Study of the Effect of Doping with Alumina and Silica the Structural and Electrical Properties of Epoxy

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Abstract. Crystalline structure of a sample of Alumina (Al2O3 – reinforced epoxy) at (4% and 8%) has two prominent peaks at point (17°) and (19.5o) with miller vectors (111) and (220), respectively. These results are corresponding to crystalline cubic and notes growing of crystallization. The spectrum of (XRD) showed increase in crystalline size which means an increase in randomization and a lack of crystallization amorphous at (6%) (Al2O3). This result showed increase in crystalline size which means an increase in randomization and a lack of crystallization amorphous. Epoxy matrix reinforced by particles of silica (SiO2) and alumina (Al2O3) with different fractions were investigated for electrical properties such as dielectric constant, conductivity and dielectric loss index. This result showed effect of additional (SiO2, Al2O3) is small concentration except (2% SiO2) has large effect.

Key words: XRD, epoxy composite, dielectric constant, conductivity dielectric loss index

1. Introduction

X-Rays are electromagnetic radiation of exactly the same nature as light but of very much shorter wave length. When fast moving electrons are incident on a metal target, the electrons beam losses its energy due to collision with the target. This results in the emission of x-ray [1].

Crystal structure of the composite epoxy:

A sample of Alumina (Al2O3-reinforced epoxy).

A sample of silica (SiO2 – reinforced epoxy).

Were characterized by X-ray diffraction (X-ray photoelectron spectroscopy) [2, 3].

Ceramic materials can be defined as inorganic materials constituted by the combination of metallic and non metallic elements whose properties depend on the way in which these elements are
Polymer-particle composites have received considerable interest in the material field because of their potential for large gains in mechanical properties however; polymers are usually combined with filler materials to improve mechanical, thermal, electrical properties and stability [5, 6]. Composites which consist of ceramic (SiO$_2$, Al$_2$O$_3$) and polymer epoxy have attracted a lot of cancer in industrial, because their theatrical advance and application, that can be synthetic in their properties by mutating their type and amount of dispersion[7,8]. Ferro electronic ceramic have very high dielectric constant are brittle and undergo from poor mechanical, strength, more over polymer having low dielectric constant in the range (2 to 5) traditionally used in low leakage capacitors are flexible easy to process and possess high dielectric strength Method. Composites connected with high dielectric strength to achieve high volume efficiency and energy strong density for application at capacitor and electric energy storage device [9, 10, and 11].

Research of the dielectric properties of polymers have increased importance because it provide an understanding to movement of molecular chains and its implementation in electrical and electronic engineering [12]. Epoxy resins are mightily cross linked unequal polymers used for insulation electric transformers; switch gear, etc [13]. The study of dielectric is main in nature and offers a consolidated understanding of many other majors in materials science [14].

In this Paper the conductivity electrical equation was studied

$$\sigma_{total} = (d |A| G)$$  \hspace{1cm} (1)

Where

(d):- is the thickness of the measured sample.

(G):- is the sample conductor iee.

(A):- is the cross sectional area.

The dielectric constant ($\epsilon$) was calculated from the equation

$$\epsilon = C/C_0$$  \hspace{1cm} (2)

Where

(C):- is the capacitance of the electrons with dielectric.

(C0):- is the geometrical capacitance of the same without dielectric.

$$C_0 = \epsilon_0 (A/d)$$

Experimental Work

Was used to synthesize the sample preparation process

1) A sample of alumina (Al$_2$O$_3$). reinforced epoxy.

2) A sample of silica (SiO$_2$) – reinforced epoxy.

In this process , the use of epoxy resin was used as a base material , which is a liquid into a solid state (2 :1 ) .the resin is added to the rein forcing material and mixed with it before herding . After the completion of the solidification and the molds all with a time of 24 hours the casting are extracted from
the molds and then the process is treated for a temperature of 50 °C and for 2 hours to complete the chemical reactions. the molds are then cut to extract the samples of the electrical tests and x-rays according to the standard specifications of the samples. In order to explain the structural characteristic of epoxy material mixing of (Al₂O₃) and (SiO₂) were analyzed by a Philips pw1050 X-ray diffract meter of (1.54 Å, voltage 30 kV, current 15 mA), from Cu- Ka radiation. The nature and crystal growth of samples prepared at different percent was characterized using X-ray diffraction (XRD). This technique was also employed by another group which gives an indication about the crystalliner size and formation material type of the prepared particles.

2. Result and Discussion
Figure (1 & 2) explains the spectrum of (XRD) pattern for epoxy material equipped with different concentrations mixing of (Al₂O₃) and (SiO₂) (0%, 2%, 4%, 6%, 8%) respectively, at room temperature as a function to the bragg angle for the range from (10°) to (60°). It is noted in figures (1 & 2) that the apparent peak [miller vector (100)] of all grafted samples is wide that, due to the nanostructure of these samples [15,16]. In addition, this figure (1-a) indicates that amorphous structure at the peak (18°) with a wide band width for a pure epoxy.

From Debage –Cherrera equation [2, 3] the value of (D) will be calculated and it equal to:-

\[ D = \frac{K \lambda}{\beta_{hkl} \cos \theta} \]

\[ D = \frac{0.94 \lambda}{\beta_{hkl} \cos \theta} \] .......................... (3)

Where:
β: The intrinsic full width at Half Maximum of the peak (Fwhm).
D: The Crystalline size.
λ: The wave length of incident X-ray radiation.
Θ: The Bragg’s diffraction angle of the respective XRD peak.
K: The shape factor =0.94.

In addition, the stress was calculating using equation:

\[ \tau = \frac{\beta \cos \theta}{4} \]  (Lines-2. m-4)  ...................... (4)

And the intensification of the interferences can by determined using this equation:-

\[ [ \sigma = \frac{1}{D^2} ] \] (Lines. m-2)  ...................... (5)

In the case of adding a different percentage of (Al₂O₃) (0%, 2%, 4%, 6%, 8%), respectively. It was observed that the crystalline size (D) was decreased but the stress and intensification of interferences were increased at (4%) and (8%) of a sample of Alumina as recorded in table (1). However, the ratio of impurity at (4%) and (8%) of (Al₂O₃ - reinforced epoxy), amulet crystalline structure has two prominent peaks at point (17°) almost and (19.5°); which match with miller vectors (111) and (220), respectively as shown in figure(1-b) and (1-d). These results are corresponding to the international specifications of the material with crystalline cubic. Additionally; during adding (6%) (Al₂O₃) to pure epoxy ;The spectrum of (XRD) showed that the structure was single crystalline and defined at peak (22°) represents
the level (111), as shown in figure (1-c). This result showed increase in crystalline size which means an increase in randomization and a lack of crystallization amorphous except the case (4%) and (8%) of (Al₂O₃- reinforced epoxy) it notes growing of crystallization however angles value constant at (17°) and (19.5°) and that are organization of crystalline structure.

Figure (1) XRD pattern of the composite epoxy, a sample of Alumina (Al₂O₃- reinforced epoxy) (a) pure epoxy (b) 4% of Al₂O₃ (c) 6% of Al₂O₃ (d) 8% of Al₂O₃.

Figure (2) shows adding silica (SiO₂) to pure epoxy according to the following percentage (0%, 2%, 4%, 6% and 8%); respectively. At (2%) of silica (SiO₂ – reinforced epoxy), nothing changed in crystalline size and the compound become amorphaces as shown in figure (2-b). While the last addition of a sample of silica about (4%), (6%) and (8%), differentiated peaks (single crystallization) by one at the same angle of (22°) and level (111) as shown in figure (2-c), (2 – d), (2 – e).

Figure (2) XRD pattern of the composite epoxy, a sample of silica (SiO₂ - reinforced epoxy) (a) pure epoxy (b) 2% of SiO₂ (c) 4% of SiO₂ (d) 6% of SiO₂ (c) 8% of SiO₂.
Table (1) Coefficient determined for the empirical equation (crystalline size, stress, intensification)

| Compounds          | $D$ (nm) | $\eta$ (lines$^{-2}$m$^{-4}$) | $\sigma$ (lines$^{-2}$m$^{-2}$) |
|--------------------|----------|-----------------------------|---------------------------------|
| pure epoxy         | 32.37636 | 0.0010702                   | 0.0009539                       |
| epoxy + alumina 4% | 1.77109  | 0.006287                    | 0.032926                        |
| epoxy + alumina 6% | 5.510934 | 0.01955                     | 0.318832                        |
| epoxy + alumina 8% | 1.77109  | 0.01955                     | 0.318832                        |

In order to understand the effect of concentration of reinforcement of ceramic on the dielectric properties. Figures (3, 4 and 5) show that relation between the dielectric constant, dielectric loss index and conductivity as a function to concentration of addition (2%, 4%, 6% and 8%) of (SiO$_2$) and (Al$_2$O$_3$) respectively, 1MHz from these figures we can find the effect of additional (SiO$_2$, Al$_2$O$_3$) is a small in concentration except (2% SiO$_2$) has large effect, this behavior clearly show that there are highly variation and non linear phenomena this can be explain because the mechanism of reaction between epoxy and (SiO$_2$), and the dielectric properties of (epoxy, Al$_2$O$_3$) composites is linear and similar behavior this because effect of many parameters come from structured formation of these material.

![Figure (3)](image-url)  
**Figure (3)** Dielectric constant with (SiO$_2$, Al$_2$O$_3$) filler weight

![Figure (4)](image-url)  
**Figure (4)** Dielectric losses index with (SiO$_2$, Al$_2$O$_3$) filler weight
3. Conclusion
   1. The summarized results from this work are as follows:
   2. The X-ray diffraction results are corresponding to crystalline cubic and notes growing of crystallization at (4% and 8%) of a sample of alumina (Al₂O₃) reinforced epoxy.
   3. Little emphasis for (SiO₂-reinforced epoxy) has a great influence on all properties.
   4. The graphs indicate an non-linear relationship due to the reactive mechanical effect between epoxy and particulate support.
   5. There is little effect of the concentration (SiO₂, Al₂O₃) on electrical properties except for the concentration (2% SiO₂).

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