Effect of Freezing Temperature and Stacking Sequence on the Mechanical Properties of Hybrid Fibre Metal Laminates Made with Carbon, Flax, and Sugar Palm Fibres

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Fibre metal laminates (FML) were reinforced with flax and sugar palm fibres in different stacking sequences and exposed to freezing conditions. The effects on the mechanical properties were explored. Both the stacking sequence and freezing condition affected the mechanical properties. The FML with flax fibres showed the highest strength and modulus under tension and bending, while the FML with sugar palm fibres showed the lowest strength and modulus. The FML with flax fibres experienced a fibre bridging effect and showed promising behaviour for aircraft applications by sustaining nearly 40000 cycles of fatigue load. Decreases in the strength, modulus, and fatigue life occurred when the FML specimens were exposed to freezing conditions. Micrographs from the tensile fractured specimens indicated delamination, fibre splitting, fibre breakage, and fibre/matrix de-bonding as the failure pattern for the pristine and conditioned laminates.

Keywords: Fibre metal laminate; Flax; Sugar palm; Freezing temperature; Mechanical properties; Fatigue life

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INTRODUCTION

Fibre metal laminates (FML) are hybrid lightweight materials that consist of alternating layers of sheet metal and composite plies arranged alternately in a 2:1 or 3:2 layup (Sinmazçelik *et al.* 2011). Commercially available FMLs are classified as GLARE, CARALL, and ARALL based on fibre reinforcements with glass, carbon, and aramid fibres, respectively. Fibre metal laminates have been successfully applied in aircraft structures because of their superior resistance to fatigue and impact loads (Chandrasekar *et al*. 2017a).

In recent years, natural fibres have emerged as a potential substitute for synthetic fibres because of their advantages, such as low density, biodegradability, and abundance (Chandrasekar *et al.* 2017b; Senthilkumar *et al*. 2018; Shahroze *et al.* 2018). Mechanical properties, such as tensile, flexural, impact, and fatigue properties, of FMLs reinforced with natural fibres including kenaf (Feng *et al.* 2017; Dhar Malingam *et al.* 2017; Subramaniam *et al.* 2017), flax (Afaghi Khatibi *et al.* 2016; Kandare *et al.* 2016), jute

(Vasumathi and Murali 2014; Vasumathi and Murali 2016), oil palm empty fruit bunch (Hussain *et al.* 2016), and sisal (Vieira *et al.* 2017) have been explored. The results from these studies demonstrated the properties and failure behaviour of such materials under various loads. The general observation from these published works was that the mechanical properties were dependent on the type of natural fibre employed and their stacking sequence in the laminate. In this study, flax and sugar palm fibres were chosen as reinforcement for the FML. Any material meant to be used in aircraft structures is supposed to have good fatigue resistance. FML such as CARALL and GLARE exhibits a fibre bridging mechanism, which is the primary reason for their superior fatigue life (Alderliesten 2017).

In a recent study, flax-based FML was reported to have similar behaviour (Kandare *et al.* 2018). This attribute makes the flax fibre an eco-friendly alternative for synthetic fibres. On the other hand, sugar palm fibres are inexpensive, unlike the synthetic fibres; they are abundantly available, have low density (1.22 g/cm^3), and could be used as reinforcement in the composites (Shahroze *et al.* 2018). So, the results from the study will help to determine the credibility of these fibres for high performance applications such as aircraft structures.

Aircraft structures are generally prone to attack from the environment through moisture diffusion and different service temperatures from -55 °C to 70 °C during their service life (Ypma and Borgonje 2001). Furthermore, natural fibres are hydrophilic in nature and are more susceptible to attack from moisture diffusion and temperature effects than synthetic fibres (Chandrasekar *et al.* 2018). The influence of freezing temperatures on the mechanical properties of FMLs with natural fibres has not been studied yet.

In this research, flax and sugar palm fibres were used as reinforcements, along with carbon prepreg, in FMLs. The fabricated specimens were also exposed to freezing temperatures and their influence on the mechanical properties was determined.

EXPERIMENTAL

Materials

Flax fibres and XA120 prepreg adhesive film were purchased from Easycomposites (Staffordshire, UK). Sugar palm fibres in their natural woven state were procured from Hafiz Adha Enterprise (Negeri Sembilan, Malaysia). The 2 × 2 twill and 300 gsm carbon prepreg was purchased from Shanghai Lishuo Composite Material Technology (Shanghai, China). The 6061-T6 bare Al sheets with a 0.5-mm thickness were supplied by Metalfort (Mumbai, India). Ethanol and tri-ethoxy(ethyl)silane solution were obtained from QREC(ASIA) (Selangor, Malaysia) and Sigma-Aldrich (Missouri, USA), respectively, for the metal surface preparation. Additionally, D.E.R. 331 epoxy resin with a 905-3S joint amine type hardener was supplied by MZI Supplies (Selangor, Malaysia).

Metal Surface Treatment

The metal surfaces were prepared by mechanical abrasion, which was followed by dipping in 5% aqueous silane solution. The first step involved sanding the metal surface with 80 grit sand paper and cleaning it with acetone to remove any residue. The sanded metal was then subsequently dipped in a 100-mL solution with a 90:5:5 volume proportion of ethanol:distilled water:tri-ethoxy(ethyl)silane for 60 s and immediately dried in an oven at 110 °C for 10 min (Zhu 2005). Sanding is the commonly used technique by researchers as a metal surface treatment due to the process simplicity; the rough surface resulting from abrasion can help the Al to bond with composite plies along with the adhesive after curing. Silane treatment further improves the interfacial adhesion, since it acts as a coupling agent between the adhesive and metal surface (Zhu 2005).

Fabrication Method

The FMLs were prepared by a hand layup and hot-press technique. Initially, composite plies with natural fibres were produced by stacking natural fibres and a resin/hardener mixture in a 150-mm × 150-mm × 2-mm mould and pressing them at 105 °C for 10 min at 10 ton of pressure. Then, the treated metal surface, carbon prepreg, and composite ply were stacked in a 2:1 layup using the stacking sequence that is shown in Table 1. The entire setup was pressed at 105 °C for 10 min with 40 ton of pressure. The hot press was switched off, and the cured FML samples were kept under pressure overnight before being removed from the mould. The samples were then cut according to ASTM standard for tests. In FML, metal volume fraction (MVF) is the parameter similar to the weight fraction in a fibre reinforced polymer composite. MVF in the FML is represented in Eq. 1 and is defined as the fractional quantity of Al sheet over the total laminate volume (Wu *et al.* 1994).

$$\text{MVF (\%)} = \frac{n{*}thickness\ of\ Al\ sheet}{Total\ laminate\ thickness}\ X\ 100\ \% \quad (1)$$

where n is the number of Al sheets in the laminate.

Table 1. Notation, Stacking Sequence, and MVF of the Fabricated FML Specimens

Symbol	Stacking Sequence	Number of Plies (Al/C/F/S)	Laminate Thickness (mm)	MVF (%)
FF	Al/C/C/C/F/F/F/F/C/C/C/Al	2/6/4/0	4.29	23.31
SS	Al/C/C/C/S/S/S/S/C/C/C/Al	2/6/0/4	4.58	21.83
FS	Al/C/C/C/F/S/S/F/C/C/C/Al	2/6/2/2	4.27	23.41
SF	Al/C/C/C/S/F/F/S/C/C/C/Al	2/6/2/2	4.48	22.32

Exposure to Freezing Conditions

A batch of FML specimens was placed in a deep freezer maintained at -40 °C for a period of 72 h. The exposed specimens were then allowed to dry at room temperature for three days prior to testing. The exposed specimens were then subjected to the following tests and their mechanical properties were studied. Pristine or dry specimens were also tested for comparison.

Tensile test

An INSTRON 3382 machine with a 100-kN capacity (Massachusetts, USA) was used to determine the tensile properties of the fabricated and conditioned FML specimens, according to ASTM D3039 (2008). The test was performed with a crosshead displacement speed of 2 mm/min and the average results were reported with the standard deviation for each configuration.

Flexural test

An INSTRON 3365 machine with a 5-kN capacity (Massachusetts, USA) was used to determine the flexural properties of the fabricated and conditioned FML specimens, according to ASTM D790-10 (2010). The test was performed with a crosshead displacement speed calculated using Eq. 2, and the average results were reported with the standard deviation for each configuration. Equation 2 is as follows,

$$F = \frac{zl^2}{6D} \tag{2}$$

where z is 0.01, l is the support span (mm), and D is the thickness of the specimen (mm).

Short beam test

The INSTRON 3365 machine of 5 kN capacity (Massachusetts, USA) was used to determine the interlaminar shear strength (ILSS) of the fabricated and conditioned FML specimens per ASTM D2344M-00 (2006). The test was done with a crosshead displacement rate of 1.27 mm/min and the maximum load before failure was recorded. The ILSS was calculated using Eq. 3, and the average results were reported with the standard deviation for each configuration. Equation 3 is as follows,

$$ILSS = \frac{3P_{\max}}{4bh} \tag{3}$$

where $P_{\max}$ is the maximum load obtained from the short beam test (N), b is the specimen width (mm), and h is the specimen thickness (mm).

Fatigue test

The tension-tension fatigue tests were performed with an MTS 810 (Minnesota, USA) at a constant frequency of 10 Hz, according to ASTM E466-07 (2007). The fatigue life or number of cycles to failure was recorded using an interface on the specimens at a stress ratio of 0.1 and between 60% to 80% of the ultimate tensile strength of the specimens.

RESULTS AND DISCUSSION

Tensile Properties

The influence of the fibre reinforcement, stacking sequence, and freezing temperatures on the tensile strength and modulus of the pristine and conditioned FML specimens were visible from the tensile stress-strain plots (Fig. 1). Both the tensile strength and tensile modulus of the specimens exposed to the combined moisture and freezing temperature were affected, as indicated by the reduction in the peak stress for failure.

The tensile strength and stiffness of the pristine and conditioned specimens are shown in Table 2. The FF arrangement had the highest tensile strength and modulus, followed by the hybrid (FS and SF) and SS configurations. Among the hybrid configurations, the FS arrangement had a better strength and modulus than the SF arrangement. A similar observation with stronger fibres in the outer layer showing higher properties has been highlighted in a previous study. According to Jawaid *et al*. (2013), stronger fibres in the outer layer helps to carry and distribute loads effectively in a hybrid composite than the inferior strength fibre in the core. Thus, the FS arrangement had stronger flax fibres in the outer layer and exhibited higher tensile properties than the SF arrangement.

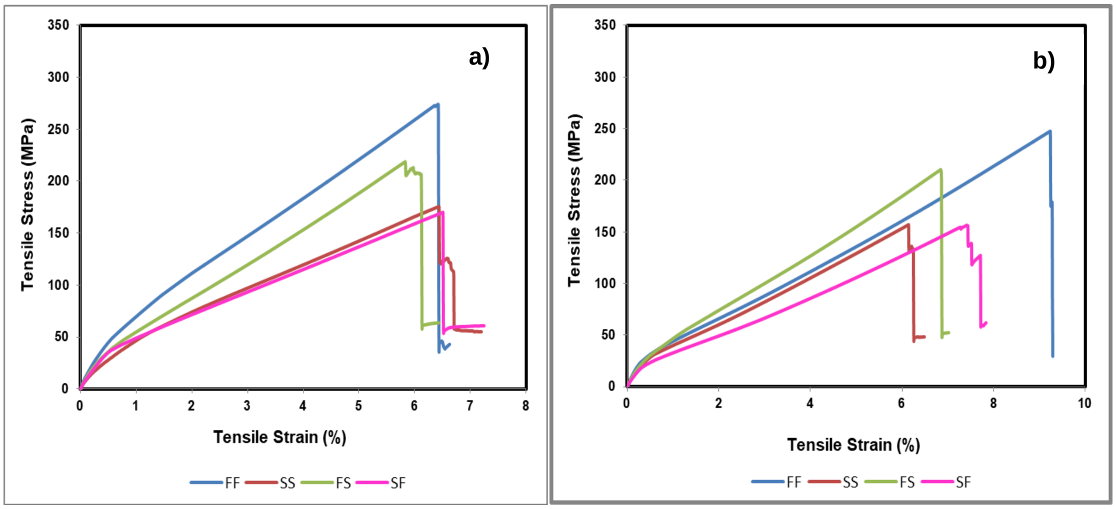

Fig. 1. Tensile stress-strain plots for the (a) pristine and (b) conditioned FML specimens

Because the flax fibres have a higher tensile strength and modulus between 500 MPa to 800 MPa and 50 GPa, respectively (per the manufacturer specification), which was higher than that of the sugar palm fibres (276 MPa and 5.9 GPa) (Ishak *et al*. 2013), the FML with the hybrid configurations possessed intermediate strength and modulus values compared with the FF arrangement and better properties than the SS arrangement.

Table 2. Tensile Properties of the Pristine and Conditioned FML Specimens

FML	Pristine		Conditioned	
	Tensile Strength (MPa)	Tensile Modulus (GPa)	Tensile Strength (MPa)	Tensile Modulus (GPa)
FF	267.20 ± 18.07	17.41 ± 1.14	250.23 ± 14.09	13.2 ± 0.40
SS	173.38 ± 2.24	3.76 ± 1.73	156.85 ± 0.33	3.06 ± 0.81
FS	221.61 ± 19.95	5.81 ± 0.76	211.02 ± 1.87	4.61 ± 0.91
SF	178.18 ± 0.61	5.56 ± 2.77	155.31 ± 15.93	4.97 ± 2.96

Table 2 shows that all of the specimens exposed to the combined moisture/freezing temperature presented a lower tensile strength and modulus than the dry or pristine specimens. The conditioned laminates exhibited a 5% to 13% decrease in the tensile strength and a 12% to 37% decrease in the tensile modulus because of exposure to freezing conditions. The natural fibres present in the laminate are hydrophilic (Chandrasekar *et al*. 2017b), and exposing them to freezing conditions further aggravated moisture absorption by the natural fibres. Thus, a considerable decrease in the strength and modulus was observed for the conditioned laminates. According to previous research (Dutta and Farrell 1988; Jones 2001; Kichhannagari 2004), polymeric laminates with natural fibres exposed to freezing conditions also underwent matrix degradation, interface weakening, and an increase in the residual stress due to the mismatch of coefficients of thermal expansion (CTE) between the constituents in the laminate from the freezing conditions. These factors, in addition to moisture absorption at freezing temperatures, also contributed to the decrease in the properties (Ray 2004).

Flexural Properties

The stress-strain plots obtained from the three-point bending tests are presented in Fig. 2. Both the flexural strength and modulus showed similar trends to those observed for the tensile properties.

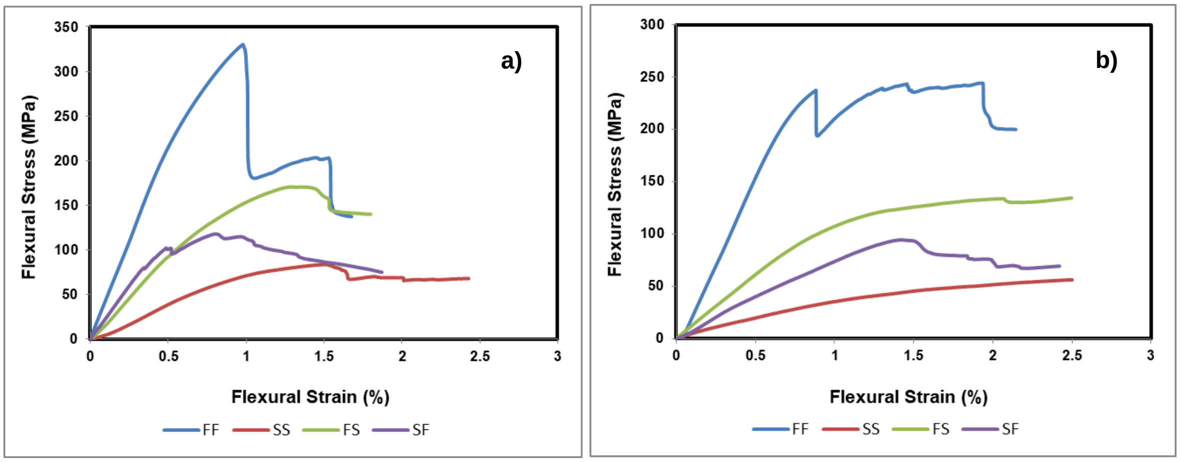

Fig. 2. Flexural stress-strain plots for the (a) pristine and (b) conditioned FML specimens

The pristine and conditioned FML specimens showed dependency on the natural fibre type, stacking sequence, and aging condition, as is shown in Table 3. Both the flexural strength and flexural modulus had the following order for the dry specimens: FF > FS > SF > SS. After exposure, the conditioned laminates showed a 22% to 37% decrease in the flexural strength and approximately 50% decrease in the flexural modulus. The SS sample showed the least resistance to freezing conditions and had the lowest flexural strength and modulus compared with the other configurations. The flexural modulus of the exposed specimens decreased more than the tensile modulus. This implied that the performance of the exposed specimens deteriorated more under bending than under tension.

Table 3. Flexural Properties of the Pristine and Conditioned FML Specimens

FML	Pristine		Conditioned	
	Flexural Strength (MPa)	Flexural Modulus (GPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)
FF	361.20 ± 60.26	44.39 ± 6.45	227.76 ± 35.08	20.09 ± 7.17
SS	80.30 ± 15.34	9.49 ± 7.39	60.85 ± 1.47	5.53 ± 1.85
FS	173.56 ± 13.94	28.75 ± 7.56	136.14 ± 17.29	10.99 ± 1.64
SF	121.54 ± 3.51	21.70 ± 2.43	90.56 ± 3.51	5.56 ± 1.53

According to Wei *et al.* (2017), interfacial bonding between the plies in a laminate is a deciding factor for the flexural strength when exposed to temperature effects. The top surface of the specimen loaded in three-point bending experiences compression, while the bottom surface experiences tension. The laminate plies in between the top and bottom surface withstand shear forces. Therefore, a possible explanation for the lower flexural strength and modulus for the exposed specimens was the accelerated degradation of the interface between the metal/prepreg/composite layer caused by moisture absorption and low temperatures.

Interlaminar Shear Properties

Variations in the ILSS for the pristine and conditioned laminates is shown in Table 4. The maximum ILSS was observed for the FS arrangement, followed by the FF, SF, and SS arrangements. Because the ILSS represents the interfacial bonding strength in a laminate, the decrease in the magnitude for all of the conditioned laminates indicated interface degradation between the individual constituents in the laminate. Among the configuratons, the SS and SF arrangements showed the highest loss of 36% and 34% in the ILSS, respectively, while the FF and FS arrangements exhibited a 3% and 18% decrease in the ILSS, respectively, because of conditioning.

Table 4. ILSS of the Pristine and Conditioned FML Specimens

FML	ILSS (MPa)	
	Pristine	Conditioned
FF	14.25 ± 1.25	13.78 ± 1.34
SS	12.26 ± 4.48	7.83 ± 0.96
FS	16.08 ± 3.55	13.26 ± 0.73
SF	12.85 ± 2.38	8.53 ± 0.55

Among the studied configurations, the FF arrangement with flax fibres had the highest strength and stiffness. Similarly, after conditioning, the FF arrangement showed a higher retention in the strength and stiffness compared with the other configurations with sugar palm fibres. A recent study also highlighted that FMLs with flax/epoxy composite plies showed a fatigue life of 10^4 cycles to 10^5 cycles between 80% to 30% of the applied load (Kandare *et al.* 2018). This demonstrated the ability of flax fibres to withstand fatigue loads, which makes them a potential candidate for applications requiring fatigue resistance. However, the material response or change in the fatigue behaviour and their properties after exposure of FMLs with flax fibres to freezing conditions has not yet been explored. Thus, fatigue properties of both the pristine and conditioned FF specimens obtained from the tension-tension fatigue test were considered.

Fatigue Properties

Tension-tension fatigue tests were performed on the pristine and conditioned FF specimens. The mean fatigue life or number of cycles to failure is reported in Table 5.

Table 5. Fatigue Life of the Pristine and Conditioned FF

FF	Stress Level (% UTS)	Maximum Stress (MPa)	Fatigue Life (cycles)	Mean Fatigue Life (cycles)
Pristine	80	213.76	8643	8560
			8522	
			8515	
	70	187.04	12633	12287
			11567	
			12662	
	60	160.32	43869	38207
			36089	
			34665	
Conditioned	80	200.18	7415	7036
			6901	
			6791	
	70	175.16	8787	9124
			9119	
			9466	
	60	150.14	16137	16127
			16475	
			15769	

The general observation was that fatigue life increased with a decrease in the load and there was limited data scattering between the specimens at all load levels. This behaviour was also observed in a recent study, and it was attributed to the damage accumulation process in a hybrid laminate with various constituents (Salman *et al.* 2016). The pristine FF specimens lasted from 8000 cycles to nearly 40,000 cycles between 80% to 60% of the applied load.

In general, the superior resistance to the fatigue loads by the FMLs was attributed to their unique fibre bridging effect. The failure mechanism occurred in the material in the following steps. The failure started with initiation of micro-cracks in the Al layer. Over time with the load being applied continuously, the micro-cracks grew and increased in length. In this phase, a part of the load was transferred to the fibre layer, which remained in contact with the Al and restricted crack growth. This is called the fibre bridging effect, and the stress transferred to the fibre layer is called bridging stress. In the second phase, delamination occurred in the metal/prepreg, followed by complete failure of the Al. The fibre reinforcements then carried the load effectively until the final failure (Homan 2006).

A similar failure pattern was observed in the pristine and conditioned FF specimens during the fatigue tests. A failed specimen from the fatigue test is shown in Fig. 3. The flax reinforcements were not as effective as the synthetic fibres at carrying bridging stress within the laminate. Thus, the pristine specimens failed at a lower number of cycles. Likewise, certain factors, such as the moisture absorption by the natural fibres, interface weakening, and residual stress because of the difference in the CTE between the constituents of the laminate caused by freezing conditions, further affected the load carrying capability of the laminate. Therefore, the conditioned laminates exhibited a lower fatigue life than the pristine or dry specimens.

Fig. 3. Failed pristine FF specimen from the fatigue test

To further study the degradation effects from the freezing conditions, the fatigue sensitivity and fractional loss in strength per decade of cycles were determined. Both parameters were calculated from the slope of the normalized S-N plot (Fig. 4) and Eqs. 3, 4, and 5 (Mandell 1981).

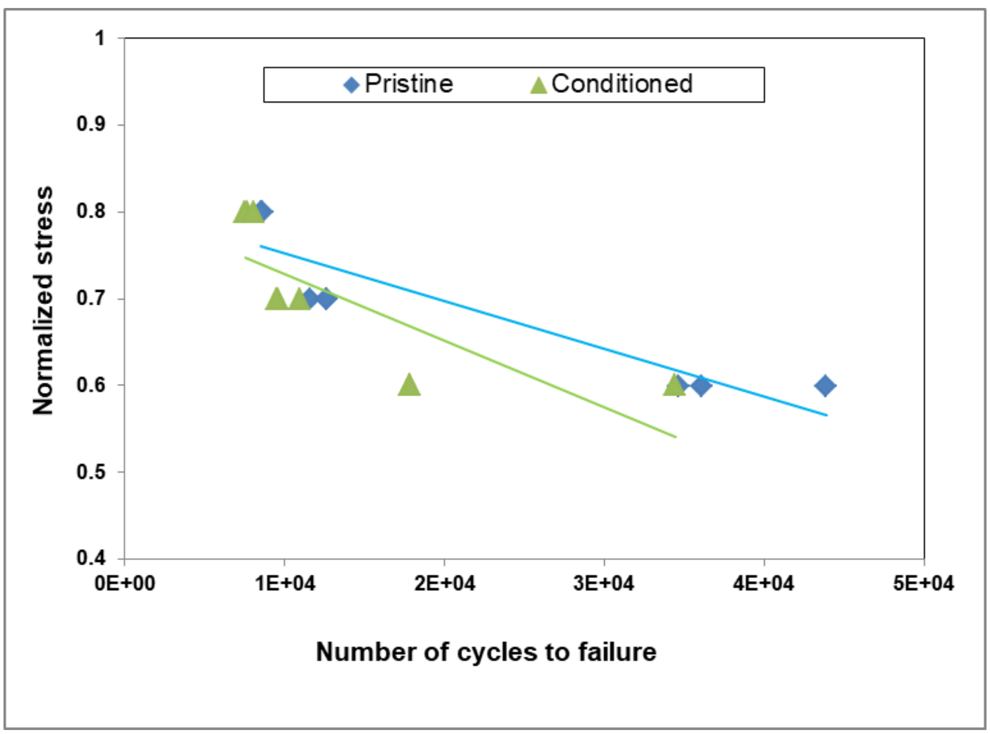

Fig. 4. Normalized S-N plot of the pristine and conditioned FF specimens

Equations 3, 4, and 5 are as follows,

$$\sigma_{\max} = \sigma_{\text{uts}} + b\log N \quad (3)$$

$$b = \frac{(\sigma_{\max} - \sigma_{\mathrm{uts}})}{\log N} \tag{4}$$

$$\frac{b}{\sigma_{\mathrm{uts}}} = \frac{\left(\left(\frac{\sigma_{\max}}{\sigma_{\mathrm{uts}}}\right) - 1\right)}{\log N} \times 100\% \tag{5}$$

where σ_{uts} is the ultimate tensile strength of each specimen from the tensile test (MPa), σ_{max} is the stress level at 80%, 70%, and 60% of the σ_{uts} (MPa), N is the number of cycles until failure, and b is the slope and fatigue sensitivity.

Table 6. Fatigue Sensitivity and Fractional Loss of Strength Per Decade of Cycles

FF	Fatigue Sensitivity			Fractional Loss of Strength per Decade of Cycles		
	80%	70%	60%	80%	70%	60%
Pristine	-12.71	-18.34	-21.83	5.09	7.34	8.74
Conditioned	-12.43	-18.13	-22.26	5.20	7.50	9.21

Table 6 shows that the fatigue sensitivity was dependent on the applied load and decreased in magnitude more for the conditioned FF specimens than for the pristine specimens. Greater magnitudes indicated a better performance of the material because the material could last a longer number of cycles before failure (Broughton *et al.* 2002). Similarly, fractional loss in the strength per decade of cycles was higher for the conditioned FF specimens at all of the load levels than for the pristine FF specimens. This suggested that the FMLs with natural fibres were sensitive to such conditions. Therefore, the strength and modulus loss because of these conditions should be accounted for when designing these materials for use in aerospace applications, where the material is expected to operate in such environments during their service life.

Failure Behaviour

Delamination between the inner plies of the FML specimens was observed in the pristine and conditioned laminates subjected to a tensile test is shown in Figs. 5 and 6, respectively. Other microscopic damages in the polymer composite, such as longitudinal micro cracks, fibre/matrix de-bonding, fibre pull-out, and fibre splitting, were also observed. These phenomena were in accordance with the observations from previous research into polymer composites exposed to freezing or sub-zero temperatures (Dutta and Farrell 1988; Jones 2001; Kichhannagari 2004).

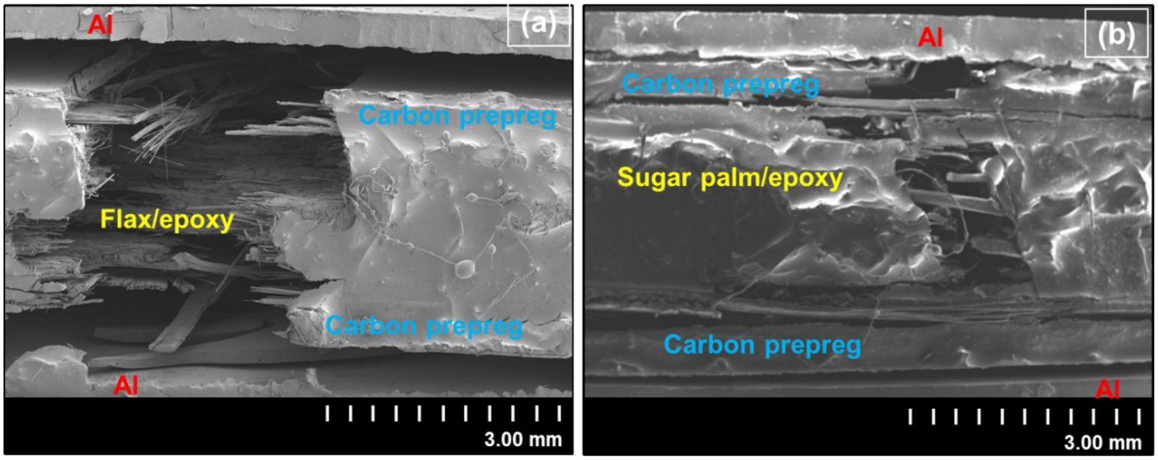

Fig. 5. Tensile failure in the pristine FML specimens (a) FF and (b) SS

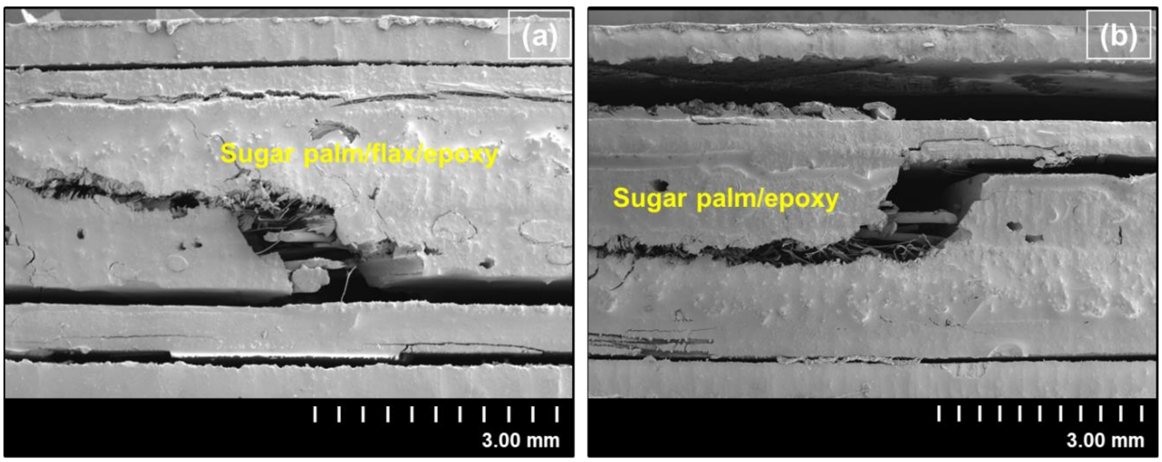

Fig. 6. Tensile failure in the conditioned FML specimens (a) SF and (b) SS

CONCLUSIONS

1. In this study, flax and sugar palm fibres were incorporated into FMLs in individual and hybrid configurations to study the influence of the stacking sequence on the laminate properties. Also, the specimens were exposed to freezing temperatures, and the impact on the mechanical properties was studied.
2. The natural fibre type and their stacking sequence within the laminates were the deciding factors for the strength and modulus in the dry or pristine specimens. Among the studied configurations, the FF arrangement with flax fibres had the highest strength and modulus, while the SS arrangement with sugar palm fibres had the lowest.
3. In the hybrid FML with flax and sugar palm, flax fibre as the outer layer and sugar palm fibre in the core showed superior strength and modulus.
4. The FMLs with flax fibres exhibited a fatigue life of approximately 10^4 cycles and displayed a fibre bridging effect similar to synthetic fibre-reinforced FMLs.
5. The laminates exposed to freezing conditions showed a substantial decrease in the strength and stiffness compared with those of the dry specimens. The degradation effects from the freezing conditions were also visible in the conditioned FF specimens, which exhibited a lower fatigue life.
6. Factors, such as the moisture absorption by the natural fibres, matrix degradation, and interface weakening, affected the mechanical properties.

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