



## Some of Optical Properties to Titanium Dioxide thin Films at different Calcined Temperatures

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### Abstract

The thin films of titanium dioxide were prepared by sol-gel technique and were deposited by using spin coating method. Some of the optical properties were diagnosed at different annealing temperatures. Films prepared have been calcined at various temperatures degree for 1 hours or less for each film. The changes of optical properties with the effect of temperatures like optical absorption, optical transmittance and optical reflectivity have been diagnosed and characterized by ultra violet-visible spectrophotometer device. By the way the optical refractive index and optical energy gap were calculated from the relationship mathematical equations that will be mentioned in the results and discussion. It is worth noting that the curves were modified by using polynomial correction. Optical absorptions have been increased with annealing temperatures, optical reflectivity and optical refractive index with increasing of annealing temperature at the wavelengths confined between ~ 600nm to ~800nm, while the optical transmittance showed an opposite results of the optical absorbance and optical energy gaps decreasing with increasing of calcined temperatures.

**Keywords:** *Sol-gel, Calcined temperatures and optical transmittance.*

### Introduction

"Thin film" technology is one of the most important technologies that contributed to the development of the semiconductor study and gave a clear idea of many physical properties [1]. The term thin films is usually called a layer or several layers of certain atoms whose thickness may not exceed one micron to many hundreds Nano produced by condensing material (atoms or molecules ) which have unique important properties that differ from whether they are a thick particle such as physical and engineering properties [2].

Thin film is used in the manufacture of electronic devices in the form of resistors, capacitors, transistors and others [3]. It is the basis for the manufacture of solar and photovoltaic cells [4]. It is also used in the manufacture of electro-optical reagents within specific spectral ranges and has many applications [5]. Thin film deposition techniques are divided into two types. Physical techniques and chemical techniques. Physical techniques includes physical vapor

depositions (P.V. D) [6], electron beam vapor deposition (E. B. P. V. D) [7], sputtering technique [8], pulsed laser deposition (P. L. D) [9], Cathodic arc deposition (Arc-P. V. D) [10] and electro hydro dynamics (E. H. D) [11]. There are many chemical techniques to deposit various thin films include, Langmuir-Blodgett method [12], spin coating technique [13], dip-coating technique [14], chemical vapor deposition technique (C. V. D) [15], plasma-enhanced chemical vapor deposition (P. E. C. V. D) [16], atomic layer deposition (ALD) [17] and sol-gel technique [18].

Let's talk about some details about Sol Gel as the technology used to prepare the important solution, Sol-gel technology is a technique that was discovered in old times, but its work began in the 1960s [19]. The increasing uses of this technique because of the advantages are not found in traditional methods of synthesis. This technique is known as a method for the formation of inorganic oxides with gel structures, which are converted to

solid glass (amorphous) at low temperatures and under atmospheric pressure. Sol-gel can be defined according to thermodynamic principles as a relatively stable solid phase formation at a certain temperature, Liquid (solution). One of the advanced ceramic materials production techniques. Sol is a colloidal solution, while gel is a gelatinous compound. In this process the solution is converted from metal compounds or suspended from very fine particles into a liquid (which is called coarse) to a very sticky mass (so-called gel).

There are two different processes for the interaction of the gel-gel, depending on whether a transformer is used in the process or colloid. In the case of salts, the resulting gel material will consist of identifiable colloid particles that have been linked to each other across surface forces to form a network. In the case of a normal solution, the solution produced by organic compounds is a typical solution (eg, metal alkoxides), the resulting gel will in most cases consist of a network of polymeric chains formed by the hydrolysis reactions (hydrolysis) and condensation.

As a method of manufacturing ceramic materials, the sol-gel process has a number of advantages. Due to the easy purification of liquids (being the initial starting material of the process), it is possible to produce materials with high purity. Materials with exceptionally good chemical homogeneity can also be produced, which are highly desirable, especially in the case of complex oxides, because the mixing of components takes place at the molecular level during chemical reactions. Low condensation temperatures are another good feature. We must not forget the existence of real disadvantages. Initial starting materials (eg, metal alkoxides) are somewhat expensive. We mentioned earlier difficulties in the usual drying phase during which cracks, sprains and significant

shrinkage are general problems. Because of these specific problems during the drying phase, the sol-gel method is used slightly in the manufacture of mono-ceramic materials. But has seen considerable use in the manufacture of small or fine products such as thin films, fibers and powders. Its use in these areas is expected to grow steadily in the future [20]. Titanium dioxides have found many, wide and important applications in several fields of materials engineering including industrial photo voltaic and dye sensitized solar cell, catalysis or photo catalysis, in electronic based devices and I.C [21].

These applications realized with micron or many Nano structured titania in form powders or films [22]. Therefore, this research has been interested in supporting the knowledge process and providing it with concepts about expanding the possible applications of this useful material, as well as the thousands of researchers who put their interest in scientific research.

### Experimental Steps

Chemically, the basic solution was prepared completely by solving 6 ml of titanium isopropoxide  $C_{12}H_{28}O_4Ti$  (TIP) adding as drops to ~40 ml ethanol  $C_2H_5OH$  then add 10 ml of concentrated acetic acid  $CH_3COOH$ . All of the solutions mentioned were supplied from Sigma -Aldrich Company with non-trivial purity arrives to 99% to some of them. Mixed solution has kept in perfect sealed dark flask and stirred for ~ 15 minutes. Next step was very important step which is included cleaning glass substrate; the substrates have been washed well by distilled water and then acetone. Thin films have been prepared by dropping chemical solution on glass substrate by spin coating technique at 3000 rpm at room temperature. The films have been annealed at temperatures (~360, ~460, ~550 and ~700) °C for (60) minutes.

**Table 1: Films codes, thicknesses and calcined temperatures at rotate speed 3 krpm**

Film code	T(°C)	(d) Thickness (nm)
.T1	~360	~ 24.7
.T2	~460	~ 23.35
.T3	~550	~ 25.70
.T4	~700	~ 24.50

## Results and Discussions

### The Optical Absorption

Fig.(1) refers to optical absorbance of prepared thin films at different annealing

temperatures and thickness, there are irregular behaviors of absorption spectra with the calcined temperature, there were founded some random crossing curves at short wavelengths. At intervals between (>

800to ~900) nm, at this point can say this is because of the presence of surface voids and some cracks on the films (K. K. Saini et al. 2007) or because of the thinness of films prepared. But through concentrated observation we founded that in the visible wavelengths between (600-800) nm that absorbance rise with increasing the calcined temperature at ~460, ~550 and ~700) oC this is may be because of attributed to the

growing crystalline phases of titanium dioxide and grains sizes as well as, this results agree with researchers results (Dongsun Yoo et al.; 2006).First figure and Figure (2) below. The highest optical absorption has been recorded at calcined temperature of ~360 o C at wavelength ~600 nm. See table no (2) and Figure (2) refer to the values of absorption at ~700 nm.

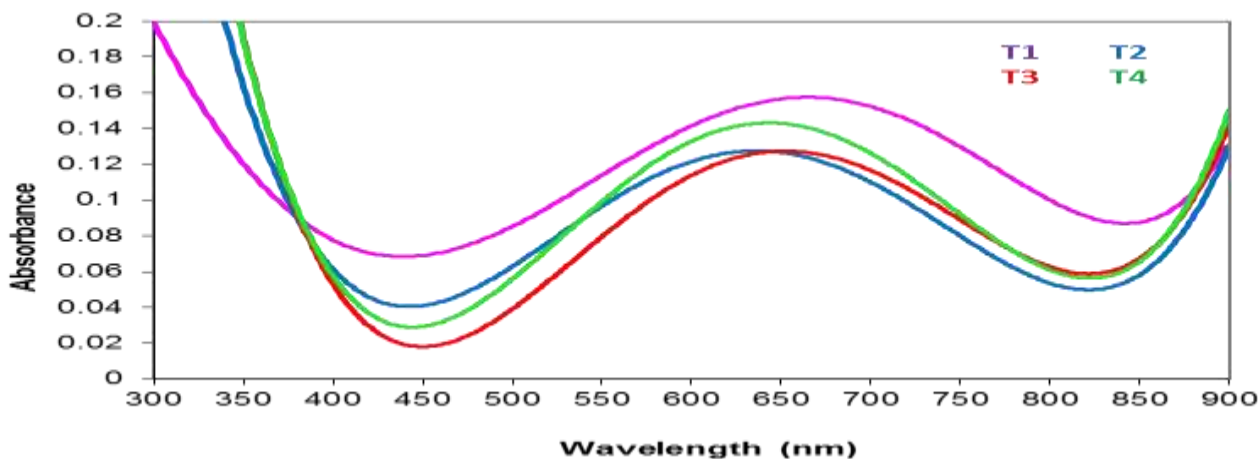


Fig. 1: absorbance with wavelength at different annealing temperatures

Table 2: Optical absorption with the various temperature at wavelength 700 nm

Films cods	T(°C)	Absorbance
.T1	360	0.13
.T2	460	0.096
.T3	550	0.100
.T4	700	0.109

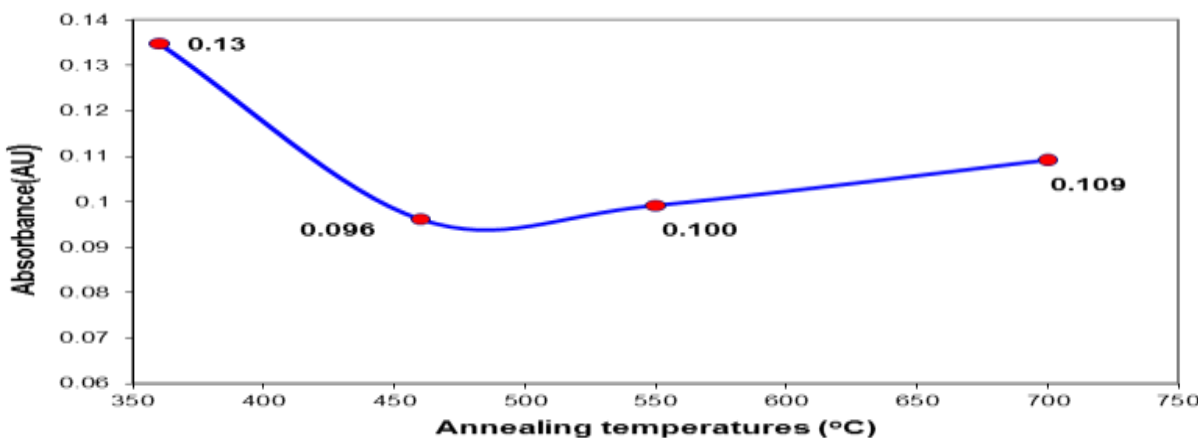


Fig. 2: Optical absorption values with various temperatures at λ=700 nm

**The Optical Transmittance**

The values of the visible optical transmittance at especially between (600 to 800) nm were normal, regular and recognizable, so at this region we can say also the visible optical transmittance rise with increasing of annealing temperatures ~460, ~550 and ~700 °C this is may be because of growing grain size and crystalline phases of TiO<sub>2</sub> like this results reported by researchers (R. Mechiakh et al.

2007) and [23]. At short wavelength there are no clear function between annealing temperatures and optical transmittance as shown in Figure (3). At ~700 nm for example transmittance percentage was about 73.0% at ~360 °C; at annealing temperature ~460°C in 700 nm transmittance percentage was about 80.00% and at ~550 °C was 80.00%. Then at ~700 °C transmittance percentage was about 78.00% more details see Figure (4) and Table (3). Finally, best transmittance was recorded at visible region at ~550 °C, this thin film can

be selected as solar cell filter because of their high optical transmittance at this

wavelengths and because of their low optical transmittance at (ultraviolet region).

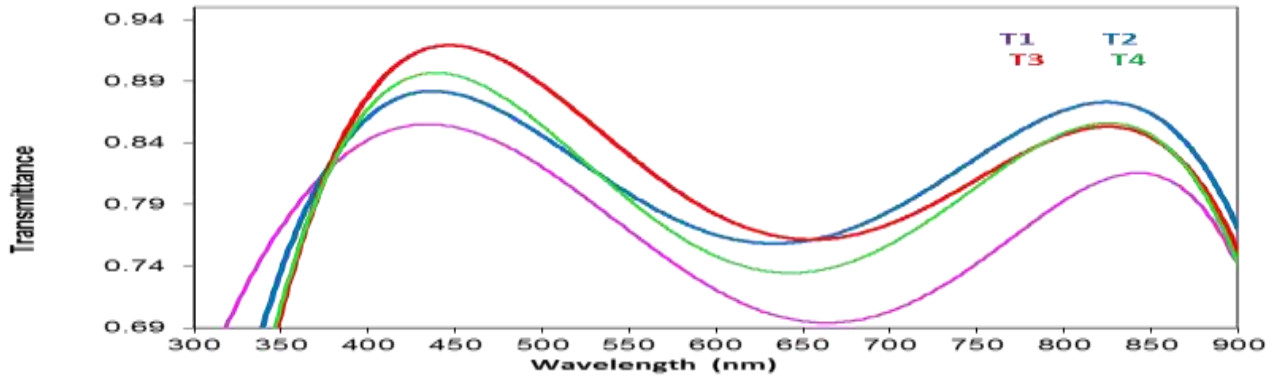


Fig.3: Transmittance with wavelength at different annealing temperatures

Table 2: Optical transmittance with the various temperature at wavelength 700 nm

Films cods	T(°C)	Transmittance
.T1	360	0.73
.T2	460	0.800
.T3	550	0.799
.T4	700	0.780

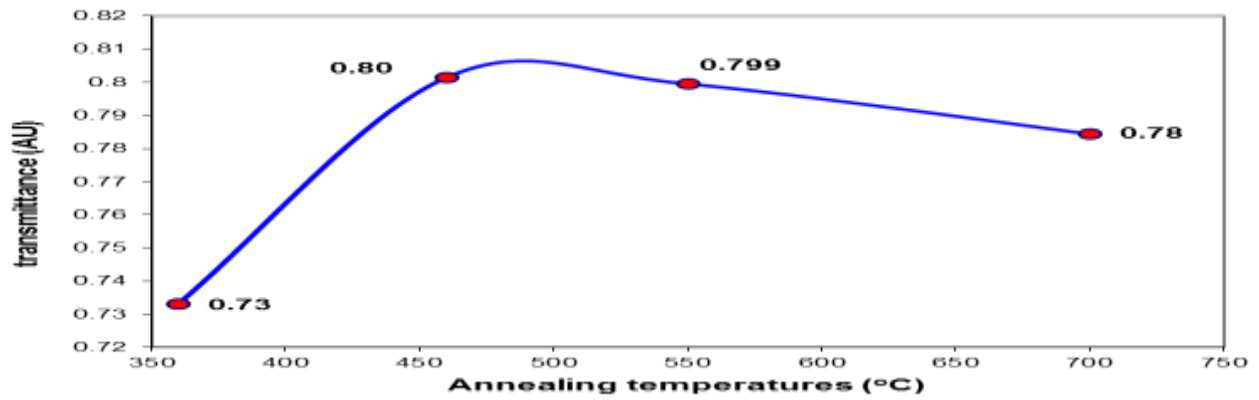


Fig. 4: Optical transmittance values with various temperatures at  $\lambda=700$  nm

### The Optical Reflectance and Refractive Index

Here we will discuss the optical reflectance and refractive indices. As we notice in Figures (5 and 6) we see the optical reflective spectrum curves have same optical absorption as a functions of the wavelengths with different temperatures because of the

same reasons obtained by (K.K. Saini ET al.2007). The optical reflectivity values were smaller than optical transmittance because of high transparency.

$$T=1-(A+R)..... (1).$$

Where R is optical reflectivity, A is optical absorbance and T is optical transmittance.

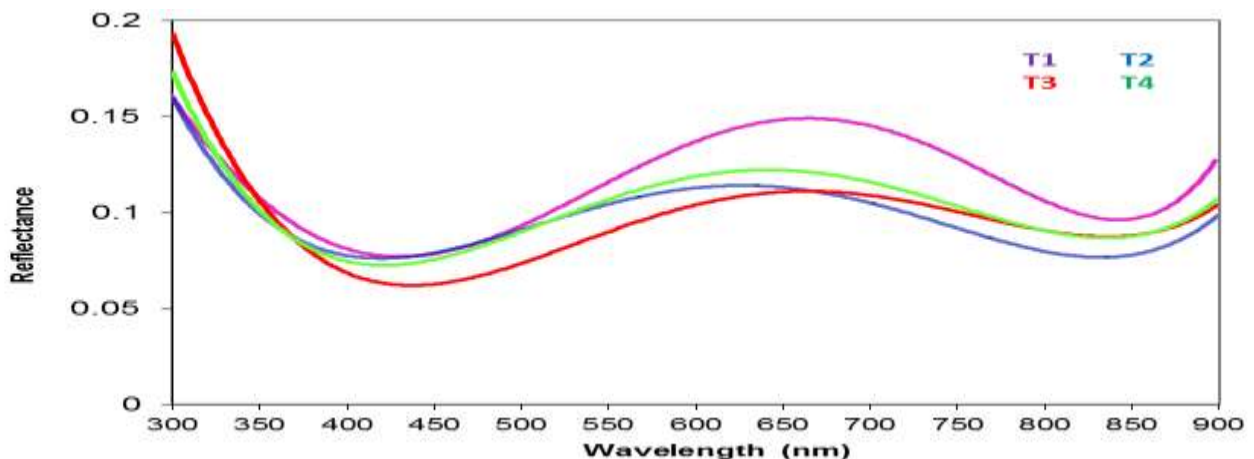


Fig.5: Reflectance with wavelength at different annealing temperatures

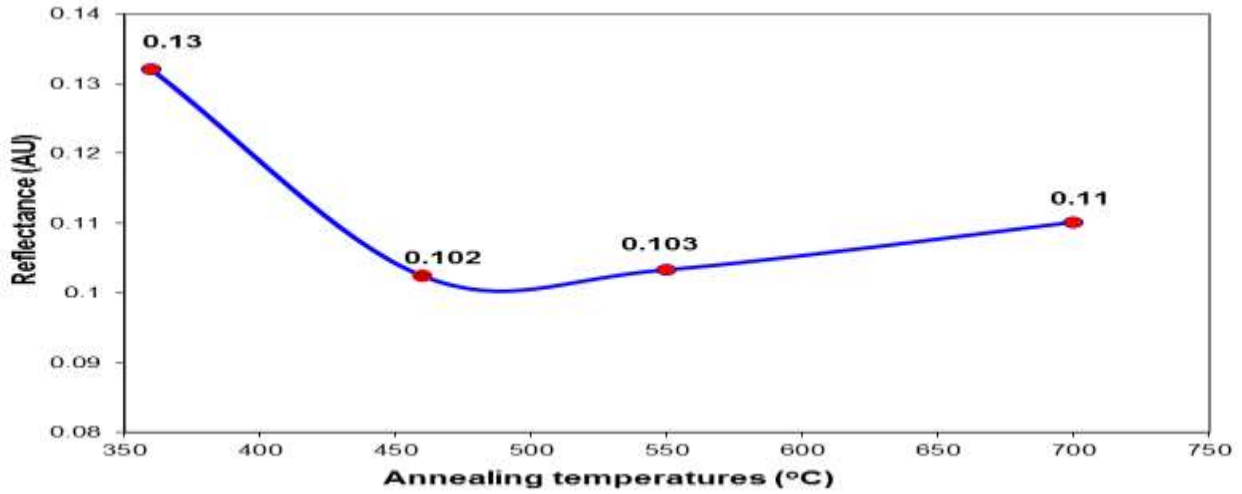


Fig. 6: Reflectance values with various temperatures at λ=700 nm

The optical refractive index rises with increasing of wavelength at UV regions and some region of the visible wavelength till to ~ 630 nm and then decreases at other parts ,

these results are degree with (Gao et al., 2004), show Figures (7) and (8) for more details. The refractive index has been calculated by using below equation

$$n = \sqrt{\frac{4R - K^2}{(R - 1)^2} - \frac{R + 1}{R - 1}} \dots\dots\dots 2$$

N is optical refractive index, R is optical reflectance and K is optical extinction coefficient.

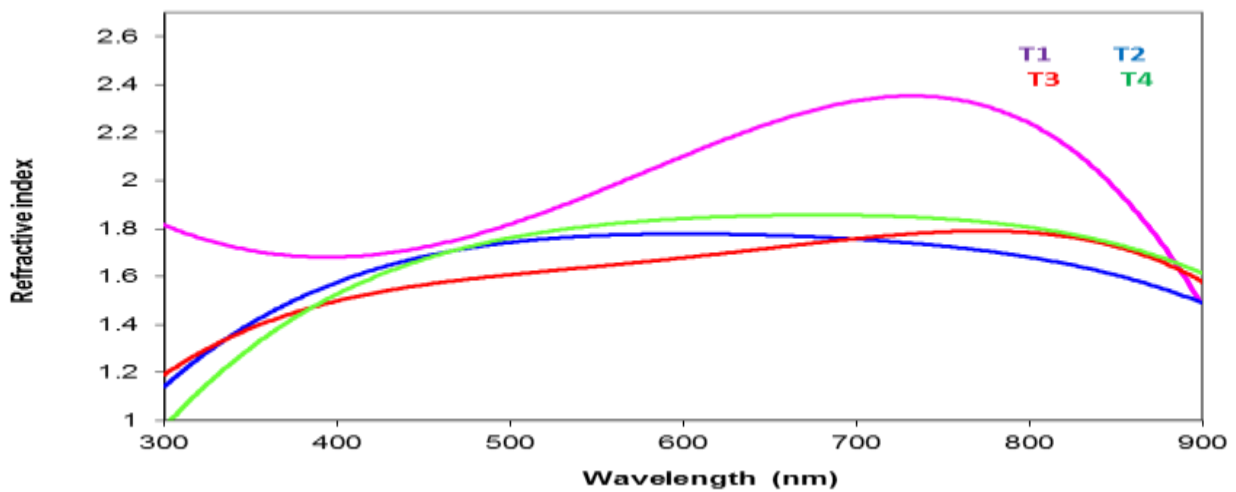


Fig.7: Refractive index with wavelength at different annealing temperatures

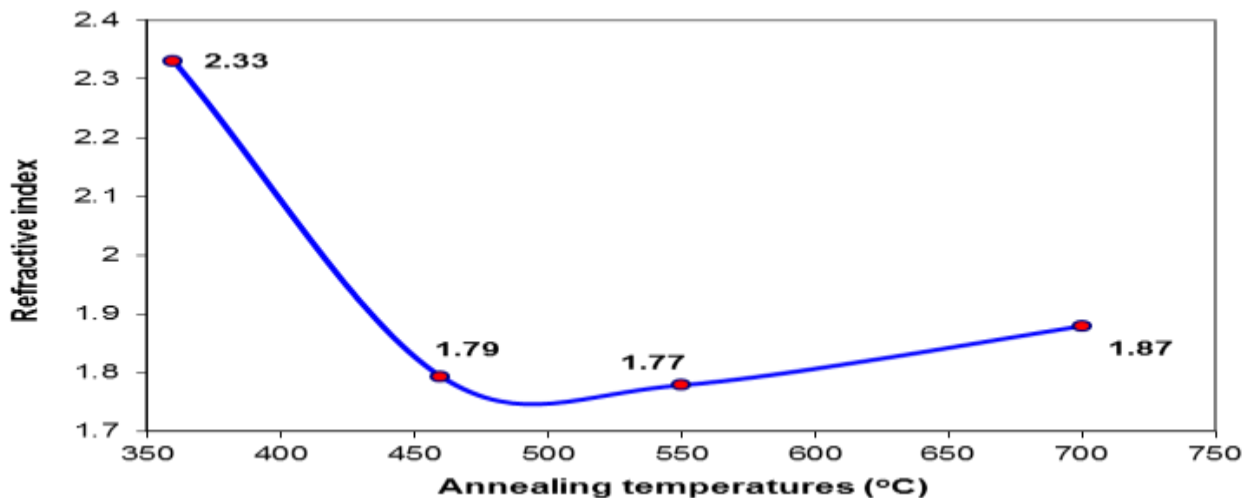


Fig. 8: Refractive index values with various temperatures at λ=700 nm

### The Optical Energy Gaps

By absorption coefficients energy gaps have been calculated by using the following equations [20].

$$\alpha = \ln \frac{1}{T}(r)^{-1} \dots\dots\dots 3$$

$$(\alpha h\nu) = S(h\nu - E_g)^L \dots\dots\dots 4$$

$$(\alpha h\nu) = cons..(h\nu - E_g)^2$$

When r is thickness ,T is optical transmittance and hv is photon energy.

By  $(\alpha h\nu)^{1/2}$  we were noting the energy gaps decrease with the rising the annealing temperatures (~450, ~450, ~700) °C [24], like this behavior can be attributed of both the crystalline phases and amorphous. See Figures (9, 10) for more details.

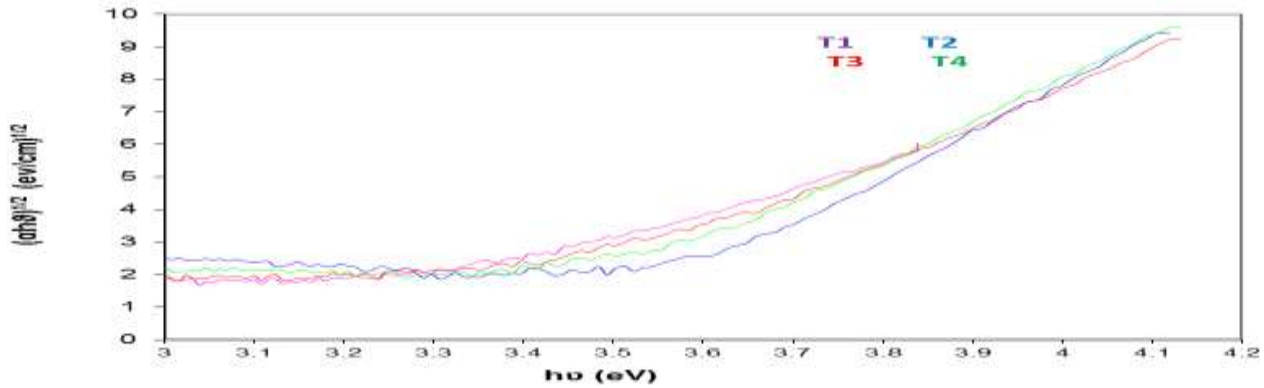


Fig. 9: Energy gap as function with the photons energies

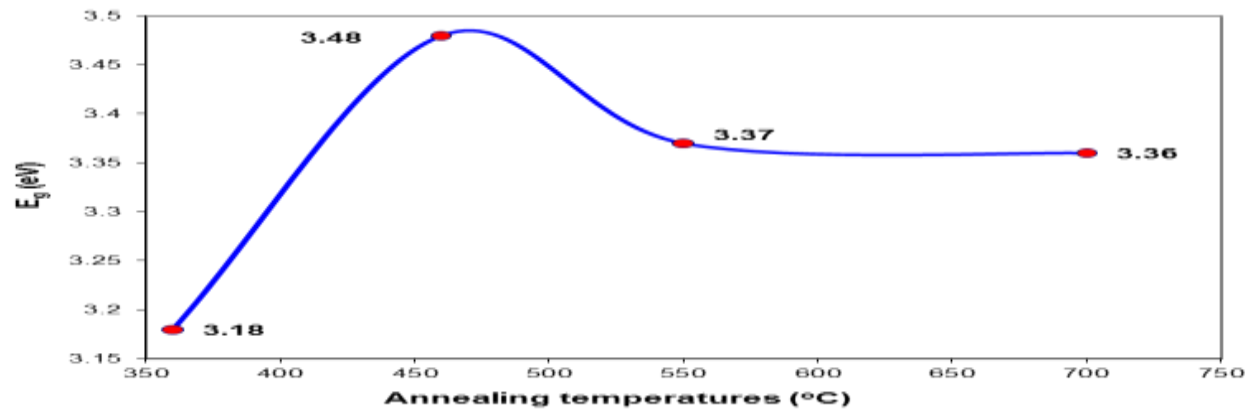


Fig. 10: Energy gaps the annealing temperatures

### Conclusions

- By concentrating observation we saw that region between the wavelengths ~(600-800) nm that optical absorbance rise with increasing the temperature (~460, ~550 and ~700) °C, and say this because growing crystalline of TiO<sub>2</sub> three phases and grain size growing as well as.
- The transmittance values at visible light were normal, especially between ~(600-800) nm was regular and so recognizable, so the transmittance decrease with rising of temperatures (~460,~550 and ~700) °C. According to the above results conclusions, like those thin films use as a filter of solar cell because of high optical transmittance visible regions and because of r low optical transmittance at uv wavelengths.

- The optical reflectivity were smaller than optical transmittance because of high optical transparency.
- The relationship between of the optical refractive index and annealing temperatures was an abnormal and different behavior in the refractive indices and their relationship with annealing temperature. However, in the region after ~630 nm, the results showed that the refractive index rising with an increase in temperature.
- Energy gaps decrease with the rise in the annealing temperature because the mixing of both the crystalline and amorphous.

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