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STUDY EFFECT THE TEMPERATURE OF PREPARATION AND ANNEALING ON TiO_2 CHARACTERIZATIONS

***Abstract**: The characterizations of TiO_2 nanoparticles which prepared on FTO conductive glass bases using the hydrothermal method with different temperatures (140,160,180) for 4 h, were studied after and before annealing. The XRD patterns were appeared that the samples preparation an increase in the intensity of the peaks and a decrease in the width of the FWHM peaks was observed due to the increase in temperature as well as an increase in the grain size as the distance between the grains decreased. UV-VIS tests demonstrated optical properties, including absorption and transmission. The increase in temperature effect at the absorption edge. It was shifted towards higher energies, that is, in its preferred direction. FESEM images were showed change an increase in the density of the substance upon increasing the temperature.*

***Key words**: TiO_2, Hydrothermal, Nanorod, Crystallographic properties.*
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Introduction

TiO_2 is one of the most common titanium compounds and is often used in many applications[1]. There are many nanostructures of titanium dioxide, including spheres, nanoparticles, and nanotubes[2]. TiO_2 exists in three different crystal stages: the rutile phase, the anatase phase, and the orthotic phase[3]. As in Fig. (1.1) both rutile and ananase have a (tetrahedral crystalline) structure, but they belong to different stages[4-6]

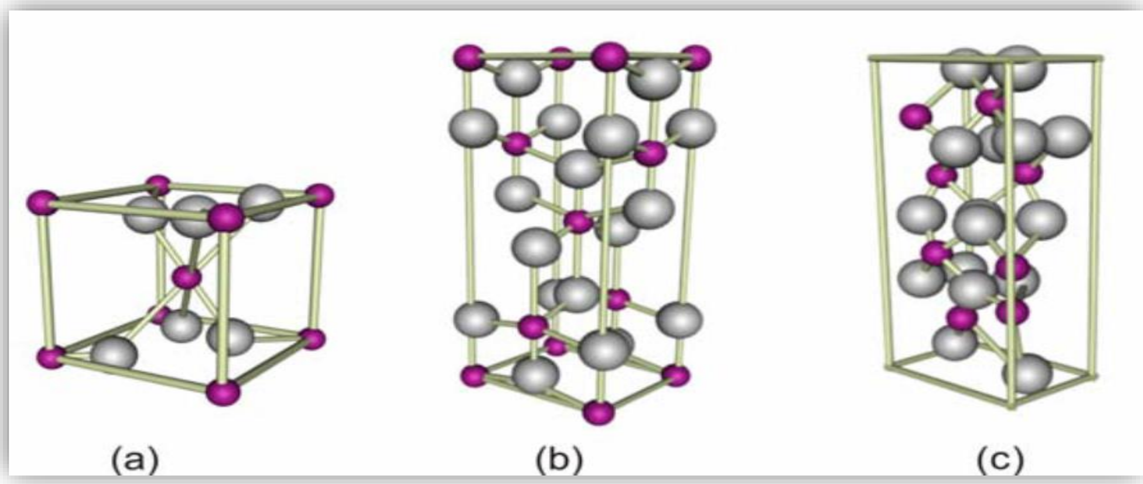

Fig. (1.1): unite (a)rutile (b)anatase (c)brookite .Grey and red spheres represent oxygen and titanium[5]

Hydrothermal Method: The Hydrothermal method was used for the first time by the British geological scientist Sir Rudrell [7]. This method is considered suitable for the environment because the reaction takes place in a closed system conditions, and it was defined as (any chemical reaction that is not homogeneous, either aqueous or aqueous, above room temperature at a pressure higher than the atmospheric pressure, and it takes this The method takes several hours to complete with the help of the microwave to collect a variety of oxides and hybrid materials.

EXPERIMENTAL

1.Preparation of Titanium tetraizobutoxide (TTB)

Two portion of titanium(IV) chloride (BDH company) ($TiCl4$) was reacted with eight parts of butanol. Titanium (IV) chloride was put into round three neck flask. Butanol was add drop by drop with applicate of the magnetic stirrer without a heating until the end of butanol and disappeared of the fog. The oiled yellow solution was obtained

2. Preparation of TiO_2 by hydrothermal method:

In a glass container, the solution consisting of (12.5) ml of distilled water is mixed with (12.5) ml of hydrochloric acid at a concentration of (35.4%) and left for 15 minutes, then placed (0.3) ml of TTB and left for 25 minutes. Mixing is done with a magnetic stirrer in Teflon, the FTO is placed at a certain angle, and then the solution is added to cover the sample, leaving a distance from the top where this portion is conductive for the measurement. Then the autoclave container is tightly closed and placed in the oven at temperatures of (140.160.180) ° C for four hours , then the hydrothermal reactor is cooled down to room temperature. Then the samples were washed with distilled water and left to dry. Then some of the samples were annealed at a temperature of 400 ° C for half an hour. These samples classified as S1,S2,S3,S4,S5 and s6 as the table 1.1

Table 1.1 Classification of samples Preparation

Preparation Temperature	Before Annealing	After Annealing
140 °C	S1	S2
160 °C	S3	S4
180 °C	S5	S6

RESULTS AND DISCUSSION
1.XRD Analysis

XRD spectroscopy was used to determine the crystal size and crystal structure of titanium dioxide prepared by the hydrothermal method that is deposited on FTO conductive glass slides. The main peaks of the glass slides FTO (110) and (200) at 2θ=(27.5) and 2θ=(37.2) This corresponds to[102]

Fig. (1.2) shows the pattern of a TiO_2 preparation samples S1,S2,S3,S4,S5 and S6. These were indicated that the crystal structure of the these films were the anatase phase which has a clear and high crystallization nature. Diffraction peaks denote (101), (004), (200), (213) (204) anatase phase (quadrangular) at 2θ = (26.56), (37.91), (51.57), (61.64) and (65.61). These results are in accordance with the card profile (JCPDS-04-0477).

Unite cell lattice constants (a=b=3.541 Å) and (c=9.4836 Å) this value are in very good approximation to standard value (a=b=3.7484 Å) and (c=9.5124 Å) and this corresponds to[8,9]. By demonstrating numbers S1, S3, and S5, we refer to new peaks that increase the temperature of the composition from 140 ° C to 160 ° C and 180 ° C. Also, when comparing these samples with annealed samples S2, S4 and S6, we observe the effect of annealing on them as it will affect the change of surface shape upon annealing with a temperature of 400 ° C. The atoms will have more activation energy for diffusion, thus it will allow the lower energy atoms to move to the appropriate location in the crystal structure and thus the crystals will grow in their preferred direction and then the crystal fusion process will start to form larger granules and within the grain boundaries, the oxygen defects will decrease. An increase in temperature leads to an increase in the size of the profile and an increase in the size of the granules, and thus an increase in roughness, an increase in the intensity and a decrease in the width of FWHM also shows us an increase in the overall stress and strain due to the annealing temperature [10-12]

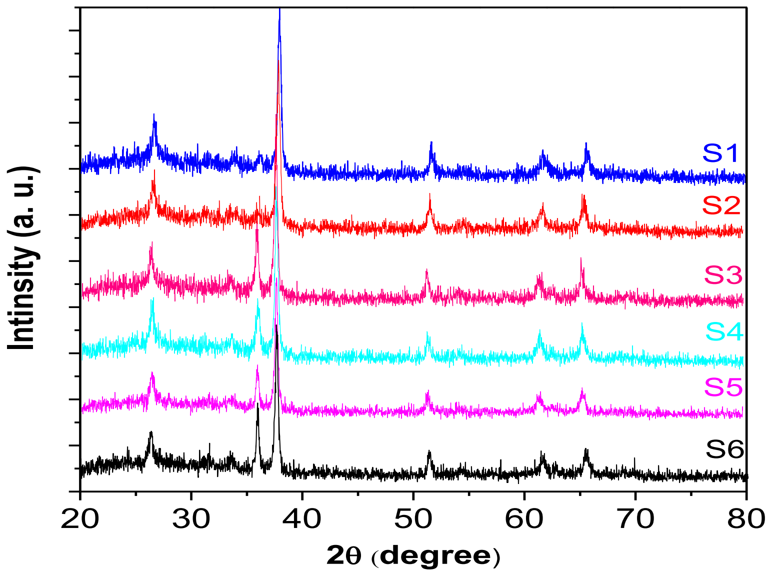

Fig. (1.2)XRD patterns for TiO2 for S1, S2 ,S3,S4,S5 and S6 samples

Table (1.2). Data on thin TiO_2 films at different temperatures with annealing and without annealing

T(K)	Sample	D (nm)	$\delta \times 10^{-3}$ (nm-2)	$\varepsilon \times 10^{-3}$
140	S1	25.22	1.57	1.37
	S2	24.2	1.7	1.43
160	S3	27.97	1.27	1.23

	S4	24.33	1.68	1.42
180	S5	28.26	1.25	1.22
	S6	27.25	1.34	1.27

The crystal size is calculated by Scherer's equation and it is found that the average crystal size is in the range (24.2-27.25 nm) as shown in Table (1.2) as well as stress and strain were calculated, as shown in Table (1.2).

2.Optical Properties

The optical properties of synthesis samples were studied by UV-VIS spectroscope. These properties include the absorbance and reflectivity measurements. Depending on reflectivity, energy band gaps were calculated of the synthesis samples.

The Absorption Measurement .a

Figure (1.3) shows the absorption spectrum of TiO_2 thin films prepared on FTO glass substrates using a different temperature hydrothermal method, with annealing and without annealing.As the absorption spectrum of S1 and S2. The absorption edge shifted from 390 nm to 400 nm. Also it has been shifted towards the red direction due to annealing and this corresponds to [15].

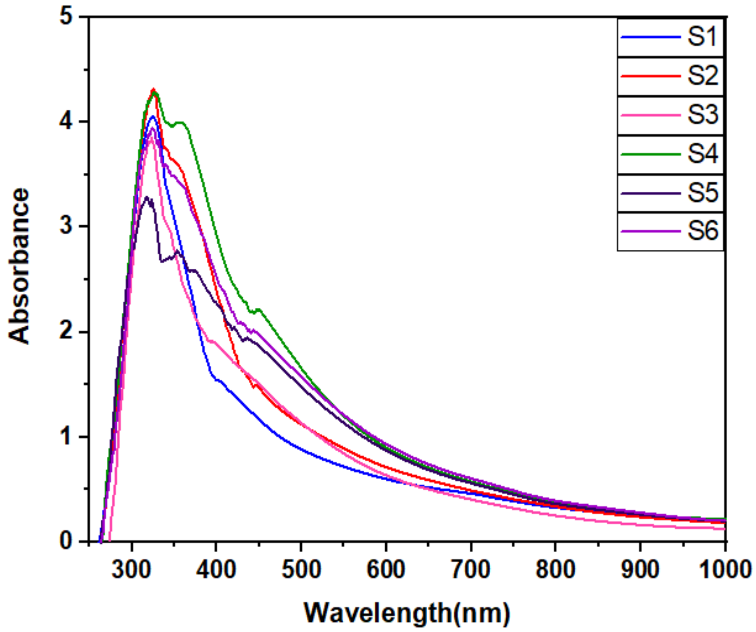

Fig. (1.3) represents the absorbance spectrumfor TiO2 for S1, S2 ,S3,S4,S5 and S6 samples

The same situation is clearly shown in the S3, S4, S5 and S6 samples shown in Fig. (1.3) where the absorption edges are slightly moved across the long wavelength (370 nm-450 nm). This is a consequence of the increase in the temperature of preparation and annealing[13,14].

c. Transmittance (T) Measurement

Optical transmittance spectra of a thin film TiO_2 prepared by the hydrothermal method deposited on FTO glass substrates at different temperatures. As a function of wavelength ranging from 390-1000 as shown in the figure (1.4), it has been observed that the permeability decreases with annealing possibly due to the decrease in the energy gap with annealing.as it showed the highest permeability at 77% and decreased to 62%. This is the reason for the effect of temperature on the surface roughness, increased grain size, and increased dislocation defects. This increases the absorption and reflection of the film

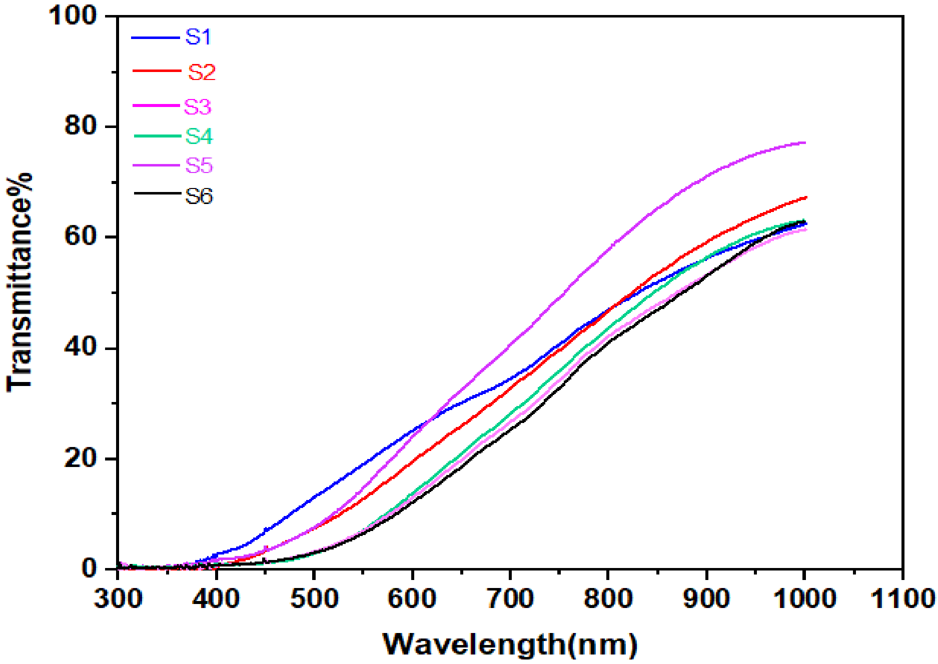

Fig. (1.4) Transmittance spectra for TiO_2 for S1, S2 ,S3,S4,S5 and S6 samples

3. FESEM Measurement

Figure (1.5) explain the FESEM images for TiO_2 samples. There was a difference between the samples in terms of density nanorod and this is due to the increase in temperature in addition to the annealing temperature and the effect of the temperature on the increase in the size of the grains and thus the surface increase significantly

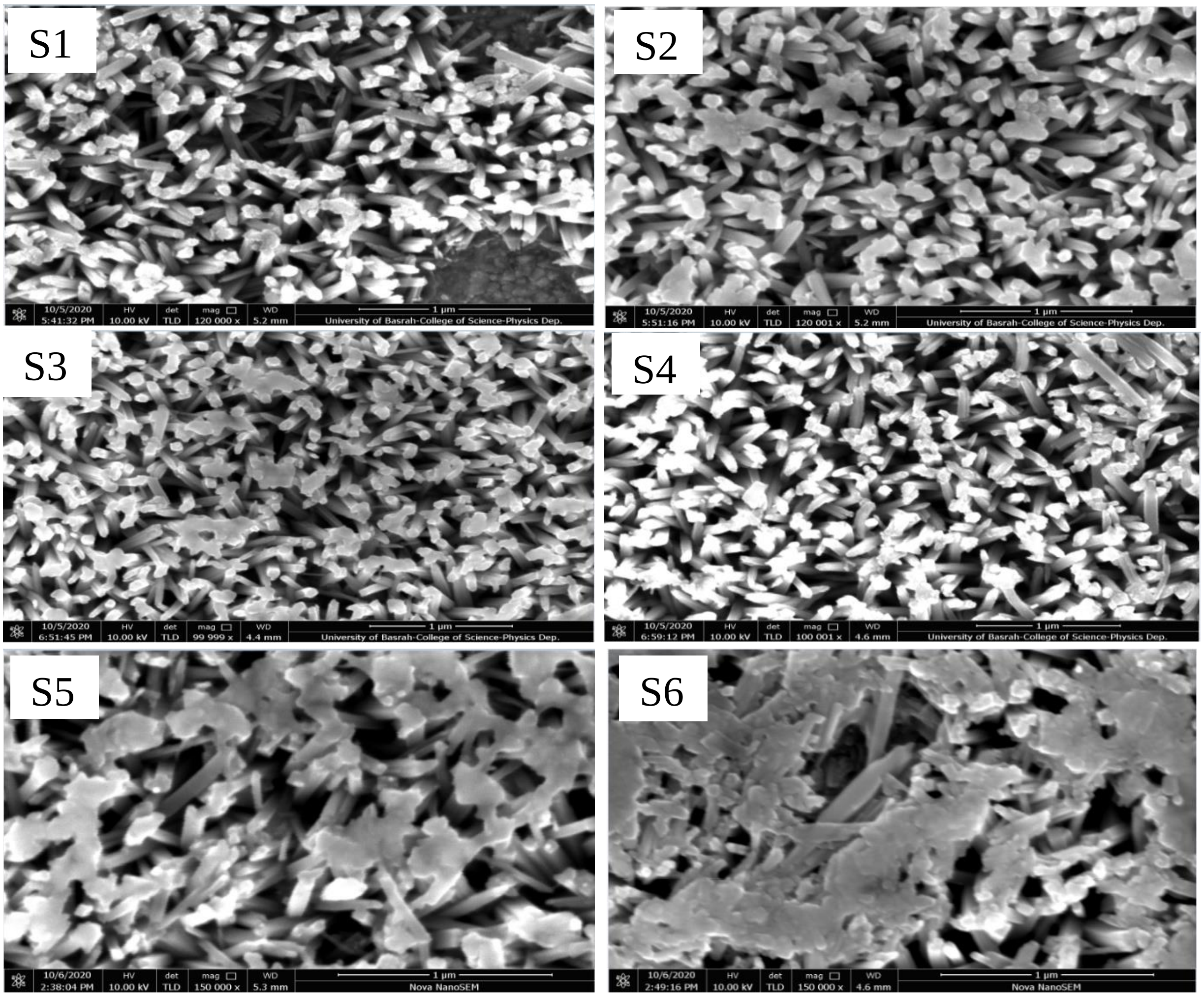

Fig. (1.5) FESEM to the TiO_2 samples

Conclusion

The characterizations of TiO_2 nanorod were changing according to change of temperature of preparation and annealing. From the XRD diffraction pattern it can be concluded that the crystal size decreases, the distance between the crystals decreases. While the width of FWMH decreases duo to these reason. The absorption edge has been shifted towards higher energies, due to the effect of temperature as it points towards the red region. FESEM examinations showed an increase in the diffusion density of nanorod result of increasing the preparation temperature from 140° C , 160° C to 180° C .

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