surface of Si, we further performed an AFM experiment. Using AFM (Dimension 300), the as formed nanorods over the surface of the Si were scanned. The AFM (flatten) image of the nanorods is shown in Fig. 1(c), an area of $10\times10\ \Omega m^2$ was scanned using the AFM microscopy in tapping mode. The scan rate of the tip was set at a frequency 1.052 Hz for better scanning image, and the data scale was selected at 18.46 nm to avoid the breaking of the AFM tip. The nanorods with different diameters are shown clearly in AFM image. Using the AFM software the height of the nanorods over Si surface was measured. The height of the nanorods above the surface of Si was found as the function of the thickness of the nanorods. We found that the maximum thickness nanorod has height 8.67 nm above the surface, while the thin nonarod has 0.76 nm height.

The electronic structure and the elemental composition of the nanorods over the Si surface were determined by HRTEM, EDX and mapping analysis during TEM measurements. The TEM Image for the plane section of the nanorod is shown in the Fig. 2; this figure shows that the nanorods are clearly of the cylindrical form. Also the Fig. 2 (a) is TEM bright field and Fig. 2 (b) is TEM dark field image. The elemental mapping was performed during the TEM characterization to detect the position of the Fe on Si substrate. From the Fig. 2 (d), it is clear that the oxygen exists all over the Si substrate, while Fe is found only at specific position as shown in Fig. 2 (f). These specific positions are corresponding to the nanorod positions. From this analysis the element Fe was detected at the position of nanorod only.

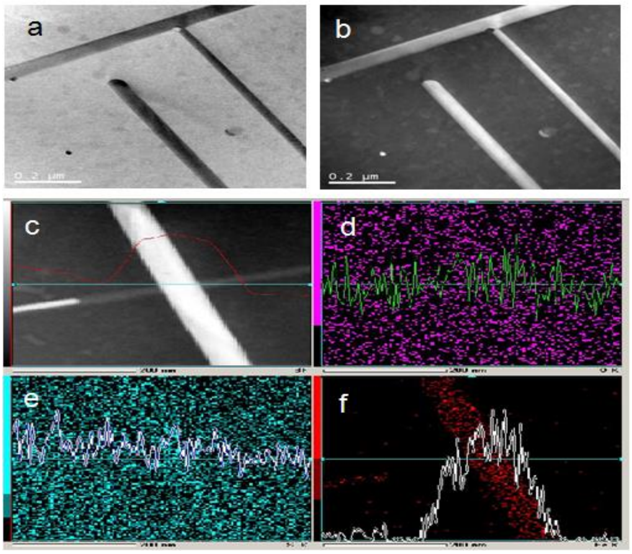

Fig. 2. (a) is transmission bright field and (b) is dark field images of plane section view of the nanorods on Si substrate. (c) Shows that oxygen is everywhere on the surface and (e) indicates the Si mapping. (f) Shows that Fe exists only at wire position.

The EDX measurements were taken at two different positions; on the nanorod and on bare substrate, respectively. The EDX at the nanorod position is shown in the fig 3. The atomic % of the Si, O and Fe were found in 18, 40 and 42, respectively, while measured over the nanorod. On the substrate the Si and O were found in ratio 1:2 forming SiO_2. Si substrate gets oxidized during the annealing of the substrate at high temperature and we found SiO_2 substrate instead of bare Si substrate. In EDX measurement the signal of Si on the nanorod position is expected to come from the Si substrate as the measurements were taken over the Si substrate.

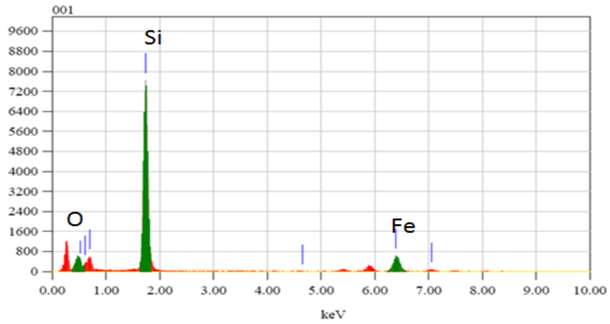

Fig. 3. EDX spectra at the nanorod position.

Further for crystallographic information the high resolution TEM was performed on the plane section TEM sample. Fig. 4 shows HRTEM image of a nanorod. The inverted fast fourier transformation is shown in top right corner of the Fig 4. This shows a hexagonal arrangement of the atoms in the crystal. Also the electron diffraction pattern is shown in the fig 4 which again shows a hexagonal pattern. The electron diffraction pattern along viewing direction (111) and high resolution TEM images shown in Fig. 4 gave the interplanar distanceequal to 2.17 Å which is quite close to the slandered value 2.3 Å (interplanar distance) of FeO along viewing direction (111). Furthermore the EDX data confirmed FeO structure formation on the Si substrate in the form of nanorod.

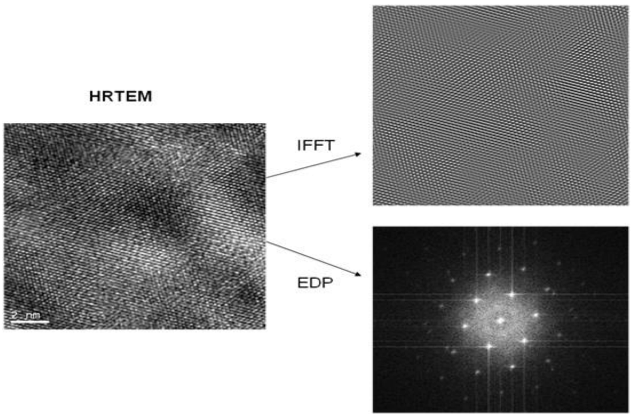

Fig. 4. HRTEM image of the nanorod with IFFT and hexagonal diffraction pattern (DP).

4. Conclusions

We have synthesized FeO nanorods on a Si substrate. Large number of the nanorods is observed on Si substrate which varies in their diameter measured in SEM for the thickest nanorods is 90.4 nm, and that of the thin nanorod 28.8nm. Also the nanorods vary in their hight above the surface of the Si. In this study we have observed that the maximum thickness wire has height 8.67 nm above the surface, while that for thin wires is measured to be 0.76nm.

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