Effect of Nano TiO$_2$ on Static Fracture Toughness of Fiberglass/Epoxy Composite Materials in Hot Climate regions

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Abstract. This work is an experimental and numerical approach to analyze the effect of Nano powder additives and temperature on the critical crack length in fiber reinforced composite materials (FRC) exposed to uniaxial tensile load. Fiberglass/Epoxy compact tension test samples (CT) are prepared according to ASTM standard E399. TiO$_2$ Nano powder is added by 2% wt to the resin to other samples. The static tensile test is applied to the samples and the results for crack length/time are discussed. ABAQUS/CAE software is used to simulate the case. The numerical and experimental results were close. The experimental results showed that the addition of 2%wt TiO$_2$ Nano powder increased the critical crack length by 32.4%. The effect of ambient temperature is studied too by applying the experimental test on the fiberglass/Epoxy samples in room temperature (30˚C) once and at outdoor (60˚C) temperature. The results showed that the effect of the temperature increase decreased the critical crack length by 51%. The numerical results determined the fracture toughness ($K_{IC}$) for the fiberglass/Epoxy samples to be 491 Pa.mm$^{0.5}$ and 517 Pa.mm$^{0.5}$ for the fiberglass/Epoxy-Nano powder samples.

1. Introduction

Fiber reinforced composites (FRC) are widely used for structural components generally in many applications and especially in aerospace applications and wind turbine blades. These structures are facing different types of loads, as regular mechanical loads or occasional like thermal loads from repeated temperature fluctuations within duty life. Temperature fluctuation may cause effective internal thermal stresses in composites, particularly in cross-ply arrangements. According to Narayanan [1], the thermal expansion coefficients exceeds $3\times10^{-5}$/˚C for a range of 0˚ and 90˚ producing 20 MPa or maybe more thermal stresses. Fatigue of composites arises by regular mechanical or thermal loads or both [2]. Researchers proposed several models for fatigue behavior analysis. Eiselun et al. [3] discussed the effect of micro-cracks in the risen generated from thermal cycling, on the tensile strength, the fatigue life, and Interlaminar shear strength. During thermal cycles, Cohen et al. [4] noticed that the cracks concentrate up to the concise cycles' number remain constant, and increased later. In their study, Herakovich and Hyer [5] dealt with thermal cycling crack density as a function of transverse plies number and layer thickness, one of their conclusions; they noticed extensive damage in the matrix (epoxy resin) that included delamination. Adams et al. [6] cleared the effect of the space environment on composites with cross-ply laminates. They concluded that the cracks try to avoid the rich areas of resin and delamination grows from transverse cracks. Kellogg et al. [7] and Dutta et al. [8] claimed elastic properties for carbon fiber composites in thermal cycles have not reduced significantly while the inter-laminar and transverse tensile strength properties were reduced significantly. Ehsan S. [9] studied the crack behavior in fiber reinforced polyester composite materials, he used Compact Tension (CT) specimens to analyze...
the effect of biaxial static loads on the crack growth rate. Three combinations of materials were used in the samples, Kevlar/polyester, glass/polyester, and hybrid glass/Kevlar/polyester. Open time tests with uniaxial and biaxial loads showed that the effect of the biaxial load decreased the static load life of the fiberglass models about 20.7% and 13.5% for the Kevlar models and 9.8% for the Hybrid Samples.

This work is experimental and numerical research for the influence of nanomaterial additives (specifically TiO₂ powder) on the crack propagation and fracture toughness of composite material. In the experimental part, (CT) group of samples constructed from Glass/Epoxy composite material were tested with open time tension load in two different thermal environments at 30°C and 60°C. Another group of samples were fabricated with adding Titanium Nano powder. In the numerical part of this work, the conception of finite element model (FEM) of the compact tension specimen fabricated from the same composition of the experimental samples. Ultimately, simulation of the (CT) tension inspection individual Arithmetically, when the crack initiation and propagation were studied, and the fracture toughness in the simulation have been performed.

2. Experimental Work

E-type glass fibers were used with Epoxy. The E-glass is most commonly glass used because it has good tensile strength up to 2.5 GPa and Elastic modulus up to (76 GPa), with good stiffness, electrical and weathering properties [10]. Table (1) gives the mechanical properties of both fiber and matrix materials that used in this work, the values are drawn from laboratory tests. And Table 2 gives the details of the nano-powder which is used in this work.

**Table 1**: mechanical properties of materials used

| Properties                  | Fiberglass E-glass | Epoxy Resin |
|-----------------------------|--------------------|-------------|
| Density k/m³                | 2540               | 1180        |
| Modulus of elasticity GPa   | 75.8               | 3.4         |
| Shear modulus GPa           | 35.42              | 1.27        |
| Poisson’s ratio             | 0.20               | 0.32        |

The specimens’ arrangement was as follows:
1- S₁: five plies of woven fiberglass/Epoxy with 50% fiber volume fraction (68%wt).
2- S₂: same as S₁ but by replacing 2% wt of the Epoxy by titanium nano-powder. Mechanical stirring and sonication methods were used for powder/epoxy mixing.

**Table 2**: specifications of Nano-powder

| Title                              | Specifications             |
|------------------------------------|----------------------------|
| Product name and formula           | Titanium Oxide Nano-particles (TiO₂) |
| Appearance                         | White Powder               |
| Purity                             | 99.5%                      |
| APS                                | 10-30 nm                   |
| Morphology                         | Nearly spherical           |
| Producer                           | Skyspring nanomaterials    |

A constant tensile load of (3 kN) is applied to the samples. The crack length measured during the test within the time interval. The test was ended when the deviation from normal crack direction occurred. The experiments were applied in two different ambient temperatures 30˚ (86 F) (room temperature) and 60˚ (140 F)
(assumed outdoor temperature). The mentioned range of temperature represents the real climate for summer in Baghdad city. Heaters were used to produce the thermal condition for the samples at test and regulated by thermostat and thermos-couples.

3. Finite Element Analysis:

The general concept of FE simulation consists three major steps (i) pre-preparing (ii) solution and analysis (iii) post-processing. It is really essential to define, carefully, the model design as the quality of the results depends on the characteristic of the input parameters. An accurate layout of the form is needed to create the problem architecture. Extended finite element method (X-FEM) is used in this research for simulating the crack growth. The (X-FEM) is created by Belytschko and Black [11], the method is based on Melenk and Babuska [12] who utilized the concept of partition of finite element unity and advancement function. In this method, the mesh update is not needed to follow the crack path [13], in other words, there is no need for mesh and re-mesh the complex discontinuity surfaces. Therefore, the fracture investigation can be traced during crack propagation without any re-meshing around the crack tip [14]. Joes et al. [15] performed (FEM) to expect the laminated fracture toughness. They compared the obtained results by results obtained from the isotropic data reduction equation (1) from ASTM standard E399 [16]. They found variation in their results compared to that of ASTM equation; and that was due to the isotropy assumption.

\[ K_{lc} = \frac{P}{h \sqrt{W}} f \left( \frac{a}{w} \right) \]  
(1)

Where \( h \) is the specimen's thickness, \( W \) is the length from the load line to the edge of the specimen as shown in figure (1). The relationship \( f \left( \frac{a}{w} \right) \) calculated according to ASTME399 as shown in equation (2)

\[
f \left( \frac{a}{w} \right) = \frac{2 + \left( \frac{a}{w} \right)}{\left( 1 - \frac{a}{w} \right)^2} \left[ 0.886 + 4.64 \left( \frac{a}{w} \right) - 13.32 \left( \frac{a}{w} \right)^2 + 14.72 \left( \frac{a}{w} \right)^3 - 5.6 \left( \frac{a}{w} \right)^4 \right]
\]  
(2)

Figure (1) the compact tension specimen [17]. \( W = 60 \) mm, \( B = 2 \) mm

With the dimensions shown in Figure (1), the (CT) specimen was formed in ABAQUS/CAE software but with a small modification to simplify the calculations. The same material combinations for the experimental work were used here. The mesh of the (CT) modeling displayed in the figure (2) where the element type was
linear hexahedral elements of type C3D8R total number of nodes: 14247 total number of elements: 9106 that was suitable for the (CT) modeling to give the perfect answer approximate with the experimental result.

![Mesh converge of CT modeling and Element shape C3D8R](image)

**Figure (2)** A) The mesh converge of CT modeling B) Element shape C3D8R

### 4. Experimental Results

Figure (3) shows the tensile test machine and the CT specimen with brackets for installation.

![Tensile test machine and CT specimen with brackets](image)

**Figure 3:** A) CT specimen with brackets  B) tensile test machine

Figures 4 and 5 gives the relationship between crack length and test time. In figure 4 , two curves are presented, the blue one is for the test on the sample S1 at 30°C and the red curve is for the test at 60°C. The critical crack length was 7.4 mm at time 60 sec in room temperature test. The critical crack length decreased to
3.6 mm at 18 seconds at outdoor temperature test. Which means that the critical crack length decreased 51% when temperature increased 30˚C.

![Crack length Vs time](image)

**Figure 4:** Experimental result for S₁ specimen at two different temperatures

The effect of adding TiO₂ Nano powder is shown in figure 5, which represents the test on specimens S₁ and S₂ at room temperature. It can be noticed that the critical crack length increased from 7.4 mm to 9.8 mm, i.e. adding 2% wt Nano powder to the resin increased the critical crack length by 32.4%.

![Crack length Vs Time](image)

**Figure 5:** Experimental result for S₁ and S₂ at 30˚C

5. **Numerical Results of FAE:**

Using the FEA of CT simulated by ABAQUS/CAE software, the crack propagation is traced as shown in the figure (6) for the two types of composite material used in this study S₁ and S₂. The applied load was 3000N at 30 C°.

A) Woven glass fiber/epoxy (S₁): The result of the crack propagation for different time periods are sketched in figure 6. The FEA also determined the maximum value of stress 847 MPa and from the equation (1) the value of fracture toughness had been calculated $K_{IC}=491$ Pa.mm⁰.⁵. Figure 7 presents the stress contours at different periods.
**Figure (6):** The Numerical result for S₁ and S₂ specimen at 30°C

**Figure (7):** The stress contours at different time
Extended Finite Element Method (XFEM) is used to investigate the crack propagation. The result of (XFEM) in ABAQUS classified into three main parameters, the first is PHILSM (ϕ); which describes the face of fracture, the second is PSILSM (ψ); which describes the progress of initial fracture, and the third is STATUSXFEM; which assigns whether the item is full, also in this part the crack growth at different times is investigated. The results as shown in Figure 8 below for glass fiber/epoxy composite material.

**Figure (8) XFEM contour a)PHILSM, b)PSLSM,c)STATUS XFEM for S1**

B) The second analysis processing when the material glass fiber reinforced epoxy with nanopowder TiO₂ (S₂) at the same conditions like load boundary condition and mesh. The results of the crack propagation for different time periods is sketched in figure 6 to be compared with the first case. The fracture toughness for glass fiber reinforced the nanopowder was $K_{IC} = 517 \text{ Pa.mm}^{0.5}$. The 2% wt Nanopowder addition made improvement in the fracture toughness by 16%. The results are shown in Figure 9 below for glass fiber/epoxy- Nanopowder composite.

**Figure (9) X-FEM contour i) PHILSM, ii) PSLSM ii) STATUS XFEM S2 specimen**
The numerical results showed that the critical crack length for the S1 samples is 5.2 mm while the length in S2 is 7.4 mm which indicates that the effect of 2% wt addition of the Nanopowder gives 42% increase in the length of the critical crack. The numerical results were harmonious with the experimental.

6. Conclusions

From the above results, it can be concluded that, the crack behavior is affected by temperature rise, the crack rate increases, the critical crack point decreases and the final failure crack length decreases too. The addition of 2%wt TiO2 made the crack rate decrease with increase in the critical crack point and also an increase in the final failure crack length.

7. References

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