Low Cycle Fatigue of Hybrid Woven Kenaf Fiber Reinforced Epoxy Composite with 1% Addition of Silica Aerogel

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ABSTRACT

This paper presented the mechanical and fatigue behavior of woven kenaf fiber reinforced epoxy composites with the composition of 16.67% kenaf and 83.33% of epoxy (1:5 weight ratio) alongside the addition of 1% of silica aerogel into the composite. There are a number of studies concentrating on natural fibers nowadays, yet their low mechanical properties when compared to other synthetic fibers had urged the addition of filler into the composite to increase the mechanical strength. Hand layup technique was used to prepare Kenaf/Epoxy (KE) and Kenaf/Epoxy/Silica Aerogel (KES) composite alongside pure epoxy (E), 100% epoxy composite. All fatigue tests were conducted at constant stress amplitude, frequency of 5 Hz, stress ratio of 0.5 and the maximum stress applied was from 90 % to 70 % of ultimate tensile strength with decrement of 5%. The results showed that the tensile properties improved as silica aerogel is added into the composite. These results showed that the tensile strength is increased by 27.3% while stiffness decreased by 78.2% when silica aerogel is added into the composite as filler. The reduction of the stiffness is associated with the brittle nature of the filler. Similar to tensile properties, the addition of silica aerogel also had resulted better fatigue life. KES composite showed more than 100% increment in number of cycles endured when compared to KE composite.

Keywords: *natural fibers, kenaf fibers, biocomposite, fatigue, silica aerogel*

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Introduction

Biocomposites had been put under the spotlight for their mechanical properties competitiveness. In general, they are used when there is the need for structural materials with good durability and mechanical properties, and also low in weight. Hence, the green and economical friendly composites are said to have found their place in many application areas. Kenaf fiber is one of the popular fibers to be implemented into biocomposite nowadays mainly due to their superior mechanical properties as well as economical and ecological advantages. Hence, a series of studies were conducted in order to better understand the properties of the fibers [1]-[4].

Bakar et al. [1] had investigated on the mechanical properties of kenaf fiber reinforced epoxy composite. They divided the composites into 2 groups: one is with 4% Sodium Hydroxide (NaOH) treated kenaf fiber while the other one is with untreated kenaf fibers, then their the mechanical properties were compared. The treated fibers turned out to have better properties than the untreated fibers. Hojo et al. [2] on the other hand, studied the tensile properties of the kenaf fiber, bamboo and jute as they were used to reinforced unsaturated polyester. They were fabricated using hand layup and compression moulding. Out of the three, kenaf composite ranked first in terms of tensile modulus and tensile strength. Focusing on tensile behavior too, kenaf fibers were also reinforced into several resins and the respective ultimate tensile strength and Young's Modulus were obtained [3]. The tensile strength of Kenaf/Epoxy, Kenaf/Polyester and Kenaf/Vinyl Ester composites increases with increasing fiber volume content. Among the three composites, Kenaf/Epoxy shows highest tensile strength and stiffness at respective fiber volume fraction. Besides, there is also study carried out to investigate the storage modulus analysis of kenaf fiber reinforced epoxy composites [4]. Storage modulus is normally known as the stored energy which include the elastic portion. The study concluded that the storage modulus of composites that utilize Epoxy B is higher than Epoxy A; while the modulus is also increased when higher fiber volume fraction (45% kenaf) is involved rather than 15% kenaf composite.

The kenaf fiber had been widely researched on and the potential is observed to be promising. Yet, few studies conducted to study the behavior of the material subjected to fatigue loading. Studies involving fatigue damage of various materials have been widely explored in these years [5]-[7]. However, unlike the research works on synthetic fibers, the works in natural fibers especially kenaf is very lacking. Other natural fibers, such as flax had been used to study the fatigue behavior as it is used to reinforce epoxy composite [8]. The flax/epoxy composites are compared to glass/epoxy composites and the biocomposites has lower fatigue strength than the glass/epoxy composites. Utilizing the same constituents, Asgarinia et al. [9] had also tested on the fatigue behavior. They concluded that the addition of glass fiber can increase the overall strength properties. This is because of the generally known high strength of glass fiber. Adding fiber of high strength to a composite will then improve the overall strength of the composite.

Silva et al. [10] had also study the topic of fatigue behavior. Sisal fibers are used in their study and they discovered that the fiber survived up to 10^6 cycles when the stress ratio is set to 0.5. Stress ratio that is higher than 0.5 would decrease the fatigue strength. The effect of varying stress ratio had been studied by Kadi and Ellyin [11]. The research focused on the fatigue behavior of unidirectional glass fiber/epoxy composite laminate by varying the stress ratio by -1, 0 and 0.5. Through a series of experiments, they concluded that the slopes of S-N curves with the stress ratio 0.5 are the lowest, followed by 0 and -1. Stress ratio is paired with fiber orientation as the variables in a study conducted by Huh et al. [12]. The study was conducted by implementing E-glass/Epoxy with unidirectional (UD), bidiagonal (BD) and triaxial (TRI) orientation alongside stress ratio of 0.5, 0.1 and -0.2. Similar to other studies, they concluded that the fatigue life is enhanced as the stress ratio decreases. Moreover, at a specific stress ratio, fatigue limit for UD composite is observed to be the highest followed by TRI and BD composite. The effect of configuration of fiber is believed to be one of the factors affecting the mechanical properties. Ekşi and Genel [13] had conducted a study to investigate the difference of properties between unidirectional and woven carbon fiber. Regardless of material, they concluded that the woven configuration will exhibit lower tensile properties than the unidirectional configuration.

The lack of knowledge on the fatigue behavior of kenaf fiber had prompt recent studies to be carried out. Abdullah et al. [14] had incorporated unidirectional kenaf fibers into epoxy to form a biocomposite and had it tested for fatigue test by varying the fiber volume fraction. Epoxy composites without any addition of kenaf fiber endured the least number of cycles for fatigue test. Adding kenaf fiber with appropriate volume fraction had increased the number of cycles the composites can endure; in other words, increasing the fatigue strength. Ismail et al. [15] studied the effect of varying fiber orientation and they reported that the fatigue life is the highest when the fiber orientation is set at 0° while 15°, 35° and 45° showed similar trend in fatigue behavior.

It is known that kenaf fibers are not the best choice to be used to bear loads as they possess lower mechanical properties than synthetic fibers [16]-[17]. Hence, there existed studies that incorporate glass fibers and kenaf fibers to produce hybrid composites that would offer better characteristics. Sharba and his team [18] incorporated glass fiber to the natural fiber to produce a hybrid composite in order to investigate the fatigue life of the composite. Berhan et al. [19] had also contributed to the studies on kenaf fibers. They studied the fatigue life of woven kenaf/glass fiber hybrid composite and concluded that the fatigue life increase with the addition of glass fiber as one of the constituents in the composite. The same combination of fibers is also utilized by Davoodi et al. [20] as they tried to investigate the mechanical properties of the composite for car bumper beam. The hybrid material presents good mechanical properties and it can be utilized in automotive structural components by enhancing the impact property. Kenaf/glass fiber composite is also produced by using polyester as the resin and the composites were subjected to water absorption test [21].

The literatures mentioned showed that the study of fatigue life of kenaf fibers is very limited, especially kenaf fiber of the woven type. It is also known that the incorporation of synthetic fibers, such as glass fibers with kenaf fibers improved fatigue life. However, other than fibers, none of the studies involves the inclusion of fillers into the kenaf polymeric composites, for instance silica aerogel.

Silica aerogel had been chosen as filler for epoxy resin by Du et al. [22]. With the weight percentage of 0%, 0.1%, 1%, 5%, 10% and 100%, they discovered that the appropriate amount of silica aerogel added into epoxy resins will help in achieving enhanced mechanical properties. The mechanical properties of epoxy with addition of 1% of silica aerogel composite were recorded to be the highest. Hence, this work implemented the addition of 1% of silica aerogel into the composite. The phenomenon is even obvious in rubbery state. With series of studies done on silica aerogel [23]-[25], the material can hence be integrated into fiber composites in order to explore more on its contributions in enhancing the mechanical properties given the outstanding properties as listed from the mentioned studies. This study is then intended to explore and compare the fatigue life of a single layer woven kenaf fiber reinforced epoxy composite with and without aerogel in 5 different levels of stresses.

Experimental Procedure

The materials used and the standards followed while conducting the experimental work are as described in details in the following sections.

Figure 1: Woven kenaf fiber used for research study

Materials

Kenaf fiber used for the experiment is of woven mat type. The fiber originated from India which came with ready-treated condition as illustrated in Figure 1. The epoxy resin EpoxAmide 103 SLOW unit and its curing agent are provided by Smooth-On Inc and are mixed the ratio of 3:1, epoxy to hardener. The specimens are fabricated through hand layup technique and the process took place on a cleaned and waxed glass surface. A single layer of kenaf fiber mat is used in this study with the weight ratio of 1:5 (kenaf fiber : epoxy).

Mass fraction of 1% of silica aerogel was added into the resin in order to fabricate KES composite. The addition of silica aerogel is done by the aid of homogenizer to promote even distribution and homogeneity. The specimens were cured at room temperature for 24 hours. Specimens were cut into dimension of $25\ cm$ long $\times\ 2.5\ cm$ width with the thickness of $0.3\ cm$ as shown in Figure 2 according to ASTM D3039. Tab ends of specimens were sanded and emery cloth tabs were glued with epoxy resin to improve gripping and to prevent slippage and premature failure during tensile and fatigue test.

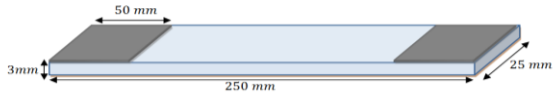


Figure 2: Coupon dimension for tensile and fatigue loading

Tensile and Fatigue Test

Tensile test was conducted to investigate tensile properties of the kenaf fiber as well as the kenaf composite specimens before performing fatigue test. For all the composites, the test was performed using the 10 kN Servo Hydraulic Instron Machine (Instron 3366). The test was conducted by setting a standard strain rate of $0.01min^{-1}$ and head displacement rate of $2mm/min$ according to ASTM D3039. Tensile test on the single kenaf fiber on the other hand is conducted according to ASTM 3822-01 with the application of paper tabbing. The diameter of the tested kenaf fiber strand is $1.45 \times 10^{-3}\ m$. The standard is used as it covers the measurement of tensile properties of natural single textile fiber which is taken from yarns or tow.

ASTM D3479 was used to conduct the fatigue test on the specimens through the tension-tension fatigue loading mode. Stress ratio used was 0.5 with frequency of 5 Hz. Note that only one stress ratio is focused in this study due to time constraint. The stress ratio was varied in order to better compare the fatigue performance of the specimens. The stress levels were varied from 90%, 85%, 80%, 75% and 70% of the ultimate tensile strength (UTS). The specimens were cycled using 810 Material Test System (MTS) machine with the number of cycles to failure recorded by data acquisition system. The materials tested for this research work were pure epoxy (100% epoxy), kenaf fiber reinforced composite (16.67% kenaf fiber, 83.33% epoxy) and kenaf fiber reinforced composite with addition of silica aerogel (16.67% kenaf fiber, 83.33% epoxy and 1% silica aerogel).

Results and Discussions

Tensile Test on Kenaf Single Fiber

After tensile test is conducted on the single fiber, the failed specimen is illustrated in Figure 3. From the data collected, it can be observed that the strength of kenaf fiber is actually higher than that exhibited when integrated into polymer composites. The observations are discussed in detailed alongside the other three composites configurations.

Figure 3: Kenaf fiber breakage

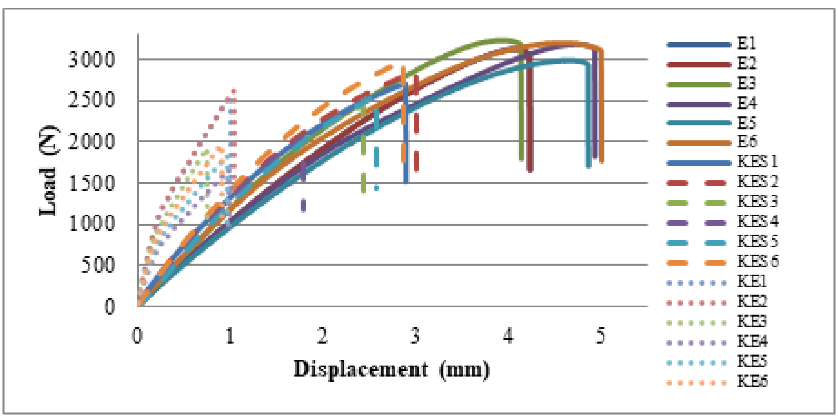


Figure 4: Load Displacement curves for all three materials

Tensile Test

Figure 4, 5 and 6 below exhibit the load-displacement and stress-strain curves of all categories of specimens. It is shown that kenaf composite that is added with 1% of silica aerogel is 27.3% higher in UTS compared to kenaf/epoxy composite that is without addition of silica aerogel. However, the Young's modulus decreases with the addition of silica aerogel. In other words, the stiffness of composites with silica aerogel is decreased. It is then believed that the silica aerogel will introduce ductility to the composite due to its nature.

By referring to the literature review done in the earlier part, natural fibers are supposed to possess high strength. However, following the experiment conducted, the tensile properties of both KE and KES composites are lower than pure epoxy. The incompatibility of fibers and matrix is the main cause in this behavior. The incompatibility hence brings inefficiency of stress transfer in the matrix [26]. Leman and Sapuan [27] also reported that the major drawback of natural fiber reinforced composites is their poor compatibility with matrix due to their hydrophilic nature; causing uneven dispersion of matrix in the fiber. The final performance of composite materials may be affected due to the compatibility issue.

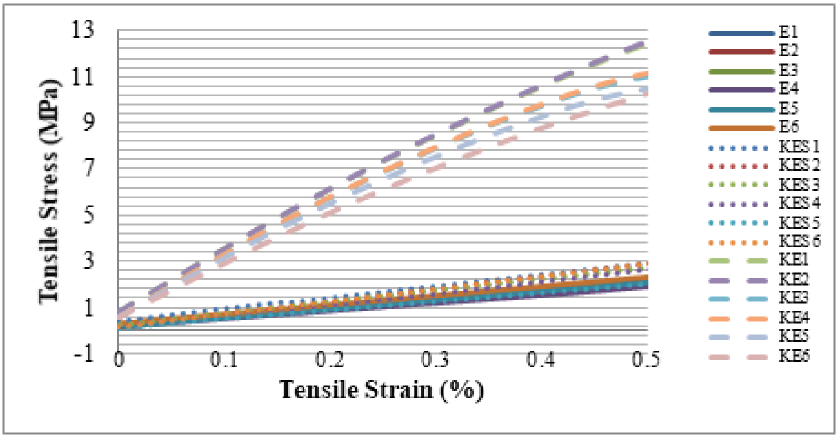


Figure 5: Stress strain curves in the linear region

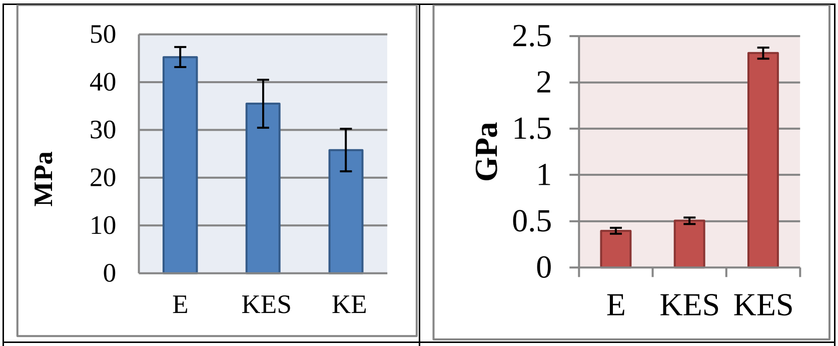
Table 1: Summary of mechanical properties

	Ultimate Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at Break (%)
E	45.26	0.397	18.16
KES	35.48	0.505	10.31
KE	25.79	2.316	2.17
Single Kenaf Fiber	48.32	9.566	8.87

The mechanical properties obtained through tensile test for all three materials alongside single kenaf fiber are summarized into the following Table 1. Note that the elongation at break is the ratio of changed length and the initial length after breakage of the test specimen. The percentage elongation at break for all types of materials are obtained through the utilized software alongside the ultimate tensile strength (UTS) and Young's Modulus.

The ultimate tensile strength of kenaf fiber is averaged to 48.32 MPa, which is higher as compared to the ultimate tensile strength of E. The composite samples showed that KES is 27.56% and KE is 75.49% lower than E. It is also indicated that the 1% of silica aerogel fillers can increase the tensile strength by 37.57%. Following the rule of mixture, the ultimate tensile strength of composite can be calculated through ultimate tensile strength of epoxy and single kenaf fiber strand as shown in Equation (1):

$$\sigma_c = \sigma_m v_m + \sigma_f v_f \qquad (1)$$



UTS	Young's Modulus

Figure 6: UTS and Young's Modulus

Whereas σ_c, σ_m, σ_f represent tensile strength of composite, matrix and fiber; v_m and v_f stand for volume fraction of matrix and fiber respectively. The calculated tensile strength for composite will range from 45.26 to 48.32 MPa by varying the volume fraction from 0 to 1. However, this only applies to unidirectional fibers. Since the fiber utilized in this research work was of woven type, the tensile strength of the woven fiber composite would be lower. The study comparing the mechanical properties of woven and unidirectional fiber composite had been included in [13].

In term of Young's Modulus, kenaf fibers displayed higher Young's Modulus compared to E, which is a brittle polymeric material. Besides, the Young's Modulus of kenaf fiber is also higher than that of KES by 94.72% and KE by 75.79%.

The mode and location of failure are recorded for all composites specimens and are shown in the Table 2, while the fractured specimens are illustrated in the following figures. Figure 7(a) and (b) showed the fractured KE specimens, Figure 8(a) and (b) showed the fractured KES specimens while

Figure 9(a) and (b) showed the fractured epoxy specimens. By observing the fractured specimens, it can be concluded that the most obvious failure type is lateral fracture, which indicates that the specimens are of brittle materials. Besides, majority of the failures occur within the gage length, located in the middle section or at the center of the specimens.

Table 2: Failure modes of E, KES and KE specimens

Pure Epoxy (E)	
Failure Modes	**Number of Specimens**
LAB	4
LGB	1
LGT	1
Kenaf/Epoxy/Silica Aerogel (KES)	
Failure Modes	**Number of Specimens**
LAB	2
LGM	3
LGT	1
Kenaf/Epoxy (KE)	
Failure Modes	**Number of Specimens**
LAT	2
LGM	4

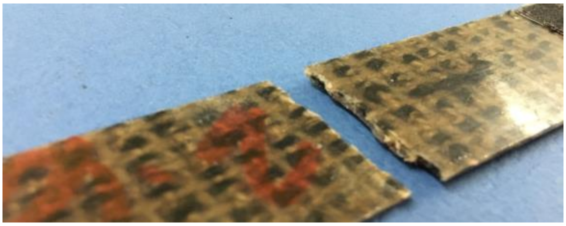


Figure 7 (a): KE composite specimen failed at gage length

Figure 7 (b): KE composite specimen that is fractured after tensile test

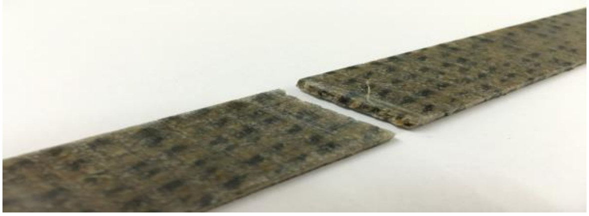


Figure 8 (a): KES composite specimen failed at gage length

Figure 8 (b): KES composite specimen that is fractured after tensile test

Figure 9 (a): Epoxy specimen failed at gage length

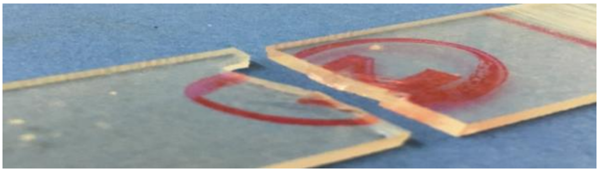


Figure 9 (b): Epoxy specimen that is fractured after tensile test

Fatigue Results

Fatigue test was conducted using different stress levels. The average number of cycles to failure for all three categories is tabulated as in Table 3.

Table 3: Fatigue life data for the specimens

Stress Level	Number of cycles to failure		
	E	KES	KE
0.90 UTS	1821.3	316.0	7.3
0.85 UTS	4593.3	751.3	112.3
0.80 UTS	5646.7	4186.0	208.7
0.75 UTS	10070.0	5632.7	1085.7
0.70 UTS	13620.3	12449.2	5177.3

The S-N curves is shown in Figure 10 and from the figure, it can be observed that the fatigue life of E is higher than KES and KE. KE composites have the lowest fatigue life compared to all three specimens. In other words, the materials can only withstand a small number of cycles upon loading before failure. However, the addition of silica aerogel into the KE composites has boosted the properties of the composites. The fatigue life increase significantly with the addition of 1% of silica aerogel. This observation can be used to conclude that the silica aerogel can act as a good filler to strengthen the fatigue strength when added into a material. This can be reasoned to the silica aerogel occupying the voids in the composite and hence help in improving the load transfer to kenaf fibers. Thus, the strength of the composite can be enhanced.

The pure epoxy exhibits better fatigue behavior compared to the kenaf composites. The incompatibility of fibers and matrix is the main cause in this behavior. The incompatibility hence brings inefficiency of stress transfer in the matrix [26].

The S-N curve established by Abdullah et al [27] was used as comparison to the generated S-N curve and illustrated in Figure 11. From the stress level of 70% UTS and higher, the trend for KE composite is similar to the curve of 15% kenaf fiber volume fraction presented by the mentioned author. Meanwhile, the curve of KES is almost identical to the curve of 45% kenaf fiber. In other words, adding 1% of silica aerogel has the same effect in improving fatigue life as in increasing the fiber volume fraction up to 45%. Abdullah et al. [27] had also reported in the same paper that the tensile properties of 45% unidirectional kenaf composite are higher than composite with 15% kenaf. Since addition of 1% of silica aerogel into 16.67% woven kenaf composite (weight fraction percentage) gives off similar trend to 45% unidirectional kenaf composite, the mechanical properties of 16.67% woven kenaf composite can also be increased with the addition of silica aerogel. This statement tallies with the finding obtained through tensile test as reported in the earlier section of this paper.

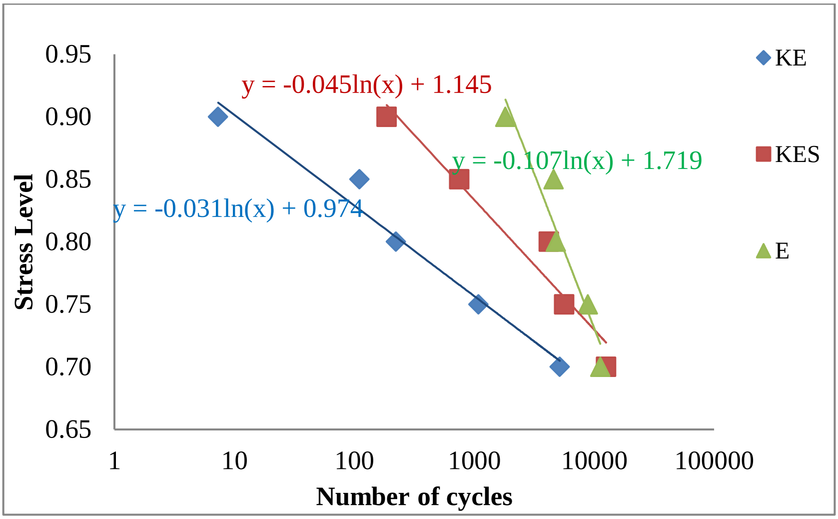


Figure 10: S-N curve for all specimens

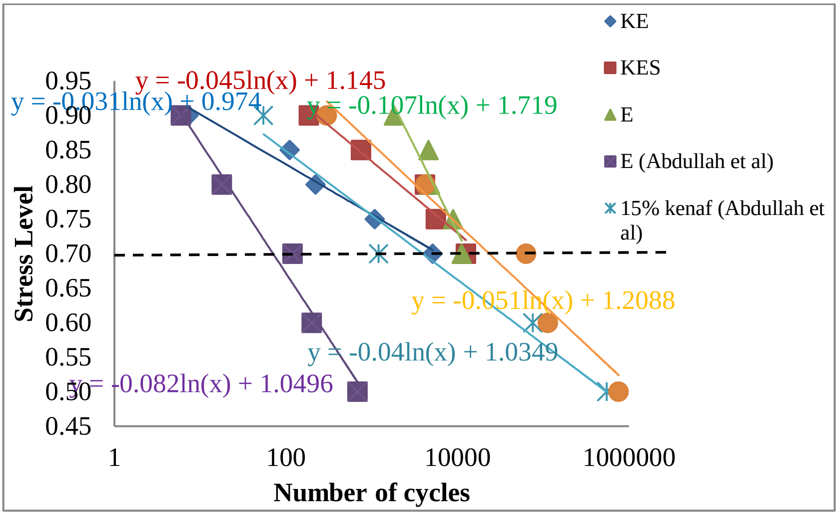


Figure 11: S-N curve comparisons with study done by Abdullah et al [29]

Observation on Specimen Failure

A high speed video camera is utilized to capture the crack formation during tension-tension fatigue test. Figure 12 shows the screenshot images from the video recorded. The position of crack that was about to take place is circled in red in the images.. It can be observed that the crack is initiated form the left side of the specimen to the right. With the frame rate of 29 frames per second, the total time taken for fracture to take place is about 3 seconds.

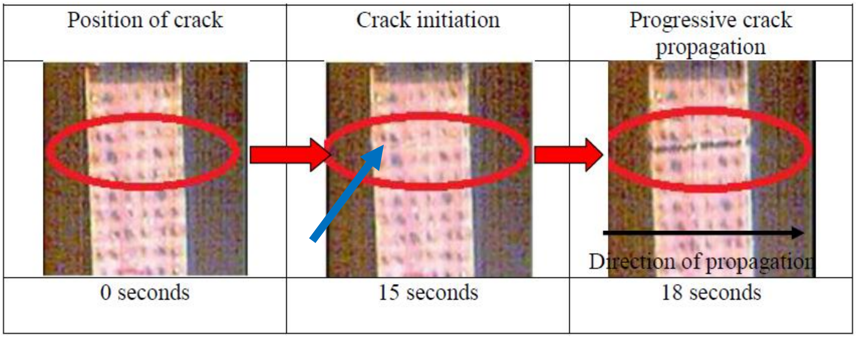


Figure 12 Crack propagation as observed using a high speed camera

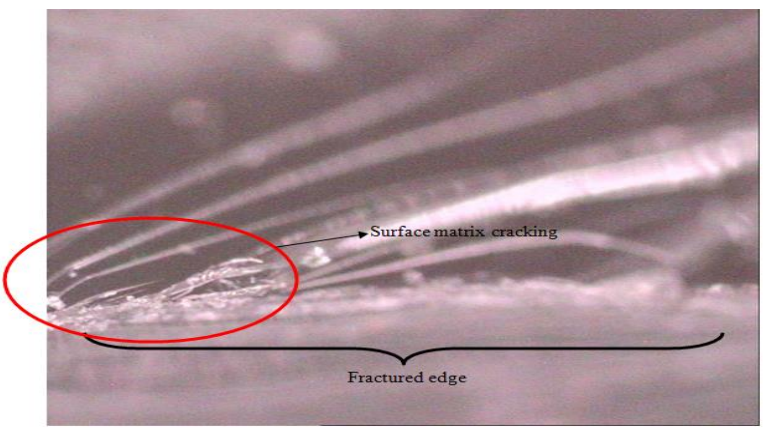


Figure 13: Surface matrix cracking of pure epoxy specimen on fractured edge ($\mathbf{10} \times$ magnification)

The fractured specimens were also observed under microscope and the fractured edges were captured and displayed into Figure 13 and Figure 14. From Figure 13, it can be observed that matrix crack initiates from the bottom left region where the intensity of crack is higher than any other region.

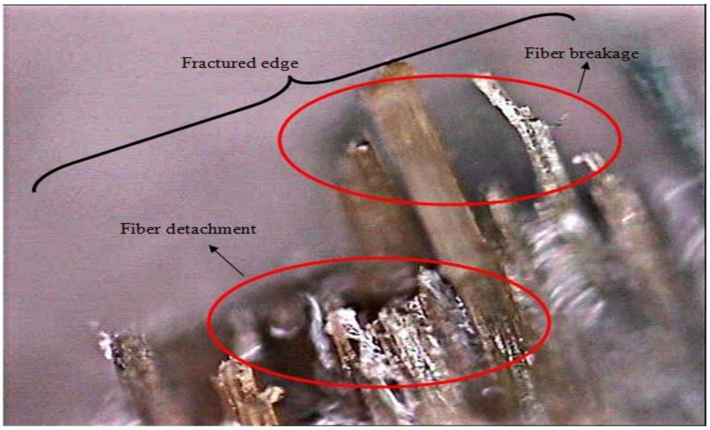


Figure 14: Fiber breakage and detachment can be observed on the fractured edge of KES specimen (10 × magnification)

Fatigue failures of fiber breakage and fiber detachment have been determined to be associated by a number of defects such as voids, resin-rich zones, misaligned fibers and regions where resin has poorly wetted the fiber [7] which can be largely due to the quality control of the manufacturing process. However, these defects are not identified as the Scanning Electron Microscopy (SEM) that is used for observation cannot be carried out due to time constraint. Fiber breakage can be observed in Figure 14. The breakage surfaces are observed to be brushier than those undergone tensile test.

Conclusion

The study had proven that the addition of 1% silica aerogel into composites improved tensile strength and fatigue properties, at the same time decreasing the stiffness owing to the ductility of silica aerogel. Hence, it can be said that there is a decrement in stiffness in exchange of better fatigue life when KES is to be implemented into any sorts of structures. KES composite can be used when higher strength is of concern, but not with application that requires high stiffness. On the other hand, the composite with silica aerogel (KES) offers better fatigue strength than composite without silica aerogel (KE). The addition of 1% of silica aerogel into the stated composite gives off similar effect with increasing fiber content to 45%, which is enhancement in fatigue strength. The composite can then be candidates for applications that are tend to be subjected to fatigue phenomenon.

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