Volume Resistivity and Mechanical Behavior of Epoxy Nanocomposite Materials

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***Abstract*-Electrical and mechanical properties of polymer composite materials are investigated through the determination of resistivity and hardness for composites samples. Epoxy composite samples have been prepared with different concentrations of certain inorganic fillers such as; Titanium dioxide (TiO_2) and Silica (SiO_2), of various size (micro, nano and hybrid) to study the electrical and mechanical behavior. The volume resistivity reaches 3.23×10^{14} ohm.cm for the micro silica composite. Surface of composite material has been mechanically examined by hardness test. The results show that the resistivity of microcomposites and nanocmposites are increased with the decrease of filler concentration. But the resistivity of hybrid composites is increased with the increase of filler concentration. Maximum hardness value was obtained from hybrid silica composite with 0.1% filler concentration.**

Keywords: Volume Resistivity; Polymer Composite Materials; Micro-Nano-Hybrid Filler; Hardnes

I. INTRODUCTION

During the last years, alternative materials (instead of porcelain and glass) for outdoor insulation have been used. The new polymer materials are presently very widely used for outdoor insulation applications. Polymer materials such as silicone rubber and epoxy composites are used in outdoor insulators. The maintenance of polymer insulators is easy at the electrical site within small period. Due to the high surface areas of the nano fillers and their molecular-level interactions with the polymer chains, there is a great interest in nanocomposites due to significant scientific questions relating to interfacial chemistry and physics as well as their greatly enhanced practical properties [1]. The incorporation of nanoparticles such as Al_2O_3, TiO_2, and SiO_2 into a polymer matrix can lead to a simultaneous improvement of different material properties [2-4]. Especially toughness and stiffness of the nanocomposite can be enhanced at the same time, which is not possible for conventional composites with micro or even macro scale fillers to the same degree and for the same low filler contents [5, 6]. Due to the high specific surface (relation of the surface to the mass) that mainly distinguishes nanoparticles from microparticles, their insertion into matrix materials can provide completely new properties compared to conventional composite materials [7–10].

In order to develop a new insulation material for heavy electric equipment, epoxy/micro- and nano- composites (EMNC) were prepared by mixing micro-sized silica with nano-sized layered silicate [11]. Epoxy resin is one of the most commonly used thermosetting materials in high voltage apparatus as insulation, due to its excellent mechanical, electrical properties and chemical stability. In recent years, the nano reinforced epoxy resin has attracted a wide interest as it significantly enhances the epoxy's properties. Researches on nano reinforced epoxy resin composites are being carried out ceaselessly. Research has shown that the epoxy/nanocomposites demonstrate some advantages in both mechanical and dielectric properties, compared to pure resin system and epoxy with micrometer-size fillers [12].

Hardness, a measure of a material's resistance to localized plastic deformation (e.g., a small dent or a scratch), is considered an important mechanical property. Early hardness tests were based on natural minerals with a scale constructed solely on the ability of one material to scratch another that was softer [13]. An analytical relation between hardness and effective strain is established on a metal during cold working. This way, its hardness can be determined from numerically obtained plastic strains without producing the part and following ASTM2240 D [14].

In this study, volume resistivity and hardness properties have been measured for different epoxy composite samples using micro, nano and hybrid titanium dioxide and silica fillers.

II. EXPERMINTAL SET-UP AND TECHNIQUES

A. Material specimen

Epoxy package contains two free liquid solvent component resins. Component A is a medium viscosity liquid (epoxy resin) and Component B (hardener) is a difunctional primary amine. The used fillers are titanium dioxide (TiO_2) and silica (SiO_2) in

micro and nano size. The micro size range is less than 20 μm and the nano size particles are less than 100 nm. A mixture of the two components, A and B in the ratio of (3:1 wt. /wt. %) were blended thoroughly using a magnetic stirrer for 3 minutes. The mixture was degassed under vacuum to remove air bubbles, and then poured into a plastic mold. Curing occurred after 24 hours at room temperature (25˚C ±1). The sample is stable to machine after 7 days.

Epoxy composites were prepared by mixing different ratios (0.1 up to 7 wt. % by weight) of titanium dioxide (TiO_2) and silica (SiO_2) as fillers, in micro and nano size with epoxy/hardener mixture [15]. Also Epoxy composites were prepared from hybrid fillers (different types and concentration of titanium dioxide (TiO_2) and silica (SiO_2)). The prepared samples were molded in disc shapes. The disc specimen is coded in Table I and Table II due to the type, size and concentration of the added filler. The titanium dioxide and silica filler has different size in micro (M), nano (N) and hybrid (M/N) and also concentration as shown in Tables I and II.

TABLE I. CODE OF EPOXY COMPOSITE SAMPLES AT DIFFERENT TYPE AND CONCENTRATION OF FILLERS.

Filler Concentration (Wt. %)	**Filler Type**			
	Micro TiO_2	**Nano TiO_2**	**Micro SiO_2**	**Nano SiO_2**
0.1	$Ti_{M0.1}$	$Ti_{N0.1}$	$Si_{M0.1}$	$Si_{N0.1}$
0.25	$Ti_{M0.25}$	$Ti_{N0.25}$	$Si_{M0.25}$	$Si_{N0.25}$
0.5	$Ti_{M0.5}$	$Ti_{N0.5}$	$Si_{M0.5}$	$Si_{N0.5}$
0.75	$Ti_{M0.75}$	$Ti_{N0.75}$	$Si_{M0.75}$	$Si_{N0.75}$
1	Ti_{M1}	Ti_{N1}	Si_{M1}	Si_{N1}
3	Ti_{M3}	Ti_{N3}	Si_{M3}	Si_{N3}
5	Ti_{M5}	Ti_{N5}	Si_{M5}	Si_{N5}
7	Ti_{M7}	Ti_{N7}	Si_{M7}	Si_{N7}

TABLE II. CODE OF HYBRID COMPOSITE SAMPLES AT DIFFERENT TYPE AND CONCENTRATION OF FILLERS.

Filler size and concentration wt%	**Filler type**		
	TiO_2/ SiO_2	**TiO_2**	**SiO_2**
Nano 0.05/Nano 0.05	$Ti_N/Si_{N(0.1)}$		
Nano 0.375/Nano 0.375	$Ti_N/Si_{N(0.75)}$		
Micro 0.05/Nano 0.05	$Ti_M/Si_{N(0.1)}$	$Ti_{M/N\ (0.1)}$	$Si_{\ M/N\ (0.1)}$
Micro 0.375/Nano 0.375	$Ti_M/Si_{N(0.75)}$	$Ti_{M/N(0.75)}$	$Si_{\ M/N\ (0.75)}$
Nano 0.05/Micro 0.05	$Ti_N/Si_{M(0.1)}$		
Nano 0.375/Micro 0.375	$Ti_N/Si_{M(0.75)}$		
Micro 0.05/Micro 0.05	$Ti_M/Si_{M(0.1)}$		
Micro 0.375/Micro 0.375	$Ti_M/Si_{M(0.75\}}$		

The samples dimensions were limited and chosen due to the limitations of the capabilities of available measuring devices in the high voltage laboratory. The dimension of each disc specimen is 50×2 mm for resistivity test and 50×6 mm for hardness test.

B. Test Apparatus

Resistivity test is applied on disc samples for measuring the volume resistivity indirectly using a resistance tester (high voltage insulation tester) and a circular ring electrode, (Figure 1) [16, 17].

Fig.1. High voltage insulation tester

Hardness test measure the ability to resist plastic deformation by penetration. This test is based on measuring the penetration of a specified type of indenter when forced into the specimen under specified conditions. The test was performed according to ASTM D 2240 standards using hardness tester (Zwick 3150, Germany) (Figure 2) [18].

Fig.2. Hardness tester

C. Measurement and uncertainty

The result of a measurement is only an approximation or estimate of the true value of the specific quantity measured. The sources of uncertainty in the results of a measurement can be affected by many factors as following:

- reference standards and measurement equipment
- measurement setup
- measurement process
- environmental conditions

Misreading of instrument results, incorrect adjustments, using the wrong instrument, errors in recording calibration data, and computational errors are also common uncertainty factors. All of these errors can be avoided with proper training and attention to detail [19].

The uncertainty of measurement associated with the input estimates is evaluated according to either a "Type A" or a "Type B" method of evaluation. The "Type A" evaluation of

standard uncertainty is the method of evaluating the uncertainty by the statistical analysis of a series of observations. The "Type B" evaluation of standard uncertainty is the method of evaluating the uncertainty by means other than the statistical analysis of a series of observations [20].

In this case the input estimate x_i is usually the arithmetic mean or the average of the individual observed values that is given by:

$$x_i = \overline{X} = \frac{1}{n}\sum_{k=1}^{n} x_{i,k} \qquad (1)$$

$$u(x_i) = s(\overline{X}) = \frac{s(X_i)}{\sqrt{n}} \qquad (2)$$

where:

$$s(X_i) = \sqrt{(\frac{1}{n-1}\sum_{k=1}^{n}(x_{i,k} - \overline{X}_i)^2)} \qquad (3)$$

When the number of repeated measurements is low (n<10), Type A evaluation of standard uncertainty is used as expressed in (2). It should be multiplied by the student, s factor (t) which is selected due to number of samples n for a required confidence level p.

$$U(X_i) = t \cdot s(\overline{X}_i) = t \cdot \frac{s(X_i)}{\sqrt{n}} \qquad (4)$$

If the probability distribution is characterized by the measurement result y and its combined standard uncertainty u(y) is approximately normal (Gaussian), and u(y) is a reliable estimate of the standard deviation of y, then the interval [y-u(y)] to [y+u(y)] is expected to encompass approximately 95% of the distribution of values [21].

Although the combined standard uncertainty u(y) is used to express the uncertainty of many measurement results, for some commercial, industrial and regulatory applications (e.g., when helth and safety are concerned), what is often required is a measure of uncertainty that defines an interval about the measurement result y within which the value of the measured Y can be confidently asserted to lie. The measure of uncertainty intended to meet this requirement is termed expanded uncertainty, suggested symbol U, is obtained by multiplying u(y) by a coverage factor (k).

$$U = k \cdot u(y) \qquad (5)$$

It is confidently believed that Y is greater than or equal to [y-U] and is less than or equal to [y+U], which is commonly written as [Y=y±U]. In general, the value of the coverage factor k is chosen on the basis of the desired level of confidence to be associated with the interval defined by the equation (5) [21].

III. RESULTS AND DISCUSSION

A. *Electrical resistivity of epoxy composites at different type for micro and nano fillers.*

The addition of an inorganic filler to epoxy means that the ionic particle increases in the polymeric composites. If the bond between the particle of filler and epoxy resin is weak, the ionic particle will be liberated at increasing the filler concentration. When filler concentration increases, the resistivity will be decreased for epoxy composites. The electrical conductivity of composites made of a conductive phase dispersed in an insulating matrix critically depends on the filler loading [22]. The resistivity test is considered nondestructive because the specimen is tested by passing current through the sample using DC high voltage supply. The color of all samples under investigation remains the same and there is no change to be found regarding the exterior surface after the test (Figure 3). The resistivity is measured for composite samples which have micro, nano or hybrid filler (Figure 4). The epoxy composite with 0.1% micro silica filler records maximum resistivity at 3.23×10^{14} ohm.cm. The resistivity for micro titanium composite is 1.3 times the resistivity of micro silica composite at 3% filler concentration. Epoxy composite loaded with 0.1% micro filler is larger than epoxy which records 2.75×10^{14} ohm.cm (Figure 4).

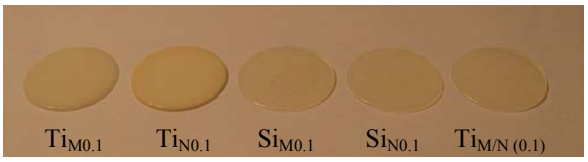

Fig.3. The specimens at different type and size of filler

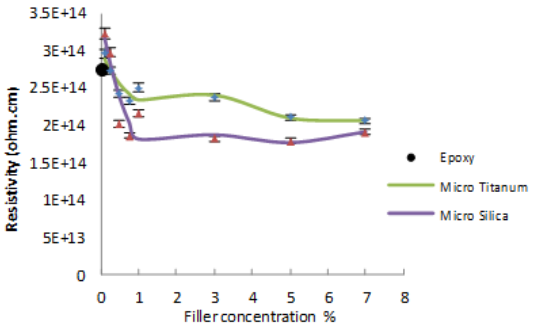

Fig.4. Electrical resistivity of micro composite loaded with different type and filler concentration.

For epoxy loaded with filler concentration larger than 3%, the resistivity for nanocomposite with titanium oxide is larger than the resistivity of microcomposite for the same filler as shown in Figure 5 due to distributed nano filler uniformly in epoxy matrix at preparation but the micro filler particle is accumulated with other particles when filler concentration increase. The behavior of resistivity for nanocomposites depends on the physical interaction between the epoxy and filler. The micro particle filler is concentrated with other particles in the mixture which gives bad filler distribution. On the other hand the distribution of nano filler in composite is uniformly distributed. Nanocomposites have good insulation properties comparing with microcomposites due to the material resistivity of titanium oxide and silica as fillers.

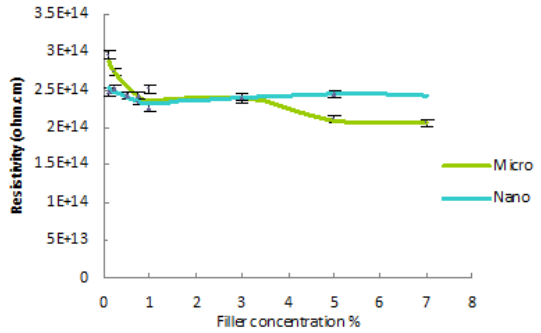

Fig.5. Electrical resistivity of epoxy composite filled with titanium dioxide at different size and concentration.

Nanocomposites resistivity has maximum value at wide range of filler concentration comparing with microcomposites (Figure 6). This is referring to the chemical structure of composites. The resistivity of nano silica composite is decreased by 24.37% when the filler increase from 1% up to 5% due to the percolation theory of material. Percolation point illustrates the characteristic of epoxy composites at increasing the filler which leads to transform the material from insulator to conductive. The sharp decreasing of resistivity for micro silica composite is referring to increase filler up to critical concentration which is called the percolation threshold point.

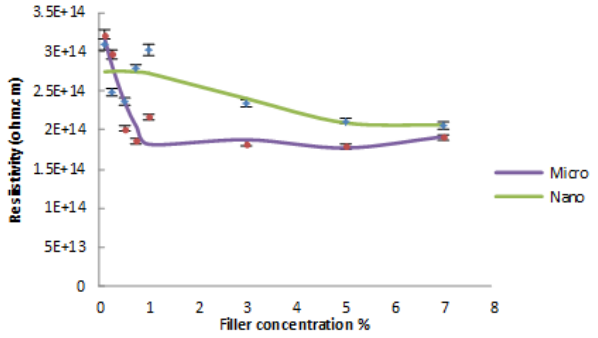

Fig.6. Electrical resistivity of epoxy composite filled with silica at different size and concentration.

B. Application of uncertainty concept in the resistivity test and hardness test.

The resistivity test is applied for polymer composites to evaluate the electrical property of composite materials and the hardness test is employed on polymer composites to illustrate the mechanical behavior of composites. The electrical test and mechanical test are repeated on four samples which have the same content. The uncertainty concept is used to detect the error in reading and how this result to be applicable and error within range. The error U is calculated with (1)-(5). If the number of similar samples (n) is equal to four and also student factor $\rightarrow$k, the coverage factor (k) will be 3.18 at confidence level 95%. Table III illustrates the final results in error calculation in value and percentage.

There is four samples with the same chemical feature, the resistivity ρ for this samples represent the mean advised value for this type and also has error U in form $\rho=\rho\pm U(\rho)$. The hardness for composite samples is detected through the mean advised value for this type in addition to error U in form Hardness= Hardness ±U (Hardness). For uncertainty principle, the suggested confidence level in 95% is suitable with the final results in error at range 5%. The calculated error is detected under uncertainty concept and in range for resistivity and hardness test.

C. Electrical resistivity for epoxy composites at different type and size for hybrid fillers.

The electrical behavior for epoxy hybrid composites depends on the added filler. Therefore the filler contains two types with different particle size (hybrid filler). The main purpose of filler addition is the improvement of electrical performance and mechanical feature. The resistivity of composite with hybrid filler is maximized comparing with nano and micro composite. Figure 7 illustrates that the maximum resistivity will be detected for composites with 0.1% hybrid filler from mixing of micro titanium dioxide and nano silica by 50:50 weight. The filler which is added to composite contains two different materials (titanium dioxide and silica) in micro size or in nano size. If this hybrid filler concentration increases, the resistivity will be decreased at any size of filler as shown in Figure 8. The hybrid filler used contains two different particle sizes for same silica material. The resistivity of hybrid composite is shown in Figure 9. If this filler concentration increases, the resistivity will decay.

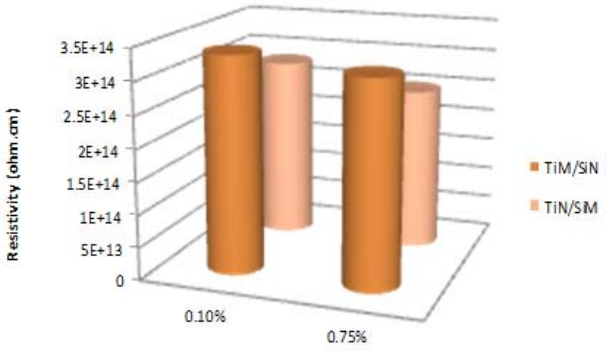

Fig.7. Electrical resistivity of hybrid composite with two different materials and size at various filler concentration.

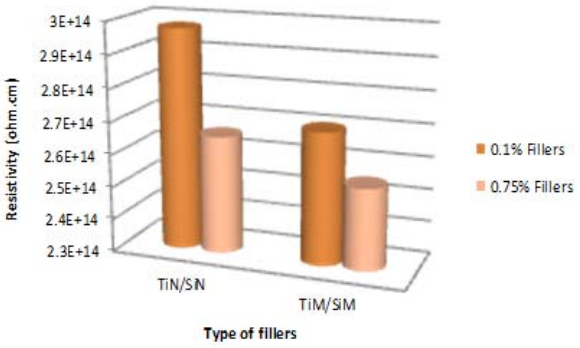

Fig.8. Electrical resistivity of hybrid composite with two different materials at same particle size

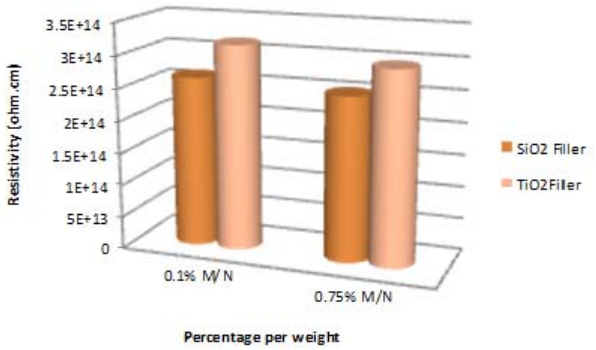

Fig.9. Electrical resistivity of hybrid composite with two materials at same type and various particle sizes.

D. Effect of filler type and size on the hardness of epoxy composites.

Mechanical properties are detected by static mechanical tests and dynamic mechanical analysis. One of the important tests in static analysis is hardness test. Hardness is indicated by a static indentation test which means that a ball, cone, or pyramid is forced into the surface of the metal being tested. The relationship of load to the area or depth of indentation is the measure of hardness, such as in Brinell, Knoop, Rockwell, and Vickers hardness tests. When the indentation is small, material hardness will be large. Hardness is a mechanical property, which can be dramatically changed by processing and heat treatment [23]. Hardness test is applied on the samples according to the ASTM D 2240 standard. Hardness of composite samples is measured by a digital hardness tester which is used to determine the hardness of soft plastic and epoxy is found on scale shore D [16].

The hardness test is applied on four samples with the same content to achieve minimum error in results and get good practical results at scale shore D. The hardness results are shown in Tables III and IV. Micro composites have hardness larger than the Nano composites. Epoxy filled with micro titanium and silica filler records maximum hardness at 85.3% and 86.7% shore D scales respectively (Table III). The average of hardness is considered for four samples with the same content and error percentage due to uncertainty concept within maximum error at 6.29%. Hybrid silica composite which contain different filler size in micro and nano has maximum hardness at 87.43% and is larger than hybrid composite (Ti_M/Si_N (0.1)) with 2.76% only at shore D scale.

IV. CONCLUSION

Micro filler is added to epoxy with minimum concentration 0.1% in order to get maximum resistivity at 3.23×10^{14} Ω.cm and other hand, Hybrid composites which contains mixture from nano titanium and nano silica filler with 0.1% concentration has at 3.35×10^{14} Ω.cm. The resistivity of epoxy composites decrease if the filler concentration increases regardless of filler size and concentration due to percolation theory. Hardness of hybrid composite material is more strength than microcomposite and nanocomposite. Hybrid silica composite using 0.1% mixture of micro and nano size gave maximum hardness at 87.43% shore D.

TABLE III. THE CALCULATION OF DIFFERENT FILLER CONCENTRATION.

Composites using titanium filler			Composites using silica filler		
Sample	**Hardness %**	**U %**	**Sample**	**Hardness %**	**U %**
Epoxy	78.5	1.97	**$Si_{M0.1}$**	80.3	3.16
$Ti_{M0.1}$	79.2	0.58			
$Ti_{M0.25}$	79.8	2.06	**$Si_{M0.25}$**	82.2	0.84
$Ti_{M0.5}$	85.3	6.08	**$Si_{M0.5}$**	83.1	1.84
$Ti_{M0.75}$	82.7	1.91	**$Si_{M0.75}$**	86.7	2.61
Ti_{M1}	82.3	1.98	**Si_{M1}**	86.7	1.11
Ti_{M3}	79.2	4.32	**Si_{M3}**	84.7	1.27
Ti_{M5}	80.2	2.66	**Si_{M5}**	84.8	1.28
Ti_{M7}	83.1	3.42	**Si_{M7}**	84.8	1.35
$Ti_{N0.1}$	75.9	2.10	**$Si_{N0.1}$**	86.15	4.04
$Ti_{N0.25}$	78.2	2.39	**$Si_{N0.25}$**	84.35	4.54
$Ti_{N0.5}$	77.4	2.24	**$Si_{N0.5}$**	85.35	3.74
$Ti_{N0.75}$	78.2	1.66	**$Si_{N0.75}$**	85.15	2.03
Ti_{N1}	78.4	5.12	**Si_{N1}**	78.83	3.42
Ti_{N3}	84	6.00	**Si_{N3}**	77.55	6.20
Ti_{N5}	81.2	1.78	**Si_{N5}**	81.75	3.29
Ti_{N7}	81.1	3.73	**Si_{N7}**	83.30	5.44

TABLE IV. THE CALCULATION OF UNCERTAINTY FORHYBRID EPOXY COMPOSITES

Samples	Hardness%	U%
$Ti_N/Si_{N(0.1)}$	81.60	6.29
$Ti_N/Si_{N(0.75)}$	76.13	3.45
$Ti_M/Si_{N(0.1)}$	85.08	2.03
$Ti_M/Si_{N(0.75)}$	76.38	3.55
$Ti_N/Si_{M(0.1)}$	80.93	1.73
$Ti_N/Si_{M(0.75)}$	76.13	4.16
$Ti_M/Si_{M(0.1)}$	82.45	1.71
$Ti_M/Si_{M(0.75)}$	75.08	2.47
$Ti_{M/N\ (0.1)}$	78.53	3.63
$Ti_{M/N(0.75)}$	82.58	6.23
$Si_{M/N\ (0.1)}$	87.43	1.01
$Si_{M/N\ (0.75)}$	77.83	1.31

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