Effect of Nano clay on the fatigue of epoxy and glass fiber composites

Khalid Mohammed Khalifah
Ministry of Education
Khalid0771188@gmail.com

Abstract. This study describes the experimental results of the fatigue behavior manifested by fatigue strength, fatigue life, and fatigue limit composites for epoxy resin and its E-glass fiber (2,4,6) laminates, nano clay prepared in laboratory reinforced in with (2%, 4%, 6%) weight fraction. The evaluation of (S-N) curves of the tested specimens was depending according to the type of E-glass fibers, laminates number, and addition nano clays small granular volume which prepared in laboratory to a polymer. The fatigue tests showed high resistance to all samples after being immersed in water. Adding the nano clay reduced the brittle nature of the Epoxy resin as it is improved drastically.

1. Introduction
The failure of metals is due to the applied loads that has become one of the engineering problems. It has been noted that metals fail when they exposed to load or periodic stress levels. This prompted many researchers in this field order to find alternatives, or to develop by finding composites materials with a metal matrix, or ceramic or polymerization. With the progress of time, the technology of composites materials has evolved to include all industrial fields, whether civilian or military [1]. Engineering parts are exposed during the period of service to Dynamic Loads periodically and in surrounding environmental conditions that cause engineering parts to fail. This phenomenon is called Fatigue Failure. It has been observed that many parts of the engineering parts fail when exposed to Alternating Stress, less than the stresses generated by the static loads. Accordingly, many of the failures that occur during use are attributable to fatigue, which increases the risk of this phenomenon is the absence of any warning before the failure [1,2]. Fatigue limit is the stress level as below which a material can be stressed cyclically for an infinite number of times without failure. Fatigue strength is the maximum cyclical stress that a material can withstand for a given number of cycles before failure occurs [3]. For polymer fatigue mechanism is completely different due to different molecular structure. If plastics articles have been machined then it is likely that this will introduce flaws capable of propagation, so the initiation phase of failure will be negligible. If the article has been molded this tends to produce a protective skin layer which inhibit fatigue initiation / propagation. In such cases it is more probable that fatigue crack will developed from within the bulk of the material [4]. In amorphous material, it is possible that cracks may develop in the voids which are formed during viscous flow. There are many variables that make the polymer fatigue a complex subject [5,6].

There are major classes of factors, which have an effect on Behavior of fatigue in engineering materials. These factors are grain size, surface finish, temperature, environment, stress concentration [7]. The fatigue mechanism is divided into three phases, as shown in Figure 1, as follows,
The formation of the crack nucleus begins on the surface of the piece due to the movement of dislocations resulting from the effect of periodic stresses to which the piece is exposed. This movement produces precise sliding beams at the surface of the material and as a result of the instability of the internal structure and the concentration of stresses at these sites, the material responds and creates new structures from permanent slide bundles and that the strain of cyclic plasticity resulting from slide bands leads to the formation of Extrusions and Intrusions on the surface of the material. This movement of dislocations and the sliding that represent areas for the concentration of stresses that can be the localized place for the beginning of the formation of the crack nucleus [9]. The crack growth can be divided into two phases: the first stage: the crack growth at an angle of (45º) compared to the stress axis. The first stage represents the deepening of the cavities formed on the surface of the material. Slow growth of the crack occurs during the crystals along the sliding levels with high shear stress. This crack extends to a distance of twice the diameter of the granule. The second stage: the crack is made at an angle of (90º) compared to the stress axis. At this stage, a growth of the crack occurs at a certain distance at each stress cycle, as this process leaves traces on the surface of the fracture and these effects are called striation. They are sites of crack peaks as it grows in stress cycles, as in Figure 2, the second stage of slide growth depends on the stress value, the size of the crack, the properties of the material, and its geometrical shape [10].

The last stage of fatigue mechanics is the sudden failure of the engineering part. As a result of the Critical Stress Intensity Factor (K) reaching the critical value (Kc), this stage is characterized by a rapid growth of the crack, which makes the remaining distance of the engineering part unable to withstand the applied load. The final fracture areas appear rough, while the first and second stage areas appear smooth. The rough areas are less than the soft areas in the case of low periodic stress, whereas in the case of high periodic stress, the rough areas are larger than the soft areas and the Figure 3 shows the fracture areas.
Figure 3. shows the fracture areas, (a) low load, (b) high load [11]

There are many Types of fatigue stress include: [12]

1. Maximum Stress ($S_{\text{max}}$)

$$S_{\text{max}} = S_m + S_a$$  \hspace{1cm} (1)

Where $S_m$:(Mean Stress), $S_a$:(Stress Amplitude),

2. Minimum Stress ($S_{\text{min}}$)

$$S_{\text{min}} = S_m - S_a$$  \hspace{1cm} (2)

3. Stress Range ($\Delta S$)

$$\Delta S = S_{\text{max}} - S_{\text{min}}$$  \hspace{1cm} (3)

4. Stress Amplitude ($S_a$)

$$S_a = \frac{\Delta S}{2} = \frac{(S_{\text{max}} - S_{\text{min}})}{2}$$  \hspace{1cm} (4)

$$S_{\text{int}} = \frac{(S_{\text{max}} + S_{\text{min}})}{2}$$  \hspace{1cm} (5)

5. Stress Ratio (R)

$$R = \frac{S_{\text{min}}}{S_{\text{max}}}$$  \hspace{1cm} (6)

There are different values for the stress ratio (R) that depend on the minimum stress value to the maximum stress value, and these values are a ($R = -1$): when the minimum stress value is equal to the maximum stress value and the load is completely opposite completely reversed stress as Figure 4. b ($R = 0$): when the minimum stress is zero ($S_{\text{min}} = 0$) and the load is alternate (Fluctuating Stress), i.e. in this case, the resulting ratio is zero and loath Regardless of the maximum stress value, ($0 < R < 1$): When the minimum stress is positive (Repeated Stress). ($-1 < R < 1$) stress ratio ranges between ($-1 < R < 1$) through the above values, the stress ratio ranges.
2. Experimental

The material used to prepare the test samples were epoxy resin (EPI0 Conbextra) as a matrix, which is supplied by Fustic Company with the hardener aliphatic amine (Hy956) E-glass fibers as reinforcements, chopped mat woven roving E-glass fibers of surface density (8 g/cm³) in the first time and addition Nano Clay of Bentonite in other times of [13].

2.1. Epoxy resin

The Matrix Epoxy resin is the thermosetting polymer in liquid stage once, and then hardened irreversibly by curing reaction called cross-linking. On completion of this reaction, the material cannot be softened again. Because of their cross-linked molecular structure, they are more resistant to heat and solvents than thermoplastics [14].

2.2. glass fibers

The reinforcing effect of the glass fibers is influenced by several factors: a- resistance to high temperatures the softening point is about 850 °C. b- transparency to visible light a composite therefore takes the colour of its matrix. c- isotropy – for example thermal expansion is identical in axial and radial directions.

2.3. Bentonite

Chemical analysis of Bentonite (Ca – Iraqi Bentonite) are shown in Table 1, the analysis was carried out in the general company for refractories -Iraq. These clays are washed with excessive amounts of distilled water to remove any soluble materials, filtered and dried at 160°C for three hours then it was

![Figure 4. Types of fatigue stress [11].](image)

![Figure 5. Woven glass fiber reinforcement[15].](image)
ground and sieved to a particle size of (53 µm), the preparing nano clays carried out by ultrasound, then used in all reinforced experiments.

**Table 1.** Chemical analysis of clays by XRD [19].

| Constituents | Wt. % Bentonite |
|--------------|-----------------|
| SiO$_2$      | 56.4            |
| Al$_2$O$_3$  | 15.39           |
| Fe$_2$O$_3$  | 5.14            |
| CaO          | 4.51            |
| MgO          | 3.43            |

![AFM Analysis Nano clays of Bentonite](image)

**Figure 6.** AFM Analysis Nano clays of Bentonite.

2.4. Fatigue test

The fatigue test was performed in the machinery and equipment engineering department at the University of Technology, and a type of device was used (Hsm19mk3, HI-TECH England Company), which works by Wohler Rotating Bending method), the standard tested sample is shown in Figure 7. The sample were subjected to stress by bending torque where the bending stress remains constant throughout the bending period (R = -1). The load is highlighted on the free end of the sample which is perpendicular to its X-axis. This, in turn, exposes the surface of the sample to tension and pressure stresses during rotation.

The bending stress $\alpha$ of the spring is determined from equation (1).

$$\sigma = \frac{mb}{W}$$  \(7\)

Where $W = \frac{b^2}{6}$

$mb$ = bending moment $b$ = the width of specimen west, $h$ = thickness of specimen

![Shape of fatigue test specimen with dimensions (mm)](image)

**Figure 7.** Shape of fatigue test specimen with dimensions (mm) [16].
3. Result

From the data obtained from the fatigue test, a curve (S-N) was created, where the y-axis represents the stress capacity and the x-axis represents the logarithm of the number of failure cycles (N.F.) For all the prepared composites, Figure 8 represents the S-N curve for those composite. It is noted that the number of failure cycles increases, the less the stress capacity. It has been observed at high stress capacity the number of failure cycles is a little due to the elastic-plastic deformation that occurs on the surface of the metal and this leads to the growth of the fissure that leads to failure, where this failure mechanism is called the flexible-plastic fracture Mechanism. When the stress capacity is low, the number of cycles leading to failure is high as a result of the flexible failure known as the Linear Elastic Fracture Mechanism (L.E.F.M).

![Figure 8. Fatigue curve for EP/G.F.](image)

S-N curves of Figure 9 shows the variation in fatigue strength with laminates number for specimens reinforced by different numbers of woven roving (W.R) E-glass fibers laminates and Nano clays. The fatigue strength for cycles were determined and listed in Figure 9, which showed a consistent increased in the fatigue strength with laminates number and weight fraction of Nano clay increased. This can be attributed to the presence of more fibers and interface area that transfer stresses from matrix to fibers and clays [17]. Figures 9, 10, 11 show the results of fatigue test of the composites material reinforced with glass fibers of different laminates (2,4,6) laminates and by weight fraction (2,4,6%), respectively.

![Figure 9. Fatigue curve for EP/G.F./Nano clay W_f(2%).](image)

It is noted from the two forms that strengthening the matrix with laminates or Nano clays led to an increase in fatigue resistance when increasing weight fraction of added particles, that the increase in
fatigue resistance values for reinforced laminates with reinforcement particles is due to the nature of these hard and distributed Nano particles in The matrix has different sizes, which increase the durability of the composites during the dispersal mechanism due to the presence of Nano particles of, especially those whose granular size is less than (100nm) which act as obstacles to deforming matrix. The foundation due to its high hardness, and therefore both mechanisms will work to impede the movement of dislocations and thus increase the durability of the material, which led to an increase in the number of failure cycles of the reinforced matrix with these particles.

![Figure 10](image1.png)

**Figure 10.** Fatigue curve for EP/G.F./Nano clay $W_f (4\%)$.

![Figure 11](image2.png)

**Figure 11.** Fatigue curve for EP/ G.F/ Nano clay $W_f (6\%)$.

To clarify the study of the effect of the weight fraction added from the stiffening particles in the failure cycles of the prepared composites, a graphical relationship was established between the number of cycles of failure and the weight fraction added to the stiffening particles of the proven stress of an adult (50,150,200,250) MPa represented by the Figure, respectively. The increase in hardness and the number of failure cycles when testing fatigue can be attributed to the presence of hard represented by Clay particles in the matrix composites with different weight fraction that will change some factors such as the distance between particles ($D_p$) in addition to the mean free path average ($m.f.p$) and according to the above-mentioned relationship and the following relationship (8) [18].

$$m.f.p = \frac{2d}{3V_p} (1 - V_p)$$

(8)

$d$: volume of particles($\mu$m).

$V_p$: Weight fraction
The presence of such minutes will hinder the movement of dislocation, and the rate of disability will be greater when increasing the weight proportions of the added particles. Accordingly, for the dislocation to pass through the dispersed particles in the composites, the stress must be sufficient for the dislocation curve. Moreover, the stress required for the dislocation curve \( (T_i) \) is inversely proportional to the distance between the particles \( (D_p) \) and according to the following relationship (9).

\[
T_i = \frac{G_m \times b'}{D_p}
\]

(9)

Gm: shear coefficient for matrix, \( b' \): Berger vector

Upon homogeneous dispersion of the stiffening particles, the distance between particles \( (D_p) \) will decrease with the increase of the percentage of added Nano clays, and accordingly, it will need more stress for the passage of dislocation during the particles, and accordingly to the increase in the hardness values, as well as an increase Fatigue resistance by increasing the number of failure cycles at constant stress, and this is shown by results of fatigue test through forms (10), (11), (12) at constant load stress of \( (50,100,150,20,250\,\text{MPa}) \), respectively.

3.1. Effect of water

The composites samples were immersed in water about (30 days) to find the effect of water on their fatigue. The reason behind this was ascribed to the degradation of the binder or weakening of the interaction between fillers lead to penetration of water to the fillers of Glass fiber, and Nano clay this would swell and plasticize that samples. The swelling causes changing in dimension and weight of samples. This changing was depended on percentage of weight gain \( (W_t\%) \) of water for sample. it is shown that the samples of EP / G.F./Nano clay have a lowest value of weight gain \( (W_t\%) \) of water due to the adhesion between matrix and reinforcement reduction penetration of water to the material. Also the changing in dimension and weight was few [18].

4. Conclusion

This work shows that successful fabrication of natural fillers filled polyester composites with different number of Glass fiber reinforced Nano clay using molding method. The Nano clay composites show better behavior at fatigue compared with Glass fiber composite alone. A few second ago the Nano clay reduced the brittle nature of the Epoxy resin as that it is improved drastically. Water has limited effect on Nano clay composites after immersed them in water relatively comparing to Glass fiber, so these materials can be used in humid environments.

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