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Predicting Fatigue and Damaged Modes Due to Defects of Bio-composite Materials

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Abstract – *In this study, fatigue failures of bio-composite materials were predicted due to manufacturing defects. Kenaf bast fibres were used to fabricate a bio-composite material with epoxy as a binding material. The bio-composites were manufactured by using a hand lay-up process. The defects in the Kenaf/epoxy bio-composite were determined by a non-destructive technique using Infrared thermal imager. Besides, the thermography analyses were verified via optical microscope and scanning electron microscope (SEM) investigations. Determinations of fatigue, as well as damage had been predicted, and it was found that the damage could be fixed with the predicted results.*

Keywords: Fatigue, Defects, Bio-composites materials, Non-destructive technique

1.0 INTRODUCTION

Nowadays, environmental-friendly composites are required to replace the conventional composites. With that, recently, the use of natural fibres, such as Kenaf fibres, as reinforcing for polymer matrices has advanced remarkably and received attention by the researchers worldwide [1-6]. Due to the increased interest in the potential of natural fibre composites or bio-composites for applications in primary structures, such as motor vehicles and building, fundamental studies of their fatigue properties are essential to allow their application where cyclic loads are experienced. Some of the applications involve components to cyclic loading. Cyclic loading will cause damage and material degradation in cumulative manner, and therefore, it is important to accurately evaluate the damage and the degraded properties to ensure that the structures of the composites could operate with high reliability during their lives. Therefore, knowledge of defects, as well as their determination, is important in cutting manufacturing cost and maintenance operation. Hence, the purpose of this study was to introduce the use of a non-destructive technique to predict the damage mode of bio-composite materials by determining the defects after cyclic loading of fatigue test. Moreover, the composites can contain a number of defects introduced during the manufacturing, which can considerably increase the likelihood of composite failure. In fact, there are many types of defects in bio-composites due to manufacturing process, such as voids, resin-rich zones,

pockets of un-dispersed cross-linkers, misaligned fibre, and regions where resin has poorly wetted the fibres [7].

Moreover, it is also essential to have a non-destructive technique (NDT) tool to predict the performance of the structures and to prove the results efficiently of experimental work done on a bio-composite material in order to schedule the maintenance or replacement of a component before its failure. Fatigue test under tension-tension load and analysis of temperature on external surface involving of thermal concept or thermal imaging technique using infra-red camera had been applied in this research. Besides, a research conducted by Toubal et al. [8] used the thermal concept or thermal imaging technique, which is an NDT, in relating damage evolution and heat dissipating in composites. Then, an analytical model based on the cumulative damage was proposed to predict the damage evolution. As claimed by Choi et al. [9], thermal imaging technique or thermography is a surface thermal radiation measurement technique that is used to detect spatial variations in the measured surface temperature pattern. Thermography reveals flaws by searching for anomalous hot-spots after thermal excitation. This robust methodology was applied to monitor the integrity of composite structures before further applications and during their operational life-cycle, as well as to anticipate failure before its occurrence to ensure reliable and safe operation. Moreover, the Infrared Thermal Imaging technique has been proved as an NDT to detect the defects in Kenaf/epoxy bio-composite materials [10].

2.0 METHODOLOGY

2.1 Materials

In this study, the material used was treated Kenaf bast fibre provided by the Institute of Tropical and Forest Product (INTROP), UPM, Serdang, Selangor, Malaysia. The raw Kenaf bast fibres were combed to disrupt and untangle the strong bonds between the individual fibres. Bernard et al. [11] reported that combed fibre exhibits stronger mechanical properties than uncombed fibre. The Kenaf bast fibres were cut to a length of 25cm using scissors. Epoxy with a density of 1.15 g/cm³was used as a binding material. Figures 1(a) and (b) show the treated Kenaf bast fibre raw materials and the unidirectional Kenaf bast after being combed and cut to 25 cm.

2.2 Preparation of Epoxy Samples

Epoxy samples were produced to control and to determine the optimum temperature, as well as the curing time for processing the Kenaf/epoxy bio-composite. The volume of epoxy used was 112.5 cm^3 for sample thickness of 0.3 cm, which was equal to 129.4 g based on the volume of the mould, in accordance with ASTM D3039 [12].

2.3 Fabrication of Kenaf /Epoxy Composites

The composite samples were prepared by combining the Kenaf bast long fibres and the epoxy resin through a hand lay-up process, and then, compressing the composite by using a hot-press compression moulding, with a mould similar to that used to fabricate the epoxy samples. One of the main issues with the Kenaf/epoxy bio-composite that needs to be addressed is uneven fibre distribution [13]. Kenaf fibres are difficult to be manually separated and visually dispersed evenly during manufacturing [14].

The composites were compressed by using a hot-press compressing machine at 70 ^oC and a pressure of 50 Bars. The mould was pre-heated for 3 minutes, followed by pressing for 20

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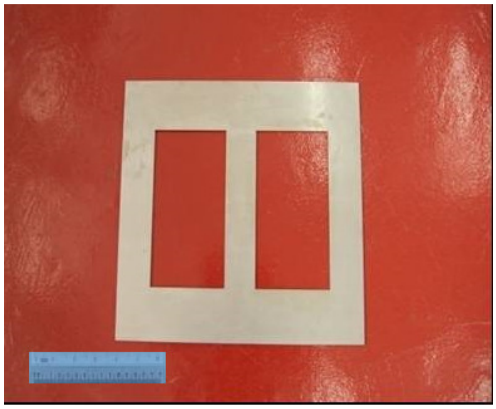
minutes, and venting for 7 times to maintain its uniform thickness, besides preventing air from entering the mould. Figures 2(a) and (b) show the mould used to fabricate the Kenaf/epoxy specimens.

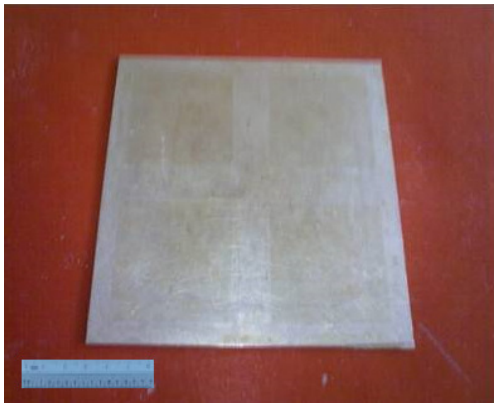
(a)

(b)

Figure 1(a) and (b): Raw materials of Kenaf bast fibres and unidirectional Kenaf bast fibres after combing, respectively



(a)



(b)

Figure 2(a) and (b): Moulds used to fabricate the Kenaf/epoxy specimens

2.4 Testing

The determination of defects on Kenaf/epoxy bio-composite was done by using IR Thermal Imager. Then, a thermography analysis via IR thermal imager was verified by using Optical Microscope and Scanning Electron Microscopy (SEM).

2.4.1 IR Thermal Imager

Infrared and thermal measurement, as well as investigation techniques, had been employed to determine the defects on the surface of the Kenaf/epoxy composite specimens by using an Infrared (IR) Thermal Imager model Fluke Flexcam. The investigation and the measurements used active thermography, whereby the Kenaf/epoxy specimens were heated in an oven at 100 ^{o}C for 60 minutes before the thermography results were recorded. In active thermography, the surface of the sample was heated by using an external heat source and the surface was

monitored [15]. The Kenaf/epoxy specimens were placed on a non-metal stand to avoid heat dissipation through conduction from the stand. These experiments were conducted for an emissivity of 0.92, with a room temperature at 29.9 ºC, and relative humidity of 70%.

2.4.2 Optical Microscope

An optical microscope was used to verify the detection of defects on the Kenaf/epoxy composite surface via thermography.

2.4.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was used to determine the defects in the Kenaf/epoxy composites and to verify the results obtained from the IR thermal imager and the optical microscope. A Hitachi S-3400N SEM was used. The Kenaf/epoxy specimens were cut to 0.5cm height x 1.5cm width at three difference thicknesses; 0.1cm, 0.3cm, and 0.5cm, with an electrical hand-saw. The surfaces of the specimens were coated with a mixture of 80% Gold and 20% Palladium.

2.4.4 Fatigue Test

Fatigue tension-tension test was carried out with ASTM D 3479/ D 3479M-96 [16], which is a Standard Test Method for Tension-Tension Fatigue of Polymer Matrix. The composite materials were tested by using the INSTRON model 8871 machine. The fatigue tension-tension tests were conducted on 90 pieces of specimens varied in fibres percentages loading and thicknesses. The load used for this test was 25kN at a room temperature of 30 ˚C.

3.0 RESULTS AND DISCUSSION

Many types of defects can be found in kenaf/epoxy composites due to manufacturing process, such as voids, resin-rich zones, pockets of undispersed cross-linker, misaligned fibre, and regions where resin has poorly wetted the fibres [17]. The defects were determined by using IR camera, optical microscope, and SEM. In this study, it had been found that the types of failures, such as fibre breakage, fibre pulls-out, and fibre irritation, had been predicted earlier due to the types of defects. As report by [17], [18], [19], and [20], composite fatigue failure is generally driven by fatigue failure in polymer matrix. Table 1 shows the prediction of the types of fatigue failures through determination of defects.

Table 1: Prediction of failures for kenaf/epoxy through determination of defects

Types of Defects	Prediction of Fatigue Failures
Voids	Fibre breakage
Resin-rich zones	Fibre irritation
Pocket of undispersed cross-linker	Fibre breakage
Misaligned fibre	Fibre breakage and pull out
Regions where resin has poorly wetted the fibre	Fibre pull out

The summary of the finding in Table 1 was verified via fatigue test and the determination of the failure had been done via SEM. It was discovered that the summary of the finding had been fixed with the results obtained from optical microscope and SEM. The specimen that contained

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a lot of voids; it was predicted that this specimen of kenaf/epoxy composites had fibre breakage when it experienced fatigue failure. Figure 3 illustrates fibre breakage.

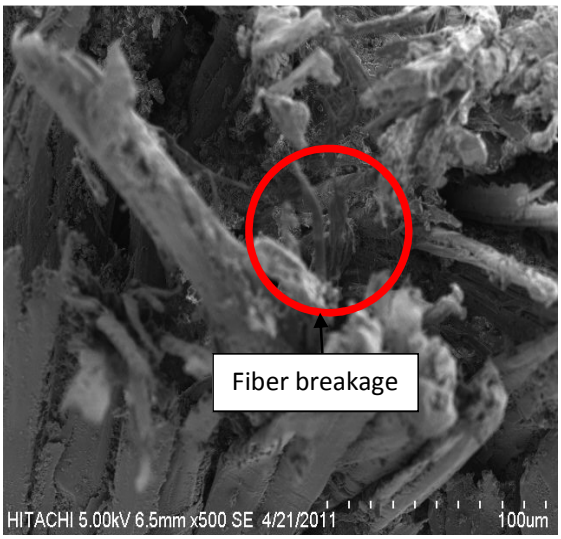

Figure 3: Fibre breakage

The specimen that showed resin-rich zones in kenaf/epoxy composite materials was predicted to have a fibre pull-out when it experienced fatigue failure. Figure 4 shows fibre irritation.

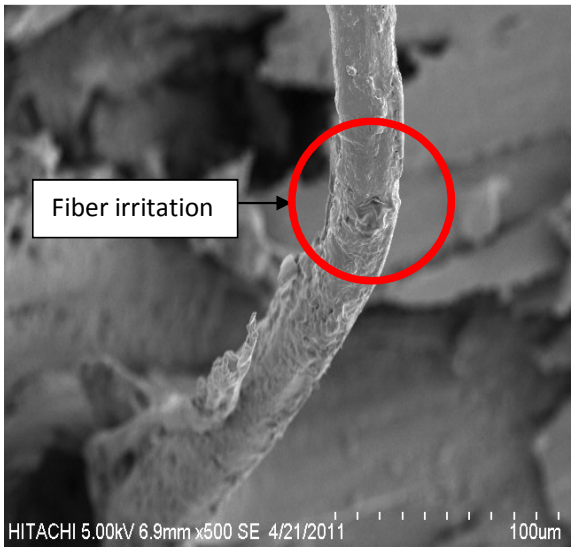

Figure 4: Fibre irritation

Pockets of undispersed cross-linker may be caused due to incomplete curing in kenaf/epoxy composite materials. It was predicted that this specimen of kenaf/epoxy composites had a fibre pull-out when it experienced fatigue failure. Figure 5 shows fibre pull-out due to pockets of undispersed cross-linker.

Journal of Advanced Research Design
ISSN (online): 2289-7984 | Vol. 9, No.1. Pages 1-12, 2015

Figure 5: Fibre pull-out

The specimen that contained misaligned fibre in kenaf/epoxy composite materials was predicted having fibre breakage when it experienced fatigue failure. Figure 6 shows fibre breakage and fibre pull-out due to misaligned fibre.

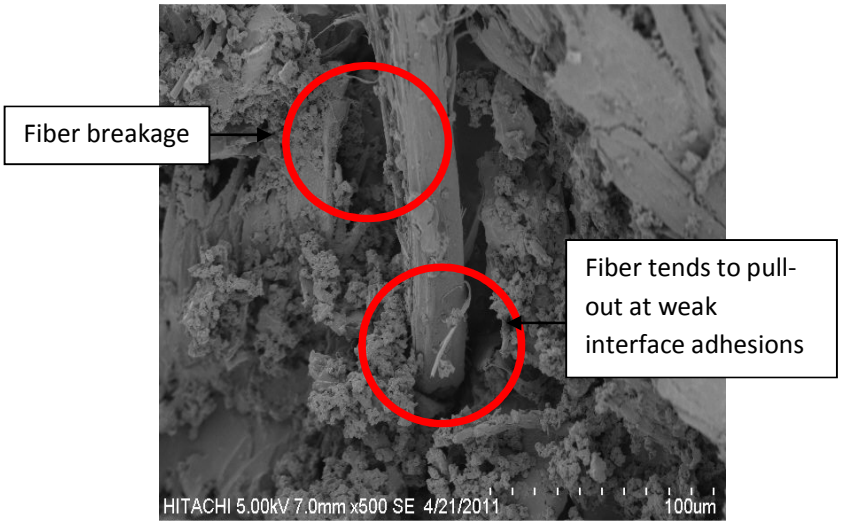

Figure 6: Fibre breakage and tended to pull-out

It was predicted that the region where resin had poorly wetted the fibre in kenaf/epoxy composite materials, this specimen would have fibre breakage when it experienced fatigue failure. Figure 7 shows fibre pull-out due to poorly resin-wetted fibre.

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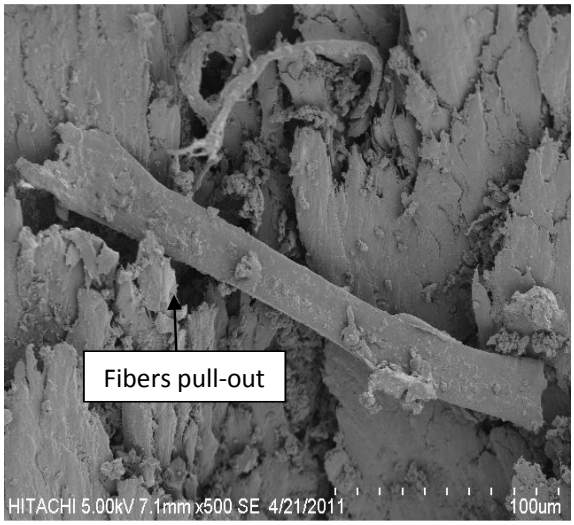
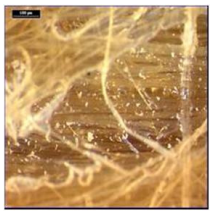
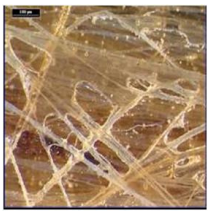
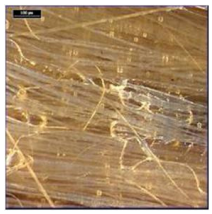
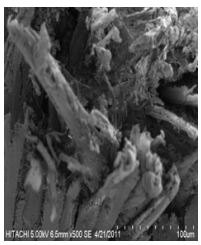
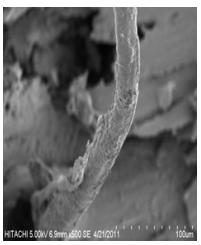


Figure 7: Fibre pull-out

Table 2 shows the summary of determination of fatigue failure in kenaf/epoxy composite due to defects depicted by SEM.

Table 2: Determination of fatigue failure in kenaf/epoxy composite due to defects via SEM

Defects	**Voids**	**Resin-rich zones**	**Pocket of undispersed cross-linker**	**Misaligned fibre**	**Regions where resin has poorly wetted the fibre**
					
Failure	**Fibre breakage**	**Fibre irritation**	**Fibre breakage**	**Fibre breakage**	**Fibre pull-out**
					

Journal of Advanced Research Design
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6.5 Fatigue Damage Determination

The results of fatigue test for all specimens are presented in Table 2. Determinations of fatigue damage had been done by using IR thermal imager and SEM. Determinations of fatigue damage were predicted (Refer Table 1) and it was found that the damages had been fixed by the predicted results, as shown in Table 2.

The specimen with 45% of kenaf/epoxy, which was determined to contain a lot of defects of pockets of dispersed cross-linker, showed mostly fibre breakage after fatigue test. It also found that the failure of specimen with 60% of kenaf/epoxy had been catastrophic without pull-out of fibre from the specimen. Meanwhile, the specimen with 75% of kenaf/epoxy showed to have the most fibre breakage. This was because; the specimen with 75% of kenaf/epoxy had been determined to contain a lot of voids.

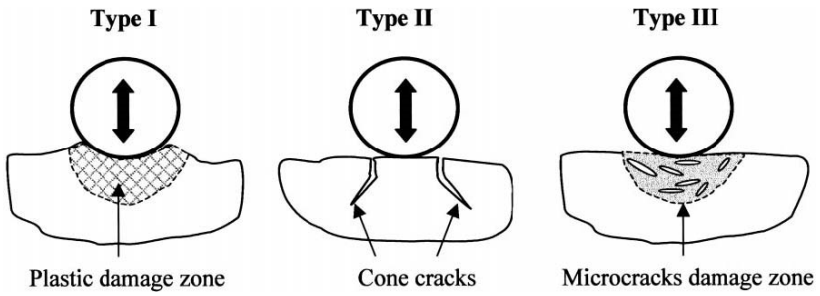

Figure 8: Schematically, the various types of surfaces and subsurface damages [21]

In order to understand the fatigue failures in biomaterials, it is essential to have some comprehension pertaining to the surface substructure of biomaterials. Figure 8 shows, schematically, the various types of surfaces and subsurface damages that were exhibited by different materials under a spherical indentor. Type I behaviour is typical of metallic materials with a high fracture of toughness and high ductility. Besides, a plastic zone built with material around the indentor had been obvious. Meanwhile, type II behaviour is typical of brittle material with high yield strength, but low fracture toughness (such as in some bioceramics). The damage zone beneath the indentor was basically elastic and a cone crack formed near the perimeter of the indentor. Lastly, the type III behaviour was quasi-brittle and was typical of materials with moderate toughness and yield strength. Mirco-cracking was often observed in the damage zone [21].

In composite materials, fatigue damage may be caused by fibre breakage, matrix cracking, or fibre/matrix debonding, as claimed by Chamis (1989) [22]. Figure 9 shows the specimens of pure epoxy, 45%, 60%, and 75% kenaf/epoxy after fatigue test, respectively. Hence, it can be concluded that by increasing fibre loading, the facture surface was rougher with brushier appearance.

Figure 10 shows the specimen with 60% of kenaf /epoxy after the fatigue test, while Figure 11 shows epoxy (matrix) cracking after fatigue tension-tension test was observed via SEM. SEM portrayed that a crack occurred in kenaf/epoxy when it experienced fatigue tension-tension loading in specimens with 60% of kenaf/epoxy. Besides, it was observed that fatigue damage development of kenaf/epoxy composites began with filler/matrix interface failure, followed by initiation and propagation of matrix, cracks from the filler matrix cracks from the filler/matrix debonding site, and subsequent development of longer matrix cracks from shorter cracks that

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caused final failure. In addition, Tang et al. (2004) [21] also found that this phenomena happened in their research on fatigue damage development via SHA/PEEK. Moreover, filler-matrix debonding contributed to stress raisers, where the local stress was intensified, leading to matrix deformation, as well as initiation, and propagation of micro cracks. As claimed by Gassan (2002) [23], the damage in natural fibre composites diffused growth by fibre-matrix debonding or crack bridging, and this is governed by fibre-matrix interaction. Therefore, kenaf/epoxy bonding is a critical issue that needs to be addressed for improvement of the performance of this composite material. Besides, the experimental studies conducted by [24-26] summarized the influence of the fibre-matrix interface on the fatigue behaviour of carbon fibre and glass/epoxy cross-ply composites. It was found that the fatigue performance was improved by increasing the interface strength.

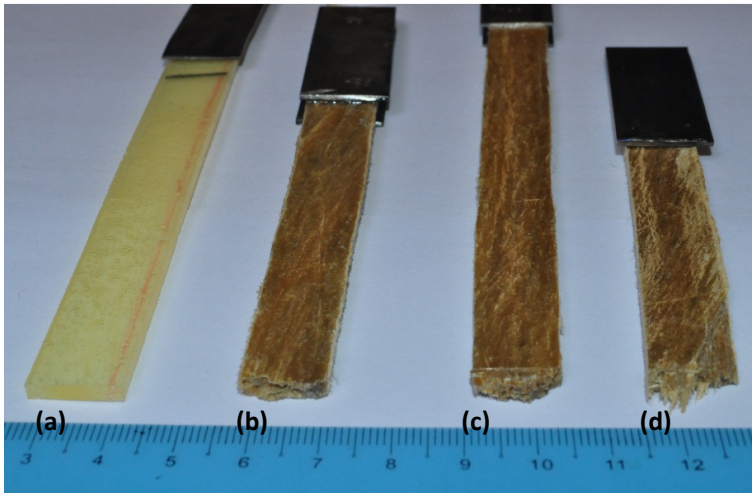

Figure 9: Specimens (a) pure epoxy, (b) 45% kenaf/epoxy, (c) 60% kenaf/epoxy, and (d) 75% kenaf /epoxy after fatigue test

Figure 10: Specimen with 60% of kenaf /epoxy after fatigue test

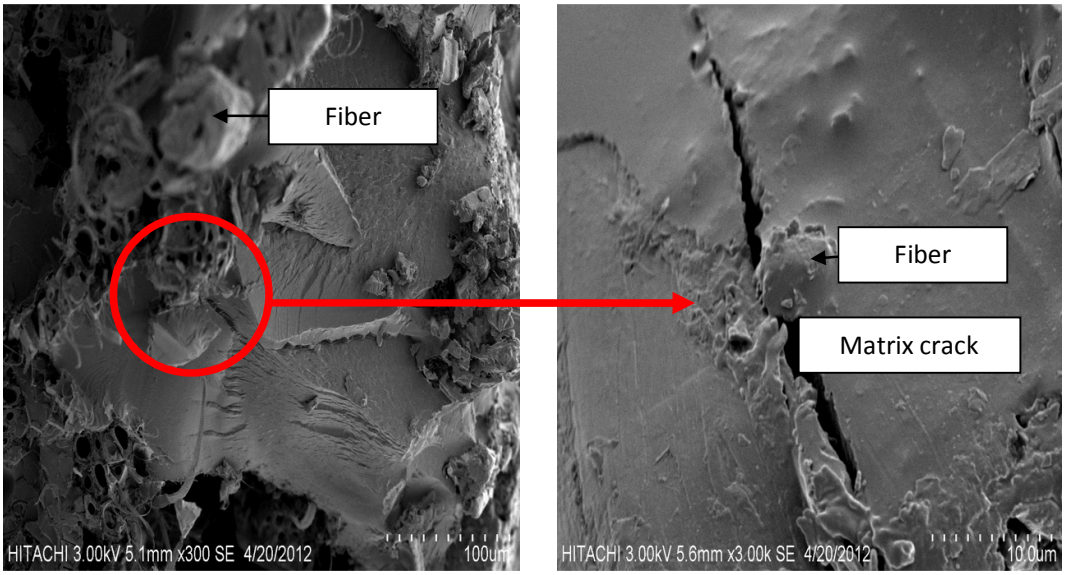

Figure 11: SEM shows crack in kenaf/epoxy specimen after experiencing fatigue tension-tension test

4.0 CONCLUSION

It can be concluded that infrared thermal imaging technique and SEM proved to be an NDT in determining the defects and the damage modes in bio-composite materials under fatigue loading. The determinations of defects via IR thermal imaging and optical microscopes had been fixed to the prediction of damage modes determined by SEM.

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