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The Effect of Heat Treatment on Growth of TiO_2 Nanorods

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ABSTRACT: This research paper intends to study the growth of TiO_2 nanorods. In designing the experiment, the growth of nanorods has been realized by Hot Filament Chemical Vapor Deposition (HFCVD). The samples have been checked by Scanning Electron Microscope (SEM) and Raman spectroscopy, which has two peaks. The 230.32 cm^{-1}, 302.23, 317.35, and 424.83 peaks are indicative of rutile phase and 134.70 cm^{-1}, 390.71, 411.68 cm^{-1} peaks show the anatase phase. The results indicate that TiO_2 nanorods have the best growth rate on Ni catalysts at 700 ℃ for 42 minutes.

***Keywords*:** Nanomaterials, titanium dioxide, TiO_2 nanorods, HFCVD, nanorods .

INTRODUCTION

Nanotechnology is the mirror of creation, the superb future technology, and the meeting point of thought and action of all scientists and researchers of science. It is also the goal and technology, new phenomenon or the molecule to molecule renovation by changing the atom-to-atom structure of the future. The molecular nanotechnology has the capacity to rearrange the materials with atomic precision. A glance through the eyes of the physics shows that in the world of nanotechnology the conventional rules of physics can no more be useful therefore, we have to shift to the quantum rules in which quantum effects play an outstanding role. [12] Research on TiO_2 began with the discovery of the phenomenon of photo induction analysis of water on TiO_2 electrodes. [1] TiO_2 belongs to the family of transition metal oxides and it is chiefly found on earth in the form of $FeTiO_3$ element and rutile. TiO_2 is biologically and chemically neutral, corrosion resistant, nonpoisonous, and relatively cheap. [11] Titanium oxide is not normally available as pure element in nature, and it must be extracted as mineral ore. The titanium containing minerals are anatase, brookite, ilmenite, leucoxene, perovskite, rutile, and sphene. [2]

Titanium dioxide is a dimorphic allotrope with the most common forms of rutile, anatase, and brookite. It has two important photocatalytic and the superhydrophilicity properties. There are widely recognized methods and techniques for producing titanium dioxide nanomaterials namely Sol-gel method, MOCVD (Metal Organic Chemical Vapor Deposition), HWCVD (hot-wire chemical vapor deposition), HFCVD (Hot Filament Chemical Vapor Deposition), hydrothermal, and vapor-liquid-solid (VLS).[3-10]

This method has not been used for the synthesis of TiO_2 nanorods. The process includes using the technique of deposition of chemical vapors and applying hot filaments to synthesize arrays of titanium dioxide nanorods with appropriate distribution and orientation. The laboratory device HFCVD has two chambers: the synthesis chamber with relative volume of 17 lit., that is the CVD reactor, where coating takes place there, and the input chamber with relative volume of 7.5 lit.,where the sample's entry and exit takes place.

Experimental procedure

In this process the undersurface of cut silicon layers are washed for 10 minutes by ethanol and acetone and distilled water to remove the impurities and corrosion substances. Then the ultrasonic and dried up sublayers are put inside the device and by using PECVD unit and a rotary pump with the initial vacancy effect of 10^{-3} torr, the current is applied with voltage of 115 mA, 1 kv. At this stage plasma is formed and the removed ions attach to the surface of silicon layers. Within 2.5 minutes the sublayer is completely coated with nickel. When the samples are cooled down, they are taken away from the unit and then the nickel-plated sublayers are etched by HFCVD unit to make it ready for heat treatment at 700 ℃, as the following.

The silicon sublayers plated with nickel are used as catalysts for the growth of titanium dioxide nanorods. When the sublayers are placed in the unit, on the heater, the system is closed and it is ready to create vacancy by rotary and diffusion pumps. The preliminary vacancy is about 10^{-3} torr. At this vacancy level, before the growth level, gas flow is used for etching the sample. The gas flow for etching, made of hydrogen and ammoniac, is carried out in this way: hydrogen with flow rate of 48.4 sccm plus ammoniac with flow rate of 48.4 are pushed into the compartment. The pressure gauge is fixed on 5 torr. When the pressure is fixed on the expected degree, the heater temperature should be 550℃. Application of 5 V power, the current stands at 13.5 A. At this stage, H_2 and NH_3 molecules hit the heated filament and then the catalyst surface and the etching is thus achieved. This operation takes 10 min and is followed by the actual growth stage. At this stage, TTIP (titanium tetra isopropoxide) is used as the precursor and Ar as the carrier gas and extenuator. After etching is completed, Ar with a flow rate of 63.2 sccm is injected into the TTIP chamber. It comes in contact with TTIP and is heated, along with the other vapors, to 60 ℃. It is then mixed with ammoniac vapor with a flow of 63.2sccm and the mixture is injected into the reaction chamber of the Hot Filament Chemical Vapor Deposition (HFCVD) device. At this stage the temperature is 700 ℃ and the pressure 1 torr, the time for growth is 42 minutes and the current stands at 13 A. After completion of the growth stage, Ar/NH_3 and TTIP gas flows are stopped and the filament and the system as a whole is left to cool. Eventually, and after the device is cool enough and the vacuum seal broken, the sample are taken out of the chamber and transferred to analysis centers to undergo SEM, Raman , EDX .

RESULTS AND DISCUSSION

I. RBS Analysis Results

The first requirement in this method is solid state of the sample. Fig 1 shows the thickness of nickel catalysts. In the following diagram, the curves within 200-1000 keV indicate Si, curves within 1000-200 keV indicate Si sublayers with nickel-plated layers and finally peaks in the curves ranging from 1400-1600 keV indicate nickel catalyst. Since the plating time was 2.5 minutes, the thickness of the nickel catalyst is 5.56 nm.

II. SEM results for Ni catalysts with TTIP as raw material at 700 ℃

As the figure shows, the result at 42min and a pressure of 1 torr, Ar and NH_3 flow being 63.2sccm, voltage and current and temperature being set at 5v, current 13A, 700℃ respectively. Pictures in small dimensions depict TiO_2 nanorods with mag. 500 nm. Fig 2 on growth nickel catalyst shows the length of these nanorods at 9.86 µm and diameter at 8.28 µm. The results show that the growth of TiO_2 nanorods on nickel catalyst has bigger diameter. With the increase in temperature and the time of growth the length and the diameter increase accordingly.

III. Raman Spectroscopy Results

Fig 3 shows Raman spec. at a temperature of 700 ℃ and growth time of 42 min. As in the diagram, in the said temperature and time we see the peaks of 424.83, 411.68, 390.71, 334.87, 317.35, 302.23, 279.32, 230.32, 134.70 cm^{-1}. The peak of 424.83, 317.35, 302.23, 230.32 cm^{-1} shows rutile phase, the peak of 411.68, 390.71, 134.70 cm^{-1} shows anatase phase, and the peak of 279.70 cm^{-1} shows Si.

IV. EDX Results

Fig 4 shows the results of EDX analysis.In this figure, too, we see signals of C،O، titanium(Ti) and Ni. Weight percentages of C and O are18.21 and 78.76 respectively. Weight percentage of the Ti on Ni catalyst is 1.18 and 1.85 respectively.The results show that the weight percentage of Ti in a bed of TiO_2 nanorods for growth on nickel catalyst shows more eight in comparison with other catalysts.

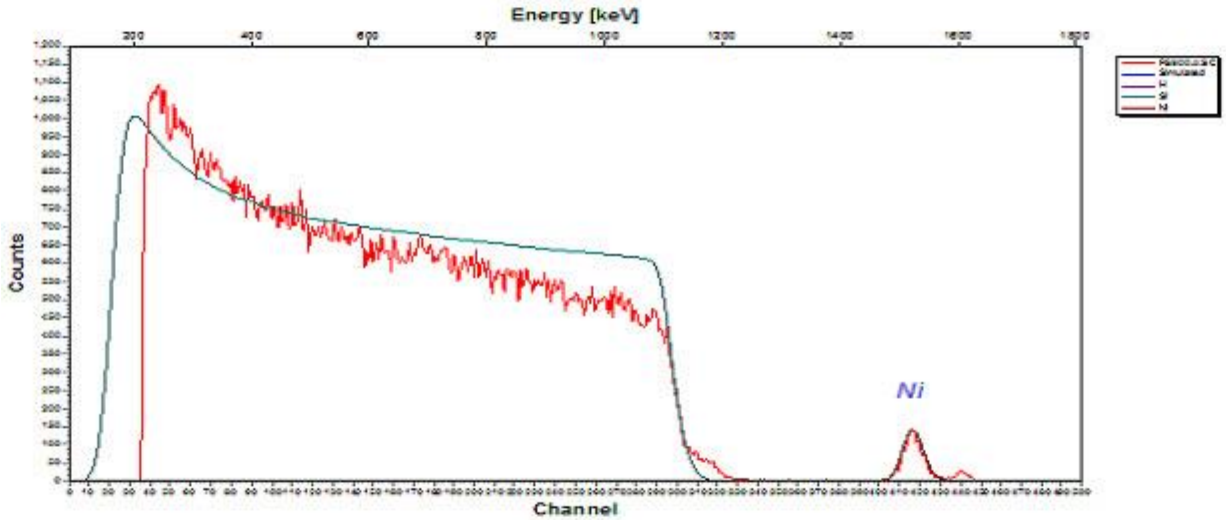

Figure 1. Diagram of Catalyst Thickness (RBS) Ni at 2.5 min

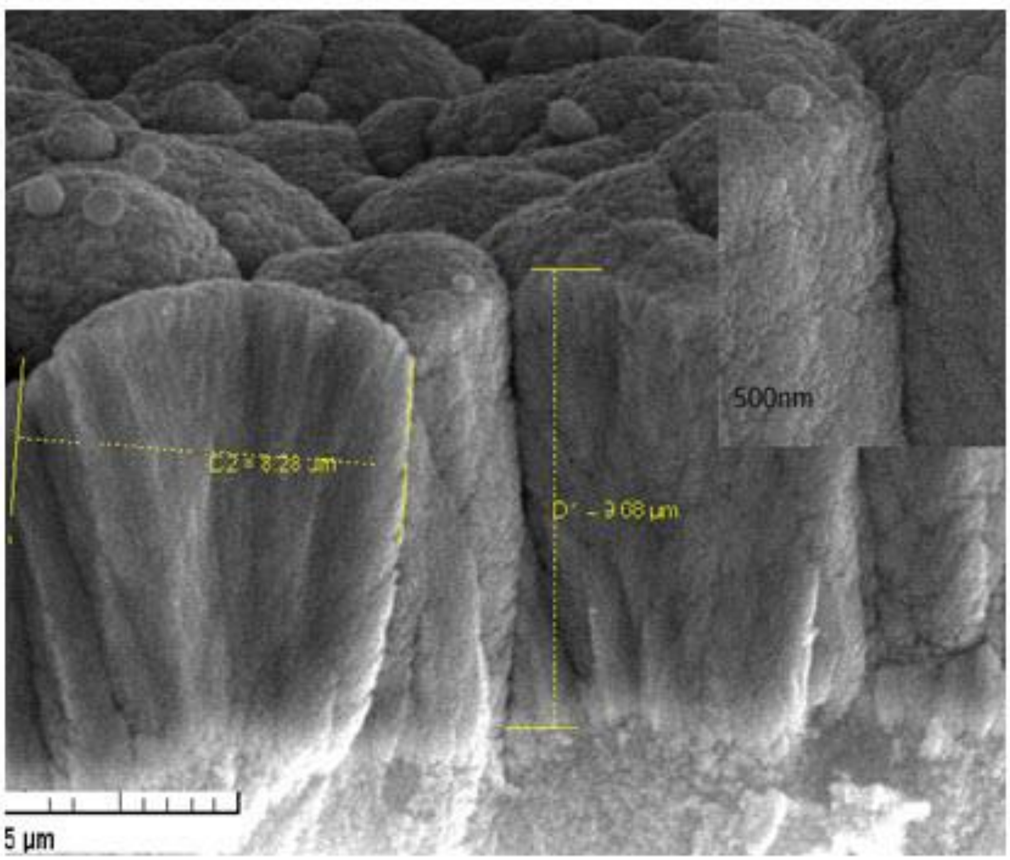

Figure 2. SEM images at temperature of 700 degrees cent

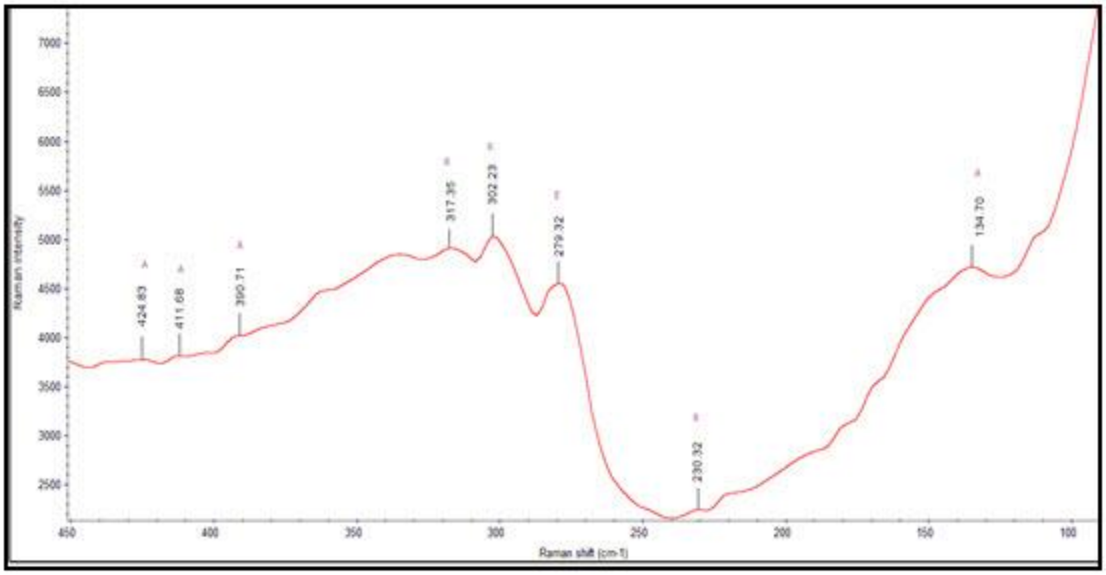

Figure 3. Raman Spec. of Nanorods at a Temperature of 700 Degrees

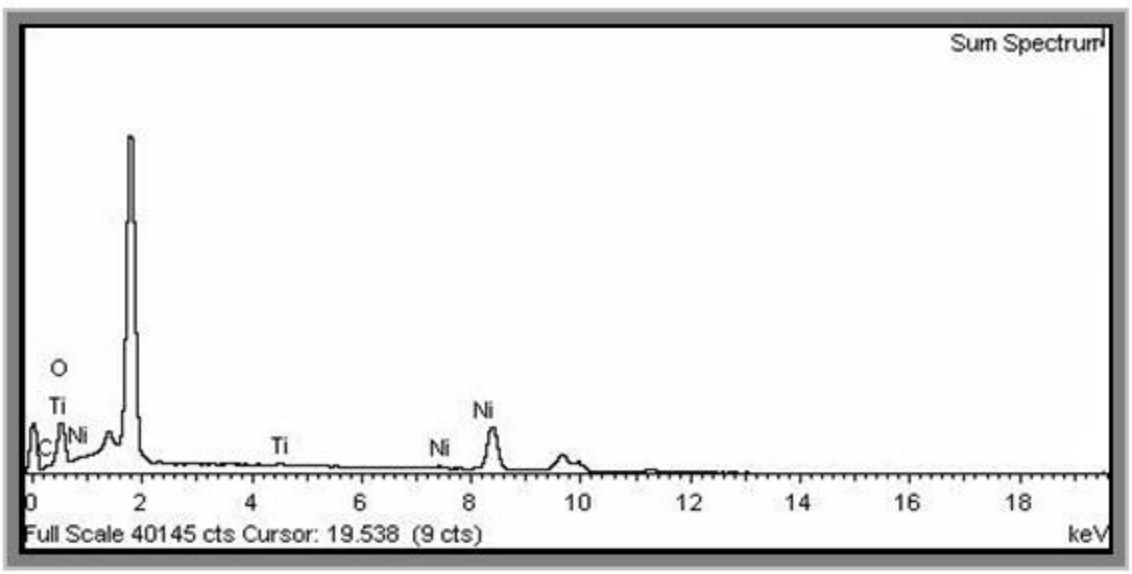

Figure 4. EDX at a temperature of 700 degrees and growth time of 42 minutes

CONCLUSION

While studying the growth rate of TiO_2 nanorods with in combination with organic metals containing titanium (TTIP) that is used as raw material for growth, as well as argon and ammoniac gases, and by applying HFCVD at a temperature of 700 ℃ for 42 minutes, we came to know that the length and diameter of the TiO_2 nanorods will be 13.34μm and 5.37 μm respectively. Using Raman spectroscopy we observed rutile and anatase peaks at this temperature.

Given the above results, the diameter on nickel catalyst increases with the increase in temperature and the growth time, so that penetration of titanium on the surface of catalyst is increased. Nickel catalyst is the best bed for the growth of TiO_2 nanorods, which are used in prevention, diagnosis and treatment of cancers, purification of aid and water, as well as in solar cells, sensors and other devices of medical use. They are also used in semi-conductive industries and vehicle manufacturing industry. Time and temperature have determining effect on the growth rate and the type of structure of TiO_2 nanorods.

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