International Journal of Pure and Applied Mathematics
Volume 119 No. 15 2018, 3761-3769
ISSN: 1314-3395 (on-line version)
url: http://www.acadpubl.eu/hub/
Special Issue

http://www.acadpubl.eu/hub/

THERMAL PROPERTIES OF HL002-TA/B EPOXY RESIN/HARDENER IN FIBRE METAL LAMINATES COMPOSITES FOR AERO-ENGINE

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***Abstract*— Fibre metal laminates (FML) composite are now widely used in many industries such as aerospace and automobile industries among others due to their superior properties compared to those of traditional materials; therefore the thermal stability and viscoelastic properties of the composite play important roles in high temperature application. The main purpose of this study is to evaluate the performance of the composites at high temperature application in fire designated zone of an aircraft engine. The composites were fabricated in a mold using hand lay-up method, compressed using a compression machine and then cured for 24 hours. The composites were heated from ambient temperature to a certain higher temperature at different heating rates using nitrogen and air gas for mass loss during thermogravimetric analysis (TGA), storage modulus, loss modulus and tan delta for dynamics mechanical analysis (DMA). The TGA result indicated a remarkable improvement in the thermal stability of all the fibre metal laminate composites where carbon fibre reinforced aluminium laminate with aluminium alloy at front and rear face had the highest percentage of residue which was 58.85%, while the smallest percentage was shown by the composite that was hybridized with natural fibre (kenaf). Dynamic mechanical results showed an increase in the storage modulus and an average glass transition temperature in the presence of polymer used; higher increment was observed on carbon fibre reinforced aluminium alloy with aluminium alloy at front and rear face. This investigation confirmed that fibre metal laminates of aluminium alloy 2024-T3, carbon fibre, epoxy and some other types of natural fibres composites possess excellent features that can be used in fire designated zone of an aircraft engine nacelle in the near future.**

Keywords-dynamic mechanical analysis; fibre metal laminate; natural/synthetic fibre; thermogravimetric analysis;

I. INTRODUCTION *(HEADING 1)*

Carbon fibre reinforced polymer composites were developed in the past decades to produce a different hybrid composite that has high stiffness and strength with low density, when compared with the traditional materials used before such as aluminium, steel, etc. The composites become more attractive in many applications such as aerospace, automotive, energy sectors and conventional energy generation due to their properties [1-5]. Therefore it is a known solution to the problems associated with aircraft parts that lead to the development of composites materials to improve the performance of different parts of aircraft in terms of corrosion resistance, thermal resistance, weight reduction, cost, etc. Due to the fact that carbon fibre reinforced aluminium alloy polymer is a form of strong composites used in aerospace sector today by virtue of properties of carbon fibre, aluminium alloy, flax, kenaf, and the polymer used has made them important materials in solving composites problems faced by the aerospace industry.

Thermogravimetric Analysis (TGA) is an analysis used in determining the thermal characteristics of materials either by mass loss or gain due to the loss of moisture, decomposition or oxidation. The mass loss of the samples prepared will be obtained in this study by determining the thermal stability of the polymeric materials. Meanwhile, Dynamic Mechanical Analysis (DMA) is the thermal analysis method that is used in measuring the properties of the deformed materials under a repeated stress and having the responses measured, which is monitored as a function of temperature or time. In addition, the relaxation phenomena in polymer that cannot be found using conventional method of measurement will also be examined and can be used in determining the elasticity and damping properties of the materials to a certain extent. The analysis become necessary owing to the importance of thermal stability in composites at risk of being exposed to fire in an aircraft fire designated zone. It is very important to understand how the materials in the composites behave thermally before any application. Previous works based on fire behaviour of polymer composites were conducted by many researchers. Tranchard *et al.* [6] reported the thermal degradation of the resin polymer, thermal delamination of the composite, thermal expansion inducing formation of cracks, internal pressure phenomenon, and gas migration through the material of T700 carbon fibre reinforced M21 epoxy resin composite laminate. The analyses were used in developing a model for the behaviour of fibre metal laminates (FMLs) composite. The thermal degradation of an epoxy resin of carbon fibre

reinforced epoxy laminate composite differ from one form to another, but in general, the thermal decomposition of carbon fibre reinforced epoxy polymers depends on the chemical nature of the hardener, the type of epoxy monomer, content and the orientation of the fibres, the thickness of the content and content used [7, 8]. Tranchard *et al.* [9] recently developed a thermal delamination model that can handle the delamination of the carbon fibre/epoxy composites in the fire test.

Hybrid materials were used to produce different composites by tailoring their properties to the required structural form by the designer [10-13]. Water absorption resistance and thermal properties of hemp natural fibre composites were improved by hybridization with glass synthetic fibres [14]. Synthetic fibres can be reinforced with natural fibres to yield a composite with required properties and also cost effective. The combination of synthetic/natural fibres produces properties which is almost the same as the combinations of synthetic fibres. The hybridization of carbon fibre with natural fibre can improve the corrosion resistance, moisture resistance, strength and stiffness of the composites. But there were few literatures on synthetic/natural fibres reinforced composites hybrid on different properties on their hybridization. Panthapulakkal and Sain [15] studied the water absorption properties of hemp/fibre glass hybrid polypropylene composites for automobile interior parts, where the properties of banana/glass fibre reinforced hybrid composite based on mechanical and interfacial properties showed increased impact strength [16]. Petit *et al.* [17] studied the thermal decomposition PPS matrix in carbon fibre composites in fire exposed laminates, and the study revealed that, at a higher temperature the melted PPS redistributes itself around the carbon fibre.

In fire conditions, the performance of polymer composites on fire had induced degradation and reduced the performance of the mechanical properties of post fire residue [18] in which the polymer matrix weakened and gradually caused the composite to fail under high temperature due to thermal degradation caused by high temperature flame. An investigation on thermoplastic laminate composites was conducted and the results showed that the composite have higher post-fire properties when compared with thermoset laminate composites under a high temperature application, since it was less susceptible to delamination cracking and produced char when burned, but its mechanical properties were still under investigation.

In order to utilize the composite for effective usage, it is necessary to determine the thermal stability and thermal characteristic of the composites. It is very important to study the thermal degradation behaviour and mechanism of the composites and evaluate their performance. This study aimed to provide information concerning the thermal degradation and other thermal properties of the composites using thermogravimetry analysis (TGA) by comparing the mass loss at a specific temperature using nitrogen atmosphere, and dynamic mechanical analysis (DMA). In the present study, hybrid composites of synthetic with aluminium alloy and natural/synthetic fibres with aluminium alloy were developed in which the ratio of synthetic and natural fibres in the composites were varied in order to understand the effect of hybridization of the materials used in the composites. The thermal stability of the composites were evaluated where mass losses of each composite with their various temperatures were determined using TGA, while storage modulus, loss modulus and tan δ of each composites were evaluated with their respective temperatures by DMA. This paper aims to provide useful information on thermal properties of different composites in terms of percentage weight loss, storage modulus; loss modulus and tan δ with their respective temperatures of each composite, and the results were compared. This paper presents the thermal stability of four composite materials for aeronautical purpose (fire designated zone of aircraft engine nacelle) viz: Carbon Fibre Kenaf Reinforced Aluminium alloy (CAKRALL), Carbon Fibre Flax Reinforced Aluminium alloy (CAFRALL), Carbon Fibre Reinforced Aluminium alloy with aluminium alloy at top and bottom (CF+AA), and Carbon Fibre Kenaf Reinforced Aluminium alloy with alternating aluminium alloy (CARALL). To meet the requirements for the certifications, it is necessary to assess how destructive a prior fire exposure can be on the residual thermal behaviour of the laminates composites.

II. MATERIALS AND METHODS

A Thermogravimetric Analyzer (TGA Q500 V20.13 Build 39, TA Instrument) was used to characterize the thermal degradation and melting temperature of the laminate composites samples in this study, using thermo-balance coupled to a mass spectrometer. The samples in the TGA were heated up to 800°C from ambient temperature (30^0C) at heating rates of 5, 10, 20, and 40°C/min under nitrogen or air atmosphere (50ml/min). The TGA was used in order to verify the carbonization-in-nitrogen procedure on the thermoset polymer, and different samples of carbon fibre and carbon with natural fibres and aluminium alloy were prepared by hand lay-up method. The experiment was carried out on samples with purge gas flow rate of 50 ml/min and an average mass of 80 mg.

The dynamic mechanical analysis was conducted using an Instrument DMA Q800 V20.24 Build 43 Module DMA Multi-Frequency – Strain InstSerial 0800-1072 Clamp Dual Cantilever with a sample size of 35 x10x3.5mm (Geometry Rectangular: Length, Width, Thickness). The experiment was conducted in bending mode of multiple frequencies with 6N dynamic force that is oscillating at fixed frequency and amplitude of 15.0 MinOscF. The relaxation spectra were recorded in the temperature range between 30 and 200 ^{0}C, at a heating rate of 5 °C/min to 200 °C from ambient temperature (30^0C).The analysis was used to find the storage modulus, loss modulus and tanδ.

Four laminate composites were used in the study: carbon fibre kenaf reinforced aluminium alloy with aluminium alloy at top and bottom (CAKRALL), carbon fibre flax reinforced aluminium alloy with aluminium alloy at top and bottom (CAFRALL), carbon fibre reinforced aluminium alloy with aluminium alloy at top and bottom (CF+AA), and carbon fibre reinforced aluminium alloy with alternating aluminium alloy (CARALL). The four samples for TGA and DMA were

cut according to the ASTM standard after fabricating the composites. The composites consisted of 0.3mm sheet of aluminium alloy 2042-T3, 0.2mm carbon fibre, 0.6mm flax, and 1.4mm kenaf and epoxy resin/hardener. Zeepoxy HL002 TA/B was used for all the composites; the epoxy has a low viscosity, good mechanical properties with high heat distortion temperature. The resin is a colourless viscous liquid with a viscosity of 5500±1000Cps at 30^0C while the hardener is a colourless liquid with a viscosity of 30±20Cps at 30^0C and epoxy resin/hardener was mixed in ratio 2:1. Table 1 shows the properties of mixed compound of resin/hardener.

The composite had the same thickness of 3.5±0.2mm with two sheets of aluminium alloy at the front and rear faces of three composites and the fourth composite consisted of six aluminium alloys in alternate with five carbon fibre. The first composite consisted of four carbon fibre layers with one layer of kenaf at the centre of the composite, the second composite contained five layers of carbon fibre and two layers of flax in alternate with the carbon fibre and the third composite contained eleven layers of carbon fibre. Tables 2, 3 and 4 shows the properties of carbon fibre, aluminium alloy and epoxy resin/hardener respectively

TABLE I. Properties of Mixed Compound of Resin/Hardener (Manufacturer, [19])

Pot life (below 30°C) (hours)	6 ± 2
Hardness (Shore D)	85 ± 2
Tensile Strength (kg/cm²)	800 ± 50
Flexural Strength (kg/cm²)	375 ± 50
HDT (°C)	70 ± 5

TABLE II. Properties of Carbon Fibre (Manufacturer, [20])

Typical Filament Properties	**Average Values**
Tensile Strength	4275 MPa
Tensile Modulus	225 GPa
Ultimate elongation	1.9%
Carbon Density	1.82 g/cm^3
Filament Diameter	7 µm
Electrical Resistivity	1380 µΩ-cm
Typical Chopped Properties	**Average Values**
Fibre Length	3, 6, 12, 25 mm
Moisture Content	<0.3%

The experiments for the four samples were conducted using TGA and DMA machine according to standard specifications as shown in **Fig. 1**.

TABLE III. Properties of aluminium Alloy 2024-T3(Manufacturer, [21])

Properties	**Value**	**Comment**
Density	2.78	AA; Typical
Mechanical		
Hardness, Brinell	120	AA; Typical; 500 g load; 10 mm ball
Hardness, Knoop	150	Converted from Brinell Hardness Value
Hardness, Rockwell A	46.8	Converted from Brinell Hardness Value
Hardness, Rockwell B	75	Converted from Brinell Hardness Value
Hardness, Vickers	137	Converted from Brinell Hardness Value
Ultimate Tensile Strength, MPa	434	AA; Typical
Tensile Yield Strength, MPa	284	AA; Typical
Elongation at break, %	16.5-17.5	AA; Typical; 1/16 in. (1.6 mm) Thickness
Modulus of elasticity GPa	73.1	AA; Typical; Average of tension and compression. Compression modulus is about 2% greater than tensile modulus.
Notched Tensile Strength, MPa	379	2.5 cm width x 0.16 cm thick side-notched specimen, kt 17
Ultimate Bearing Strength,MPa	855	Edge distance/pin diameter = 2.0
Bearing Yield Strength, MPa	524	Edge distance/pin diameter = 2.0
Poisson's Ratio	0.33	
Fatigue Strength, MPa	138	AA; 500,000,000 cycles completely reversed stress; RR Moore machine/specimen
Machinability, %	70	0-100 Scale of Aluminium Alloys
Shear Modulus, GPa	28	
Shear Strength, MPa	283	AA; Typical
Electrical		
Electrical Resistivity, ohm-cm	5.82e-006	AA; Typical at 68°F
Thermal		
CTE, linear 68°F, µm/m-°C	23.2	AA; Typical; average over 68-212°F range
CTE, linear 250°C, µm/m-°C	24.7	Average over the range 20-300°C
Specific Heat Capacity, J/g-°C	0.875	
Thermal Conductivity, W/m-K	121	AA; Typical at 77°F
Melting point, °C	502-638	AA; Typical range based on typical composition for wrought products ¼ inch thickness or greater. Eutectic melting is not eliminated by homogenization.
Solidus, °C	502	AA; Typical
Liquidus, °C	638	AA; Typical

TABLE IV. Properties of natural Fibres [22]

Fibre	**Density (g/cm^3)**	**Tensile strength (MPa)**	**Young's Modulus (GPa)**	**Ultimate elongation (%)**	**Diameter (µm)**
Kenaf	1.4	930	53	1.6	150-900
Flax	1.5	345-1500	27.6	2.7-3.2	40-600

Figure 1. Characterization equipments (TGA and DMA)

III. RESULTS AND DISCUSSION

Two different thermal analyses were performed in this study which was thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA), and their results are presented in this section.

A. Thermogravimetric Analysis (TGA)

TGA became necessary due to the non-oxidative nature of the surface where fire impinges on the samples because of oxygen depletion, and it was performed using inert atmosphere and their thermal properties were evaluated and analysed for the four different composites (CAKRALL, CAFRALL, CARALL and CF+AA).Their TGA and DTG results are shown in Fig. 2-3.

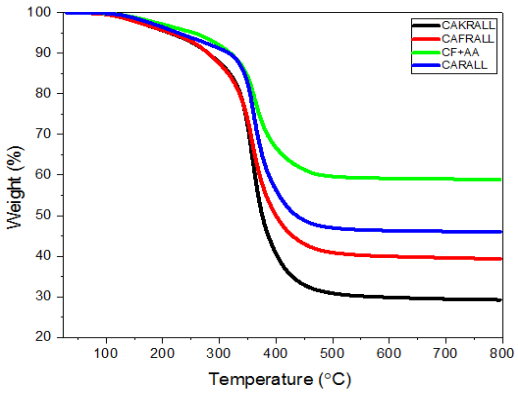


Figure 2. Thermogravimetric curves for the composites

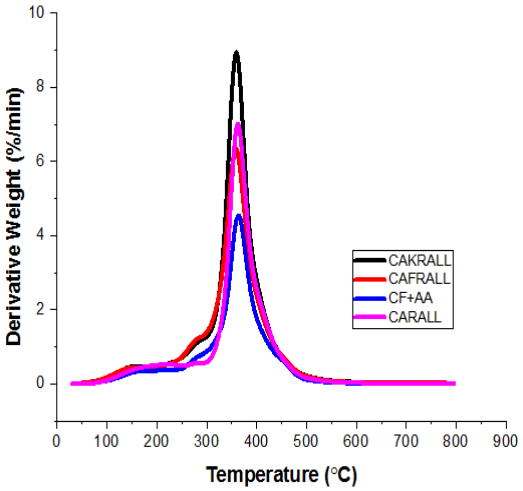
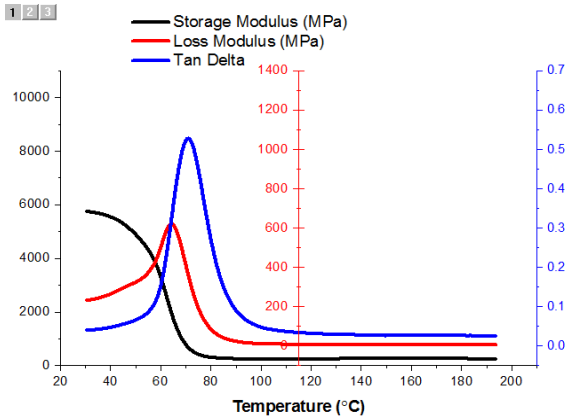


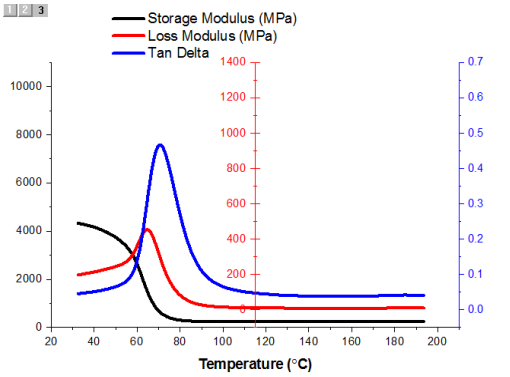
Figure 3. DTG curves for the composites

B. Dynamic Mechanical Analysis (DMA)

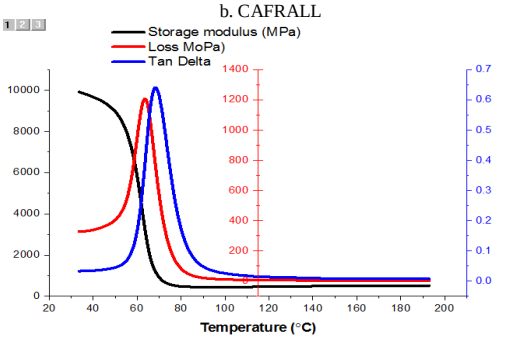
Dynamic mechanical analysis (DMA) was the second thermal test conducted for the FMLs, as it became necessary to know the changes in the stiffness of the laminate composites that can be subjected to high heat temperature as a function of temperature. The test was performed using inert atmosphere and their thermal properties were evaluated and analysed for the four different composites considered. The results are shown in Fig. 4.



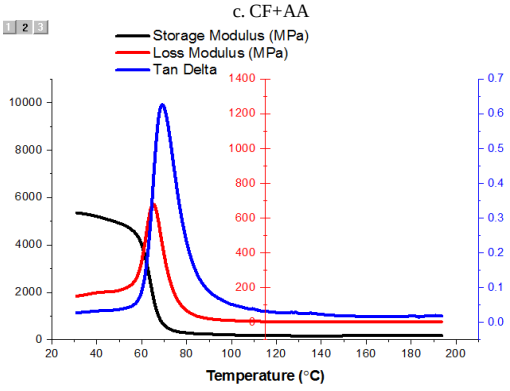
a. CAKRALL



b. CAFRALL



c. CF+AA



d. CARALL

Figure 4. Dynamic mechanical analysis for the Composites

From the curves of Fig. 2-3 of TGAs and DTGs, all four FMLs samples shows almost the same characteristic in terms of percentage weight loss and temperature range for the TGA and also almost similar DTG curves. The mass of the sample were lost as the temperatures of the analysis increased, and an early degradation was observed at a lower heating rate. Almost 3 weight % represented the total degradation was observed around 150^0C of the samples which was mostly due to the low boiling additive in the composites as reported by Brazier and Nickel [23], Sircar and Lamond [24], and Yanget al. [25], also due to the loss of moisture contents in the samples. While 15 weight % of the total degradation was observed around 350^0C, 20 weight % around 380^0C, 52 weight % represented the total degradation around 480^0C and a small degradation of 55 weight % around 800^0C. The residual compound was the only components that remained, whereby different samples had different residue. The samples with only synthetic fibres had the highest residue when compared with the hybrid composites of natural/synthetic fibre of FML. Carbon Fibre Reinforced Aluminium Laminates with aluminium alloy at top and bottom (CF+AA) had the highest residue of 58.85%, while the one with alternate aluminium alloy (CARALL) had 45.93%. The least residue was observed for Carbon Fibre Kenaf Reinforced Aluminium Alloy (CAKRALL) with 29.22%, while Carbon Fibre Flax Reinforced Aluminium Alloy (CAFRALL) produced residue of 39.32%.

Therefore it was clearly observed that a natural fibre undergoes more decomposition than the synthetic fibres, that the degradation of the matrix was delayed by the carbon fibre in the composites, and this result was in agreement with the results obtained with composites using thermoplastic matrices such as glass fibre reinforced with polycarbonate (PC)/acrylonitrile butadiene styrene (ABS) blends and glass fibre reinforced with polyether ether ketone [26, 27]. This indicates that TGA analysis is a method of finding the polymer and fibre contents in an experiment. It was observed that, moisture in the samples escaped during melting and drying, which resulted in thermal decomposition of the samples. The major decomposition of all the composites sample were observed between a temperature range of 350^0C to 480^0C and the compound main degradation was between 350^0C to 380^0C. Different peak temperature values were obtained from different types of samples from their DTG curves, which ranges between 375^0C to 390^0C, whereas the DTG peak temperature is the most important character in the TG analysis programmed temperatures. The main residue that was left after the analysis was the carbon fibre and some small portion of aluminium alloy, while the polymer used in preparing the samples underwent a complete degradation during the analysis. Therefore, the analysis of the composites has indicated a slightly lower thermal stability than their polymer counterparts. Thermal stability of the composites considered was based on the composition and morphological factors (cohesion of the matrix with fibre). The percentage weight loss from DTG curves from Fig. 3 is shown as a function of temperature. Peak DTG temperatures and thermal decomposition temperatures from Table 5 indicate the range of temperatures of the FMLs composites.

TABLE V. 5The DTG peak temperatures and thermal decomposition temperatures for the four composites

Samples	DTG Peak Temperatures (°C)	Thermal Decomposition Temperature (°C)
CAKRALL	395	333.03
CAFRALL	385	326.42
CF+AA	390	335.32
CARALL	385	342.76

The results obtained from TGA showed that all the laminates composites are suitable for use in high-temperature applications.

The results of the DMA analyses of the fibre metal laminates (FMLs) for four composites (CAKRALL, CAFRALL, CF+AA and CARALL) and epoxy based resin and hardener of Fig. 4 were analysed and discussed in this section. The ratio of the loss to storage modulus, a measure of the damping within the system (tan δ) shows the glass transition temperatures of the laminates composites as 71.01^0C, 70.63^0C, 68.22^0C, 69.02^0C, of an epoxy for CAKRALL, CAFRALL, CF+AA, and CARALL respectively. It is clearly indicated that their glass transition temperature is approximately 70^0C. The tests were performed in different range of temperature and frequencies and responded to various transitions and relaxation action of epoxy resin in the laminate composites, which gives more details about the fibre–matrix interfaces. The result indicated a significant decline in the storage modulus among all the laminate composites between 60^0C and 75^0C which corresponded to glass transition temperature of the samples, but at higher temperature a very small differences in the storage modulus were observed among the composites samples. At about 80^0C for all the laminate composites, the stiffness of the composites was well expressed than the softening point of the matrix as there was a slight or no change in the modulus with increase in temperature. The results obtained indicated more increase in the matrix stiffness above than transition glass temperatures of the composites were almost the same with the result obtained on influence of carbon nanotubes on the thermal, electrical and mechanical properties of polyether ether ketone/glass fibre laminates and DMA studies of vapour grown carbon nano fibre [26, 28].

From the DMA curves, it was observed that all the four curves for storage modulus look alike. A remarkable fall was noticed in the section between 50^0C and 70^0C. Therefore, the lower decrement observed was due to the incorporation of polymer matrix with the reinforced fibres that increased the stiffness of polymer matrix; more fibre to the epoxy meant more stiffness to the composites; this phenomenon was observed in [29, 30]. The composition and morphology of the composites affected the elastic energy stored in the materials; as a result of this, the storage moduli of the composites were affected. At room temperature (30^0C), the carbon fibre reinforced aluminium alloy with aluminium alloy at top and bottom (CF+AA) produced an increase in the modulus over the other composites of synthetic with natural fibres of aluminium alloy at top and bottom that is almost 50%;and likewise on 60^0C, 90^0C and 120^0C as indicated in Table 6. The differences between the synthetic composites and synthetic combined with natural fibres and aluminium alloy at top and bottom composites were due to several factors. Among the factors are; more cohesion forces exists between the polymer matrix and synthetic fibre, uniform load were more distributed on synthetic fibre with matrix, more efficient stress transfer exist in synthetic/matrix combination improves more interfacial adhesion among the fibre/matrix. Another factor that affects the performance of the composites is the areas where there is weaker interfacial adhesion between the fibres and matrix, likewise higher internal porosity affect the performance of storage modulus.

TABLE IV. Storage modulus (È) at different temperatures, glass transition temperature (T_g) and tan δ_{max}

Samples	$È_{30^oC}$ (Gpa)	$È_{60^oC}$ (Gpa)	$È_{90^oC}$ (Gpa)	$È_{120^oC}$ (Gpa)	T_g	tan δ_{max}
CAKRALL	5.60	3.00	0.25	0.25	71.01	0.463
CAFRALL	4.50	2.50	0.28	0.25	70.63	0.470
CF+AA	9.90	7.50	0.50	0.60	68.22	0.620
CARALL	5.30	4.20	0.25	0.20	69.02	0.613

From the result obtained of Fig. 4, there was only one peak value for each composite which corresponded to glass transition temperature (T_g) as indicated in Table 6. The T_g of different laminate composites has slight temperature differences between one another due to the interfacial interaction of the polymer matrix with the reinforced fibre; this condition was observed in Warrier *et al.* [31]. Also the T_g of the laminate composites was lower due to the fact that the composites used thermoset polymer and no addition of any binder that would restrict the mobility of the polymer, therefore there was an increase in polymer movement in the composites at lower temperature. Also, the result indicated that synthetic fibre had the highest peak value of tan δ and lower T_g values than the natural fibre sandwich with synthetic fibres by almost 30%; this shows that synthetic fibres allows more mobility of the epoxy than its counterpart (natural fibre). Therefore, elastic nature and shear stress concentration of the fibres together with visco-elastic energy dissipation in polymer matrix of the composite affected tan δ.

The peak values of temperature differences of loss modulus of the four laminate composites were very negligible with carbon fibre reinforced aluminium laminates having the least value with only one transition peak which corresponded to epoxy relaxation. The peak temperature values of the laminate composites ranged from 63°C to 65°C, while their loss modulus values were 610 MPa, 460 MPa, 1230 MPa and 680 MPa for CAKRALL, CAFRALL, CF+AA and CARALL respectively. This indicated that there is more polymer mobility in CF+AA than the other three composites. All the composites can be used in fire designated zone of the aircraft engine due to their thermal stabilities.

IV. CONCLUSION

The performance characteristics of laminate composites of synthetic fibre and natural/synthetic fibre hybrid composites have been evaluated in this study based on their thermal stability in high temperature application. The aim of the study was achieved by evaluating the thermal degradation of the composites. A TGA study showed a remarkable thermal stability improvement in the presence of epoxy resin/hardener (HL002 TA/TB). The incorporation of metal with reinforced fibres yields an intense increment in

the thermal stability of the polymer. Synthetic fibre metal laminates shows a higher percentage of residues than the hybrid of synthetic with natural fibre metal laminates after the composites underwent TGA test, the percentages residues of the laminates composites obtained were: CF+AA produced 58.85%, CARALL 45.93%, CAFRALL 39.32% and CAKRALL 29.22%. The DMA test showed a strong increment of magnitude on storage modulus and an average glass transition temperature of the laminate composites which resisted a temperature of about 200^0C. It showed that higher increment of storage modulus was achieved by increasing fibre while the damping property decreased, which indicated an increase in thermal stability of the composite. CF+AA composite showed a higher storage modulus, therefore had the highest thermal properties compared to the other three types of composites; also the highest peak values of tan δ was observed on the synthetic fibres with less T_g by almost 30%. More epoxy mobility was observed on synthetic fibre composite than in combination of synthetic fibre with natural fibre.

ACKNOWLEDGMENT

The authors would like to thank the Universiti Putra Malaysia for providing the financial support through the IPS Grant scheme no. 9538500. The authors would also wish to thank assistant engineer Mohamad Nasir Johari and Saffairus Salih for their technical support given.

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