Mechanical Properties of a Novel Fibre Metal Laminate Reinforced with the Carbon, Flax, and Sugar Palm Fibres

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Concerns regarding the disposal, degradability, and recycling of synthetic fibres used in composite materials have highlighted the need for eco-friendly materials. This article focuses on fabrication and characterization of the fibre metal laminate (FML) reinforced with carbon, flax, and sugar palm fibres in order to reduce the environmental impact without compromising the strength requirements. Out of autoclave (OOA) manufacturing processes, including hand lay-up and hot compression molding, were employed to fabricate the FML. Tensile, compressive, inter-laminar shear strength (ILSS), and fatigue properties of the fabricated FML were studied. The results indicate that tensile properties and compressive strength for flax based FML (CFC) was superior and 23% higher than CSC while 5% higher than the hybrid CFSSFC configuration. CFSSFC outperformed CFC and CSC in the inter-laminar shear strength by showing 6.5% and 25% increment in magnitude. In case of fatigue, CFC showed excellent fatigue resistance by withstanding high fatigue loads and lasted up to 10^4 cycles before failure. Delamination between the metal/composite plies was observed in fractured samples under all the mechanical loads.

Keywords: FML; Flax; Sugar palm; Natural/synthetic fibre; Mechanical properties

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INTRODUCTION

Fibre metal laminate (FML) is a high-performance material made up of alternating layers of metal alloy and composite bonded together with an adhesive. Commercially available FML include ARALL, CARALL, and GLARE based on the synthetic fibres such as aramid, carbon, and glass, respectively (Asundi and Choi 1997; Sinmazçelik *et al.* 2011). These synthetic fibre-based FML have found successful applications in aircraft structures including tail skin, wing skin, fuselage panels, engine pylon, *etc*., due to their high strength to weight ratio, excellent fatigue and impact resistance, corrosion resistance, and reduced moisture absorption (Chandrasekar *et al.* 2017a). The problem with the present synthetic based materials is their impact on the environment due to difficulties in recycling (Begum and Islam 2013), production of greenhouse gas associated with the fibre production, and the increased demand for the synthetic carbon fibres due to its extensive applications in various industries (Roberts 2011). Other disadvantages of the FML, including the long processing time and high production cost associated with the vacuum bagging using an autoclave, also pushes the need for a low cost alternative (Salve *et al.* 2016).

Recently, the focus has been on the development of environmentally friendly materials that could reduce the usage of synthetic fibres, though the latter cannot be completely eliminated due to its superior mechanical properties that are required for the high-performance applications. Also, there is new focus on "out of autoclave" (OOA) based fabrication techniques to produce FML and composites. Various types of natural fibres abundantly available in nature could substitute for synthetic fibres in applications involving the composite and FML. Natural fibres obtained from the crops, plants, and trees are burnt to residues (Sahari and Sapuan 2011), but could instead be used as reinforcements in the composite/FML materials, depending on the strength requirements of the applications. Other significant benefits of using natural fibres are their bio-degradability, lower density, non-abrasive characteristics to equipment, and lower health risk compared to that of the synthetic fibres (Ku *et al.* 2011). Flax, sisal, kenaf, jute, and hemp fibre based composites have already found commercial applications in the automotive structures (Karus and Kaup 2002). Research on the use of flax/epoxy prepreg in the aircraft structures is gaining interest among the aircraft manufacturers as per the blog post in the Composites World (CW) (Black 2015). Very limited work on mechanical properties of the natural fibre reinforced FML such as oil palm (Dhar Malingam *et al.* 2016; Hussain *et al.* 2016; Sivakumar *et al.* 2017), kenaf (Mohammed *et al.* 2018; Subramaniam *et al.* 2017), jute (Vasumathi and Murali 2014, 2016a, 2016b; Vasumathi *et al.* 2014), and sisal (Vieira *et al.* 2017) could be found as per the author's knowledge. For natural fibre based FML to be commercialized, extensive research is needed on the mechanical properties, failure behavior, and formability.

This work examined the tensile, compressive, inter-laminar shear strength (ILSS), and fatigue properties of the natural/synthetic fibre-based FML. Among the various natural fibres, flax fibre has high tensile properties, marginally near to that of the E-glass fibres with less density, while the sugar palm fibre also has very low density (1.22 g/cm^3 to 1.26 g/cm^3) (Sanyang *et al.* 2016). This was the motivation behind the use of flax and sugar palm fibres in this work. However, natural fibres, which are generally hydrophilic in nature (Chandrasekar *et al.* 2017b), could easily cause internal stresses in FML due to the moisture absorption. Other limitations of natural fibres include variation in material properties based on location, poor fibre/matrix adhesion and inferior mechanical properties compared to the synthetic fibres (Senthilkumar *et al.* 2018). Thus, carbon prepreg was used along with the natural fibre based FML to add strength to the material, as it is well known that natural fibre based composite materials have mechanical properties inferior to that of the synthetic materials.

FML used in high performance applications such as aircrafts could be prone to structural instability due to compressive buckling and inter-laminar de-bonding, leading to failure of the structure (Remmers and De Borst 2002). The FML used in wing structures are subjected to bending loads due to the fuel weight and compression due to the pressure difference between the top and bottom surface of the wing, which creates lift. Thus, it is important to study the compressive and ILSS properties of FML, which is comprised of heterogeneous materials with a metal and prepreg/composite layer. The failure phenomenon observed from the fractured samples will help to understand the response of the material to such loads.

EXPERIMENTAL

Materials

Materials including flax fibre (F), sugar palm fibre (S), carbon prepreg (C), D.E.R 331 epoxy resin with 905-3S joint amine type hardener, and 2024-T3 aluminium sheet metal (Al) of dimension 150x150x1mm^3 were used to fabricate the FML. Naturally woven sugar palm fibres (S) known as "ijuk" formed around the trunk of the tree were taken from Negeri Sembilan, Malaysia. Unidirectional sugar palm fibres were extracted from its natural woven form through a comb nail (Fig. 1a). The unidirectional flax fibres were supplied by the Lineo^{TM} (Mont St Aignan, France) in tape form (Fig. 1b). The materials and their specifications used in this research were as highlighted below in Table 1.

Fig. 1. Unidirectional long fibres a) Sugar palm and b) Flax

Table 1. Materials and its Specifications Used for Fabrication of the FML

Material	Specifications
Flax fibre	Unidirectional long fibres in tape form (110gsm)
Sugar palm fibre	Unidirectional long fibres
Epoxy resin: hardener	2:1
Carbon prepreg	Unidirectional & thickness of 0.1mm (fibre:resin = (50:50)
2024 T3 Al alloy sheet metal	Thickness-1mm

Table 2 provides the properties of sugar palm, flax fibre and 2024-T3 Al sheet. The properties reported for flax fibre and Al sheet were provided by the suppliers.

Table 2. Fibre and Metal Properties

	Sugar palm fibre (Sahari *et al.* 2012)	Flax fibre	2024-T3 Al
Density (g/cm^3)	1.20	1.45	2.78
Tensile strength (MPa)	276.60	1499.3	400-430
Tensile modulus (GPa)	5.90	79.75	70-80

Fabrication Method

The natural/synthetic fibre based FML was fabricated by the hand lay-up and hot compression molding procedure, as described in Fig. 2.

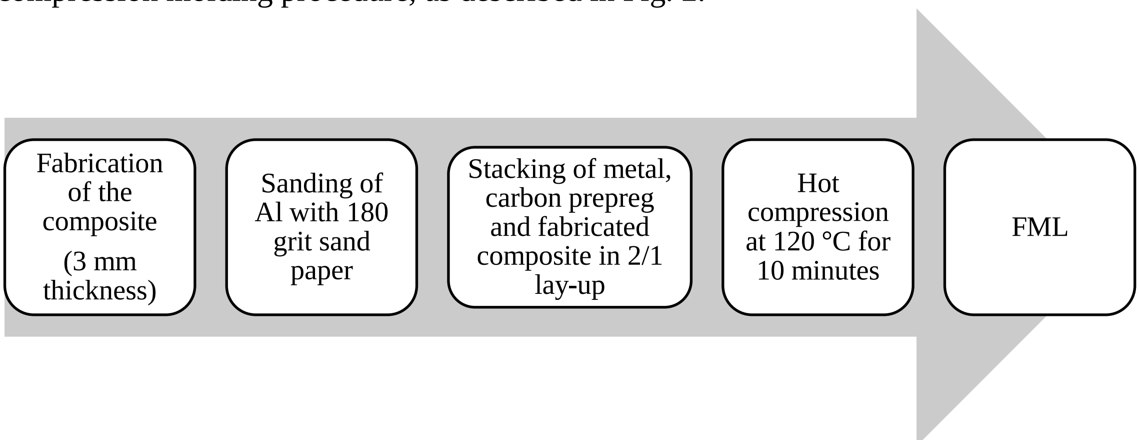

Fig. 2. Fabrication of FML using the hand lay-up and hot compression moulding process

The first step was the fabrication of individual/hybrid composite with flax and sugar palm composite using the hand lay-up and hot compression molding method at 120 °C for 10 min. The fibre loading was kept constant to 30% weight for both the flax and sugar palm fibre-based composites, with a 15% weight for each fibre (50/50 fibre weight ratio) in the overall 30% fibre loading for the hybrid configuration. The second step was the sanding of Al metal surface with 180 grit sand paper to make the surface rough, to enable better adhesion between the metal, prepreg, and composite. The metal was washed with acetone to remove the dirt and burs from the sanding process. Finally, the metal, prepreg, and flax/sugar palm based composite plies were stacked in 2/1 lay-up, as shown in Fig. 3. The number of layers used in each configuration is shown in Table 3. The composites were then wrapped with the commercial Al foil and hot pressed at 120 °C for 10 min with a pressure of 40 tons to form the final FML.

Fig. 3. Schematic of 2/1 layup of the fabricated FML a) CFC, b) CSC, and c) CFSSFC

The metal volume fraction of fabricated FML configuration is the fractional quantity of Al sheet per unit total laminate volume (Wu *et al.* 1994) and is calculated as in Table 3.

Table 3. Metal Volume Fraction of the Fabricated FML Configurations

2/1 Layup	Stacking configuration	Total metal thickness (mm)	Total laminate thickness (mm)	Metal volume fraction %
CFC	$Al_2C_2F_4$	2*1	5.05	39.60
CSC	$Al_2C_2S_4$	2*1	5.13	38.98
CFSSFC	$Al_2C_2F_2S_2$	2*1	5.09	39.29

Mechanical Testing

The tensile, compressive, ILSS, and fatigue properties of the fabricated FML samples were tested as per the ASTM D3039 (2008), ASTM D3410 (2016), ASTM D2344M (2000), and ASTM E466 (2007) standards, respectively. Five specimens were tested in each configuration for the tensile, compression, and ILSS, and the average values were reported. The tension-tension fatigue tests were carried out on an Instron MTS793 to determine the fatigue life. The natural fibre based FML was also subjected to a fatigue test at 90% ultimate tensile strength and stress ratio of R=0.1 until failure. Three specimens were tested and the average values were reported. The fatigue life and load *vs.* displacement curves could be directly obtained from the user interface after the tests. The material and testing specifications used to determine the mechanical properties are given in Table 4.

Table 4. Material Dimension and Specifications for Testing Mechanical Properties

Testing Method	Specifications	Material Dimensions	ASTM standard followed
Tensile	Crosshead speed – 2 mm/min Gauge length – 50 mm	120 × 15 × ~5 mm^3	ASTM D3039
Compression	Crosshead speed – 1.27 mm/min Gauge length – 40 mm	80 × 15 × ~5 mm^3	ASTM D3410
Short beam test (ILSS)	Crosshead speed – 1.27 mm/min Gauge length – 30 mm	60 × 15 × ~5 mm^3	ASTM D2344
Tension-tension fatigue	Grip pressure – 250 psi Stress ratio, R = 0.1 Frequency – 10 Hz 90% ultimate tensile strength	120 × 15 × ~5 mm^3	ASTM E466

The maximum peak load obtained from the short beam test was used to calculate ILSS values of the tested FML samples from Eq. 1,

$$\sigma_{ILSS} = 3P_{max}/4bh \quad (1)$$

where σ_{ILSS} is the inter-laminar shear strength or short beam strength (MPa), P_{max} is the maximum peak load (N) obtained from the short beam bending test, b is the width (mm), and h is the thickness of the tested FML sample (mm).

RESULTS AND DISCUSSION

Tensile and Compressive Properties

Figure 4a-b shows the tensile and compressive behavior of all the tested FML configurations. Yielding could be observed in case of all the compression tested samples. The observed trend was a typical behavior of the thick FML, and the yield was primarily due to the presence of Al in the FML (Krishnakumar 1994).

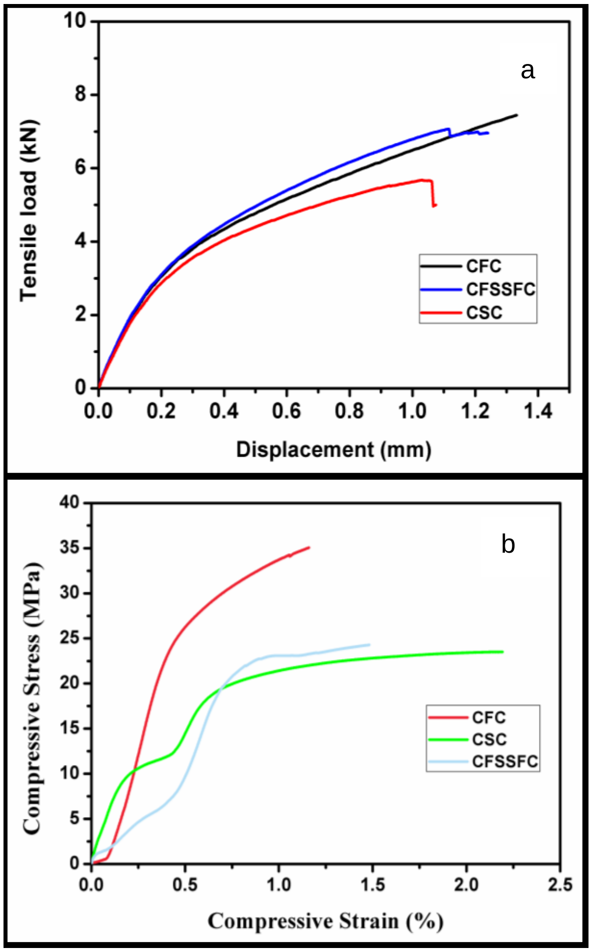

Fig. 4. a) Load *vs.* Displacement from the tensile tests; b) Stress *vs.* strain % from the compression tests

Table 5 shows that pure flax based FML (CFC) possessed the highest tensile strength stiffness and compressive strength followed by the hybrid (CFSSFC) and sugar palm based FML (CSC). It is to be noted that flax fibre has tensile strength equivalent to E-glass fibre (Zhu *et al.* 2013) which enables them to fail at higher loads compared to sugar palm and the hybrid configuration. It could also be observed that the properties can be varied by changing the stacking sequence of the fibers in the composite. The sequence with flax as the outer core and sugar palm as the inner core showed intermediate strength. Using the high strength fibres such as carbon/flax on the outer core helped the CFSSFC to withstand high load and thereby greater mechanical properties than CSC. The dependence of mechanical properties on fibre type was also reflected in a recent study on flax and kenaf based FML where the carbon/flax based FML outperformed carbon/kenaf with superior tensile and compressive properties (Mohammed *et al.* 2018) and as well as on Carbon/Jute/epoxy/Al based FML (Vasumathi and Murali 2014).

Table 5. Tensile properties and Compressive Strength of the Tested FML Configurations

FML Configuration	Tensile Strength at Maximum Load (MPa)	Tensile Modulus (GPa)	Compressive Strength at Maximum Load (MPa)
CFC	99.30 (SD: 5.87)	11.18 (SD: 2.47)	35.72 (SD: 3.19)
CSC	75.55 (SD: 8.23)	10.66 (SD: 4.89)	24.49 (SD: 6.89)
CFSSFC	93.88 (SD: 3.98)	12.81 (SD: 5.76)	27.65 (SD: 4.80)

Figure 5a indicates the scanning electron microscope (SEM) image of "as fabricated" CFC. The reason for lower magnitude of tensile and compressive strength for the studied FML configurations in comparison to their monolithic counterpart 2024 T3 Al alloy (Table 2) and individual fibre/polymeric composites was due to weak interfacial bonding between the metal/composite plies, which resulted in angle ply failure in the Al layer and delamination between the metal/composite, as shown in SEM images (Fig. 5b-c). The observed tensile failure pattern was a typical behavior of thermoset based FML and was similar to that obtained in previous study on the flax based FML (Chandrasekar *et al.* 2016). The weak interface which caused the compressive strength to decline was also highlighted in the previous literature (Botelho *et al.* 2006).

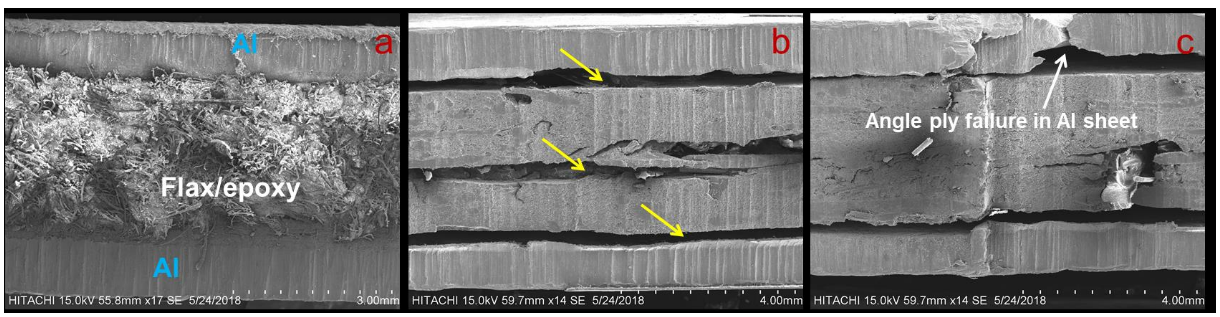

Fig. 5. CFC a); as fabricated; b) Inter-layer delamination after tensile test; c) Delamination/angle ply failure in Al sheet after tensile test

In the case of compression, buckling and delamination between the metal and composite layer was the main cause of failure; for instance, failure behavior of CSC as shown in Fig. 6. As per Remmers and de Borst (2001), the typical failure mode in the compression tested FML started with the initial delamination followed by local buckling, delamination growth, and final failure. This trend was observed in all FML samples subjected to compression, as depicted in Fig. 6. However, the initial delamination, which starts with the crack initiation and propagation between the metal and composite layer, was not visible in the FML subjected to compression (Botelho *et al.* 2007).

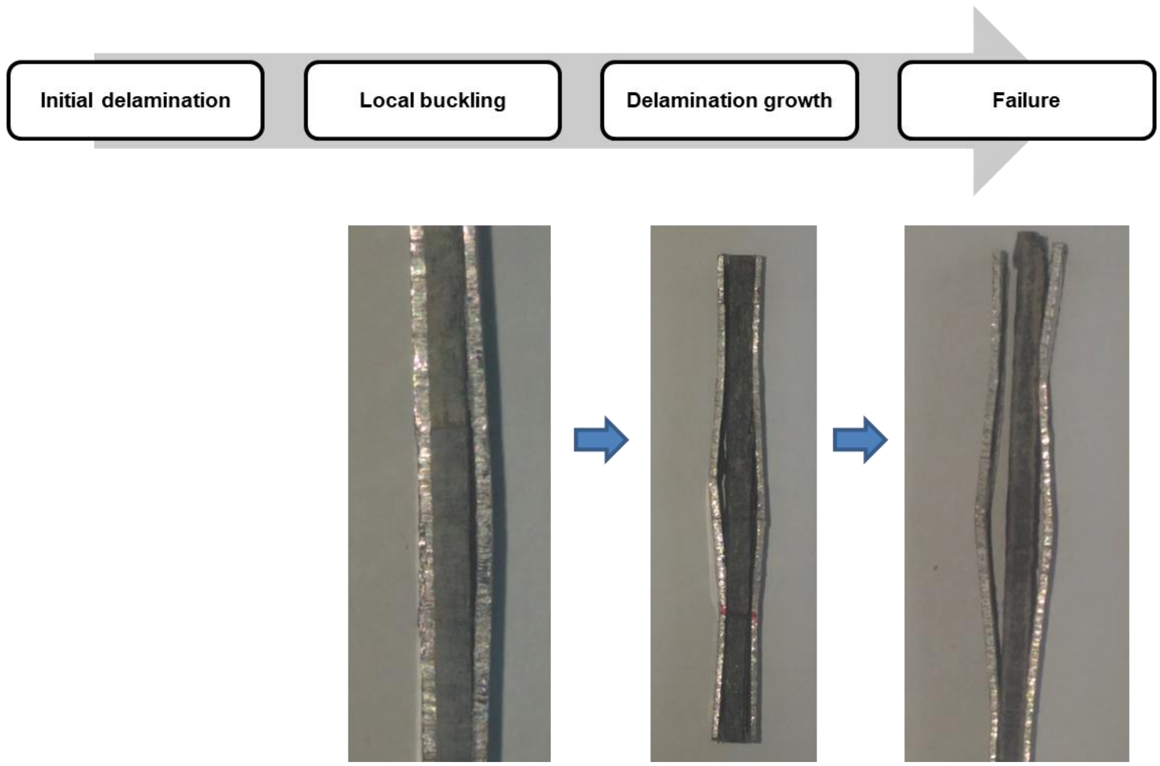

Fig. 6. Failure behaviour observed in CSC under compresssion

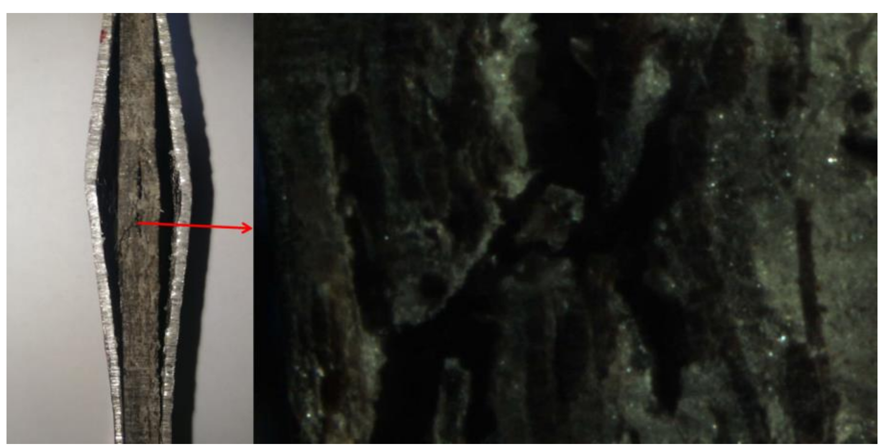

Fig. 7. Optical micrograph showing fibre breakage & matrix crushing in CFC due to compression

In addition to delamination between the metal/composite layer and buckling of Al layer, compression tested CFC samples showed damage to the polymeric composite laminate in the form of fibre breakage and matrix crushing (Fig. 7).

ILSS

The ILSS values of the studied FML configurations were determined from the short beam test, in which the samples were subjected to 3-point bending with the span short enough to indicate the interfacial bonding strength. Figure 8 shows the ILSS values of all tested FML configurations from the short beam test.

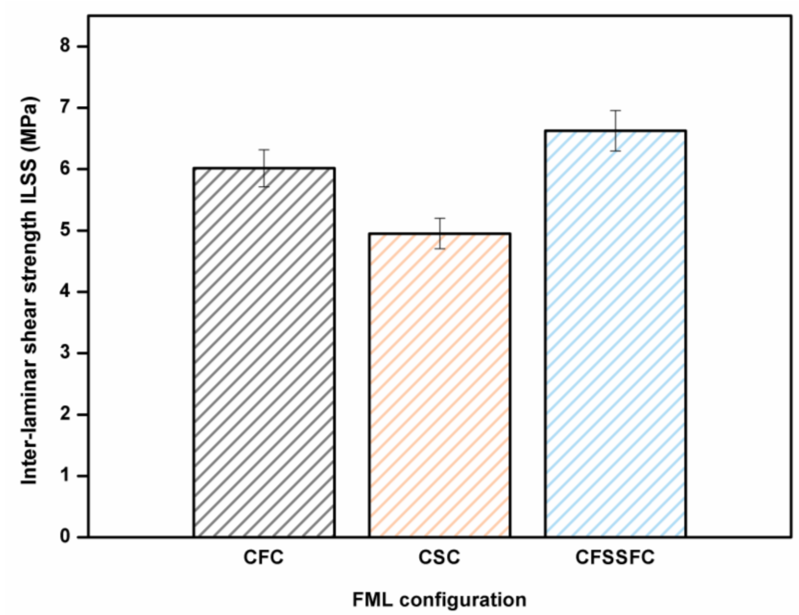

Fig. 8. ILSS values of the FML configurations obtained from short beam test

The highest value was observed for the CFSSFC, followed by CFC and CSC. In the case of composites, ILSS is governed by the bonding strength between fibre/matrix, whereas for FML, it depends on interaction between metal/composite and fibre/matrix as well. Furthermore, the values for FML could be significantly lower than the composite counterpart, since the magnitude highly depends on the interfacial bonding between the metal and composite layer (Botelho *et al.* 2006).

The FML samples subjected to short beam test showed inter-laminar shear and delamination between the metal/carbon prepreg/composite, as shown in Fig. 9. This type of failure behavior, which is the characteristic of FML subjected to short beam testing, has been reported widely in the literature (Botelho *et al.* 2004).

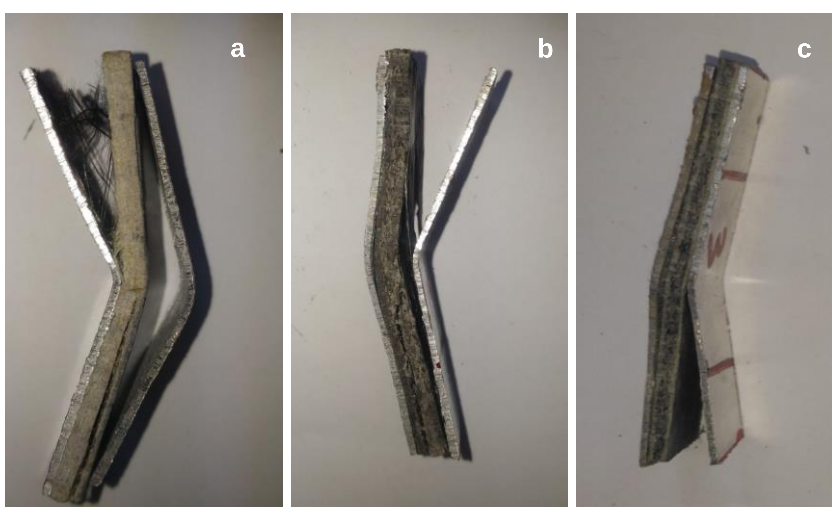

Fig. 9. Failure modes in a) CFC; b) CSC; and c) CFSSFC after short beam test

Other failure modes such as fibre splitting and inter-layer delamination between the composite plies could also be observed, as shown in Fig. 9. The main difference in the failure mechanism between the FML and composite was that delamination occurred between the metal/composite inter-layers for the former, while delamination occurred between the fibre/matrix for the latter. The higher ILSS values for the CFSSFC configuration could be due to the delamination between the composite inter-layers (Fig. 10).

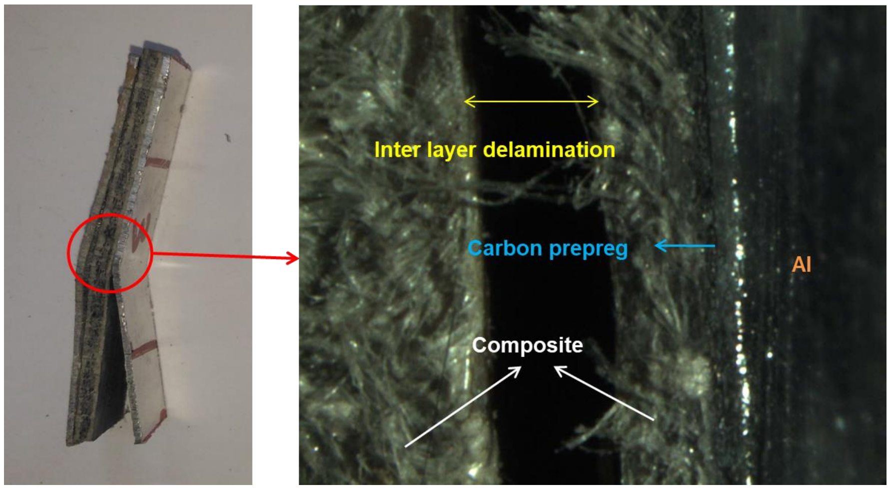

Fig. 10. Inter layer delamination in CFSSFC after short beam test

Fatigue properties

FML have superior resistance to fatigue loads, which makes them highly suitable for aircraft structures. Fatigue life of a material indicates the number of cycles at which the material fails and cannot withstand fatigue load any longer. The typical fatigue life of a material subjected to low cycle fatigue tests with high load is up to 10^4 or 10^5 cycles (Collins 1993). The results in Table 6 indicated that only flax based FML (CFC) showed superior fatigue resistance by withstanding high fatigue loads and failed at 10^4 cycles, while the CSC and CFSSFC configuration had lower fatigue life.

Table 6. Fatigue Life of the Tested FML Configurations

FML Configuration	Fatigue Life (cycles)
CFC	17152
CSC	344
CFSSFC	890

The failure in all FML configurations subjected to fatigue loads started with the crack initiation in the Al layer followed by delamination and failure in the composite laminate. The observed failure pattern was similar to that reported by (Sivakumar *et al.* 2017) on the natural fibre based FML.

The fractured CFC samples from the fatigue test indicated angle ply failure in Al (Fig. 11a). SEM images also showed delamination between the Al and carbon/flax plies (Fig. 11b) and fibre splitting in the flax/epoxy composite (Fig. 11c).

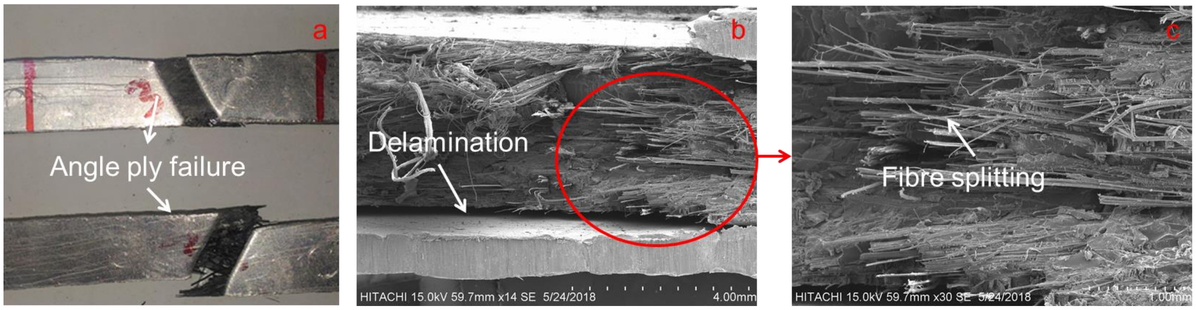

Fig. 11. Fatigue failure in CFC: a) Angle ply failure; b) Delamination; c) Fibre splitting

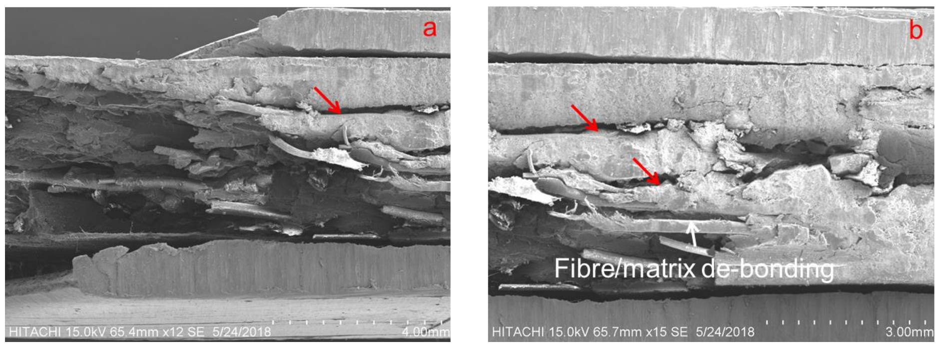

Fig. 12. Fractured CSC from samples fatigue tests showing a) Inter-ply delamination; b) Fibre/matrix de-bonding

The failure behavior was in congruence to that reported by Kandare *et al.* 2018 on flax based FML tested under tension-tension fatigue. According to them, flax, unlike synthetic fibres, could not sustain bridging stresses leading to crack tip opening and breakage in Al layer.

The low strength sugar palm fibre in CSC could not withstand the bridging stress due to the high fatigue load. This in turn led to multiple delaminations between the composite plies (red arrows in Fig. 12a-b), fibre/matrix de-bonding and matrix breakage in the composite layer, which all together contributed to least fatigue life.

The results of this preliminary study highlighted the response of natural fibre based FML to mechanical loads and its failure behavior. Further research will be focused on achieving even higher fatigue resistance and superior mechanical properties by the use of metal surface treatments to improve the bonding characteristics and further increasing number of carbon prepreg layers.

CONCLUSIONS

1. In this study, tensile, compressive, inter-laminar shear strength (ILSS), and fatigue properties of the fibre metal laminate (FML) reinforced with unidirectional flax, sugar palm, and carbon fibres were studied. The composite and FML was fabricated by the out of autoclave manufacturing process using hand lay-up and hot compression molding.

2. Flax based FML (CFC) showed superior tensile, compressive, and inter-laminar shear strength over the hybrid configuration (CFSSFC) and sugar palm based FML (CSC).
3. Buckling phenomenon and delamination between the metal/composite plies were observed in samples subjected to compression. Delamination between the metal/ composite plies were also the main cause of failure for samples subjected to short beam test.
4. Similarly, fatigue life or the cycles to failure was highest for CFC and lowest for CFSSFC and CSC. The fatigue life range for CFC at high loads looks promising for applications in the aircraft structures, which requires high fatigue resistance.
5. Fatigue failure in the FML followed fibre bridging mechanism with the crack initiation in Al layer subsequently followed by crack propagation and fibre pull-out in the composite layer.

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