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## Effect of TiO<sub>2</sub> Nanomaterials on the Fatigue Properties of Composite Materials with Cracks

Ruqayyah H. Ghani<sup>1</sup> and Mundher A. Dookhi<sup>1,2</sup>

<sup>1</sup>College of Dentistry,  
National University of Science and Technology,  
Thi-Qar, Iraq

<sup>2</sup>Department of Cooling and Air Conditioning Engineering Techniques,  
Imam Ja'afar Al-Sadiq University,  
Baghdad, Iraq

The present study examines the impact of TiO<sub>2</sub> nanoparticles on the fatigue behaviour of composite materials with internal or exterior cracks. In order to guarantee that the TiO<sub>2</sub> nanoparticles are completely dispersed throughout the base material, the nanocomposite material is created by combining them with epoxy using an ultrasonic mixer, varying weight percentages of particles as 1%, 2%, and 3%. The epoxy is supplemented with TiO<sub>2</sub> nanoparticles. A single layer of woven roving glass fibre is added to the synthesized epoxy nanoparticles to create various hybrid nanocomposite materials, which have a consistent weight of 10%. After that, lamellar hybrid composite nanomaterials are made by hand with a vacuum device. Experimental specimens for tensile and fatigue tests are produced in compliance with ASTM guidelines. According to the data, the hybrid nanocomposite with the highest fatigue-stress limit is with 3% of particles. Additionally, it is generally noted that the stress behaviour of this material is improved by the inclusion of TiO<sub>2</sub> nanoparticles.

У цьому дослідженні розглядається вплив наночастинок TiO<sub>2</sub> на утомну поведінку композитних матеріалів з внутрішніми або зовнішніми тріщинами. Щоб гарантувати повне розподілення наночастинок TiO<sub>2</sub> по всьому основному матеріалу, нанокompозитний матеріал було створено шляхом поєднання його з епоксидною смолою за допомогою ультразвукового змішувача, змінюючи на рівні 1%, 2% і 3% вагові відсотки наночастинок TiO<sub>2</sub> (й епоксидну смолу доповнювали ними). До синтезованих епоксидних наночастинок було додано один шар плетеного скловолоконного ровінгу (джгута зі скловолокна, що одержується шляхом зрощування кількох ниток) для створення різних гібридних нанокompозитних матеріалів, які мають стійку вагу у 10%. Після цього вручну за допомогою вакуумного пристрою було виготовлено пластинчасті гіб-

ридні композитні наноматеріали. Експериментальні зразки для випробувань на розтяг і втому виготовлено згідно з рекомендаціями ASTM (Американського товариства з випробування матеріалів). Згідно з даними, гібридний наокомполит з найвищою межею втоми містить 3% частинок. Крім того, внутрішня міцність (під час напруження) цього матеріалу поліпшується завдяки додаванню наночастинок  $\text{TiO}_2$ .

**Key words:** polymer, composite materials, crack, fracture mechanics, fatigue.

**Ключові слова:** полімер, композитні матеріали, тріщина, механіка руйнування, втома.

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## 1. INTRODUCTION

One of the major challenges in the marine energy industry is the ability to predict fatigue failure of composite structures. High-cycle fatigue occurs in many marine energy conversion devices; in particular, tidal turbine blades, which are subject to an environmentally affected fatigue load and, therefore, the corrosion fatigue capability is a life-limiting factor. Glass fibre-reinforced epoxy-resin composites are frequently used in the design of tidal turbine blades, and the primary fatigue failure modes are delamination and fibre breakage [1, 2]. Other researchers say that incorporation of a very small volume fraction of nanofiller into the polymer enhances fatigue properties of some materials. Their data were supported by the high-cycle fatigue test by Grimmer and Dharan, which were able to extend the high-cycle fatigue life of the epoxy and glass fibres with the use of multi-walled carbon nanotubes [3]. Khan *et al.* [4] asserted that adding nanoclay to carbon fibre-reinforced plastic (CFRP) composites could improve the materials' fatigue life under cyclic loads, mechanical residual qualities after a specific cycle-fatigue period, and mechanical properties under static loading. The studies according to Pinto *et al.* [5] have shown that the addition of  $\text{TiO}_2$  nanoparticles can improve the fatigue fracture propagation in the epoxy.

According to Wetzel *et al.* [6], enhancement of the epoxy fatigue-fracture resistance can be improved by the addition of  $\text{Al}_2\text{O}_3$  nanoparticles. Research indicates that, in comparison with pure epoxy, the fracture in dynamically loaded nanocomposites tends to crack at a slower pace. Because of the silica-nanoparticles' inclusion to the epoxy polymer resin, the maximum fatigue stress was noted by Ajaj *et al.* [7], while, at the same time, with reducing the brittleness of the resin, fatigue life, and the composites' surface roughness. The impact of adding nanoclay on the fatigue behaviour of carbon fibre-reinforced polymers (CFRPs) was investigated by Khan *et al.* [4].

The collected results showed that adding nanoclay to CFRP composites enhances the composites' fatigue behaviour for a given cycle-load level, mechanical properties under static loading, and mechanical features after a specific cyclic-fatigue period. Borrego *et al.* [9] investigated the fatigue properties of composites composed of nanoscale clay and multi-walled carbon nanotubes. Under tension-tension loading, it has been discovered that both nanoclay and multi-walled carbon nanotubes raise the fatigue ratio, indicating that they may inhibit the spread of fatigue cracks. Ansari and colleagues [9] examined how the fatigue-life behaviour of fibre-reinforced polymer composites was influenced by factors such as fibre volume percentage, fibre type, and fibre orientation. As concluded, fatigue frequency first increases and then it decreases as the volume of the fibre fraction increases up to a certain point.

At a length of 500 meters, C. Capela *et al.* [10] investigated the fatigue life of an epoxy-based composite reinforced with carbon fibres in different volume fractions ranging from 2% to 10%. The fibre volume fraction increased up to 17.5% enhances stiffness, tensile strength, and fatigue resistance by 140%, 52%, and 400%, respectively, according to the data. Nevertheless, after that, mechanical qualities are slightly decreased. The behaviour of carbon/epoxy was examined by Amore and Grassia [11] in order to predict static strength, fatigue limit, and residual strength of composite under cyclic loadings of constant amplitude. It has been observed that the model captures the hierarchical damage into specific insights development as well as other important characteristics of the composites' reaction. By altering the fibre direction at three distinct angles 0°, 45°, and 90°, Lee *et al.* [12] investigated the high-cycle fatigue behaviour of glass fibre with a 30 wt.% implant in polyamide 6,6. The results indicated that the glass fibre with a 0° direction had the highest fatigue life strength across all testing conditions. Debonding, vacancies, and the formation of microcracks at the fibre ends were blamed for the failure. Utilizing 60% long carbon fibre-reinforced nylon 6,6 oriented at three distinct flow angles 0°, 45°, and 90°, Bondy *et al.* [13] looked into how fibre direction affected the materials' fatigue life. The results showed that the intensity of fatigue stress decreased as the fibre flow angle increased. The effects of epoxy resin and activated carbon powder at various weight fraction ratios of 0, 5, 10, 15, 20, 25, 30, 35, and 40 wt.% on tensile properties were investigated experimentally by Mustafa B. Hunain *et al.* [14]. The results showed that the tensile strength magnitude increased up to 15 wt.%, when the activated-carbon content was increased, but then decreased at 40 wt.%.

The current study examines how composite materials with internal or exterior fissures behave during fatigue and TiO<sub>2</sub>-nanomaterials'

impact. In order to guarantee that the  $\text{TiO}_2$  nanoparticles were completely dispersed throughout the base material, the nanocomposite material was created by combining them with epoxy using an ultrasonic mixer and varying weight percentages of particles as 1%, 2%, and 3%. The epoxy was supplemented with  $\text{TiO}_2$  nanoparticles too. A single layer of woven roving glass fibre is added to the synthesized epoxy nanoparticles to create various hybrid nanocomposite materials, which have a consistent weight of 10%.

## 2. METHODOLOGICAL APPROACH

### 2.1. Materials

The polymeric substance, which was utilized for this study, is Ren floor HT 2000 formulated in a transparent epoxy. One plate short-fibre E glass E6-CR was used for reinforcement, and kraft adhesive tape and wax were used for creation of the interior crack inside the plastic.

### 2.2. The Method of Work

After placing a glass beaker and a pot of hot water in epoxy beaker and hardener, epoxy resin (Ren floor HT 2000) and hardener (HT 2000) are mechanically stirred for 10 minutes in a weight ratio of 2:1. The mixture was mechanically mixed using a mechanical mixer (Model No. HE-133). The mound should then contain the mixture and unidirectional fibreglass added. The inside crack dimensions were of  $10 \text{ mm} \times 1 \text{ mm} \times 1 \text{ mm}$ , and two different techniques were used to repair the crack, plastic sticky tape and wax. The manual techniques involve cutting the tape or wax apart and then combining all the parts including pouring the sample with the inner crack



Fig. 1. Epoxy type (Ren floor HT 2000).



Fig. 2. Fibre E glass (E6-CR).



Fig. 3. Wax.

that is allowed with 36 hours of curing time. The moulder crack (*i.e.*, the dimension of the inner crack) is done concomitantly with their hardening, which is done with a CNC-operated machine. Using a CNC machine, the plastic moulds were in compliance with ASTM.

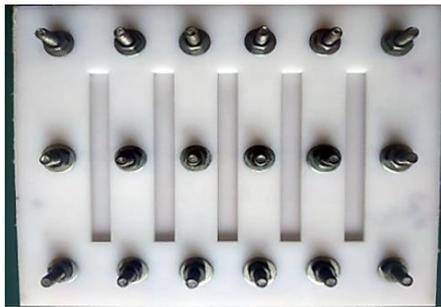
### 2.3 Fatigue Test

The HSM20-type Alternating Bending Fatigue Machine (Hi-Tech) [15] was used. It was found that the instrument was situated at the Department of Mechanics in the College of Engineering at the University of Technology (in the location shown in Fig. 5).

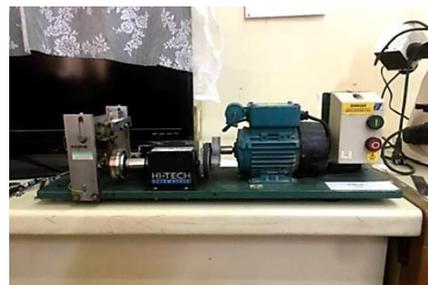
The specimen dimensions were created based on the instrument guide. Six samples were evaluated for each composite, and the test frequency was kept constant at 20 Hz with  $R=1$ , where  $R$  is the ratio of the maximum to minimum stress. The materials were tested at six different maximum stress values, starting from 80% to 20% of the ultimate tensile stress [16]. Finally, in every situation, the  $S-N$ -curve is plotted by charting the stress against the number of cycles, and Fig. 6 shows the fatigue specimens after testing.

The fatigue tolerance of polymer composites is influenced by several factors including, which type of matrix and which type of reinforcing fibre have been used, and how the structure among the two metals is arranged as these are influential over the stress applied, temperature and these afore-mentioned variables [17]. Seven replicas of the same type are tested, and multiple loads are applied in order to perform the fatigue test of the fabricated composite materials. All the loads are the same, whereas the structures of 42 specimens differ and are being tested for fatigue in multi-tester setup.

The outcomes have been represented in the form of the  $S-N$ -curves. These curves have been formed using the fatigue-test data, which have been gathered, and a curve-fitting methodology, which



**Fig. 4.** 5-mm-thick fatigue specimens moulded using an HSM20 machine [16].



**Fig. 5.** Bending fatigue machine (University of Technology).



Fig. 6. Samples from fatigue tests following failure.

has been applied to these curves.

### 3. RESULTS AND DISCUSSIONS

The estimation of how long the composite material has been in use is an essential part; so, a Univariate Linear Model (ULM) analysis is conducted. The results for the samples of epoxy (Ren floor HT 2000) and a

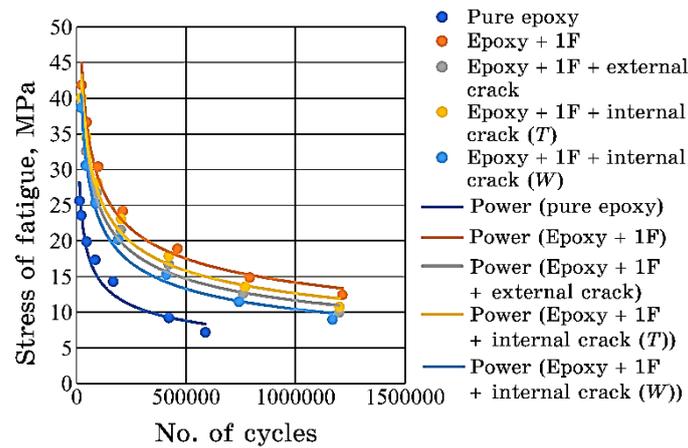


Fig. 7.  $S-N$ -curve without  $TiO_2$ .

TABLE 1. Fatigue properties without  $TiO_2$ .

Samples	$R^2$	$a$	$b$
Free EP	0.9509	579.76	-0.319
EP + 1F	0.9847	965.7	-0.306
EP + 1F + external crack	0.9837	1165.7	-0.333
EP + 1F + internal crack ( <i>Tape</i> )	0.9792	1082.4	-0.322
EP + 1F + internal crack ( <i>Wax</i> )	0.9853	1289.4	-0.349

single short E glass fibre plate (E6-CR) in the graphs illustrate the changing number of cycles *vs.* fatigue-stress values. The tests were performed in two cases to understand, if there were any nanomaterials in this material, and in both cases as integrity.

We document the percentages of expansion and enhancement that resulted after addition of TiO<sub>2</sub> nanoparticles to understand how the incorporation of nanomaterials affects the composite materials, which have internal or external cracks. These are evidenced by the images in Figs. 7–11 and Tables 1–4.

Dynamic stress can be alleviated through TiO<sub>2</sub> nanoparticles. The fatigue fracture propagation increase for the epoxy is discussed in details with diagrams and tables. The table tops show the increase of dynamic stress by 3%. This is seen as a result of the addition of 3% scaffolding to the epoxy. The increased fatigue strength of the hybrid nanocomposite material is enabled due to the interaction of materials and components between added nanoparticles as well as the slurry due to their huge surface area.

However, such adhesive strengthening can be combined with the increase of volume concentration of the nanoparticles, which would in turn have a negative effect on the fatigue strength of the product.

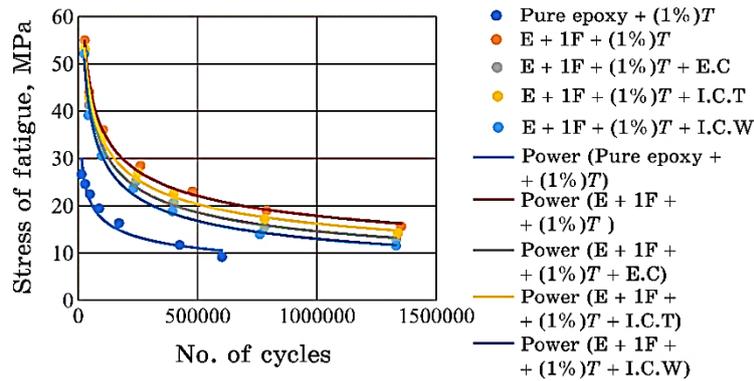


Fig. 8. *S-N*-curve with 1% of TiO<sub>2</sub>.

TABLE 2. Fatigue properties with 1% of TiO<sub>2</sub>.

Samples	$R^2$	$a$	$b$
Free EP + 1% TiO <sub>2</sub>	0.9888	266.84	-0.233
EP + 1F + 1% TiO <sub>2</sub>	0.9903	1252.4	-0.299
EP + 1F + external crack + 1% TiO <sub>2</sub>	0.995	1875.4	-0.345
EP + 1F + internal crack ( <i>Tape</i> )+1% TiO <sub>2</sub>	0.9935	1522.5	-0.321
EP + 1F + internal crack ( <i>Wax</i> )+1% TiO <sub>2</sub>	0.9948	2378.5	-0.37

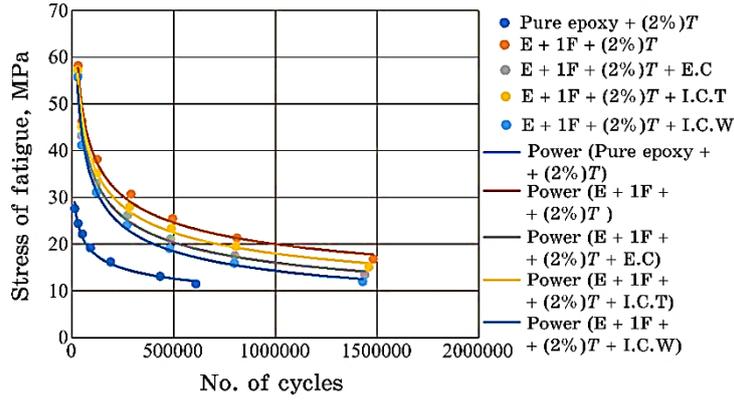


Fig. 9. *S-N*-curve with 2% of  $\text{TiO}_2$ .

TABLE 3. Fatigue properties with 2% of  $\text{TiO}_2$ .

Samples	$R^2$	$a$	$b$
Free EP + 2% $\text{TiO}_2$	0.9459	416.24	-0.277
EP + 1F + 2% $\text{TiO}_2$	0.994	1249.1	-0.308
EP + 1F + external crack + 2% $\text{TiO}_2$	0.9956	1787.3	-0.348
EP + 1F + internal crack ( <i>Tape</i> ) + 2% $\text{TiO}_2$	0.9939	1435	-0.325
EP + 1F + internal crack ( <i>Wax</i> ) + 2% $\text{TiO}_2$	0.9946	2155.9	-0.37

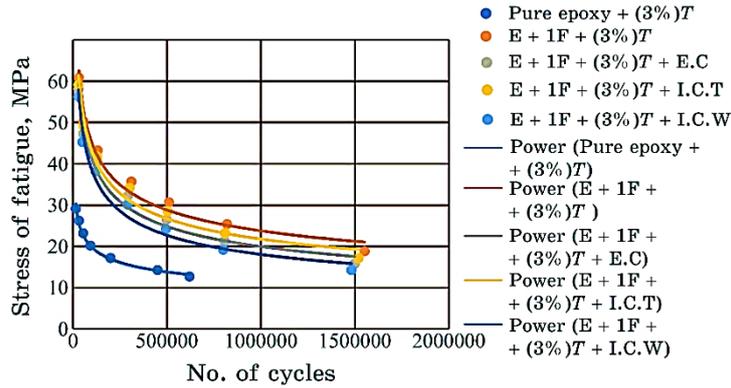


Fig. 10. *S-N*-curve with 3% of  $\text{TiO}_2$ .

TABLE 4. Fatigue properties with 3% of  $\text{TiO}_2$ .

Samples	$R^2$	$a$	$b$
Free EP + 3% $\text{TiO}_2$	0.9927	261.74	-0.224
EP + 1F + 3% $\text{TiO}_2$	0.9713	1111.2	-0.278
EP + 1F + external crack + 3% $\text{TiO}_2$	0.9754	1549.5	-0.315
EP + 1F + internal crack ( <i>Tape</i> ) + 3% $\text{TiO}_2$	0.9723	1327.5	-0.298
EP + 1F + internal crack ( <i>Wax</i> ) + 3% $\text{TiO}_2$	0.9765	1743	-0.331

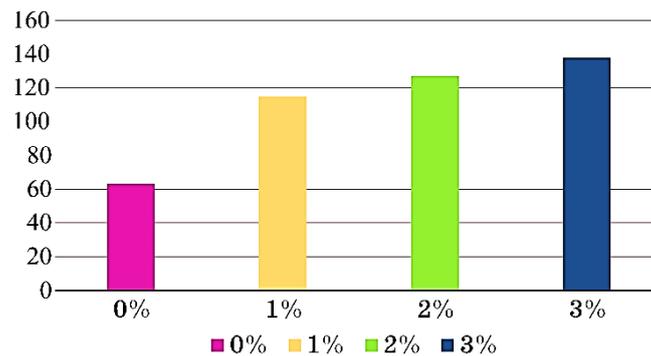


Fig. 11. Fatigue-improvement percentages.

#### 4. CONCLUSION

Results, which are regarded as less efficient than composite materials without manufacturing flaws or internal cracks, show that the existence of the internal or exterior cracks in composite materials decreases their mechanical capabilities. We discovered that adding nanoparticles improves the composite-materials' ability to withstand fatigue stress.

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