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Research Article

MEASUREMENT OF OPTICAL ENERGY GAP AND OPTICAL CONSTANTS OF EPOXY–BASED MICROCOMPOSITES FILLED WITH TiO_2 PARTICLES

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ABSTRACT

In this work, the pure epoxy reinforced by TiO_2 microparticles with weight percentage (2, 4, 6, and 8%) in particle size (50μm) by using molding method with shearing mixer at room temperature in order to prepare microcomposites for examined and measured their optical energy gap and optical constants of prepare specimens. The system used to measure Transmittance and Absorbance consisting laser semiconductor with wavelength (650nm), power (5mW) equipment with a power meter. The values of absorption coefficient less than $10^4 Cm^{-1}$which indicated that the electronic transition in pure epoxy resin and epoxy /TiO_2 microcomposites was indirect electronic transitions. The results showed that the energy gap of epoxy is 4.7eV and this value decreased within range of (4.7-2.05)eV when concentration of TiO_2 microparticles increased from (2-8)wt.% respectively, Also the results showed that the optical constants which include the extinction coefficient (k), the refractive index (n), real part (ε_r) and imaginary part (ε_i) of dielectric constant increased with increasing the weight fraction of TiO_2 microparticles at wavelength 650nm.

INTRODUCTION

As part of what is witnessing our world today of technological development astonishing in various fields led industries requirements of modern applications and looks forward eagerly tremendously to new types of non-traditional materials, so-called advanced materials name, to being a modern materials are employed in advanced technological applications were not known than before, so the advanced applications that require materials to meet the unique properties of multiple may not be combined in a particular one of the known traditional materials such as metallic, ceramic and polymeric materials[1]. Composites are mixtures consisting of at least two phases of different chemical compositions with fillers or reinforcing fibers and a appropriate matrix, has been of great interest from both fundamental and practical standpoints, composites was used in making solar cells, optoelectronic device elements, laser diodes and light emitting diodes (LED), modern applications in aircraft, military and auto industry [2].

Polymer material such as a polar thermosetting epoxy resin was becomes a promising material for high performance and wide variety used for applications such as: coatings, electrical, automotive, marine, aerospace, and civil infrastructure as well as tool fabrication, pipes and vessels in chemical industry. Selective modification of epoxy matrices with filler particles has been successfully applied to adapt the materials to different physical, chemical or technical needs [3].

Reinforcing materials, for example, Titanium dioxide (TiO_2) has been generally utilized as a pigment and as a part of sunscreens, paints, ointments, toothpaste, and so on. it has been investigated recently for its interesting optical properties, electronic properties and stability in the adverse environmental for its high refractive index, wide band gap and chemical stability filler, A typically micron-sized, is incorporated into composite materials to enhance their properties [4].

One interesting aspect of filler content effect is on optical properties of the resin. When electromagnetic radiation or a ray of light interacts with composite surface, some of the light may be partly reflected and some partly refracted. The density of the filler determines how strongly the light is scattered within the material.

The study of optical absorption and especially the absorption edge is a useful method for investigate of optically induced the transformations and for the provide information about the band structure and energy gap in both crystalline and non crystalline materials [5].

The optical constants describe an electromagnetic wave in the medium of propagation; the refractive index n gives the phase shift of the wave, and the extinction coefficient or attenuation index k gives the attenuation of the wave. In practice, one often

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uses the absorption coefficient a instead of k because of the Beer's low formalism describing the absorption [6].

Absorption is defined as the relative rate of decreases in the light intensity along its propagation path which is expressed in terms of absorption coefficient (α) [7]. The fundamental absorption edge takes place when a photon of known energy excites an electron from valance band to the conduction band, which can be used to determine the energy gap of the semiconductor and estimated from absorption edges which are given approximately by the equation [8].

$$\lambda(nm) = \frac{1240}{E_g^{opt}(eV)} \quad \ldots\ldots\ldots\ldots\ldots (1)$$

Two types of optical transitions are direct and indirect transitions, both involve the interaction of an electromagnetic wave with the electron in valence band which may cross the forbidden gap to the conduction band [9]. The value of absorption coefficient was determined by the equation

$$\alpha = 2.303\left(\frac{A}{t}\right) \quad \ldots\ldots\ldots\ldots (2)$$

(Bougurs-Beer relation)

Where A is the absorbance, $A = \log\frac{1}{T}$,

T represents the transmittance $(\frac{I}{I_\circ})$ where I and $I_\circ$ are the intensities of the incident and transmitted beams respectively
t is the thickness of the sample

The extinction coefficient $\boldsymbol{K}$ (imaginary part of the refractive index) can be calculated by the relation [10].

$$K = \frac{\alpha\lambda}{4\pi} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where:

λ is the wavelength of the incident radiation.

As well as the refractive index of the sample can be determined from the relation when the reflectance (R) and the extinction coefficient ($\boldsymbol{K}$) are known [11].

$$n = [\frac{(1+R)^2}{(1 \quad R)^2} \quad (K^2 \quad 1)]^{\frac{1}{2}} \quad \frac{(1+R)}{(1 \quad R)} \quad \ldots\ldots\ldots (4)$$

The complex index of refraction is defined as [12].

$$n_c = n \quad iK \quad \ldots\ldots\ldots\ldots (5)$$

The complex dielectric constant is define as

$$\varepsilon = \varepsilon_r \quad i\varepsilon_i \quad \ldots\ldots\ldots\ldots (6)$$

Where ε_r is the real part of the dielectric constant and given by

$$\varepsilon_r = n^2 \quad K^2 \quad \ldots\ldots\ldots\ldots (7)$$

ε_i is the imaginary part of the dielectric constant and given by

$$\varepsilon_i = 2nK \quad \ldots\ldots\ldots\ldots. (8)$$

The optical energy gap can be determined when the refractive index is known (n) by using the relation

$$\frac{(n^2 \quad 1)}{(n^2+2)} = 1 \quad \sqrt{\frac{E_g^{opt}}{20}} \quad \ldots\ldots\ldots\ldots (9)$$

The Properties of Materials

In this experimental works the raw materials used to prepare the samples are listed below:

Matrix Materials

Low viscosity epoxy as a matrix (SBI) specialty Building Industries from (RAY ET Al EAMA' AR Company). The mixing ratio for resin and hardener is 3:2 and specific density 1.05 g/cm^3.

Reinforcing Particle

The reinforcing particle used to improve the optical properties of pure epoxy is Titania microparticles by (Cristal Globalpharma) with mean diameter of 50µm, purity99.99% and density3.9 g/cm^3.

Preparation of the Samples

The preparation steps of the neat epoxy and epoxy microcomposites for optical test were prepared by hand layup technique by high shear mechanical mixing process which can be summarized as follows:

Neat epoxy preparation

Epoxy resin and hardener are weighted by using Electronic balance type (Sartorius BL 210S/Germany) with sensitivity of 10^{-4}g and with rang up to 2Kg for suitable mixing ratio, then manually mixing the epoxy resin and hardener by shearing mixer for 15 minutes in container to have good homogeneity between epoxy resin and hardener. After that it was using vacuum system to remove the bubble before molding the epoxy. The samples were left for 48 hours before pulling out from molds and then they left for 7 days before any test to get better curing conditions.

Epoxy microcomposites preparation

Microocomposites were prepared with different weight percentage (2, 4, 6 and8%) of micro TiO_2 particles, the particles weighted with electronic balance of four digits type (Sartorius H51). A mixture of Epoxy resin with TiO_2 microparticles were mixed in a clean container using high shear mechanical mixer to obtain a fine dispersion of the fillers in the matrix, hardener was added to the mixture of epoxy resin and micro titania particles After that it was using vacuum system to remove the bubble before casting it as sheets (of dimensions 10x10cm^2), by using clean glass mould for casting .The casting was cured at room temperature for 24 hours. Sheets were casted in an oven for 1hour with temperature 50 ^{o}C, it was left for 48 hours before pulling out from molds and then left at room temperature for 7 days before processing further and kept then in vacuum chambers after that the specimens were cut for optical test.

RESULTS AND DISCUSSIONS

The optical energy gap and optical constants of epoxy and epoxy/TiO_2 (2, 4, 6 and8) wt. % at room temperature have been determined by using laser semiconductors with wavelength (650nm) from the measuring of the transmittance and absorbance of specimens, a pulsed laser was used to directly irradiate onto surface of the specimen and the direction of the laser beam is always perpendicular to the surface. Figure (1) represents absorption coefficient α (measured in cm^{-1}) which is calculated by using equation (2) verses the weight fraction of TiO_2 for Ep/TiO_2 microcomposites, the results show that the absorption coefficient increase with increasing the concentration of TiO_2 particles, the increased in absorption is revealed to the smooth reflecting surfaces of the specimens and there was not much scattering loss at the surface [13]. The results obtained shows that the values of absorption coefficient of Ep/TiO_2 micro- composites less than $10^4 Cm^{-1}$which indicates that the electronic transition was indirect, This agrees with results reported Bahaa *et al*[14].

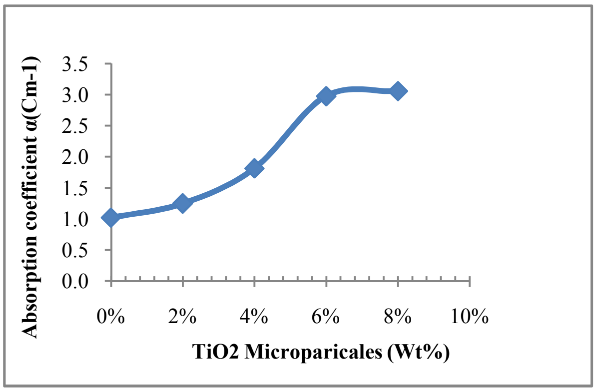

Figure 1 absorption coefficient vs. the weight fraction of $TiO_2$for EP/TiO_2microcomposites

Figure (2) illustrates the variation of the extinction coefficients (K) for epoxy and TiO_2/epoxy at the (650nm) with variation of TiO_2 concentration. The extinction coefficients can be calculated from equation (3) it is found that the values K increasing with increase concentration of additives TiO_2 in prepared samples, it increases from $0.52*10^{-5}$ for pure epoxy resin to $1.58*10^{-5}$ for 8 wt. % TiO_2concentration as shown in Figure (2). A result indicates that the dopant atoms of micro TiO_2 will modify the structure of the pure epoxy resin [15].

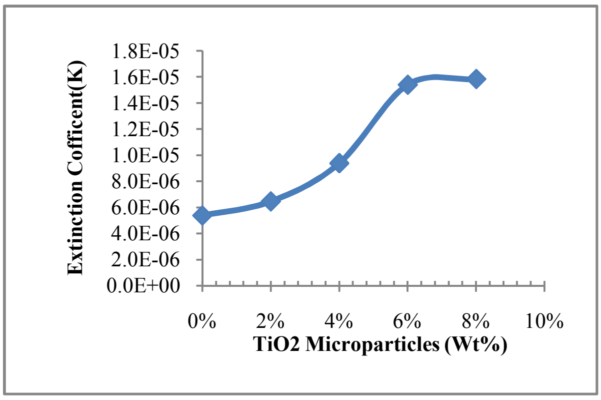

Figure 2 extinction coefficients vs. the weight fraction of $TiO_2$for EP/TiO_2microcomposites

The refractive index was measured for all specimens from equation (4) it was found that the value of the refractive index increases with increasing the weight percentage of the added TiO_2 to the epoxy as shown in Figure (3), it increases from 2.07 for pure epoxy resin to 2.7at 8wt. % of TiO_2 microparticles as illustrated in Table (1) which is a result of increasing the number of atomic refractions due to the increase of the linear polarizability in agreement with Lorentz - Lorentz formula [15]. As well as the reason of this result is from the increasing intensity of the resulting composite materials, in other wards as a result of increasing the number of free electrons [16].

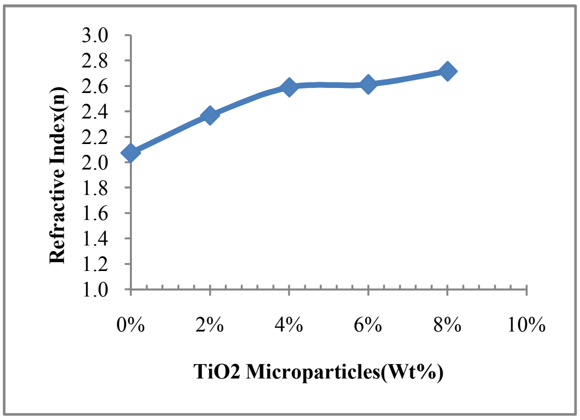

Figure 3 refractive index vs. the weight fraction of TiO_2forEP/TiO_2microcomposites

Figures (4and5) show the variation of real and imaginary parts of dielectric constants ($\varepsilon_r = n^2 - k^2$ and $\varepsilon_i = 2nk$) of Ep/TiO_2 microcomposites with variation of TiO_2 concentration .It is concluded that the variation of ε_r mainly depends on (n^2) because of small values of (k^2), while ε_i mainly depends on the (k) values which are related to the variation of absorption coefficients (α) [17].

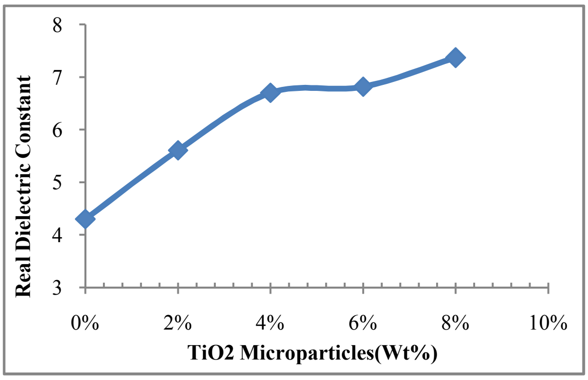

Figure 4 real part of dielectric constant (ε_r) vs. the weight fraction of $TiO_2$for EP/TiO_2microcomposites

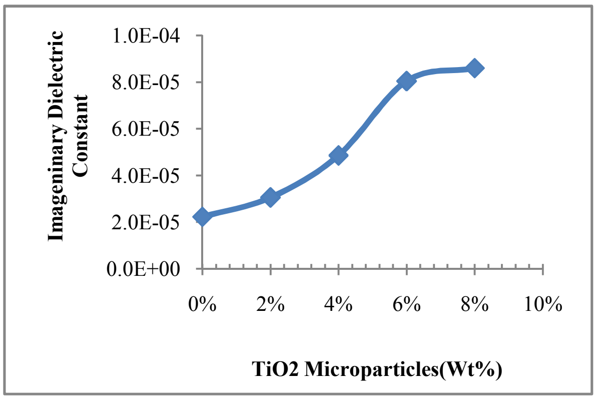

Figure 5 imaginary part of dielectric constant (ε_r) vs. the weight fraction of $TiO_2$for EP/TiO_2microcomposites

The optical energy gap calculated according to the relationship (9). The Table (1) shows the value of optical energy gap for pure epoxy and epoxy/TiO_2 composites and Figure (6) represents the variation of optical energy gap for pure epoxy and epoxy/TiO_2microcomposites with the variation of TiO_2 content; it has been found that the value of energy gap decreases with increasing the concentration of TiO_2 microparticles , thus the increase additive causes the reduction of E_g values since the structure will be approach from that of bulk material[18]. We note higher value obtained of optical energy gap for the pure epoxy resin about (4.5eV), the results obtained agree with results reported by other works [14, 19].

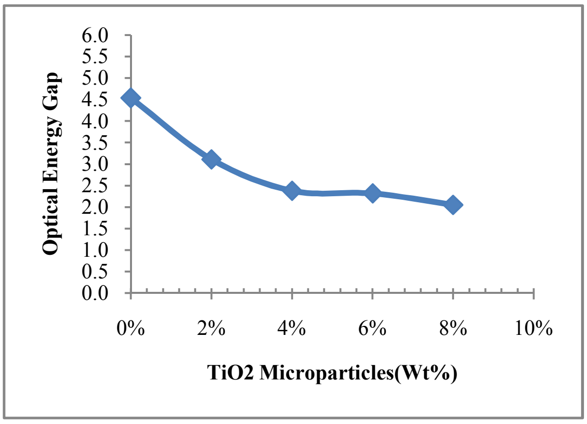

Figure 6 Optical energy gap E_g^{opt} vs. the weight fraction of $TiO_2$for EP/TiO_2microcomposites

Table 1 The values of (α, n, ε_r and ε_i) and E_g^{opt} for epoxy and epoxy/TiO_2 at λ=650 nm

Material	α(Cm^{-1})	n	ε_r	ε_i*10^{-5}	$E_g^{opt}(eV)$
EP	1.019	2.07	4.3	2.2	4.53
EP/2%TiO_2	1.248	2.36	5.6	3.0	3.1
EP/4%TiO_2	1.813	2.58	6.7	4.8	2.37
EP/6%TiO_2	2.977	2.61	6.8	8.0	2.31
EP/8%TiO_2	3.058	2.71	7.3	8.6	2.05

CONCLUSION

1. The absorption coefficient of pure epoxy increases with increasing of micro titanium oxide (TiO_2) concentrations.
2. From the results obtained shows that the values of the absorption coefficient (α) of Epoxy/ TiO_2microcomposites less than ($10^{-4}Cm^{-1}$) this indicates indirect electronic transitions.
3. The optical constants which include the refractive index (n), the extinction coefficient (k), real (ε_r) and imaginarily (ε_i) part of dielectric constant at different concentration of TiO_2 particles. It increased with increasing of microTiO_2 concentrations and the values take at λ=650nm.
4. The optical energy gap of pure epoxy decreases with increasing of microTiO_2 concentrations.

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