Fabrication of Fibre Metal Laminate with Flax and Sugar Palm Fibre based Epoxy Composite and Evaluation of their Fatigue Properties

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ABSTRACT

Fibre metal laminate (FML) with the carbon, flax and sugar palm fibres were prepared by hand layup and hot press technique. Their tensile and fatigue properties were studied. Results indicate that both the tensile properties and fatigue behaviour was dependent on the natural fibre type and their stacking sequence in the laminate. Flax based FML showed promising results with superior tensile properties and fatigue life. It also displayed fibre bridging mechanism, a phenomenon responsible for the higher fatigue life. Introducing flax in combination with sugar palm fibre resulted in slightly higher fatigue life than the sugar palm fibre based FML. FML with the sugar palm fibres showed the least fatigue life below 1000 cycles and did not exhibit fibre bridging effect. This behaviour indicates that sugar palm fibre may not be a suitable fibre for applications requiring fatigue resistance.

KEY WORDS : *Fibre metal laminates, Tensile properties, Fatigue life, Fatigue sensitivity, Natural fibres*

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INTRODUCTION

Fibre metal laminates (FML) are high strength materials made up of alternating layers of the metal alloy and composite layers adhesively bonded together. The commercially available FML such as CARALL, ARALL and GLARE used in aircraft structures constitutes Al alloy, carbon, aramid and glass fibre based composites respectively[1]. FML is a lightweight material that combines the advantages of both the metal and composite due to their higher strength to weight ratio and resistance to impact loads[2].

During the service life of aircrafts, their structures are highly susceptible to fatigue loads. One of the reasons for successful application of FML like GLARE and CARALL in aircrafts is their superior resistance to fatigue loads[3]. Number of studies have been undertaken to explore the fatigue properties of such materials. Their response to fatigue load and failure mechanism has been well documented. It was found that GLARE and CARALL has fatigue life in the range of $10^5 – 10^6$ cycles [4]. Despite the successful usage of commercially available FML in aircrafts, some of the drawbacks like difficulties in recycling, disposal requirements, demand for the synthetic fibres and high production cost of the synthetic fibres have urged the need to develop environment friendly or bio-degradable material[5]. Over the years, natural fibres from the plants have been emerging as a potential substitute for synthetic fibres. The main advantages of using natural fibres include abundance, low cost, non-toxicity and lower extent of damage to tools due to their non-abrasive nature [6]. Use of natural fibres as reinforcement in FML is slowly gaining attention. In general, the FML used in high performance applications such as aircraft structures is fabricated using autoclave. In the autoclave, FML specimens stacked in the layering arrangements is cured under the pressure and temperature in the vacuum atmosphere. However, this process is highly expensive and time consuming. Therefore, out of autoclave (OOA) curing process such as hand lay-up, hot press moulding, vacuum assisted resin transfer moulding (VARTM), etc. have been developed by researchers as an alternative. VARTM process involves infusing resin within the stacked layers of metal and composite in FML through the flow distribution system that has tubes and vacuum pump[7]. In hand lay-up method, the fibre layers along with the resin was placed between the metal layers followed by curing at room temperature [8]. The main drawback of this method is the difficulty in obtaining required thickness and higher void content due to the lack of applied pressure. Hot press moulding is the most commonly used method other than the conventional autoclave curing due to their simplicity, ease of handling, cost effective and fast curing abilities. FML with the sisal [9], oil palm empty fruit bunch fibres [10] and kenaf fibre kenaf [11, 12] were fabricated using the composite plies and epoxy adhesive layers stacked between the metal layers followed by compression of the stacked layers at high temperature under pressure for few hours. FML reinforced with the flax and kenaf fibres fabricated from the hot press moulding. Recent studies on the fatigue properties of FML reinforced with the flax fibres and kenaf fibres showed promising results with the specimens exhibiting failure beyond 10^4 cycles [13, 14]. So, this signifies the suitability of some natural fibres as potential reinforcements in FML.

In this study, the natural fibres such as flax and sugar palm were introduced into the laminate along with the carbon prepreg in 2/1 layup sequence and the FML was fabricated by hot press moulding. The FML specimens with various stacking sequence were subjected to fatigue loads and their fatigue life was recorded. According to the author's knowledge, fatigue behaviour of FML with sugar palm fibres have never been addressed. Studies on fatigue properties of the natural fibre reinforced FML were limited to determination of the number of cycles to failure. In addition to the fatigue life, fatigue sensitivity and fractional loss of strength per decade of cycles was also evaluated.

2. MATERIALS AND METHODS

2.1 Materials

6061-T6 Al alloy sheet (Al) of thickness 0.5mm used in this study was procured from Metalfort, India. 2 x 2 twill carbon prepreg (C) with 60:40 fibre and resin content was purchased from Shangai Liso Composite Technology Co. Ltd., China. 150 gsm Biotex flax fibres (F) in the unidirectional tape form and XA120 prepreg adhesive film (E) were procured from Easycomposites, UK. Sugar palm fibres (S) were obtained from Negeri Sembilan in Malaysia through Hafiz Adha Enterprise, Malaysia. D.E.R. epoxy resin with 905-3S joint amine type hardener for composite fabrication, ethanol & Tri-ethoxy(ethyl)silane for metal surface preparation was supplied by MZI supplies, Malaysia.

2.2 Fabrication method

2.2.1 Metal surface pre-treatment

6061-T6 Al obtained from the supplier was initially cleaned with acetone to remove surface contaminations. The surface preparation of Al substrate involves mechanical abrasion followed by the silane treatment. In mechanical abrasion, Al substrate was scrubbed with the 120 grit silicon carbide sand paper to create a rougher surface texture. Abraded Al sheets were then cleaned with acetone to remove residues and subsequently treated with 5% volume aqueous alcoholic silane solution. The solution was prepared by mixing 90/5/5 volume proportion of ethanol/distilled water/tri-ethoxy(ethyl)silane and stirred for 2 minutes. Al sheet metal was then dipped in the prepared solution for 60 seconds and dried in an oven at 110 °C for 10 minutes [15]. The treated sheet metals were used to fabricate FML within 24h of treatment to avoid the aging effects.

2.2.2 Fabrication procedure

Prior to the fabrication, the naturally woven sugar palm fibres were washed with water, dried in an oven at 80 °C for 24h and hot pressed at 80 °C for 10 minutes at 10 ton pressure to form a compressed fibre mat. This provides even thickness and good distribution of fibres compared to the random and fluffy structure of the "as received" fibres.

FML was prepared by hand lay-up and hot press moulding in 2 steps. Firstly, natural fibre reinforced composites (NFC) were fabricated by stacking fibre layers and epoxy/hardener mixture into the 150*150*2mm^3 mould followed by compression in hot press at 105 °C for 10 minutes with 10 ton pressure. Secondly, the surface treated Al sheets, epoxy film, carbon prepreg and prepared NFC were stacked in 2/1 lay-up and cured in hotpress at 105 °C for 10 minutes with 40 ton pressure as shown in Figure 1. The machine was switched off and FML sample was left overnight in the compressed position. Cured FML was then cut with the band saw into coupons for assessment of the tensile and fatigue properties.

2.3 Tensile test

Uniaxial tensile test was performed as per ASTM D3039 standard to determine the ultimate tensile strength of pristine and conditioned specimens. Specimens of dimension 150*15* ~ 4.5 mm^3 were loaded in INSTRON 3382 and a tensile load was applied at a cross head displacement rate of 2mm/min. 5 specimens were tested in each configuration and average results were reported.

2.4 Fatigue test

Constant amplitude tension-tension fatigue tests were carried out on FML specimens as per ASTM E466 to

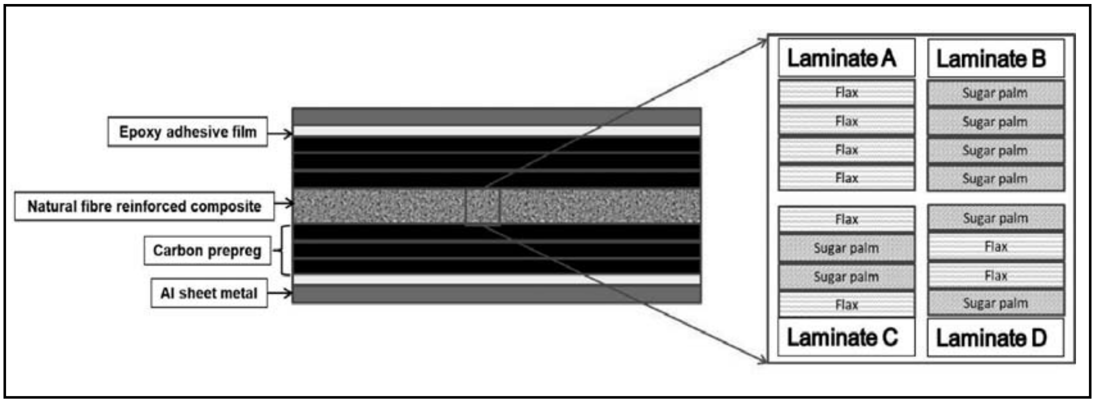

Figure 1. Schematic of 2/1 layup FML specimens with notation

determine the fatigue life under laboratory conditions using MTS 810. Fatigue tests were performed at 80%, 70% and 60% of the ultimate tensile strength. A stress ratio of 0.1 (Equation (1)) and frequency of 10Hz was maintained. 3 specimens were subjected to fatigue test in each configuration and the results were reported. Fatigue performance was evaluated by normalized S-N plot, fatigue sensitivity (Equation (3)) and fractional loss of strength per decade of cycles (Equation (4)).

$$\text{Mean stress} = \frac{S_{max} + S_{min}}{2} \qquad (1)$$

$$S_{max} = S_{ult} + k \, logN \qquad (2)$$

$$\text{K}= \frac{S_{max} - S_{ult}}{\log N} \qquad (3)$$

$$\frac{k}{S_{ult}} = \left(\frac{\frac{S_{max}}{S_{ult}} - 1}{\log N} \right) x \; 100\% \qquad (4)$$

where S_{ult} is the ultimate tensile strength of each specimen from the tensile test (MPa), S_{max} is the maximum stress level such as 80%, 70% and 60% of S_{ult}(MPa), N is the number of cycles to failure and “b” is the slope or fatigue sensitivity.

2.5 Fractography

The failure behaviour of fractured specimens from the tensile and fatigue test was studied through the visual images and microstructure from the scanning electron microscope (SEM). Specimens were sputtered with gold coating prior to the observation under Hitachi S-3400N SEM.

3. RESULTS AND DISCUSSION

3.1 Tensile properties

The ultimate tensile strength and tensile modulus of FML specimens obtained from the tensile test is given in Figure 2.

It could be observed from Figure 2 that the ultimate tensile strength and tensile modulus of the FML with the hybrid natural/synthetic fibres was dependent on the natural fibre type and their stacking sequence within the laminate. Laminate A with the unidirectional flax fibre showed maximum strength compared to the laminate D with the sugar palm fibres. In this study, FML with flax fibres exhibited 267 MPa which was only 7 % lower than the monolithic 601-T6 counterpart. The superior strength for F/F is credited to the high tensile strength and stiffness for the unidirectional flax fibre. The unidirectional fibres also carry and distribute the load effectively in the loading direction [12]. Sugar palm fibres have 50% lower tensile

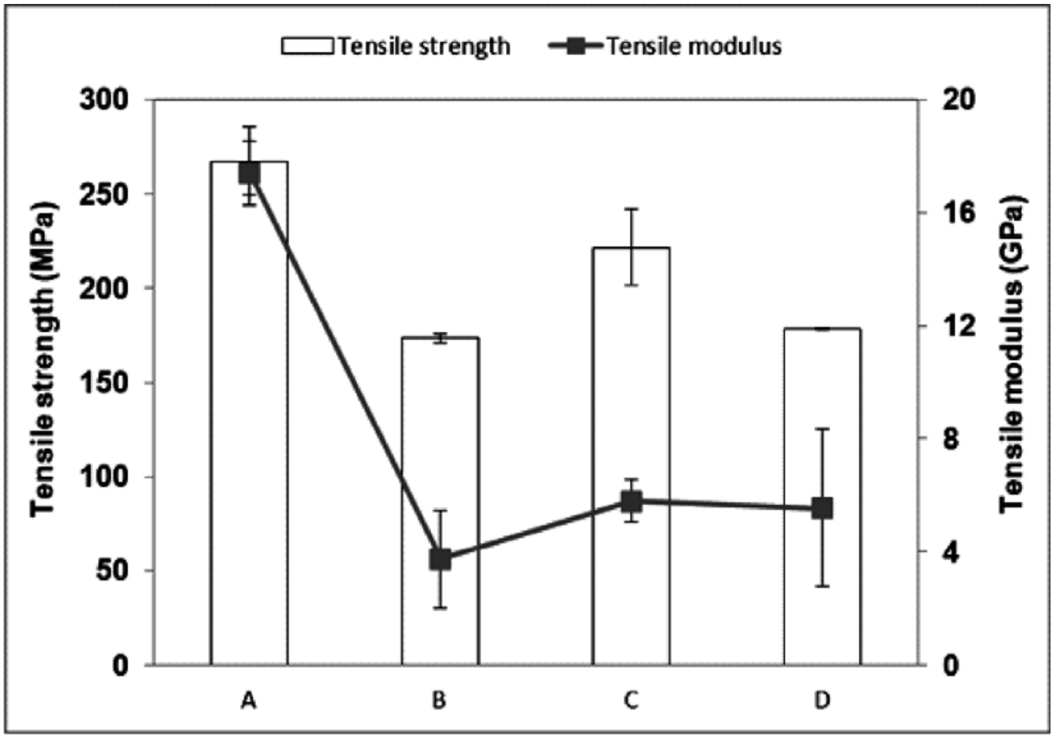

Figure 2. Tensile properties of the studied laminate

strength and nearly 10 times lower tensile modulus than the flax fibres. Thus, all the studied configurations with sugar palm showed lower tensile strength and tensile modulus. The advantage of hybridizing flax with sugar palm fibres was beneficial in terms of strength and modulus. Hybrid configurations, laminates B & C showed intermediate properties compared to the individual flax and sugar palm based FML. According to Feng et al[16], the outer fibre layer is the main load bearing component in a FML with the hybrid fibre configuration while the inner fibre layer contribution is less significant. Similar pattern was noticed in hybrid configurations. Laminate C presented a greater strength and modulus due to the presence of stronger flax fibre in the outer layer than the laminate D with sugar palm fibre as the outer layer. The magnitude of tensile modulus for hybrid and sugar palm based configuration was approximately between 4GPa – 6GPa. These values though lower than their counterparts like composite and monolithic metal alloy was in the similar range as reported by other researchers on FML with the natural/ synthetic fibres (Table 1).

3.2 Fatigue properties

3.2.1 Fatigue life

The number of cycles to failure at 80 - 60 % of ultimate tensile strength recorded from the

TABLE 1.Comparison of tensile properties with the previous studies on FML with natural/synthetic fibres

Properties	Al, carbon, flax	Al, carbon, sugar palm	Al, carbon, and jute	Al, carbon, and flax	Al, carbon, and kenaf
	Author		[17]	[8]	
Fabrication method	Hand layup and hot press technique		Hand lay-up & compression molding at 80°C for 4h.	Hand layup & compression moulding	
Tensile strength (MPa)	267.20	173.38	~174	375	310
Tensile modulus (GPa)	13.83	3.12	~3.8	2.68	2.65

user interface of MTS 810 is presented in Table 2 and the normalized S-N plot is shown in Figure 3.

The fatigue life was found to be dependent on the type of natural fibre in the laminate configuration and magnitude of applied load. At 80% load, the fatigue failure occurred at 10^3 cycles for laminate A with flax fibres and the number of cycles to failure increased to 10^4 cycles at 60% load. The general observation was that lower the stress level, higher the fatigue life. This trend was also observed in the other laminates. However, the fatigue failure occurred at shorter cycles for laminates B, C and D.

TABLE 2. Mean fatigue life, fatigue sensitivity and fractional loss of laminates A – D

Laminate	Stress level (%)	Maximum (MPa)	Mean cycles to failure	Fatigue sensitivity "k"	Fractional loss k/S_{ult} (%)
A	80	213.76	8560	-12.71	5.09
	70	187.04	12287	-18.34	7.34
	60	160.32	38207	-21.83	8.74
B	80	138.7	535	-13.39	7.45
	70	121.37	1313	-17.28	9.62
	60	104.03	1808	-22.06	12.28
C	80	177.29	3629	-11.59	5.62
	70	155.13	5393	-16.59	8.04
	60	132.97	6583	-21.62	10.48
D	80	142.54	2575	-11.79	5.86
	70	124.73	2940	-17.40	8.65
	60	106.91	4366	-22.10	10.99

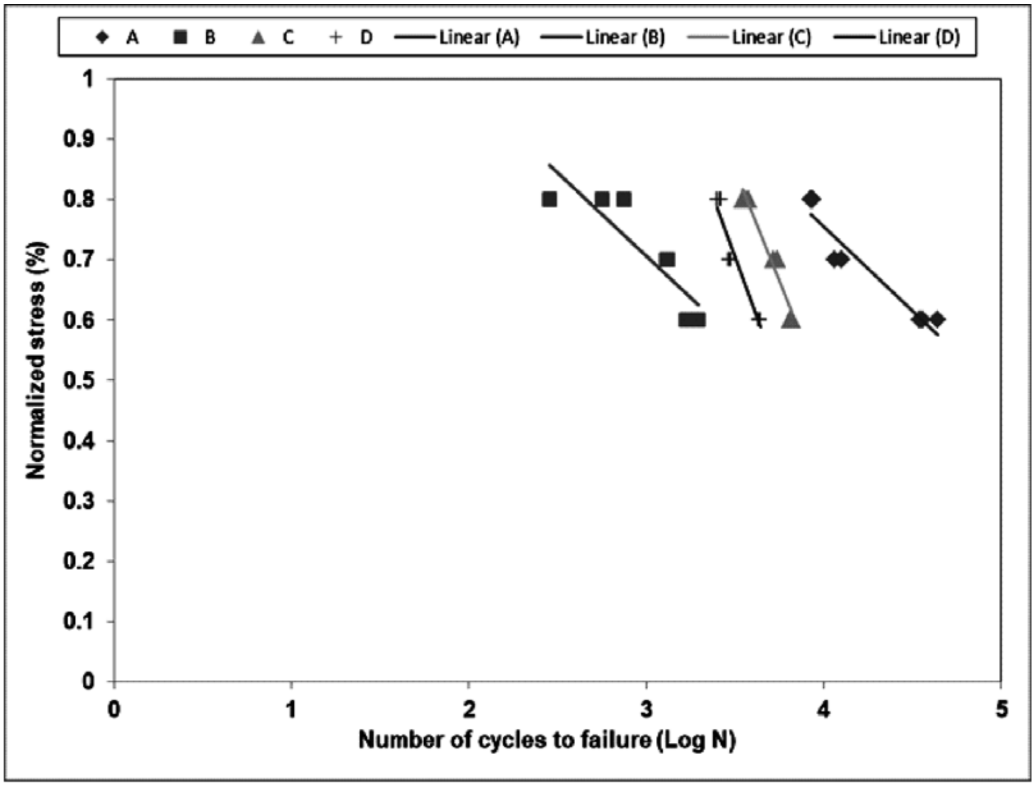

Figure 3. Normalized S-N plot of the laminates A - D

Laminate B with sugar palm fibres showed the least fatigue life between 10^2-10^3 cycles at 80 - 60 % load. Laminates C and D showed higher resistance to fatigue load than laminate B. Their fatigue life was in the range of 10^3 cycles. The reason for superior performance of laminate A over the laminate B is attributed to the superior tensile strength of flax fibres and unidirectional tape form which exhibits maximum strength in the loading direction. The advantage of FML over the monolithic Al alloy and composites is their fibre bridging mechanism.

FML in this study is an adhesively bonded structure that consists of adhesive film, carbon fibre prepreg, NFC, and metal sheet bonded together. According to Mandell [18], when the fatigue failure occurs between $10^2 – 10^6$ cycles in a bonded structure, normalized S-N curve can be linearized to a straight line as shown in Figure 3. Slope of the linearized S-N curve between each stress level (S_{max}) and S_{ult} can be calculated from the Equation (4) and Equation (5).

The slope represents fatigue sensitivity or fatigue resistance of the adhesively bonded structure. Similarly, the fractional loss of fatigue strength per decade of cycles gives an assessment of the degradation in fatigue strength at each stress level. According to Broughton et al., [19], the fatigue resistance of the adhesively bonded structure depends on the geometry of the structure and applied fatigue load. Similar inferences on the slope or fatigue resistance were observed for the FML specimens (Table 2). The fatigue sensitivity of FML specimens improved with the decrease in stress level. At 60% load, all the tested specimens regardless of the fibre configuration showed the highest slope. This implies better performance as the material can withstand longer cycles before failure. Similarly, laminate A with the flax fibres exhibited the least fractional loss value compared to the laminates with sugar palm fibres. A lower fractional loss for a material indicates better fatigue performance. However, the fractional loss was found to increase with the decrease in stress level. Both the fibre type and fibre architecture or fibre orientation in the laminate was found to influence the fatigue sensitivity and fractional loss. Similar observations were also made in the recent study on glass/kenaf based FML [16].

3.4 Failure behaviour

It is well known that FML exhibits fibre bridging effect when subjected to the fatigue load. On the application of fatigue load, the fatigue damage starts as crack initiation in Al followed by nucleation and growth until failure of Al layer. Following the failure of Al layer, the fatigue load is transferred to the fibre reinforcement which is known as the fibre bridging effect. This ability of the glass and carbon fibre reinforcements to sustain the bridging stresses post crack growth and failure in the Al layer provides superior performance under the fatigue loads [4]. Among the studied laminate configurations, fibre bridging effect was observed only in case of laminate A with flax fibres at all stress levels as per the visual observation during the test.

The failure initiates in Al layer as crack on both the sides referred to as “through the crack” or “surface crack” with crack on one side while the other side remains intact [3]. In this study, the surface crack was observed followed by delamination of the metal/composite plies and fibre bridging until failure during the test. Initially, a single crack occurred in Al followed by the fibre bridging and the second crack as final breakage. So, cracks in Al layer on either side of the specimen could be seen as shown in Figure 6. Multiple cracks in the Al layer of FML was also reported in a previous study [13].

During the test, fibre bridging effect could not be noticed for laminate B regardless of the stress level. Signs of the sudden fracture like fibre breakage, fibre splitting, composite ply failure and brittle fracture in Al layer could be observed (Figure 4(a)). This implies that the sugar palm fibres have poor resistance to fatigue loads and cannot sustain the bridging stress transferred from the Al. Bridging effect could not be noticed in case of laminates C and D (Figure 4(b) & 4(c)) as well. However, the presence of high strength and stiffer flax in the hybrid configurations helped the FML to withstand few hundred cycles higher than the laminate B.

On the other hand, since the density of the flax and sugar palm fibres were different, fibre distribution in the NFC could also have an impact on the material behaviour. In order to study the fibre distribution, the fractured specimens of laminate A, B, C and D at 80%

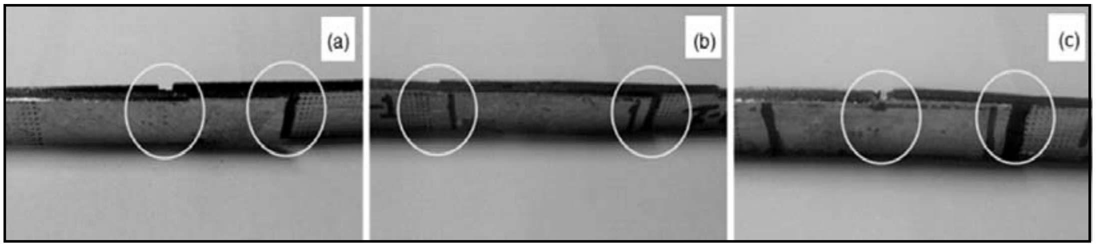

Figure 4. Fatigue failure in laminate A showing multiple cracks in Al layer a) 80% load b) 70% load and c) 60% load

load from the fatigue test was observed under SEM. The specimens tested at 80% load exhibited complete delamination between the metal/prepreg/NFC exposing the inner composite layer. Thus, these specimens were taken for fibre distribution analysis. From the microstructure shown in Figure 5, it could be noticed that unidirectional flax fibres were of identical size and had uniform distribution within the composite. This helps to withstand and

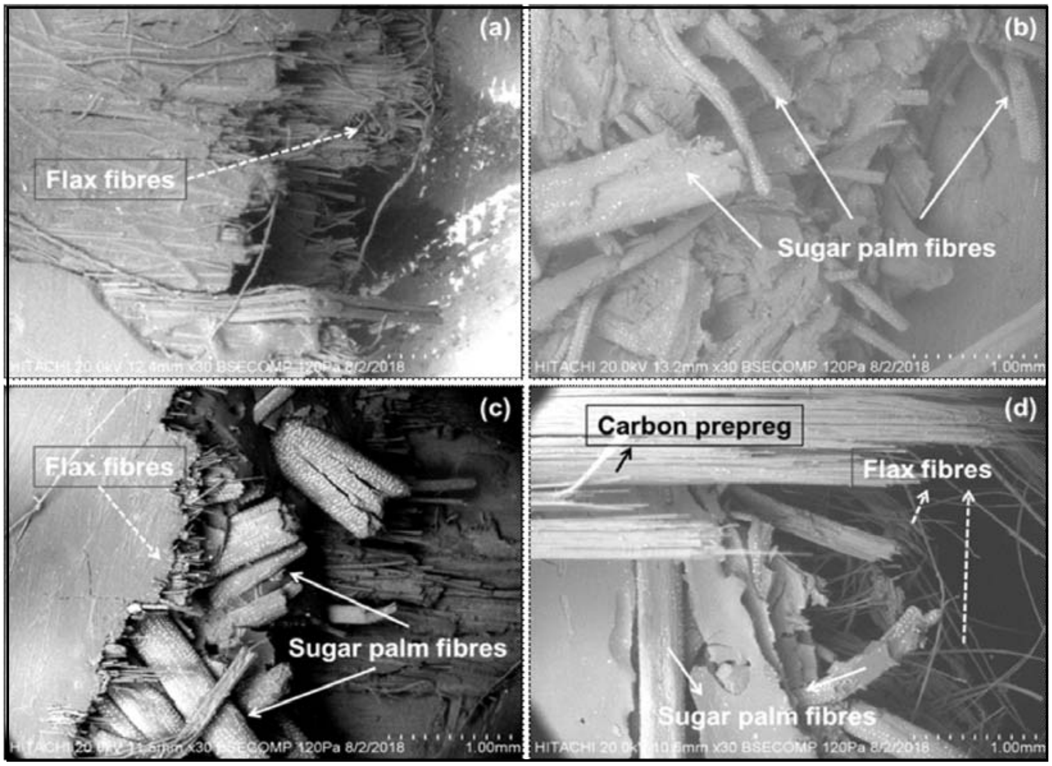

Figure 5. Fibre distribution in the NFC within the FML a) Laminate A, b) Laminate B, c) Laminate C and d) Laminate D

distribute the load effectively throughout the specimen. However, in case of naturally woven sugar palm, the fibres were randomly distributed and were of uneven thickness. The uneven thickness within the natural woven fibre layers could have impact on the overall fibre weight %. Also, the random distribution could easily lead to dense and inadequate fibre regions within the NFC. This in turn affects the load transfer, thereby lower strength and fatigue life apart from their inherently lower tensile strength of the sugar palm fibre.

CONCLUSION

This study investigated the tensile and fatigue properties of the FML with flax and sugar palm fibres prepared by hand layup and hotpress technique. The major findings from this research were that the tensile properties and fatigue behaviour were influenced by the fibre reinforcement and their stacking sequence in the laminate.

- Flax based FML was found to have the highest tensile strength, modulus and toughness than the sugar palm based FML. In hybrid configuration, use of stronger flax fibres in the outer layer with sugar palm in the core was highly beneficial in terms of tensile properties.
- Flax based FML presented longer fatigue life in the range of 10^4 cycles and exhibited typical fibre bridging effect on failure. However, the flax fibre reinforcements were not as effective as synthetic fibres to bear the bridging stress for longer period of time, this caused the failure at shorter number of cycles.
- Similar to the tensile properties, hybridizing flax with the sugar palm resulted in slightly longer fatigue life than the sugar palm based FML. However, fibre bridging effect was not visible in the other configurations.
- Fatigue sensitivity and fractional loss of fatigue strength per decade of cycle was dependent on the fibre type and applied load. FML with flax fibre had the highest fatigue sensitivity and least fractional loss among the studied configurations.

The preliminary study on the FML with flax and sugar palm fibres indicated slightly lower tensile properties than the monolithic Al. However, longer fatigue life displayed by FML with the flax fibres makes them a potential candidate for aircraft applications where such loads are prominent. On the other hand, the least fatigue life exhibited by FML with the sugar palm fibres shows their poor resistance to fatigue loads. This behavior indicates that the sugar palm fibres cannot be employed in high performance applications.

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