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Effect of annealing and Mo , V and Ni Oxides as Doping Elements
on Optical and Electrical Properties of BaTiO₃ thin films
Ass.Professor Dr. Sabah A. Salman

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Abstract

In this work, BaTiO$_3$ thin films pure and doped with oxides (MoO$_3$,V$_2$O$_5$ and NiO) were deposited using pulsed laser deposition (PLD) technique with thickness equal to (300nm) on glass substrates at temperature equal to (573K). The effects of annealing at temperatures (673K,773K) and doping on the optical and electrical properties have been investigated. UV-VIS transmittance measurements showed that the films are highly transparent in the near-infrared region for as deposited films and in the visible region for annealing films at temperatures (673K,773K),while the absorption edge of BaTiO$_3$ films doped with (MoO$_3$,V$_2$O$_5$ and NiO) was shifted to the visible region. The optical energy gap was calculated and was found to be (3.6eV) for as deposited films, and this value increases from (3.69eV) to (3.8eV) with increasing annealing temperature from (673K) to (773K), and decreases with different elements oxides dopants. The optical constants such as refractive index, extinction coefficient and real and imaginary part of dielectric constant have been calculated. The electrical properties of these films were studied under different annealing temperatures and different doping materials. The d.c conductivity for all deposited films decreases from (1.36 × 10^{-7}) (Ω.cm)$^{-1}$ to (6.18 × 10^{-6}) (Ω.cm)$^{-1}$ and increase in activation energy with increase of annealing temperature, while the d.c conductivity increase and activation energy decreases with different elements oxides dopants .

Keywords: BaTiO$_3$ thin films, pulsed laser deposition, optical and electrical Properties.

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تأثير التلدين والتشويب بأكاسيد العناصر Mo , V و Ni على الخصائص البصرية والكهربائية لأغشية BaTiO₃ الرقيقة

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الخلاصة

تم في هذا العمل تحضير أغشيــة الباريوم تيتانيت النقية والمشوبة بأكاسيد (المولبدينوم,الفناديوم والنيكل) باستخدام تقنية الترسيب بالليزر النبضي بسمك مقداره (300nm) على قواعد زجاجية بدرجة حرارة أساس مقدارها (573K). تم دراسة تأثير التلدين بدرجات حرارة (773K,673K) والتشويب على الخصائص البصرية والكهربائية. درست الخصائص البصرية بواسطة قياسات مطياف النفاذية للأشعة المرئية وفوق البنفسجية , حيث أظهرت النتائج بأنّ الأغشية شفّافة جداً في منطقةِ الأشعة تحت الحمراء القريبة للأغشية كما تم ترسيبها وفي المنطقة المرئية للأغشية الملدنة بدرجات حرارة (773K,673K) .وعند أشابة الباريوم تيتانيت بلاكاسيد (المولبدينوم,الفناديوم و النيكل) , نلاحظ حدوث إزاحة بحافة الامتصاص نحو المنطقة المرئية . تم حساب قيمة فجوة الطاقة البصرية ووجدت قيمتها (3.6eV) للأغشية كما تم ترسيبها وتزداد هذه القيمة مع زيادة درجة حرارة التلدين من (3.69eV) إلى (3.8eV) وتقل عند التشويب بمختلف عناصر الاكاسيد . حسبت الثوابت البصرية مثل معامل الانكسار, معامل الخمود وثابت العزل بجزأيه الحقيقي والخيالي. درست الخصائص الكهربائيــة للأغشية المحضرة عند درجات حرارة لتلدين مختلفة وعناصر تشويب مختلفة,حيث لوحظ انخفاض التوصيليـــة الكهربائيـــة المستمـــرة لجميــع الأغشيــة المرسبة مــن (1.36*10⁻⁷) (Ω.cm)⁻¹ إلـــى (6.18*10⁻⁶) (Ω.cm)⁻¹ وزيادة بطاقة التنشيط بزيادة درجة حرارة التلدين,بينما تزداد التوصيـــلية الكهربائيـــة المستمـــرة وتقل طاقة التنشيط عند التشويب بمختلف عناصر الاكاسيد .

الكلمات المفتاحية: الأغشية الرقيقة لباريوم تيتانيت , الترسيب بالليزر النبضي , الخصائص البصرية والكهربائية.

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Introduction

Barium titanate ($BaTiO_3$) was one of the first ferroelectric materials discovered, and also one of the first recognized photorefractive material. It is, to date, the most extensively investigated ferroelectric material [1].

$BaTiO_3$ is a member of the perovskites family, of which the parent member is the mineral $CaTiO_3$, called perovskite. The perovskite family includes other well-known materials such as $KNbO_3$, $KTaO_3$, $PbTiO_3$ and $SrTiO_3$. The general formula of any compound belonging to this family is ABO_3, where A is a monovalent, divalent or trivalent metal and B a pentavalent, tetravalent or trivalent element. From the point of view of practical applications this material is very interesting because it is chemically and mechanically very stable and it exhibits ferroelectric properties at and above room temperature [2]. From the optical applications point of view, $BaTiO_3$ is very interesting because of its high linear and nonlinear electro optic coefficients. It is, a well-known dielectric material which has been used as an insulating material to fabricate MIS structures. It exhibits several advantages and properties such as high charge storage capacity, good insulating property, low leakage current density and high dielectric breakdown strength [3].

Barium titanate ($BaTiO_3$) and other perovskite-type materials are being extensively studied for their potential commercial applications in dynamic random access memories (DRAMs) [4, 5]. Ferroelectric thin films attract wide interest due to its good characteristics of dielectricity, piezoelectricity and pyoelectricity. And surface acoustic wave (SAW) device [6,7].

It has become one of the most important electro ceramics since the discovery of its versatility in multilayer ceramic capacitors (MLCC), positive temperature coefficient of resistance (PTCR), thermistors, piezoelectric sensors, transducers, actuators and ferroelectric random access memories (FRAM) and electro-optic devices[8,9]. Its dielectric maximum is shifted towards room temperature by the compositional substitution and its dielectrics were sensitive to temperature, field strength and frequency, especially near the Curie temperature. $BaTiO_3$ thin films can also be used as dielectrics in an amorphous form, bringing the advantage of a much lower process temperature that is required for crystalline $BaTiO_3$ films [10,11].In case of thin films $BaTiO_3$ have been prepared by sputtering [12] PLD [13] , sol-gel,

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MOD [14] and MOCVD [15] .PLD is currently the most important method for preparing epitaxial structures for device physics studies.

Experimental procedure

The $BaTiO_3$ was prepared by using solid state reaction method by mixing BaO and TiO_2 compounds in [1:1] ratio, $BaTiO_3$ doped with ratio (0.1wt%) of (Mo,V and Ni oxide) by using solid state reaction method also.

$BaTiO_3$ thin films pure and doped with oxides (MoO_3,V_2O_5 and NiO) were deposited using pulsed laser deposition (PLD) technique.

The pulsed laser deposition experiment was carried out inside a vacuum chamber generally in (10^{-3}Torr) vacuum conditions. The focused Nd:YAG SHG Q-switching laser beam incident on the target surface makes an angle of ($45°$) with it. The films were deposited on glass at substrate temperature (T_S=573K) with rate of deposition equal to (0.5nm/sec). The deposition was carried out using a Q switched Nd:YAG laser with a frequency second radiation at (532nm) (pulse width (10 nsec) repetition frequency (6 Hz)), for (250) laser pulse.

Films thickness was measured by using optical interferometer method and found to be (300nm), as deposited $BaTiO_3$ films have been annealed at temperatures (673K,773K) in air.

A double –beam UV-VIS-NIR 210A Spectrophotometer was used to measure the transmittance and absorbance of $BaTiO_3$ films deposited at different conditions in the range of (200-1100 nm). The background correction was taken for each scan.

In order to measure the electrical properties, ohmic contacts are needed. It was obtained by indium wire of high purity (5 nines) under vacuum. The evaporation process was started at a pressure of (10^{-5}Torr). The best condition for good ohmic contact was satisfied by a layer of (250nm).

The electrical measurements of $BaTiO_3$ films deposited at different conditions have been done. The D.C electrical conductivity has been measured as a function of temperature over

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the range (R.T – 453K) by using the electrical circuit (sensitive digital electrometer type keithley (616) and electrical oven).

Results and discussions

Figure (1) shows the spectral optical transmittance as a function of wavelength in the range of (300–1100nm) for $BaTiO_3$ films deposited at substrate temperature (T_S=573K) and annealed at different annealing temperatures (673K,773K). This figure reveals that the transmittance depends on the annealing temperature. The maximum transmittance observed for $BaTiO_3$ films deposited at (T_S= 573K) equal to (42%) in the near-infrared region, while for the annealing films, the maximum transmittance equal (58%) at annealing temperature (673K) and (81.7%) at annealing temperature (773K). The behavior of the transmittance spectra is opposite completely to that of the absorption spectra. In general, we can observe from this figure that transmittance increases with increasing of annealing temperature and this may be due to improving of the crystallite size which means a decrease in the absorption. The films were found to be highly transparent in the visible wavelength region with an average transmittance in excess of (80%). This is probably ascribed to the increase of particle size and surface roughness.
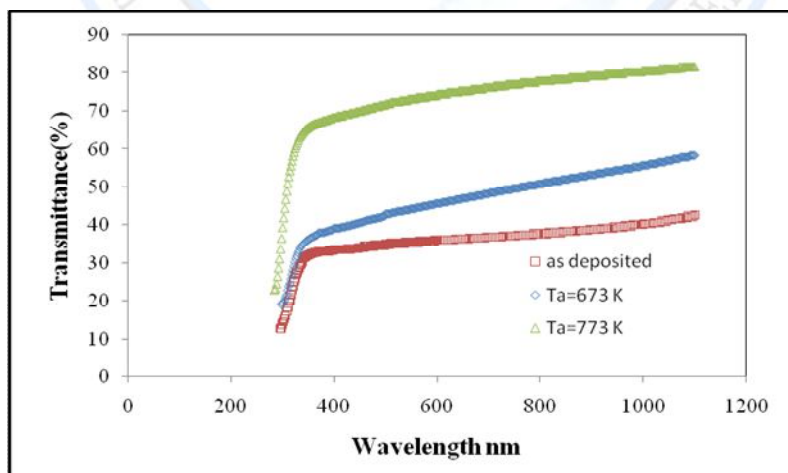

Figure (1): UV-VIS transmission spectra of the BaTiO₃ films at substrate temperature (573K) with thickness of (300 nm) and different annealing temperature (673K,773K).

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Figure (2) shows the spectral optical transmittance as a function of wavelength in the range of (300–1100nm) for BaTiO₃ films doped with different elements of mixed oxides (MoO₃ ,V₂O₅ and NiO) with doping ratio (0.1wt%). An increase in the transmittance is observed as the doping oxides were changed from MoO₃ to NiO to V₂O₅. The films were found to be highly transparent in the visible wavelength region. The maximum transmittance observed for BaTiO₃ films was almost (42%), while for the doped films, the maximum transmittance was equal to (85%) for BaTiO₃: MoO₃ and (75% and 56%) for both BaTiO₃: NiO and BaTiO₃: V₂O₅, respectively.
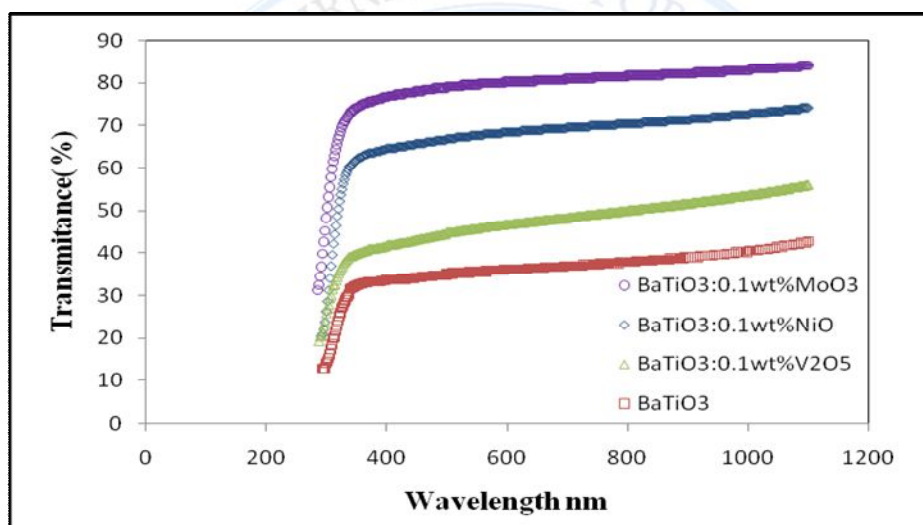
Figure (2): UV- VIS transmission spectra of the BaTiO₃ films at different doping (MoO₃ ,V₂O₅ and NiO) with thickness of (300 nm) at substrate temperature (573K).

The absorption coefficient (α) is given by [16]:

$$\alpha = 2.303\frac{A}{t} \qquad \text{.. (1)}$$

Where (A) is the absorbance and (t) is the sample thickness.

The variation of the absorption coefficient (α) for BaTiO₃ films deposited at substrate temperature (T$_S$=573K) and annealed at different annealing temperatures (673K,773K) is shown in Figure (3). It is observed that the absorption coefficient decreases with decreasing photon energy. We deduce that the absorption is not attributed to the free carriers only, but to

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impurities or localized electronic states. Also, we can notice from this figure that (α) in general decreases with the increase of annealing temperature. This is due to the increase of energy gap with annealing temperature.

Figure (4) shows the variation of the absorption coefficient (α) for BaTiO₃ films doped with different elements of mixed oxides (MoO₃,V₂O₅ and NiO) with doping ratio (0.1wt%). It is observed that the absorption coefficient decreases with decreasing of photon energy. Also, it can be noticed from this figure that (α) in general decreases as the doped oxides change from V₂O₅ to NiO to MoO₃.
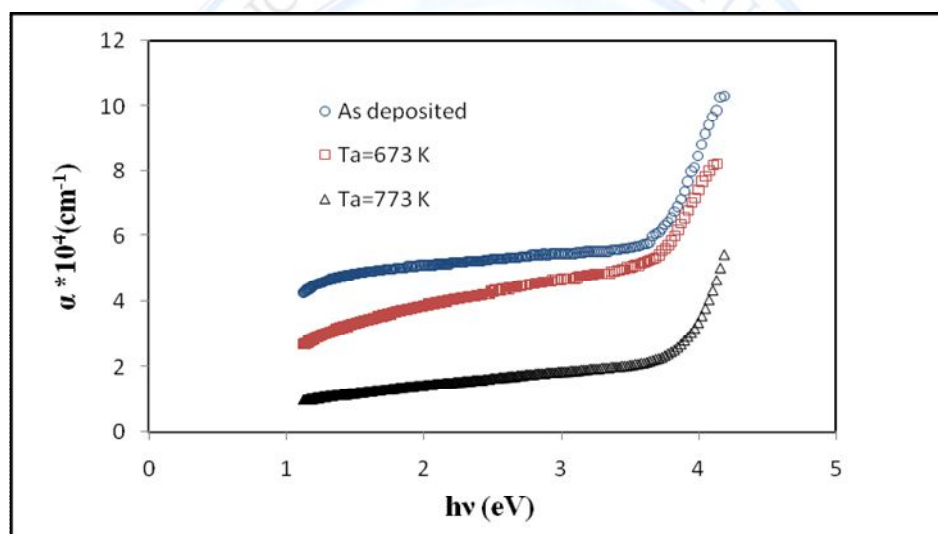
**Figure (3): the variation of the absorption coefficient (α) with photon energy of the
BaTiO₃ films for as deposited (Ts=573K) and annealed at (673K,773K).**

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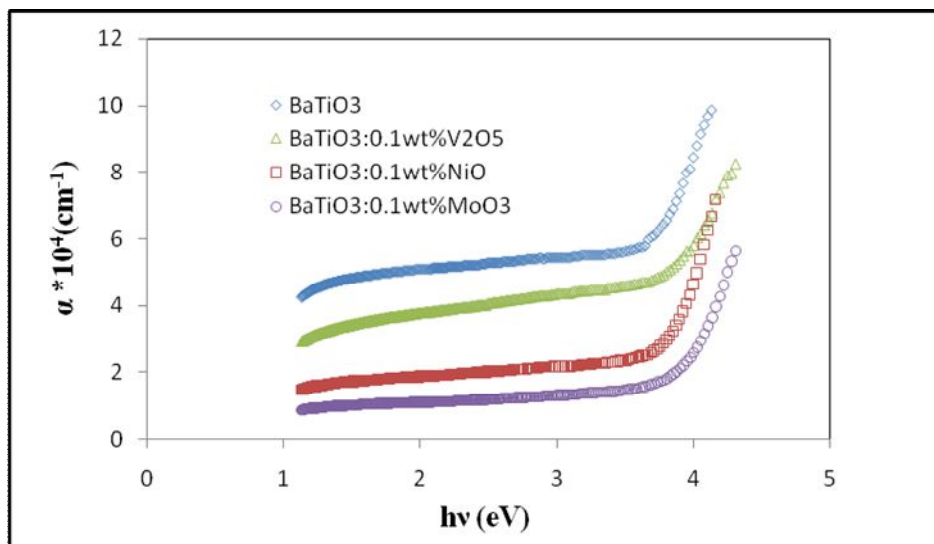Ass.Professor Dr. Sabah A. Salman

Figure (4): The variation of the absorption coefficient (α) with photon energy of the BaTiO₃ films at different doping (MoO₃ ,V₂O₅ and NiO) at substrate temperature (573K).

The optical energy gap values (E_g^{opt}) for BaTiO₃ films have been determined by using Tauc formula by plotting the relations of $(\alpha h\nu)^2$ vs E (eV) for direct energy gap as shown in Figure (5).The energy gap is obtained from intercept of the extrapolated linear part of the curve with the energy axis. The direct energy gap of the pure BaTiO₃ films increases from (3.6eV) to (3.8eV) when annealing temperature increases from (573K) to (773K). We can see that the annealing shifts the optical energy gap from approximately (3.69eV) for (673K) and (3.8eV) for (773K) as represented in the table (1). Our results are in agreement with S.S.Kim et *al* [17]. The annealing temperature for BaTiO₃ films having different effect leads to increase in the optical energy gap values, because the increasing of annealing temperature process decreases from the secondary levels and the structure defects which lead to the contract tails region. This leads to expand in the optical energy gap.

The optical energy gap values (E_g^{opt}) for BaTiO₃ films doped with different elements of mixed oxides (MoO₃,V₂O₅ and NiO) with doping ratio (0.1wt%) are in the range of (3.59–3.5

eV), as shown in the Figure (6). It is also observed that the direct energy gap decreases as the doped oxides change from MoO_3 to NiO to V_2O_5 as represented in the table (1) .
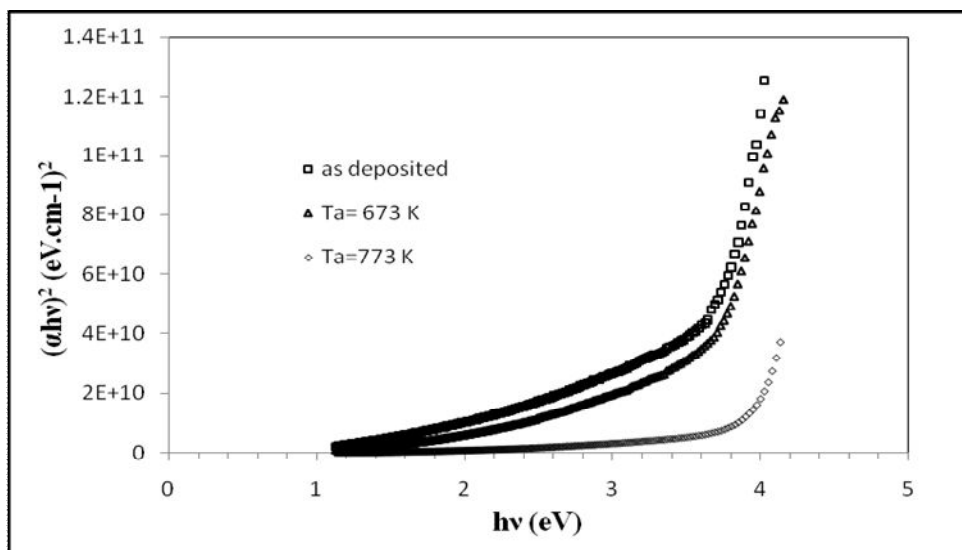

Figure (5): $(\alpha h\nu)^2$ versus photon energy (hν) of the BaTiO₃ films at:(a) Substrate Temperature(573K). (b) Annealing temperature (673K). (c) Annealing temperature (773K).
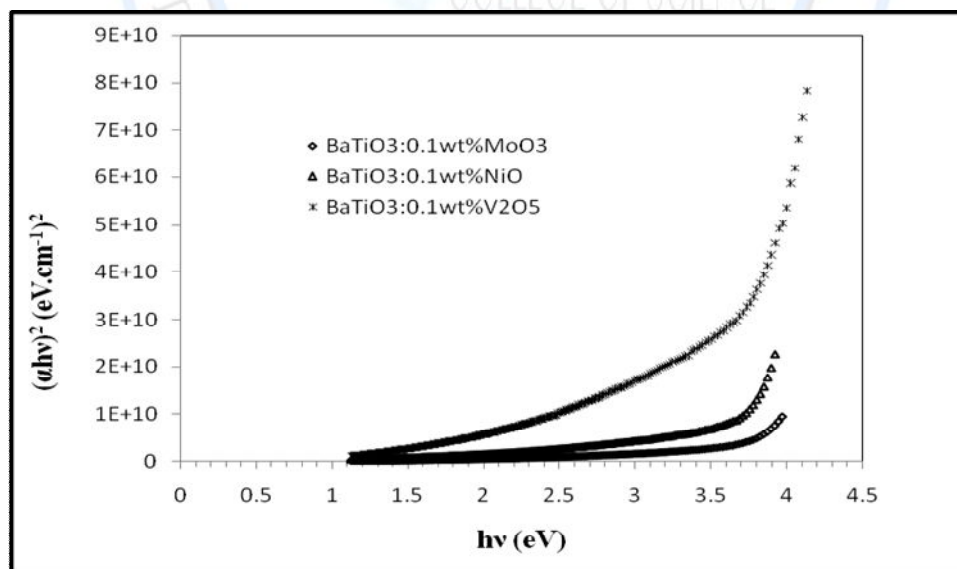

Figure (6): $(\alpha h\nu)^2$ versus photon energy (hν) of the BaTiO₃ films at different doping elements (MoO₃,V₂O₅ and NiO) at substrate temperature (573K).

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Table (1) optical energy gap for direct transition of different annealing temperatures and doped BaTiO₃ films for different elements of mixed oxides.

Sample	Optical energy gab (eV) (direct)
BaTiO₃ at Ts=573 K	3.6
BaTiO₃ at 673 K	3.69
BaTiO₃ at 773 K	3.8
BaTiO₃:0.1wt% MoO₃ at 573 K	3.59
BaTiO₃:0.1wt% NiO at 573 K	3.55
BaTiO₃:0.1wt% V₂O₅ at 573 K	3.5

The refractive index (n) value can be calculated from the formula [18]:

$$n = \left(\frac{4R}{(R-1)^2} - k^2 \right)^{\frac{1}{2}} - \frac{(R+1)}{(R-1)} \dots\dots\dots\dots\dots (2)$$

Where (R) is the reflectance

   Figure (7) shows the variation in refractive index (n) with wavelength in the range of (300-1100nm) for BaTiO₃ films deposited at substrate temperature (T_S=573K) and annealed at different annealing temperatures (673K,773K). It is observed that the refractive index, in general decreases slightly with increasing of annealing temperature. The values of the refractive index varies from (2.4) to (2.2) at absorption edge depend on annealing temperature. Our results are in agreement with Y.Avrahami [19]. This behavior is due to the increase in energy gap which is due to decrease of the defect density which means decreasing of the reflection where the refractive index depends on it.

   Figure (8) shows the variation in refractive index (n) with wavelength in the range of (300-1100 nm) for BaTiO₃ films doped with different elements of mixed oxides (MoO₃,V₂O₅ and NiO) with doping ratio (0.1wt%). The refractive index decreases as the doped oxides change

from V_2O_5 to NiO to MoO_3. The values of the refractive index varies from (2.4) to (2.18) at absorption edge.
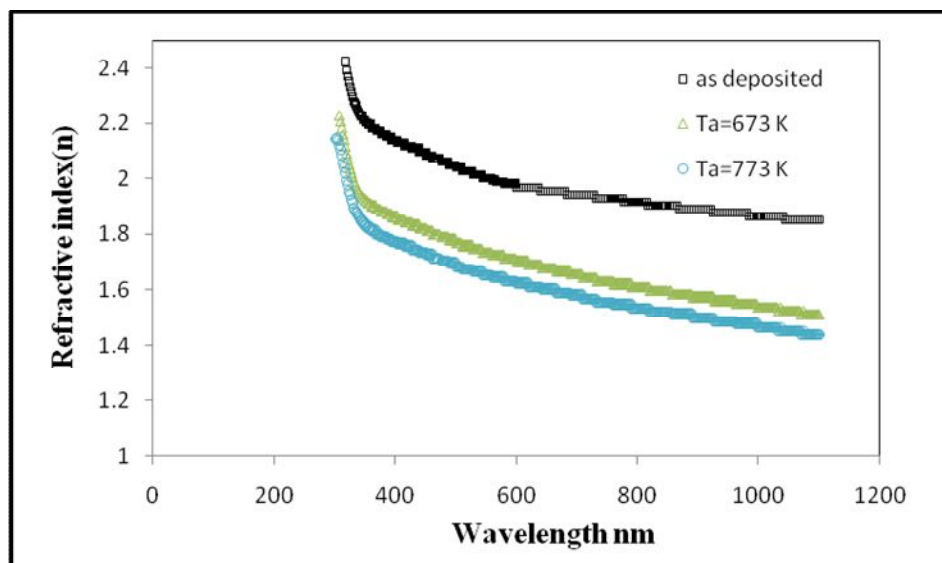

Figure (7): the variation of the refractive index (n) with wavelength for as deposited (Ts=573K) and annealed BaTiO₃ films at (673 K,773K).
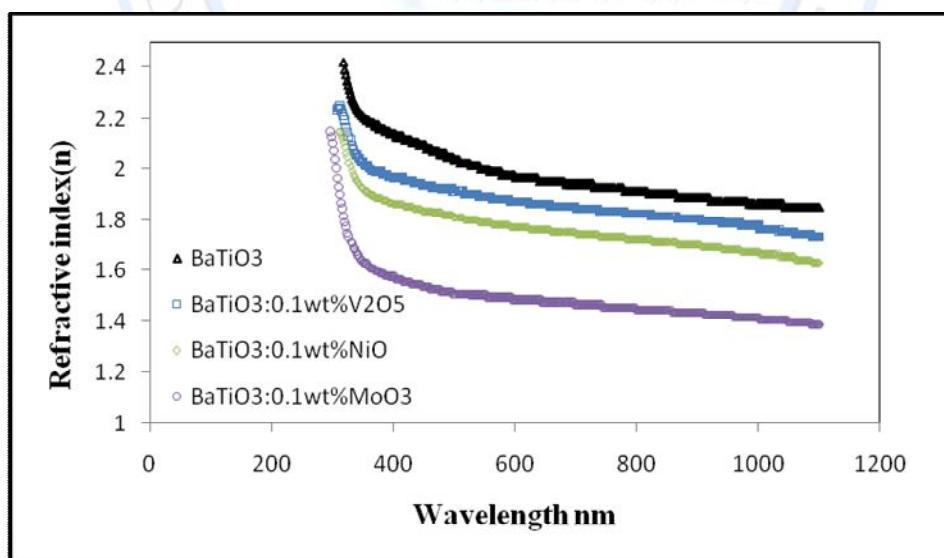

Figure (8): the variation of the refractive index (n) with wavelength of the BaTiO₃ films with different doping (MoO₃,V₂O₅ and NiO) at substrate temperature (573K).

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The extinction coefficient (k), which is related to the exponential decay of the wave as it passes through the medium, is defined as [20]:

$$k = \frac{\alpha\lambda}{4\pi} \ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \ (3)$$

Where (λ) is the wavelength of the incident radiation

Figure (9) shows the variation in extinction coefficient (k) with wavelength in the range of (300-1100nm) for BaTiO₃ films deposited at substrate temperature (Ts=573K) and annealed at different annealing temperatures (673K,773K). It is observed that the extinction coefficient decreases with the increase of annealing temperature, and have the same behavior of absorption coefficient, because mainly depend of extinction coefficient on absorption coefficient.

Figure (10) shows the variation in extinction coefficient (k) with wavelength in the range of (300-1100nm) for BaTiO₃ films doped with different elements of mixed oxides (MoO₃,V₂O₅ and NiO) with doping ratio (0.1wt%). The extinction coefficient decreases as the doped oxides change from V₂O₅ to NiO to MoO₃.
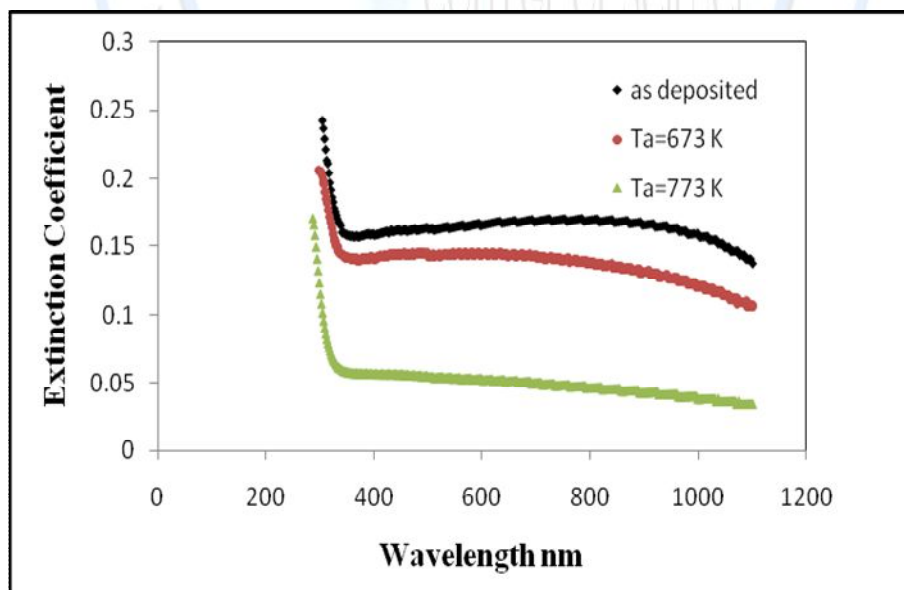

Figure (9): the variation of the extinction coefficient (k) with wavelength for as deposited (Ts=573K) and annealed BaTiO₃ films at (673K,773K).

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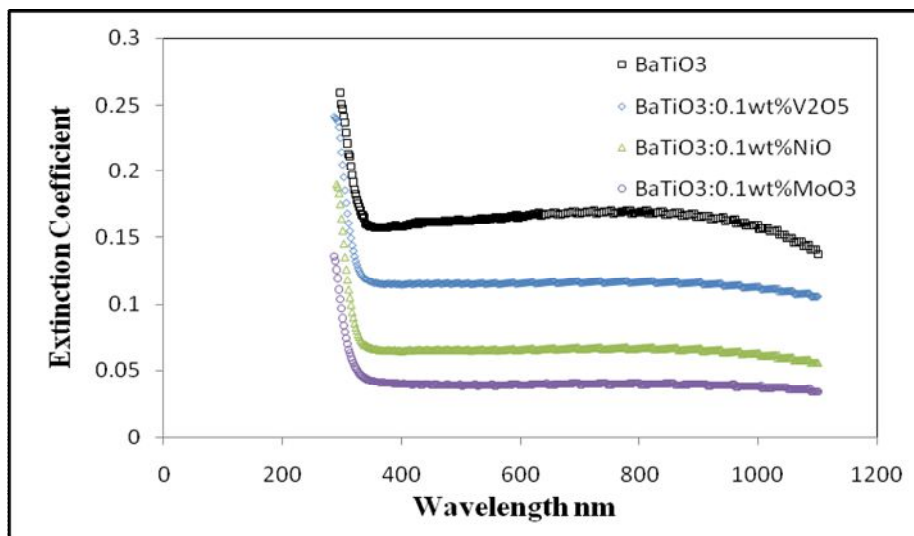
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Fig (10): the variation of the extinction coefficient (k) with wavelength of the BaTiO₃ films at different doping (MoO₃,V₂O₅ and NiO) at substrate temperature (573K).

Real and imaginary parts of dielectric constant were determined using the equations (4) and (5), respectively [21].

$$\varepsilon_r = n^2 - k^2 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

And $\quad \varepsilon_i = 2nk \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$

The plots of real and imaginary (ε_r and ε_i) parts of dielectric constant with wavelength in the range of (300-1100 nm) for BaTiO₃ films deposited at substrate temperature (Ts=573K) and annealed at different annealing temperatures (673K,773K) are shown in Figures (11 and 12) respectively. The variation of the real part of dielectric constant depends on the value of the refractive index. By contrast, the imaginary part of dielectric constant depends mainly on the extinction coefficient values which are related to the variation of absorption coefficient. This means that real part decreases and the imaginary part decreases with the increase of annealing temperature.

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Also, Figures (13) and (14) shows the variation of real and imaginary (ϵ_r and ϵ_i) parts of dielectric constant with wavelength in the range of (300-1100 nm) for BaTiO₃ films doped with different elements of mixed oxides (MoO₃,V₂O₅ and NiO) with doping ratio (0.1wt%). It is observed that real part and imaginary part of dielectric constant decrease with doped oxides change from V₂O₅ to NiO to MoO₃ due to decrease of the refractive index and extinction coefficient.
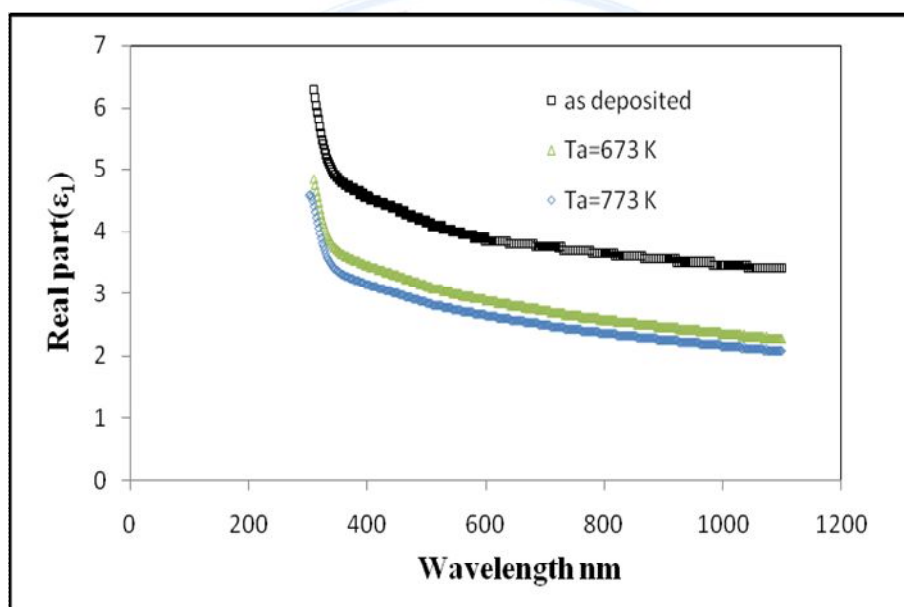

Figure (11): the variation of the real part (ϵ_r) with wavelength for as deposited (Ts=573K) and annealed BaTiO₃ films at (673K,773K).

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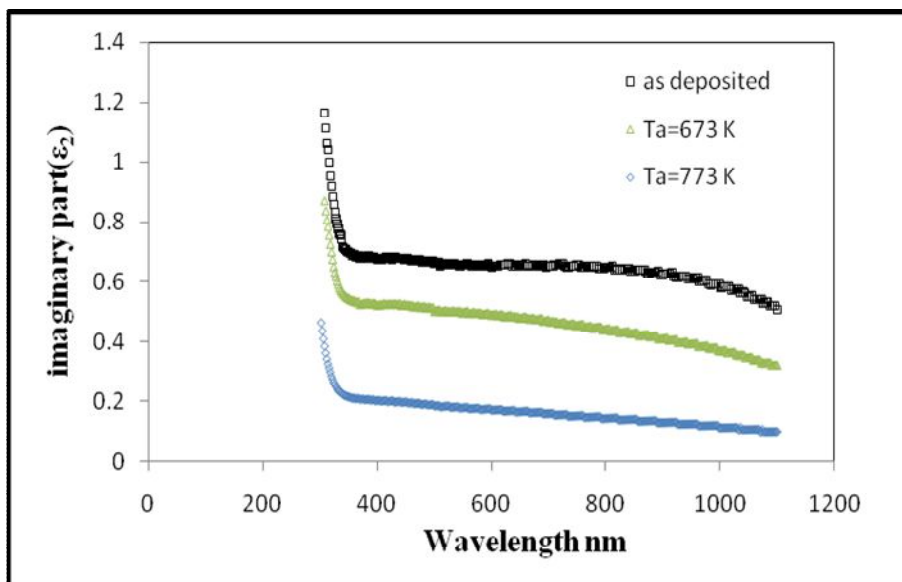
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Figure (12): the variation of the imaginary part (ϵ_i) with wavelength for as deposited (Ts=573 K) and annealed BaTiO₃ films at (673K,773K).
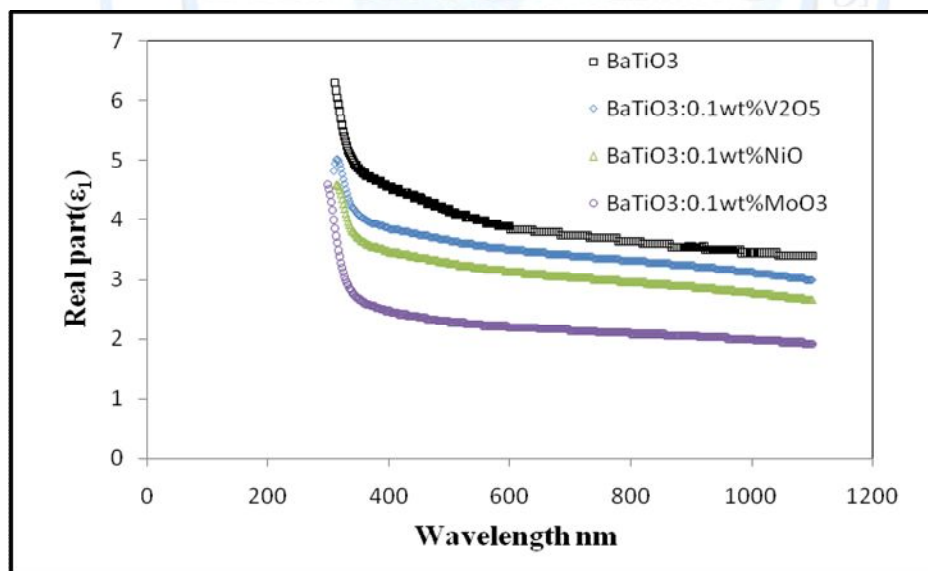

Figure (13): the variation of the real part (ϵ_r) with wavelength of the BaTiO₃ films at different doping (MoO₃,V₂O₅ and NiO) at substrate temperature (573K).

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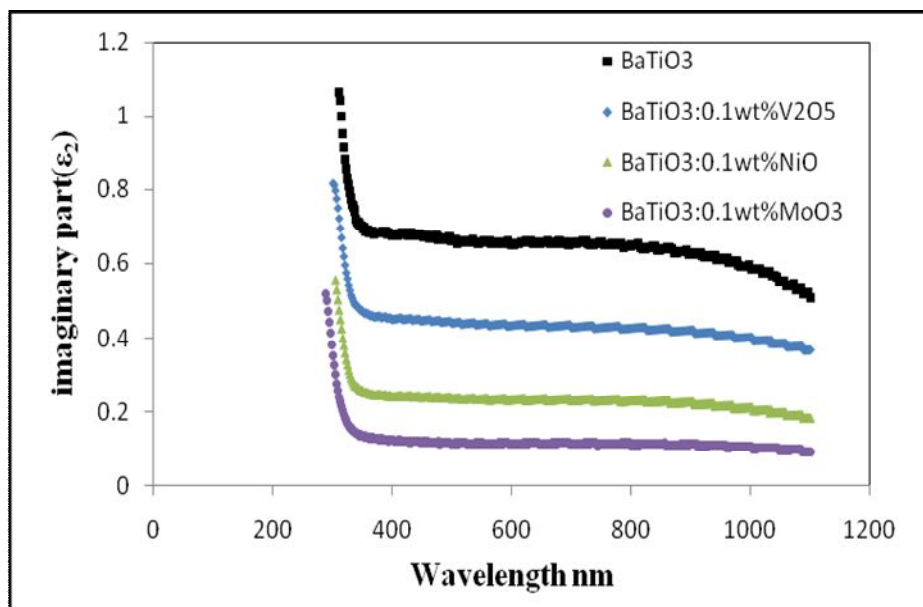
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Figure (14): the variation of the imaginary part (Є$_i$) with wavelength of the BaTiO₃ films at different doping (MoO₃,V₂O₅ and NiO) at substrate temperature (573K).

In order to study conductivity mechanisms, it is convenient to plot logarithm of the conductivity (Ln σ) as a function of (1000/T) in the range of (303–453K) for BaTiO3 films deposited at substrate temperature (TS=573K) and annealed at different annealing temperatures (673K,773K) as shown in Fig (15) and (16). It is clear from these figures that there are two transport mechanisms, giving rise to two activation energies (Ea1) and (Ea2). At higher temperatures range (413–453K), the conduction mechanism is due to carrier excited into the extended states beyond the mobility edge and at lower temperatures range (303–403K); the conduction mechanism is due to carrier excited into localized states at the edge of the band [22]. It is observed that the activation energies increase while (σRT) decreases with increasing of the annealing temperature as represented in the table (2) .Also, figure (17) shows the variation of logarithm of the conductivity (Ln σ) as a function of (1000/T) in the range of (303–453K) for BaTiO3 films doped with different elements of mixed oxides (MoO3,V2O5 and NiO) with doping ratio (0.1wt%). It is clear from these figures that there are two transport mechanisms, giving rise to two activation energies (Ea1) and (Ea2) in the

same ranges. It is also observed that the activation energies decrease while (σRT) increases as the doped oxides change from MoO3 toV2O5 to NiO as represented in the table (2) .
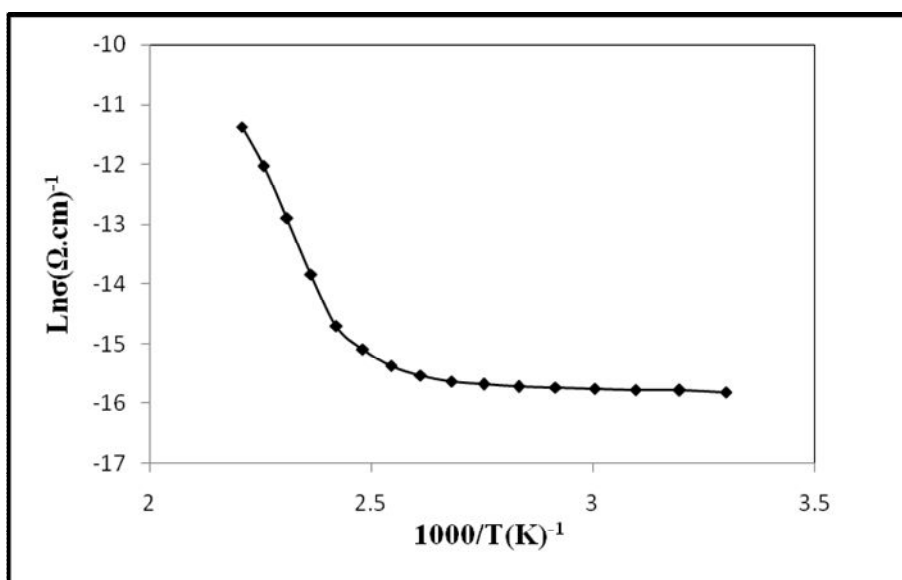

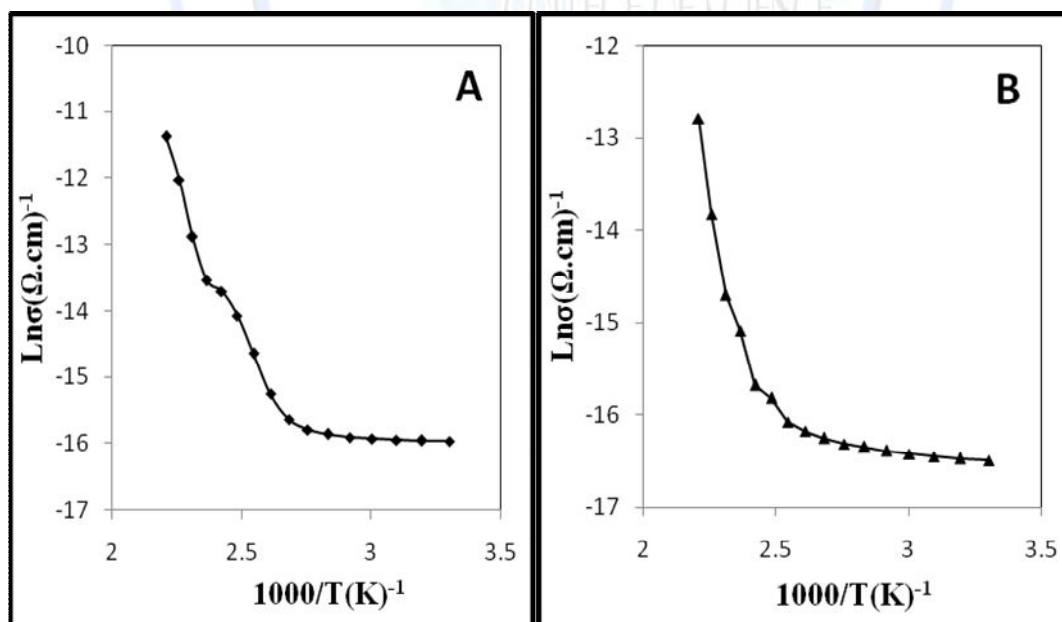**Figure (15): (Ln σ) versus (1000/T) of BaTiO₃ films at as deposited (Ts=573K).**

Figure (16): (Ln σ) versus (1000/T) of BaTiO₃ films at different annealed temperatures (A) 673K (B) 773K.

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Figure (17): (Ln σ) versus (1000/T) of BaTiO₃ films at as deposited (Ts=573K) and different doping ; (a) MoO₃, (b) V₂O₅, (c) NiO.

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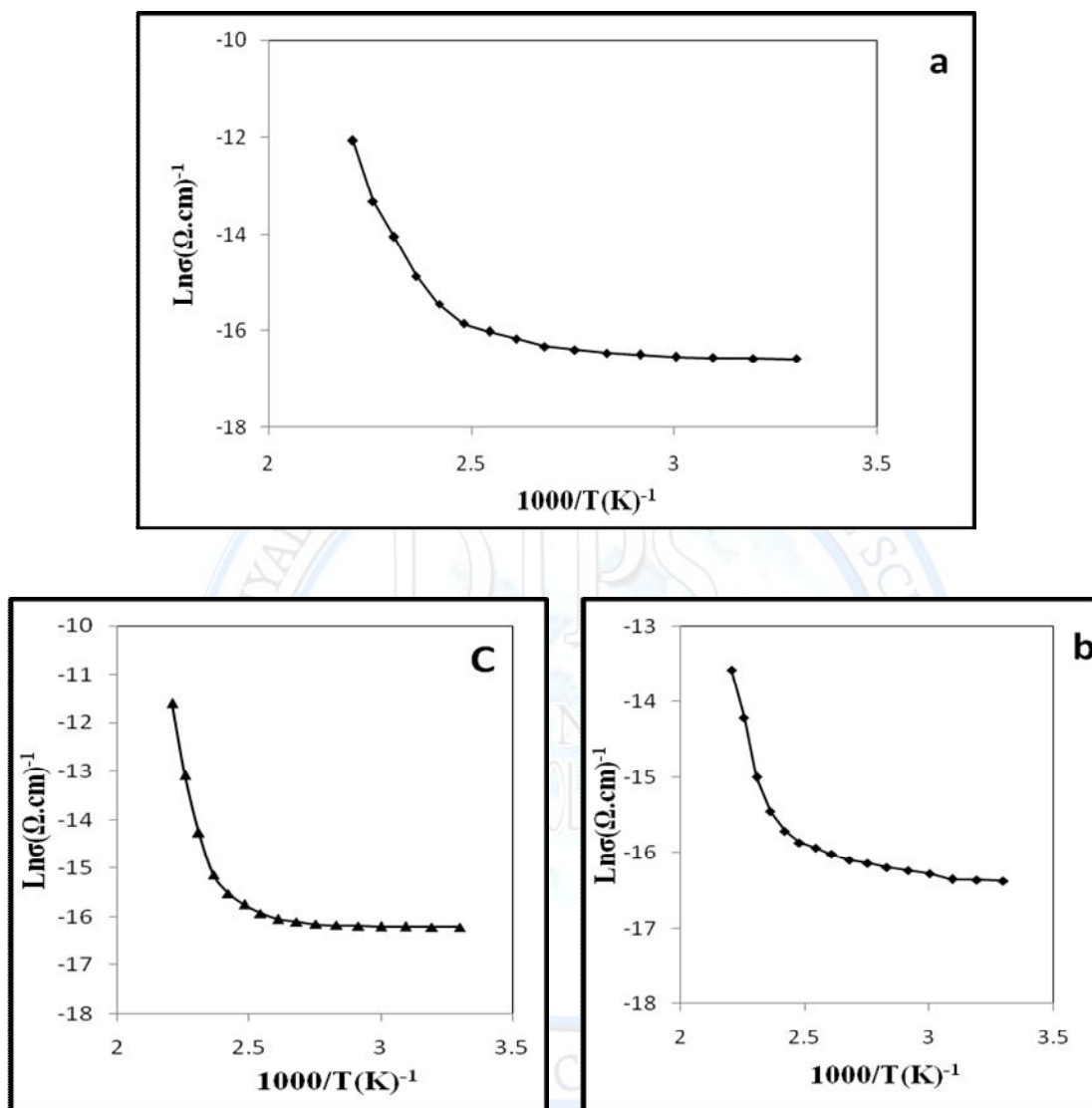
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Table(2) D.C conductivity parameters of different annealing temperatures and doped BaTiO$_3$ films for different elements of mixed oxides.

sample	T$_a$ (K)	(303 – 403)K	(413 –453)K	σ$_{R.T}$ (Ω.cm)$^{-1}$
		E$_{a1}$ (eV)	E$_{a2}$ (eV)	
BaTiO$_3$	Ts=573	0.038	1.062	$1.36*10^{-7}$
	673	0.044	1.150	$1.16*10^{-7}$
	773	0.046	1.209	$6.18*10^{-6}$
BaTiO$_3$:0.1wt%MoO$_3$	Ts=573	0.036	0.855	$6.28*10^{-8}$
BaTiO$_3$:0.1wt%V$_2$O$_5$	Ts=573	0.032	0.839	$7.74*10^{-8}$
BaTiO$_3$:0.1wt%NiO	Ts=573	0.016	0.354	$9.09*10^{-8}$

Conclusions

The transmittance spectrum for BaTiO$_3$ films shows strong attenuation in the short wavelength region and shows strong attenuation in the visible region after annealing. The transmittance increases with increasing annealing temperature. The optical energy gap is direct energy gap and increases with increasing annealing temperature, while the refractive index, absorption coefficient, extinction coefficient, real and imaginary parts of dielectric constant decreases slightly with increasing annealing temperature. The d.c conductivity for all deposited films decreases and increase in activation energy with increase of annealing temperature, there are two transport mechanisms of the charge carriers over the range of (290-503 K) for as deposited and annealed films. For BaTiO$_3$ films doped with different elements of mixed oxides (MoO$_3$,V$_2$O$_5$ and NiO) with doping ratio (0.1wt%), the transmittance spectrum shows strong attenuation in the visible region and all the optical parameters are changes with doping, while the d.c conductivity increase and activation energy decreases with different elements oxides dopants .

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