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Fire Resistance and Thermal Performance of Sustainable Hybrid carbon/ Flax Fibre Reinforced Aluminium Laminates with Silicon Carbide Filler for Aircraft Engine Fire-Designated Zones

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ABSTRACT

This study evaluates the effects of incorporating flax fibers and silicon carbide (SiC) nanoparticles in carbon/aluminum fiber metal laminates, addressing a critical research gap in sustainable, fireproof composites for aerospace fire-designated zones. Unlike previous studies that examined plant fiber or SiC-filled composites separately, this work investigates their combined impact within fiber metal laminates under ISO 2685 fire conditions. Four laminate variants, CARALL, CARALL + SiC, CFFRAL, and CFFRAL + SiC were fabricated and tested using fire exposure, Thermogravimetric Analysis (TGA), Dynamic Mechanical Analysis (DMA). All the laminates were classified as fireproof, enduring 1100°C for 15 minutes. TGA showed marginally higher weight loss (~2%) in CFFRAL, while SiC addition increased thermal conductivity by 6.5% in CFFRAL and 6.75% in CARALL, reducing fire insulation. DMA revealed enhanced stiffness in SiC-reinforced laminates, with CARALL + SiC showing the highest storage modulus (10.23 GPa at 30 °C). FESEM analysis confirmed resin burnout in CARALL but improved char retention and ceramic residue in SiC-reinforced laminates, while CFFRAL variants displayed micro-cracks, voids, and bio-char formation. These findings demonstrate the viability of flax fibers in aerospace-grade composites, highlight biochar's role in sustainability, and emphasize the need to optimize SiC content for balanced mechanical and thermal performance.

摘要

本研究评估了在碳/铝纤维金属层压板中掺入亚麻纤维和碳化硅 (SiC)纳米粒子的效果，解决了航空航天防火指定区域可持续防火复合材料的关键研究空白. 与之前分别研究植物纤维或SiC填充复合材料的研究不同，这项工作研究了它们在ISO 2685火灾条件下在纤维金属层压板中的组合影响. 制备了四种层压板变体，CARALL、CARALL +SiC、CFFRAL和CFFRAL+SiC，并使用火灾暴露、热重分析 (TGA)、动态力学分析 (DMA)进行了测试. 所有层压板均被归类为防火，耐1100 在°C下保持15分钟. TGA显示CFFRAL的重量损失略高 (约2%)，而添加SiC使CFFRAL和CARALL的导热系数分别提高了6.5%和6.75%，降低了防火隔热性能. DMA显示SiC增强层压板的刚度增强，CARALL +SiC显示出最高的储能模量 (30℃时为10.23 GPa °C). FESEM分析证实了CARALL中的树脂烧坏，但改善了SiC增强层压板中的炭保留和陶瓷残留，而CFFRAL变体显示出微裂纹、孔隙和生物炭形成. 这些发现证明了亚麻纤维在航空航天级复合材料中的可行性，突出了生物炭在可持续性中的作用，并强调了优化SiC含量以实现平衡机械和热性能的必要性.

KEYWORDS

Fire resistance; char morphology; fibre metal laminates; plant fibre composites; aerospace materials; sustainability

关键词

耐火性; 炭形态; 金属纤维层压板; 植物纤维复合材料; 航空航天材料; 可持续性

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Introduction

The aerospace industry demands materials that not only meet stringent safety and thermal performance requirements but also align with global sustainability goals. Aircraft engine fire-designated zones require materials capable of enduring extreme temperatures while maintaining structural integrity and fire resistance (Mohammed 2022b; Talib and Mohammed 2019). Traditional materials, such as carbon fiber composites and aluminum alloys, have long been employed due to their superior mechanical properties and thermal performance (Mazlan et al. 2022; Tiwary, Kumar, and Chohan 2022). However, the environmental impact of synthetic fibers and their energy-intensive production processes has prompted growing interest in greener alternatives. Among these, plant fibers have emerged as promising candidates due to their renewability, biodegradability, and lower carbon footprint. Integrating plant-based reinforcements into advanced composite systems may offer a balanced pathway between high-performance and environmental responsibility. In this context, this study aims to systematically evaluate the combined effects of flax fibers and silicon carbide (SiC) nanoparticles in carbon/aluminum fiber metal laminates (FMLs), with the objective of enhancing fire resistance and thermal performance in aircraft engine fire-designated zones while promoting sustainability through the incorporation of plant fibers in aerospace materials.

Currently, CARALL (Carbon Reinforced Aluminium Laminates) are widely utilized in aerospace applications due to its exceptional strength-to-weight ratio, fatigue resistance, and suitability for critical components like fuselages and engine nacelles (Gao et al. 2023; Y. Kim et al. 2023) (Zhu et al. 2021). investigated the fabrication and thermal-mechanical properties of mesophase pitch-based carbon fiber (MPCF) reinforced aluminum (Al) composites using vacuum hot pressing. The composites with 20–50 vol% MPCF, processed at 650°C, 45 MPa for 60 min, exhibited high thermal conductivity (230.3–288.3 W/m·K), low longitudinal coefficient of thermal expansion (2.28 to −0.22 ppm/K), and high elastic modulus (147.5–324 GPa). The presence of a 2–5 nm amorphous interface layer with minimal carbide formation contributed to their superior thermal and structural properties, making them promising for thermal management applications. Although CARALL offers superior mechanical and thermal performance, the environmental concerns associated with synthetic fibers and energy-intensive production processes underscore the need for greener alternatives. Partial substitution of carbon fibers with plant fibers can contribute to lowering the environmental impact of composites (Ismail, Akpan, and Dhakal 2022). This is achieved by reducing the energy demand during fiber production and introducing renewable, biodegradable materials into the system.

Plant fiber-based materials, known for their inherent char-forming ability, have gained attention as sustainable flame-retardant solutions due to their capacity to reduce heat release and enhance fire resistance through the formation of protective char layers (Morsy et al. 2025; Muralidharan et al. 2024). Several studies have investigated this mechanism and its influence on the flammability performance of plant fiber-based materials. For instance, by treating cotton fabric with a natural blend of pomegranate rind extract (PRE) and 3% sodium tri-polyphosphate (STPP) (Basak and Ali 2019), created a fabric that could put out its own flames, leaving only a short 20 mm char. The treated fabric reached an LOI of 35% and retained over 10% of its mass at 550°C, compared to just 1–2% for untreated cotton. It also burned more slowly, stopped afterglow in its tracks, and produced less smoke and flammable gases, due to the way the treatment encouraged char formation and shifted decomposition to a lower temperature. According to another study by (Bhowmick, Basak, and Samanta 2022), degummed liquor obtained from alkali-treated ramie fiber waste has been reported to contain polyphenols, pectin, and tannin-based compounds, with TG analysis showing ~ 50% mass retention at 500°C. When applied to polyester fabric, this bio-based treatment increased the LOI by ~ 40%, eliminated melt dripping, reduced shrinkage during combustion, and improved the char yield by 20–25% compared to the untreated fabric. These enhancements were attributed to the presence of high-molecular-weight phenolics, pectin, and tannin-based polyphenols, which promote char formation and improve thermal stability (El Gazi et al. 2021). examined a wide range of thermally thin materials, including cotton fabrics, flax fabrics, jute fabrics, polypropylene (PP) knitted fabrics, PP sheets, aramid-based fabrics, and balsa wood sheet, to explore differences in effective heat of combustion (~40 kJ/g for PP vs ~12 kJ/g for plant fiber fabrics) and initial mass influence flammability. They reported that the critical heat flux remained constant at ~20 kW/m^2 for plant fiber fabrics, while time-to-ignition increased with sample weight. Furthermore, peak heat release rate was found to scale proportionally with heat flux, sample weight, and effective heat of combustion. The authors explain that charring materials, such as plant fiber fabrics, improve fire resistance by producing carbon-poor gases during burning, which lowers the amount of heat released, and by forming a protective surface char layer that slows down heat and mass transfer. This char barrier can make thin materials behave more like thick ones in a fire, helping to reduce the peak heat release rate by both limiting the heat produced and decreasing the amount of fuel available to burn.

Plant fibers have also emerged as promising substitutes for synthetic fibers in fiber metal laminates (FML), offering environmental benefits such as renewability, biodegradability, and reduced carbon footprint (More 2022). Previous research has investigated plant fibers such as kenaf in fiber metal laminates, focusing on their thermal, physical, and mechanical properties. In this context (Mohammed et al. 2018), assessed the thermal stability and viscoelastic properties of carbon and kenaf FML composites for high-temperature applications in aircraft engine nacelles. TGA results showed that carbon fiber-reinforced aluminum laminate had the highest residue at 58.85%, while kenaf-based composites had the lowest, indicating superior thermal stability in synthetic fiber composites. DMA analysis confirmed enhanced storage modulus and glass transition temperature, making carbon and hybrid kenaf FML suitable for fire-designated zones in aerospace applications (Ishak et al. 2021). We examined the effect of water absorption on the tensile properties, thickness swelling, and formability of plant fiber metal laminate made of kenaf-polypropylene and aluminum 5052-O. Results showed that the kenaf metal laminates absorbed 0.67% water with 0.91% thickness swelling, but water had minimal impact on its tensile properties. Additionally, the composite exhibited higher formability than aluminum sheets, making it suitable for complex-shaped lightweight automotive components.

Flax fiber is a plant fiber derived from the stems of the flax plant (Linum usitatissimum). It is classified as a bast fiber, which comes from the inner bark (phloem) of the plant. The fibers are known for their high strength, low density, biodegradability, and excellent thermal stability, making them a sustainable alternative to synthetic fibers in composite materials. Flax fiber offers excellent thermal stability and produces high char residue after thermal degradation, which acts as a protective barrier against heat, making it a promising reinforcement in FML for high-temperature applications (Khiari et al. 2021). Its strong mechanical properties, fire resistance, and insulating char layer enhance the structural integrity of components in aircraft engine fire-designated zones (N. K. Kim et al. 2020). Previous studies on the properties of FML incorporating flax fibers, such as FLAx-REinforced aluminum (FLARE), revealed their potential for improved environmental sustainability and balanced performance. FLARE demonstrates a reduced specific energy absorption (25% lower than glass fiber laminates) and a significant decrease in damping loss factor with aluminum addition, making it suitable for aerospace and broader transportation applications while offering recyclability and a lower carbon footprint (Alcaraz, Alderliesten, and Mosleh 2024) (Mohammed 2023). We investigated the thermal stability of hybrid plant-carbon FML using flax and kenaf composites, fabricated via hand lay-up and post-cured at 80°C for 3 hours. TGA results showed carbon/flax composites had 29.75% higher residue than carbon/kenaf, while DMA revealed carbon/kenaf composites had a 0.5% higher storage modulus at higher temperatures, indicating their suitability for fire-designated zones in automotive and aerospace applications. Another study by (Mohammed 2022a) revealed that carbon/flax FML exhibited a 21.43% higher thermal conductivity and withstood flame temperatures for 15 minutes, meeting the ISO 2685 standards when compared to carbon/kenaf FML. Study by (Mohammed 2019) shows that carbon fiber-reinforced aluminum alloy laminates (CF+AA) with aluminum layers on the top and bottom outperform other configurations, resisting flame temperatures 7.86% longer than CARALL (alternating aluminum and carbon layers), 18.2% longer than flax-carbon laminates, and 23.43% longer than kenaf-carbon laminates, making them highly suitable for aircraft engine fire-designated zones.

Despite the advantages of plant fiber composites, concerns remain regarding their thermal stability and mechanical performance under the extreme conditions typical of aerospace applications. The incorporation of silicon carbide (SiC) nanoparticles into hybrid composites has shown potential to enhance their thermal conductivity, stiffness, and fire resistance. Based on a study by Vaggar et al. (Vaggar et al. 2022) silicon carbide nanoparticles (SiC-NPs) exhibited greater thermal stability than copper nanoparticles (Cu-NPs) in glass fiber reinforced epoxy composites. The weight loss ranges from 44.2% to 76.8% for SiC-NPs composites compared to 64.5–94.5% in Cu-NPs composites at 750°C. The study proved that thermal conductivity increased with filler percentage, with SiC-NPs composites showing higher values than Cu-NPs composites. According to (Ganesh, Anand, and Degife Mammo 2022), inclusion of 10 vol% silicon carbide (SiC) in kenaf fiber-reinforced epoxy composites significantly enhanced thermal properties, with thermal conductivity reaching 0.71 W/mK (97.22% higher than pure kenaf composites), and heat deflection temperature (HDT) increasing to 119.25°C (34.89% higher than composites without SiC). Additionally, the coefficient of thermal expansion (CTE) decreased by 21.32%, and thermal stability improved, with a maximum safe working temperature of 330°C and 22.16% higher residues at 700°C compared to composites without SiC. Additionally, Cheng et al. (2021) developed a three-dimensional (3D) functional silicon carbide nanowire/natural rubber (f-SiCnw/NR) thermal interface material (TIM) using a freeze-casting method to enhance heat transfer efficiency. The composite achieved a thermal conductivity of 0.856 $W\ m^{-1}\ K^{-1}$ at 8.89 vol% filler content, demonstrating excellent heat dissipation, electrical insulation, and mechanical properties. This approach offers a promising strategy for creating high-performance rubber-based materials for advanced thermal management applications. Kang et al. (2021) evaluated the mechanical properties of woven SiC fiber fabric using the KES-FB system, confirming its suitability for high-heat environments above 1500°C. The SiC composite yarn fabric exhibited high compressive energy-to-thickness (WC/T) ratios, indicating bulkiness, and passed KFI criteria for carbonation length and cumulative flame time, proving its effectiveness as fire-resistant work clothing. Although there are several studies recording, the potential of SiC to enhance thermal conductivity and fire resistance, the effects of combining flax fibers with SiC fillers in FML remain underexplored, particularly for aircraft fire designated zone applications.

This research aims to systematically investigate the combined effects of flax fiber reinforcement and silicon carbide (SiC) nanoparticles on the fire resistance, fiber and char morphology of burnt laminates, and thermal stability of Fiber Metal Laminates (FMLs), with a specific focus on Carbon/Flax Fiber Reinforced Aluminium Laminates (CFFRAL) for use in aircraft engine fire-designated zones. While prior studies have focused separately on either synthetic or plant fiber composites and the inclusion of ceramic fillers like SiC, the impact of integrating carbon/flax hybrid fibers with SiC fillers remains largely unexplored. This study fills that gap by examining how SiC inclusion influences thermal conductivity, structural integrity, and fire behavior, with the objective of optimizing sustainable composite materials for aerospace safety applications. The fire test, conducted in accordance with ISO 2685 standards, is significant for evaluating the real-time flame resistance and burn-through capability of the laminates under high-temperature exposure, reflecting operational aircraft engine scenarios. Thermogravimetric Analysis (TGA) provides precise quantification of thermal degradation, weight loss, and residual char content, which are essential indicators of fire-retardant performance and long-term stability. Dynamic Mechanical Analysis (DMA) evaluates the stiffness, damping, and thermal transition behavior of the composites, offering insights into their viscoelastic properties across a temperature range relevant to aerospace environments. Furthermore, Field Emission Scanning Electron Microscopy (FESEM) of burnt laminates enables detailed observation of microstructural changes, fiber-matrix interaction, and char morphology, which are crucial for understanding failure mechanisms during combustion. Collectively, these evaluations contribute to the development of next-generation aerospace materials that are not only fireproof and mechanically robust but also environmentally sustainable and safer for cabin environments.

Materials and method

Materials

The materials utilized for fabricating the specimens in this study include aluminum alloy sheets 2024-T3, woven carbon fiber, woven flax fiber, silicon carbide nanoparticles, and epoxy resin. Some of these materials require procurement as they are not commonly available for experimental and practical applications. The aluminum sheets underwent mechanical treatment, alkaline etching, and anodizing to ensure strong interfacial bonding between the aluminum and FRP layers. Aluminum alloy is the most used material in aircraft due to its lightweight nature and high strength-to-weight ratio. Furthermore, aluminum alloys exhibit high corrosion resistance due to the protective oxide layer that naturally forms on their surface. In this study, Aluminum 2024-T3 (Figure 1a) sheets are selected due to their wide adoption in aircraft

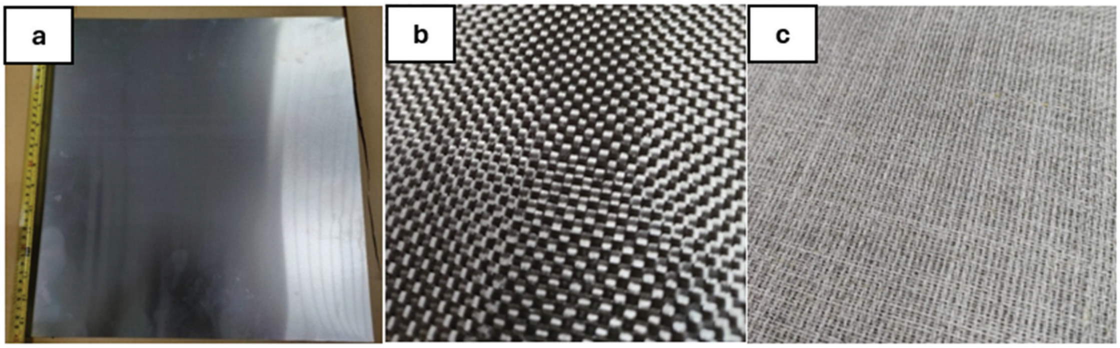


Figure 1. (a) Aluminium alloy 2024-T3 sheet (400mm x 400mm); (b) woven carbon Fibre (400mm x 400mm); (c) woven flax Fibre (400mm x 400mm).

Table 1. Properties of woven carbon Fibre.

Properties	Value
Grade	HTS40 (3K)
Density (g/cm^3)	1.76
Areal Density (GSM)	220
Filament Diameter (μm)	7.0
Tensile Strength (MPa)	4200
Tensile Modulus (GPa)	240
Elongation (%)	1.8
Filament Count	3000

materials, high strength-to-weight ratio, excellent damage tolerance, and superior fatigue resistance, making them ideal for ensuring structural integrity and performance in the extreme conditions of aircraft engine fire-designated zones.

Woven carbon fiber (Figure 1b) is one of the most popular materials in nearly all industries, particularly in the aerospace sector. It is used both individually and in combination with other fibers to form hybrid composites tailored for various applications based on specific requirements. This popularity stems from its exceptional mechanical and thermal performance compared to other synthetic fibers. Table 1 lists the properties of plain-woven carbon fiber used in this study according to the supplier's technical data sheet.

Flax fabrics, belonging to the Linaceae family, are applied in various industries as substitutes for synthetic fibers, thanks to their lightweight and high strength. In this study, plain woven flax (Figure 1c) was selected due to its mechanical properties, which closely resemble those of glass fiber and outperform those of other plant fibers. Flax fibers are inherently hydrophilic and tend to absorb moisture, which can adversely affect the composite's dimensional stability and interfacial bonding. To mitigate this, the flax fibers were treated with a 5% sodium hydroxide (NaOH) solution, which removes surface impurities and hemicellulose, enhances fiber-matrix adhesion, and reduces water absorption. The areal density of the plain-woven flax fiber used in this study is 260 gsm. Flax has a density of 1.5 g/cm^3, an elongation of 2.7–3.2%, a tensile strength of 500–1500 MPa, and an elastic modulus of 27.6 GPa.

The epoxy resin system used in this study was Smooth-On EpoxAmite® 100 (Part A), a high-performance laminating epoxy known for its excellent bonding properties and durability. It was combined with EpoxAmite® 102 Medium Hardener (Part B), a slow-curing hardener that provides extended working time, allowing for more precise lay-up and improved wet-out of reinforcing fibers.

Table 2. Properties of silicon carbide (SiC).

Purity	Content (%)					
SiC	F.C	Fe	Al	Ca	O	Others
99.95%	<0.1	<0.005	<0.005	<0.01	<0.2	<0.5

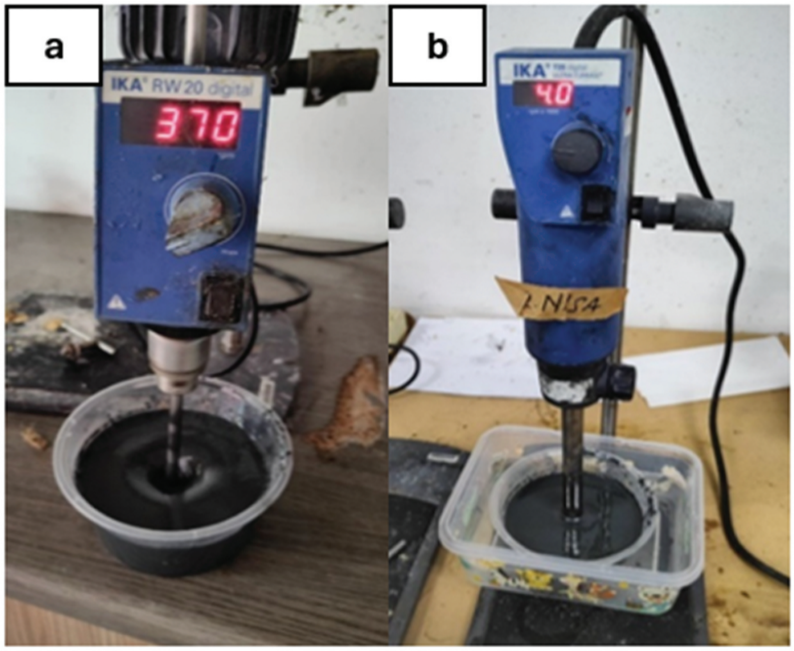


Figure 2. Preparation of epoxy/SiC mixture; (a) mechanical stirring of SiC in epoxy; (b) dispersion of SiC in epoxy.

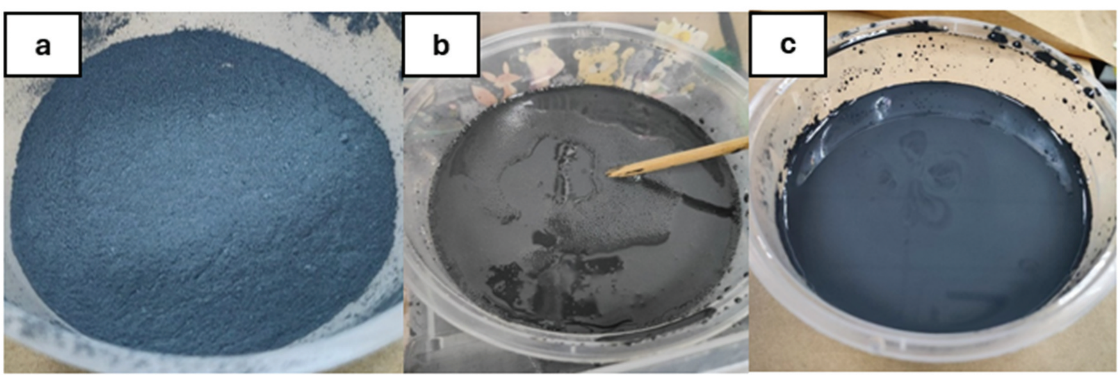


Figure 3. Homogenization of epoxy/SiC mixture (a) SiC 50nm particle size powder used in this Experiment; (b) epoxy/SiC before dispersion; (c) epoxy/SiC after dispersion.

Silicon carbide (SiC), is a compound of silicon (Si) and carbon (C), known for its exceptional hardness, high thermal conductivity, and excellent resistance to wear, corrosion, and extreme temperatures, which contributes to enhancing the overall performance of the fabricated composites. SiC was chosen as the filler for epoxy resin due to its high melting point and thermal stability, which can reach up to 2700°C. However, SiC is also known for its high thermal conductivity, which may not be favorable for fire-retardant applications. In this research, the effect of SiC on the overall fire resistance and thermal performance of the Hybrid FML is studied. Table 2 shows the properties of SiC (50 nm particle size) according to the technical data sheet provided by supplier. The density of SiC ranges from 1.7 to 2.5 g/cm^3.

To incorporate SiC nanoparticles into the laminates (applicable to laminates 2 and 4), an exact amount of SiC powder (50 nm particle size) was first added to the epoxy resin and mechanically stirred for 5 minutes, as shown in Figure 2a. This initial mixing helps to evenly distribute the nanoparticles and prevent agglomeration. Following this, the mixture underwent further dispersion using a probe homogenizer for 30 minutes to achieve uniform nanoparticle dispersion at the nanoscale level, as demonstrated in Figure 2b. The homogenization was conducted in an ice-water-salt bath to prevent temperature rise, which could otherwise degrade the epoxy resin's molecular structure and reduce the composite's mechanical integrity. The effectiveness of the dispersion process is illustrated in Figure 3,

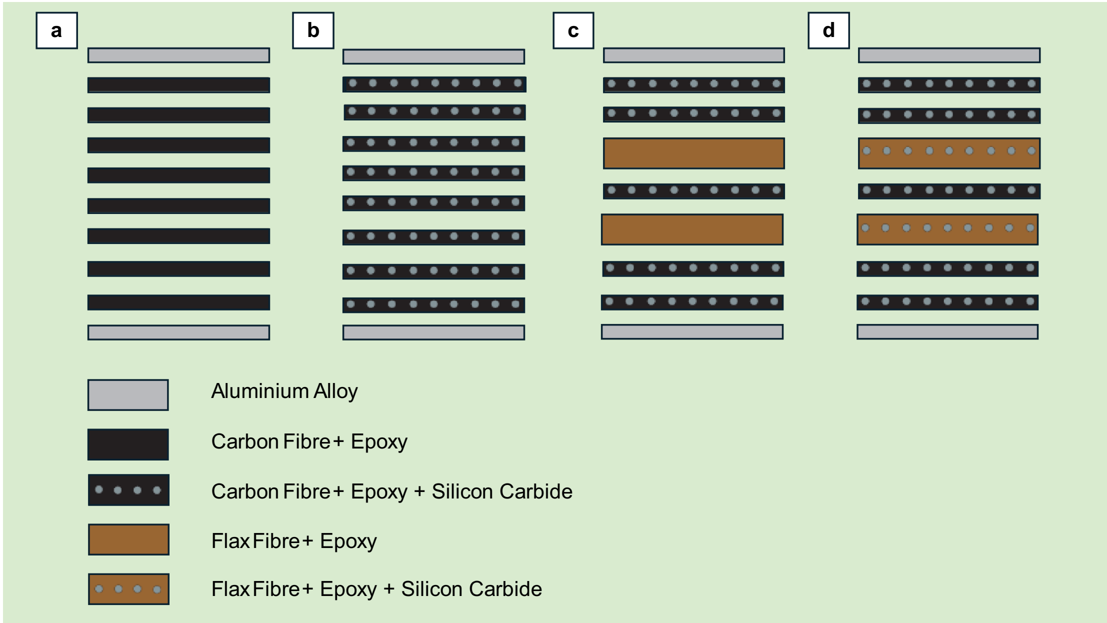


Figure 4. Laminate composition and stacking sequences in 2D View; (a) CARALL; (b) CARALL + SiC; (c) CFFRAL; (d) CFFRAL + SiC.

which shows the SiC powder used (Figure 3a), the epoxy/SiC mixture before homogenization (Figure 3b), and the improved homogeneity after dispersion (Figure 3c). The well-dispersed epoxy/SiC mixture was then used as the matrix in the fabrication of laminates CARALL + SiC and CFFRAL + SiC. The incorporation of SiC nanoparticles is aimed at enhancing the thermal conductivity, stiffness, and fire resistance of the laminates by forming a more thermally stable and structurally robust polymer matrix.

Fabrication

Four variants of aluminum metal-fiber hybrid laminate samples were fabricated using hand layup method followed by compression molding. Some materials required pre-processing before fabrication, such as mechanical treatment for the aluminum alloy sheets and the combination of silicon carbide with epoxy resin. A total of four laminates were fabricated with different stacking sequences and compositions but nearly identical thicknesses, as shown in Figure 4.

The fabricated laminates in this study were approximately 3.26–3.40 mm thick, with each variant comprising distinct layering sequences and material proportions. The laminates are reinforced with epoxy resin mixed with a hardener in a ratio of 100:28.4, meaning that for every 100 g of epoxy, 28.4 g of hardener is added, according to the manufacturer's instructions. Table 3 shows the number of layers and weight percentage of each material used to fabricate the variants.

CARALL and CARALL + SiC laminates consisted of two outer aluminum alloy (2024-T3) sheets sandwiching eight layers of woven carbon fiber, all oriented in a plain weave configuration, with the latter incorporating 1 wt% silicon carbide (SiC) nanoparticles uniformly dispersed in the epoxy matrix. The first

Table 3. Number of layers and weight percentage of each element in laminates.

	Number of Layers			Weight Percentage (%)				
Laminate	Aluminium Alloy	Carbon Fiber	Flax Fiber	Aluminium	Carbon Fiber	Flax Fiber	Polymer	Silicon Carbide
CARALL	2	8	–	23.55	26.45	–	50	–
CARALL + SiC	2	8	–	23.55	26.45	–	49	1
CFFRAL	2	5	2	24.7	17.3	8	50	–
CFFRAL + SiC	2	5	2	24.7	17.3	8	49	1

Table 4. Size and mass of each material used.

Material	Size (mm)	Mass (g)	Thickness (mm)
Woven Carbon Fibre	400 x 400 + 5%	36.5 ± 0.5	0.30 ± 0.02
Flax Fibre	400 x 400 + 5%	42.0 ± 3.0	0.50 ± 0.05
Aluminium Alloy Sheet	400 x 400	130.0 ± 0.5	0.33 ± 0.02

Table 5. Fibre and polymer mass of each laminates.

Laminate	Aluminium Alloy (g)	Carbon Fibre (g)	Flax Fibre (g)	Epoxy (g)	Hardener (g)	Silicon Carbide 1% (g)	Total Mass (g)	Total Thickness (mm)
CARALL	260	292	–	429.90	122.09	–	1104	3.26 ± 0.5
CARALL + SiC	260	292	–	421.30	119.65	11.04	1104	3.26 ± 0.5
CFFRAL	260	182.5	84	410.05	116.50	–	1053	3.40 ± 0.5
CFFRAL + SiC	260	182.5	84	401.84	114.12	10.53	1053	3.40 ± 0.5

sample also serves as a benchmark specimen to compare the properties of the other specimens that incorporate plant fiber and silicon carbide in addition. In contrast, the CFFRAL and CFFRAL + SiC laminates featured two aluminum sheets, five layers of carbon fiber, and two layers of woven flax fiber, also in a plain weave, arranged in alternating sequence to ensure structural integrity and balance. The CFFRAL + SiC laminate included 1 wt% SiC nanoparticles within the epoxy resin. Fiber mass proportions were approximately 17.3% carbon fiber and 8% flax fiber in the hybrid variants, while the polymer matrix (epoxy and hardener) accounted for around 49–50% of the total weight, ensuring consistency across the laminates.

Table 4 presents the size, mass, and thickness of the woven carbon fiber, flax fiber, and aluminum alloy sheets used in fabricating the laminates, showing that flax fiber is slightly heavier and thicker than carbon fiber. Table 5 outlines the mass distribution of each material within the four laminate variants (CARALL, CARALL + SiC, CFFRAL, and CFFRAL + SiC). One percent silicon carbide (SiC) is incorporated in CARALL + SiC and CFFRAL + SiC. The total mass and thickness of the laminates remain consistent across their respective categories, with CARALL variants being slightly heavier (1104 g) than CFFRAL variants (1053 g).

Fire test analysis

The fire resistance of one laminate from each variant were evaluated using a calibrated propane burner compliant with ISO 2685. Burner calibration was conducted prior to testing to ensure compliance with the standard, achieving a flame temperature of 1100°C ±80°C and a heat flux density of 116 ± 10 kW/m^2 ("ISO 2685:1998," 1998). Specimens measuring 300 × 300 mm were cut and prepared using a bandsaw, spray-painted black, and allowed to dry as shown in Figure 5a. To ensure consistent emissivity and accurate thermal imaging during the fire test, all specimens were spray-coated with a thin layer (approximately 30–50 μm) of high-temperature-resistant matte black paint (Rust-Oleum High Heat, rated up to 650 °C).

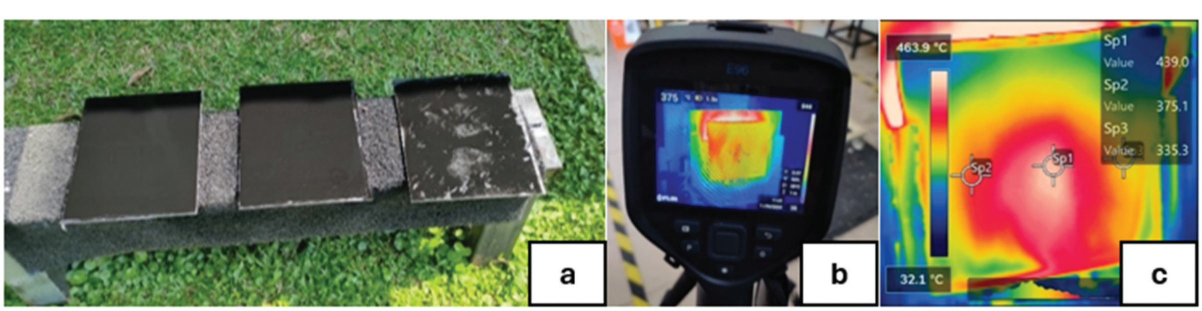


Figure 5. (a) Black paint sprayed specimens; (b) advanced thermal imaging camera taking image of burning specimen (c) temperature analysis using FLIR software.

This coating was selected due to its stable performance at elevated temperatures and its low reflectivity, which enhances the reliability of infrared thermographic measurements. The uniform matte finish reduces glare from the aluminum surface, enabling accurate detection of temperature gradients on the cool face of the specimen using a thermal imaging camera.

A thermal imaging camera calibrated for a range of 0°C to 600°C was used to monitor the cool face temperatures. Following flame stabilization, specimens were exposed to the burner flame at a 3-inch distance for 15 minutes, with thermal images captured every 2 minutes, as shown in Figure 5b. FLIR software was used to analyze the back-face temperatures at three points as shown in Figure 5c, calculating mean temperatures for thermal conductivity assessment. From the fire test, each laminate was classified as either fire-resistant, fireproof, or neither. According to ISO 2685, the composite is considered fire-resistant if it can withstand 5 minutes of standard flame exposure, and fireproof if it can endure 15 minutes under standard flame conditions without being burned through. The thermal conductivity of each laminate was calculated using equation (1), Fourier's law of heat conduction:

$$q = -\lambda \frac{\Delta T}{\Delta x}(1) \quad (1)$$

Where:

- q = Heat Flux in W/m^2,
- λ = Thermal conductivity of the specimen, W/m·K
- ΔT = Temperature difference between the front face and back face of the specimen,
- Δx = Distance between front face and back face of the specimen (thickness) in m.

Thermogravimetric analysis (TGA)

TGA was conducted using a Mettler Toledo TGA/DSC 1 STAR System, with a small ceramic furnace capable of achieving temperatures up to 1100°C with a controlled heating rate ranging from 0.1°C to 50°C/min. The system features an ultra-microbalance with a resolution of 0.1 μg, ensuring highly accurate weight measurements during thermal degradation. The furnace chamber is designed for rapid heat transfer and uniform temperature distribution, with continuous purge gas flow (typically nitrogen or air) to simulate inert or oxidative environments. This setup allows simultaneous recording of mass loss and thermal events, making it suitable for evaluating thermal stability, decomposition behavior, and residual content of composite samples under precisely controlled heating conditions. This test provides results on residual weight and weight loss over time, allowing for an analysis of thermal stability. In the TGA machine, the rate of weight loss and the percentage of residual sample are determined by comparing the sample's weight before and after heating. The samples were prepared by cutting the laminates using a band saw. This testing method is not influenced by the sample size of each specimen, allowing for variation in sample weight according to the cutting size. The weights of the samples used in TGA testing were as follows: CARALL – 63.72 mg, CARALL + SiC – 42.22 mg, CFFRAL – 58.89 mg, and CFFRAL + SiC – 38.50 mg. The samples were heated from 30°C (ambient temperature) to 800°C at a rate of 15°C per minute.

Dynamic mechanical analysis (DMA)

The testing was conducted in accordance with the ASTM D7028 standard, which employs a flexural three-point bending mode on a dual cantilever setup. According to the standard, the samples were prepared to dimensions of 60 × 12 × 6 mm using a band saw. During the test, the samples were heated from 30°C (ambient temperature) to 200°C at a heating rate of 5°C per minute. The DMA test was performed using the DMA Instrument Q800 (V20.24 Build 43) with Instrument Serial Number 0800–1072. The tests involved collecting data on storage modulus, loss modulus, displacement, and the force applied during the flexural tests. This evaluation aimed to analyze the viscoelastic properties of each specimen under bending loads and high temperatures, with the goal of assessing their durability and fatigue resistance.

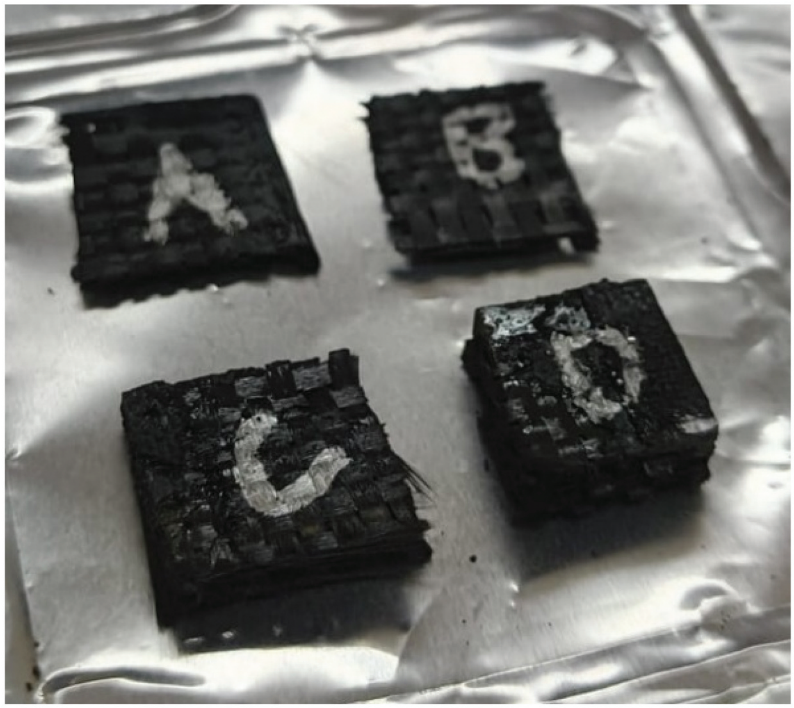


Figure 6. Burnt specimens for FESEM analysis; (a) CARALL; (b) CARALL + SiC; (c) CFFRAL; (d) CFFRAL + SiC.

Field Emission Scanning Electron Microscopy (FESEM) analysis

To evaluate the microstructural morphology and degradation characteristics of the developed fiber metal laminates (FMLs) post-fire exposure, burnt specimens (Figure 6) were subjected to Field Emission Scanning Electron Microscopy (FESEM) analysis. The study aimed to assess the char structure, fiber degradation, and ceramic residue distribution after the ISO 2685 fire test. Due to the extensive thermal damage and matrix decomposition, only the CFFRAL and CFFRAL + SiC laminates retained sufficient polymer-char material for direct FESEM observation. These charred samples were sectioned and mounted for surface analysis to investigate the morphology of the degraded matrix, fiber–matrix interface, and the potential reinforcement effects of silicon carbide (SiC) nanoparticles in the polymer residue.

For CARALL and CARALL + SiC laminates, the exposure to 1100°C flame caused severe delamination and complete resin burnout, resulting in the recovery of only the burnt woven carbon fiber mats. These fiber mats were analyzed to examine surface damage, thermal degradation signs, and the presence of any residual deposits or SiC remnants on the fiber surface. Although matrix information was not available for these samples, the carbon fiber condition provided insight into fire-induced structural changes. All specimens were mounted on aluminum stubs using conductive carbon tape and sputter-coated with a thin layer of platinum (approx. 10 nm) to enhance conductivity. Imaging was performed using a JEOL JSM 7600F FESEM operated at an accelerating voltage of 5–10 kV, with magnifications ranging from 100× to 50,000× depending on the surface features of interest. High-resolution images were captured to examine surface porosity, char continuity, and the presence of microcracks, delamination fronts, and fiber pull-out zones.

Results and discussion

Fire test

The burner calibration for the fire test ensured compliance with ISO 2685 standards, achieving a stabilized flame temperature of 1163°C with a heat flux of 116.39 kW/m^2. After igniting the burner, the calibration process was carried out until the temperatures recorded by the thermocouples stabilized for 2 minutes. The burner took approximately 120 seconds to reach 1000°C, and the flame began to stabilize after 300 seconds. The flame temperature increased slowly from 0 to 80 seconds because the propane gas pressure was gradually adjusted to ensure safety throughout the process. Once it was confirmed that there were no issues with the burner, both the propane gas pressure and the cool gas pressure gradually increased, raising the temperature from around 450°C to 1100°C within 30 seconds. From 120 seconds to 300 seconds, the pressure of the valves was continually adjusted until the flame temperature stabilized within the range specified by the ISO 2685 standard. Between 300 and 500 seconds, the temperature exhibited some fluctuations but remained relatively high and stable, with a lowest recorded temperature of 1097°C and a highest temperature of 1195°C. The average temperature during this period was 1163°C. These temperature fluctuations were attributed to inconsistent air velocity and slight instability in the supply of the three different gases. However, the average temperature was maintained within the range required by ISO 2685 for more than 2 minutes. Gas pressures used were 260 Pa for propane, 4071 Pa for mixing gas, and 2870 Pa for cooling air, aligning with findings by (Talib and Mohammed 2019).

Each laminate was subjected to burning until either burn-through occurred or the test reached a time limit of 15 minutes. The hot surface (the front surface facing the burner) was assumed to have a consistent temperature of 1163°C, as recorded during the burner calibration. Meanwhile, the temperatures at three selected points on the cool face (the surface facing the thermal camera) were recorded every 2 minutes. The average temperature of these three points was then calculated depicted in Figure 7. CARALL + SiC and CFFRAL + SiC achieved higher temperatures more quickly than the other laminates during the first 100 seconds. The distinguishing factor for CARALL + SiC and CFFRAL + SiC is the inclusion of silicon carbide nanoparticles in their polymer matrix. According to (Hapuhinna, Gunaratne, and Pitawala 2022) the addition of SiC enhances the cross-link density of the epoxy, thereby improving the thermal conductivity of the composite. This improved thermal conductivity facilitates faster heat transfer through the laminate structure. Although direct porosity measurements were not conducted, it is plausible that enhanced matrix

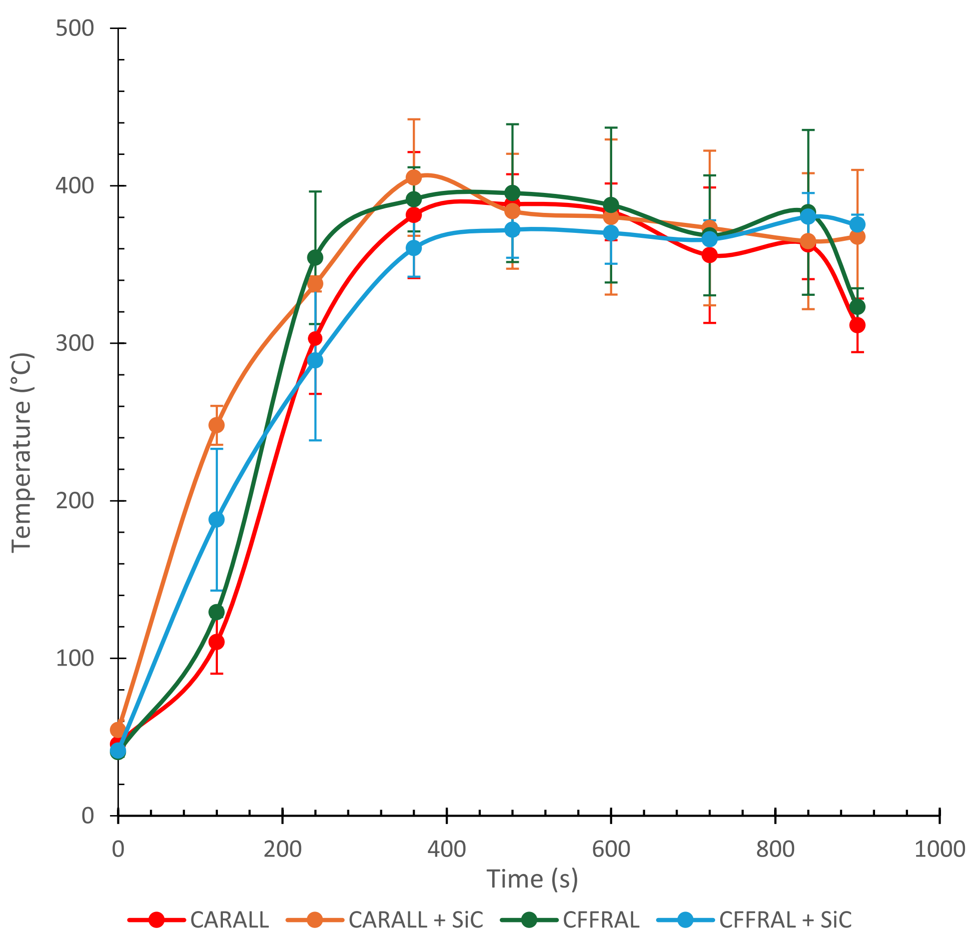


Figure 7. Temperatures vs Time plot for fire test.

Table 6. Fire test result of each laminate.

	Average Temperature (^{0}C)			
Time (Seconds)	CARALL	CARALL + SiC	CFFRAL	CFFRAL + SiC
0	45.42	54.43	40.37	41.30
120	110.24	247.90	129.20	188.00
240	302.88	337.73	354.30	289.07
360	381.40	405.23	391.40	360.37
480	388.33	383.83	395.33	372.03
600	383.47	380.17	387.77	370.00
720	355.93	373.20	368.53	365.93
840	362.73	364.83	383.13	380.30
900	311.40	367.63	323.03	375.37

homogeneity due to SiC addition may have contributed to reduced thermal insulation, leading to the observed increase in heat transmission rate (Li et al. 2023).

After 200 seconds, all four laminates exhibited nearly identical increments in temperature, and by 350 seconds, the temperatures began to stabilize, maintaining a range between 350°C and 400°C. The highest temperature recorded was 405°C at 360 seconds for the CARALL + SiC laminate. This sudden temperature spike was attributed to flames slipping through the laminate from the side causing the thermal camera to detect the flame temperature rather than the temperature of the side area of the laminate. At 900 seconds, the lowest temperature recorded was 311°C for CARALL, followed by CFFRAL at 323°C, CARALL + SiC at 367°C, and CFFRAL + SiC at 375°C.

Based on the results in Table 6, laminates containing flax fibers demonstrated higher temperatures compared to those with only carbon fibers; however, the differences were minimal. Specifically, CFFRAL recorded only 3.66% higher temperatures than CARALL, while CFFRAL + SiC recorded 2.08% higher temperatures than CARALL + SiC. These results highlight the potential of flax fibers in providing fire insulation and performing well in high-temperature environments, suggesting that some carbon fiber layers could potentially be replaced with flax fiber layers. Additionally, the laminates enhanced with silicon carbide nanoparticles consistently exhibited higher temperatures than those without additives. The introduction of silicon carbide nanoparticles increased the temperature of CARALL by 16.56% and CFFRAL by 15%. This indicates that while adding silicon carbide nanoparticles improves temperature tolerance. However, it may not be ideal for thermal insulation applications, as the reduced air pockets lead to faster heat transfer.

According to Table 7, all laminates were able to withstand the flame for over 900 seconds without experiencing burn-through, classifying them as fireproof materials according to the ISO 2685 fire test standard. However, each laminate exhibited slightly different characteristics during the burning process. Qualitative observations during the ISO 2685 fire tests provide valuable insights into the combustion behavior of the developed laminates. All specimens exhibited surface flaming primarily due to epoxy matrix degradation; however, those incorporating silicon carbide (SiC) nanoparticles demonstrated visibly reduced flame intensity and shorter flame duration. This effect is attributed to increased crosslink density in the matrix, which helped suppress volatile release and flame propagation. Carbon fiber played a dominant role in thermal shielding, contributing to higher thermal residue and lower back-face temperatures due to its inert, high-temperature-resistant structure. In the hybrid variants containing flax fibers (CFFRAL and CFFRAL + SiC), flax contributed to char formation and partially improved fire retardancy, though with slightly higher back-face temperatures. This is due to the partial combustion of lignocellulosic components in flax under high-temperature conditions. However, flax also acted as an effective insulator, delaying heat transfer during early-stage combustion.

Although direct Limiting Oxygen Index (LOI) testing was not conducted, the thermal performance of the laminates can be indirectly assessed using TGA data and burn-through resistance under ISO 2685

Table 7. Fire resistant characteristics of each specimen.

Specimens	Burn Through Time (s)	Result
CARALL	>900	Fireproof
CARALL + SiC	>900	Fireproof
CFFRAL	>900	Fireproof
CFFRAL + SiC	>900	Fireproof

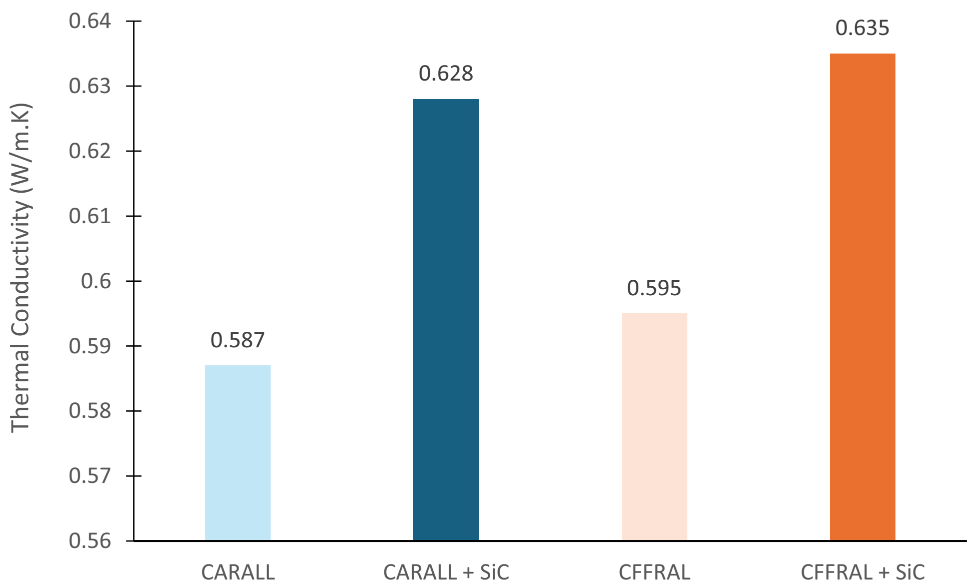


Figure 8. Thermal conductivity.

conditions. Laminates with higher thermal degradation onset and residue percentage such as CFFRALL with 344.78°C onset and ~ 48.5% char residue demonstrated comparable thermal stability to carbon fiber-dominant composites. Moreover, all the laminates withstood direct flame exposure at 1100°C for 15 minutes without burn-through, further confirming their classification as fireproof. These results are consistent with materials that typically exhibit LOI values above 26%, suggesting that the developed hybrid laminates possess strong resistance to ignition and sustained burning under oxygen-limited conditions.

After obtaining the cool face temperature of each laminate, the thermal conductivity was analyzed using Equation (1) and the results were visualized in Figure 8. Since the final temperature of the cool face is directly proportional to the thermal conductivity, the thermal conductivity results exhibited a similar trend to the temperature versus time results observed during the fire test. Laminates containing silicon carbide nanoparticles demonstrated overall higher thermal conductivity compared to those without the additive, while replacing some carbon fiber layers with flax fibers slightly increased the thermal conductivity of the laminates. Specifically, adding silicon carbide nanoparticles to CARALL increased thermal conductivity by 6.75%, and for CFFRAL, the increase was 6.5%. Similar phenomenon has also been reported by Mikociak et al. (Mikociak, Rudawski, and Blazewicz 2018). In contrast, replacing carbon fibers with flax fibers resulted in only about a 1% increase in thermal conductivity.

High thermal conductivity is generally undesirable for thermal insulation applications, as it facilitates greater heat transfer through the material, which contradicts the primary goal of maintaining a stable internal temperature. In this study, the addition of silicon carbide (SiC) nanoparticles increased the thermal conductivity of the laminates. While this may enhance heat dissipation in certain contexts, it is not advantageous for applications requiring effective thermal insulation, such as fire-resistant aerospace structures. Conversely, flax fibers exhibited fire-retardant characteristics that were comparable to those of carbon fibers. At first glance, this result may seem unsurprising when considering typical ambient conditions; however, it becomes more significant in the context of ISO 2685 fire-designated zone simulations, where laminates were exposed to direct flame at temperatures exceeding 1100°C. In these tests, flax fibers were embedded within aluminum-based fiber metal laminates and subjected to high thermal loads with limited oxygen supply on the back face due to the metallic layer conditions that differ significantly from open atmospheric exposure. The performance of flax fibers in this confined, high-temperature environment in terms of thermal stability and insulation suggests their potential as a sustainable alternative in hybrid composites for high-temperature aerospace applications.

Figure 9 illustrates the different characteristics of the laminate before and after the fire test respectively. During the test, the first layer of aluminum alloy sheet exposed to the flame began to expand and melt at the center shortly after the laminate was positioned three inches away from the

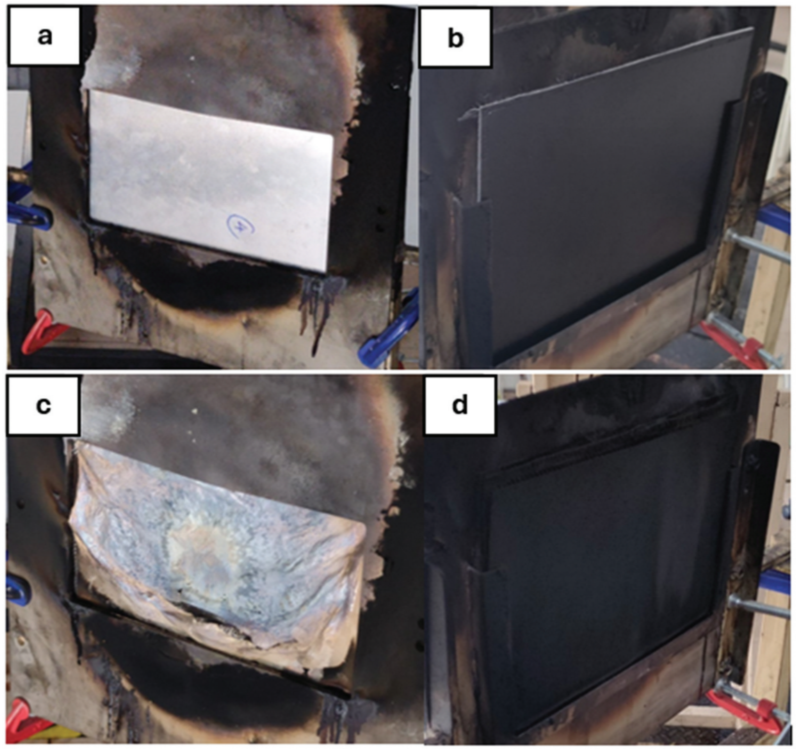


Figure 9. The hot and cool surface of laminate; (a) hot surface before fire test; (b) cool surface before fire test (c) hot surface after fire test (d) cool surface after fire test.

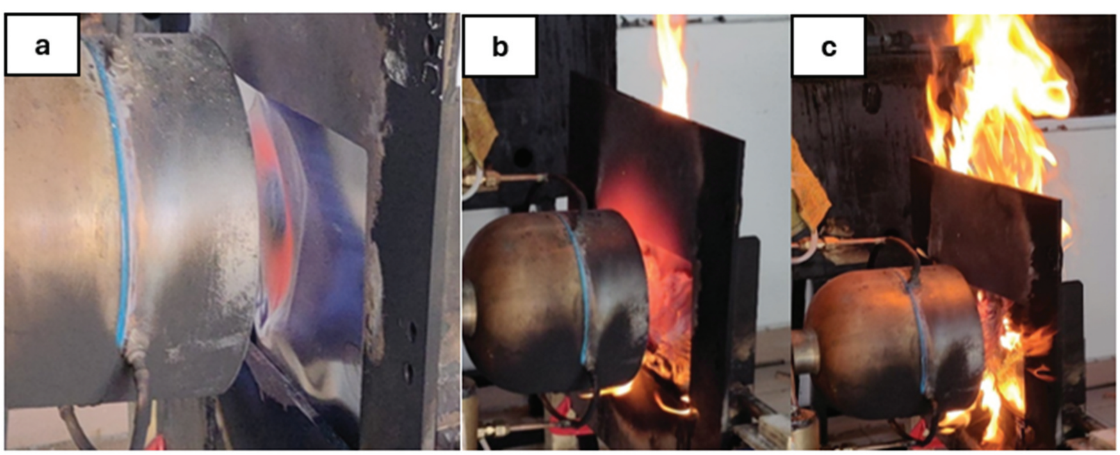


Figure 10. Flame intensity (a) at 30 seconds; (b) at 120 seconds; (c) at 240 seconds.

burner, as shown in Figure 10a. This drastic change in the aluminum alloy sheet's surface lasted for 30 seconds and remained in that state until the fire test ended. The aluminum alloy reacted with oxygen during the burning process to form aluminum oxide (Al_2O_3), which is a white, powdery substance that develops on the surface of the aluminum alloy as it burns, as shown in Figure 9c. The formation of aluminum oxide creates a protective barrier that prevents further oxidation of the underlying fiber layers, thus minimizing flame spread and reducing excess heat transfer (Mosina et al. 2020). This protective barrier offers a degree of thermal insulation, which further enhances the fire resistance capability of the laminate. A small flame began to develop on the surface of the laminates at 120 seconds, worsening by 240 seconds, as shown in Figures 10b and 10c, respectively. The flame on the laminate surface persisted for approximately 300 seconds before its intensity decreased and ultimately extinguished after 10 minutes of burning. The flames were primarily attributed to epoxy resin, a phenomenon also observed in the studies by Chulikavit et al. 2023. Although cured epoxy resin is generally more heat-resistant than uncured resin, it can still undergo pyrolysis when exposed to high temperatures, as in this experiment. Based on visual observation during the fire tests, combustion of the developed composite generated moderate amounts of dark smoke and soot, primarily from epoxy resin decomposition. The smoke release was more pronounced in resin-rich areas, while regions with higher fiber and filler content appeared to produce less visible smoke due to enhanced char formation.

The flame extinguished around 10 minutes after the burning began, primarily due to the complete consumption of the epoxy resin in the laminate, which resulted in significant delamination of the individual layers. Once delamination occurred, air containing oxygen was able to circulate between the layers, further inhibiting heat transfer. According to Wang et al. (Wang and Zhang 2019), the carbon fiber layers contracted and compressed tightly together with the burnt epoxy resin, forming a carbon layer on the fiber surface that restricted further oxygen flow to the underlying fiber of the laminate. All four laminates exhibited similar characteristics during the fire test; however, hybrid laminates that contained carbon and flax fiber layers demonstrated higher thermal conductivity (Figure 8) and back-face temperatures (Figure 7) when compared to pure carbon fiber composites. This difference can be attributed to the partial combustion of the flax fiber layers during the fire test, unlike the carbon fiber layers, which remained more intact.

Figure 11 displays the burnt surface condition of the four laminate specimens after undergoing fire testing at 1100°C for 15 minutes in accordance with ISO 2685 standards. All specimens experienced delamination of the aluminum alloy sheets on both sides, with the heated face showing clear signs of aluminum melting and oxidation, forming a whitish aluminum oxide layer. On the cool face, soot deposition was observed across all samples, indicating combustion by-products permeated through the laminate structure. In images (a) and (b), representing CARALL and CARALL + SiC, the woven carbon fiber mats were completely delaminated, with no visible resin

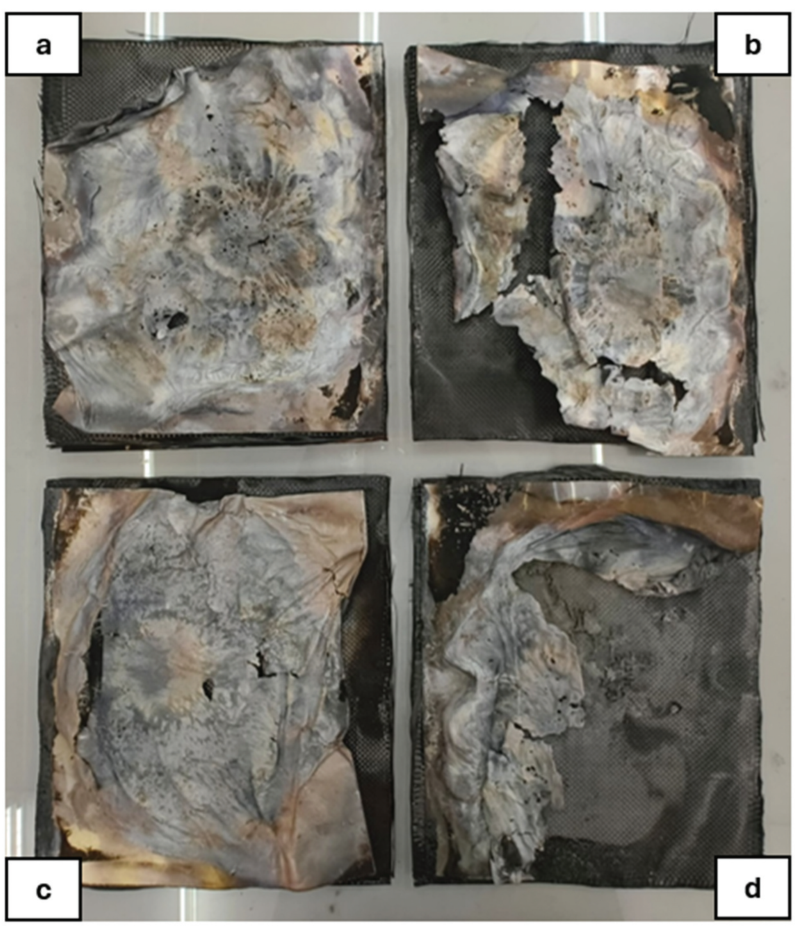


Figure 11. Burnt laminates after fire test; (a) CARALL; (b) CARALL + SiC; (c) CFFRAL; (d) CFFRAL + SiC.

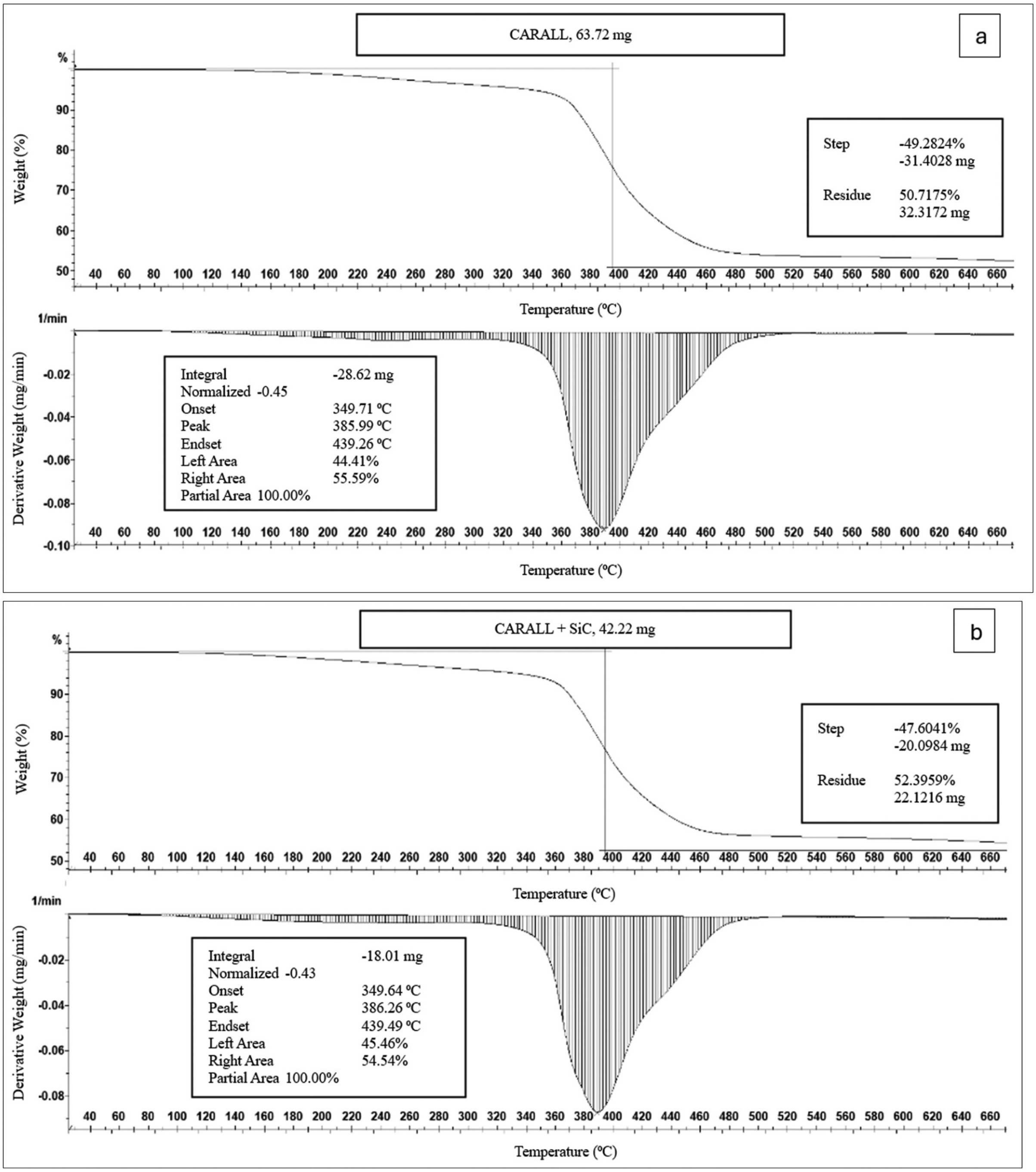


Figure 12. TGA and DTG result Curves; (a) CARALL; (b) CARALL + SiC; (c) CFFRAL; (d) CFFRAL + SiC.

remaining due to extensive thermal degradation. In contrast, images (c) and (d), corresponding to CFFRAL and CFFRAL + SiC, also showed delamination, but it was less severe compared to (a) and (b). In these hybrid laminates, the presence of flax fibers promoted char formation, which helped to hold the flax and carbon fiber layers together even after resin decomposition. Notably, in samples (b) and (d), the melting of aluminum sheets appears more critical, likely due to the increased thermal conductivity from the inclusion of SiC nanoparticles, which facilitated faster and more intense heat transfer through the laminate. This observation supports the thermal conductivity results shown in Figure 8, where SiC-reinforced samples exhibited significantly higher conductivity, ultimately accelerating the thermal degradation of the aluminum layers.

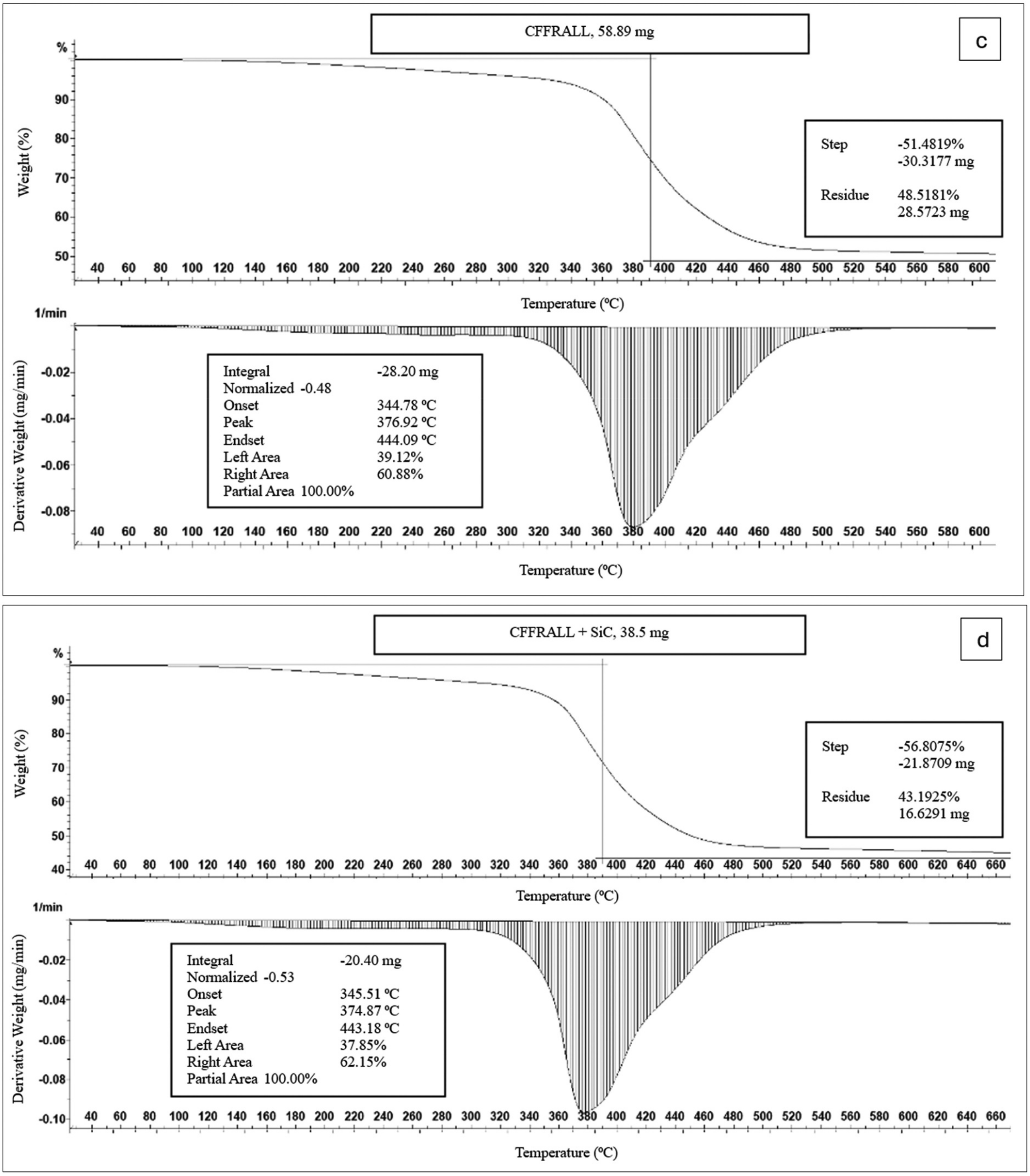


Figure 12. (Continued).

Table 8. Overall TGA results.

Laminates	Initial Weight (mg)	Residue Weight (mg)	Weight Loss (%)	Onset Temperature (^{0}C)	Peak Temperature (^{0}C)	Endset Temperature (^{0}C)
CARALL	63.72	32.32	49.28	349.71	385.99	439.26
CARALL + SiC	42.22	22.12	47.60	349.64	386.26	439.49
CFFRAL	58.89	28.57	51.48	344.78	376.92	444.09
CFFRAL + SiC	38.5	16.63	56.81	345.51	374.87	443.18

Thermogravimetric analysis (TGA) results

TGA was performed to assess the volume changes in the fiber and polymer content present in the composite. As the composites underwent melting and drying, moisture was released, leading to thermal degradation. All the variants were heated from 30°C to 800°C at a heating rate of 15°C per minute. The initial weight, residue weight, weight loss, onset temperature, peak temperature, and endset temperature for each laminate were obtained from the tests and recorded in Table 8.

In thermogravimetric analysis, T_5, T_{10}, and T_{25}% refer to the temperatures at which 5%, 10%, and 25% weight loss of the sample occur, respectively. These parameters indicate the progression of thermal degradation, where a lower T_5 or T_{10} reflects earlier volatile release and reduced initial thermal stability, while higher values demonstrate delayed degradation and improved heat resistance of the composite. According to Figure 12, the T_5, T_{10}, and T_{25}% values provide clear evidence of the effect of fiber type and SiC addition on the thermal stability of the laminates. For CARALL, T_5, T_{10}, and T_{25}% occurred at approximately 340°C, 375°C, and 395°C, respectively, while the addition of SiC lowered these points slightly by ~ 5°C, suggesting a faster onset of mass loss but without major impact on the overall stability. In contrast, CFFRAL exhibited lower T_5 (≈320 °C), T_{10} (≈370 °C), and T_{25}% (≈390 °C) values, reflecting the more thermally volatile nature of flax fibers. With SiC incorporation, these values decreased more sharply to ~ 270°C, ~340°C, and ~ 380°C, indicating an accelerated degradation process in the early stages. The larger downward shift in CFFRAL compared to CARALL can be explained by the different fiber – matrix interactions. In CFFRAL, the flax fibers are more susceptible to thermal breakdown, and the presence of SiC particles disrupts the protective char-forming ability of the fibers, leading to higher weight loss and faster volatile release. On the other hand, in CARALL, the SiC addition appears to stabilize the matrix by enhancing char density, which results in reduction in weight loss despite the small decrease in T_5, T_{10}, and T_{25}% values.

A similar study conducted by Gapsari et al. (Gapsari et al. 2022), where the addition of iron powder (IP) filler to timoho fiber-reinforced polyester composites resulted in enhanced thermal stability as evidenced by increased char residue from 9.72% in the neat composite 0 to 25.78% in the 10% filler incorporated composite. Despite no significant shift in the initial decomposition temperature, the overall residual mass increased, indicating improved resistance to thermal breakdown, similar to the TGA results in the current study, where SiC nanoparticles contributed to higher residual mass and improved thermal endurance in hybrid Carbon/Flax laminates. Another study by Andoko et al. (Andoko et al. 2023), demonstrated that hybridizing Carbon Fibres (CF) with Ceiba Petandra Fibers (CPF) in hybrid composites exhibited higher initial and final degradation temperatures, compared to pure CPF composites, indicating superior resistance to thermal decomposition. These findings affirm that both synthetic reinforcement and inorganic fillers contribute to enhanced thermal durability in hybrid composite systems by promoting char formation and delaying total decomposition, which is critical for aerospace fire-designated zones.

There is a clear correlation between temperature generation and the depolymerization of composite materials, as demonstrated by TGA results. For instance, all tested laminates began significant thermal degradation between 344°C and 350°C, marking the onset of depolymerization. Onset temperature is the temperature at which the material begins to lose weight, indicating the start of thermal degradation. A higher onset temperature indicates better resistance to decomposition at elevated temperatures. CARALL exhibited the highest onset temperature at 349.71°C indicating superior thermal stability followed closely by CARALL + SiC at 349.64°C. In contrast, the hybrid CFFRAL laminate began degrading slightly earlier at 344.78°C, suggesting flax fibers initiate depolymerization at a lower temperature. Compared to CARALL, which had a weight loss of 49.28%, the CFFRAL + SiC laminate experienced a significantly greater loss of 56.81%, indicating increased susceptibility to thermal decomposition. These results indicate that the inclusion of flax fibers and SiC nanoparticles affects the depolymerization behavior by influencing the thermal stability of the matrix. The depolymerization process, driven by increased thermal energy, is thus directly observable through the earlier onset and greater mass loss in TGA profiles, establishing a quantitative correlation between temperature and material degradation.

Furthermore, hybrid laminates containing flax fibers (CFFRAL and CFFRAL + SiC) exhibited slightly higher endset temperatures (~444 °C) compared to the carbon fiber-dominant laminates (CARALL and CARALL + SiC), which ended thermal degradation at ~ 439°C. Endset temperature is the temperature where major weight loss concludes, representing the end of the active decomposition phase. After this point, the material transitions into a more thermally stable residue or char, depending on its composition. Despite having slightly higher onset temperatures, the hybrid flax-carbon laminates sustained thermal degradation over a longer temperature range. This behavior may be attributed to the delayed decomposition of lignocellulosic components in flax and the formation of a protective char layer that prolongs mass retention at elevated temperatures. The extended degradation window could be indicative of complex thermal breakdown mechanisms involving both the flax/carbon hybrid fibers and the polymer matrix, which may enhance fire retardancy under certain conditions.

In terms of peak temperature, at which the maximum rate of weight loss occurs, is often used to identify the main degradation mechanism and is influenced by the type of polymer matrix and reinforcement. Laminates containing all carbon fiber layers achieved a higher peak temperature of approximately 386°C, compared to laminates with flax fiber layers, which registered around 375°C. This suggests that laminates with all carbon fiber layers have slightly higher maximum energy absorption compared to those with flax fiber layers. The reduction in performance due to replacing carbon fiber layers with flax fiber was also reported by Dhakal et al. (2013). The inclusion of SiC nanoparticles exhibited some degree of thermal stability improvement in CARALL laminates which adding SiC nanoparticles into CARALL laminate obtained lower weight loss than CARALL laminate alone with 47.5% weight loss compared to 49.3%. This revealed that SiC enhances the thermal stability of the laminate and ability to absorb and dissipate heat. However, the inclusion of SiC nanoparticles in the CFFRAL laminate appears to reduce its thermal stability compared to the CARALL and CARALL + SiC laminates. The higher weight loss and lower residue observed in the TGA results suggest that the SiC nanoparticles contribute to greater thermal degradation. This could be due to the interaction between SiC nanoparticles and the organic flax fibers potentially leading to increased thermal degradation. The SiC nanoparticles might not be as effective in forming a protective barrier or improving heat dissipation in the presence of flax fibers.

Dynamic mechanical analysis (DMA) results

Each of the variants were heated from 30°C to 200°C at a heating rate of 5°C per minute while undergoing dual cantilever 3-point bending to evaluate their stiffness under various temperature conditions. The storage modulus, glass transition temperature, and tan delta were recorded in Table 9, and the thermal performance of each laminate was analyzed based on these results. The storage modulus ($\boldsymbol{E}$) indicates a material's stiffness in relation to increasing temperature, tan delta (Tan δ_{max}) represents the damping factor that reflects the damping characteristics of the material, and the glass transition temperature ($\boldsymbol{T_g}$) is the point at which the material transitions from a rigid to a flexible state.

Based on Table 9 and Figure 13, the results for CARALL (Figure 13a) appear to be abnormal, as they are all extremely low compared to those of the other samples. It is suspected that the CARALL sample has a significantly different thickness compared to the other samples, leading to these inaccurate results. Therefore, the results for CARALL are not directly comparable with those of the other laminates in this DMA test. In contrast, CARALL + SiC (Figure 13b) exhibited the highest thermal stability under varying temperatures among all samples, showing storage moduli of 10.23 GPa at 30°C, 6.49 GPa at 60°C, 0.164 GPa at 90°C, and 0.162 GPa at 120°C. All samples experienced a significant drop in storage modulus between 90°C and 120°C, indicating a loss of stiffness as temperature increased.

From Figure 13, CARALL + SiC underwent a substantial loss of stiffness at 53°C, while CFFRAL showed a similar loss at 65.4°C and CFFRAL + SiC at 66.5°C. When comparing CFFRAL laminates, the inclusion of SiC nanoparticles notably enhanced the thermal stability of the laminate, particularly at lower temperatures. A similar behavior was reported by (Diharjo et al. 2024), where the addition of fillers to the matrix increased rigidity and toughness by promoting a broader interfacial area between the matrix and the fillers, whether of micro or nanoscale. At 30°C, CFFRAL with SiC exhibited a 5% higher storage modulus than CFFRAL without SiC, and at 60°C, it outperformed CFFRAL without SiC by 13%. This suggests that the SiC nanoparticles promote a better interfacial bond between the fibers and the epoxy resin polymer matrix. Between 90°C and 120°C, the differences in storage modulus become minimal as all samples approach their lower limits of stiffness. The enhancement of storage modulus in the DMA test due to the inclusion of SiC

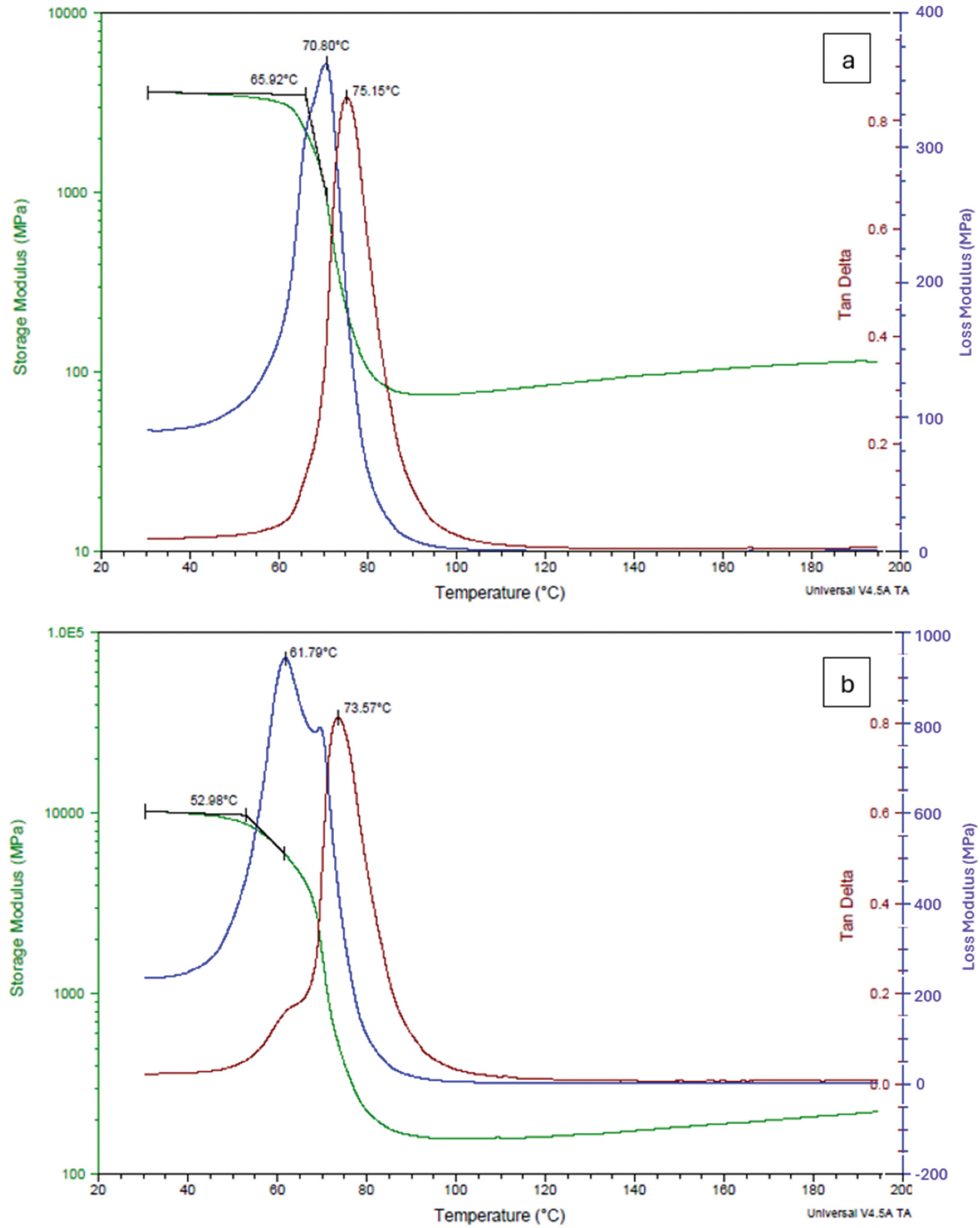
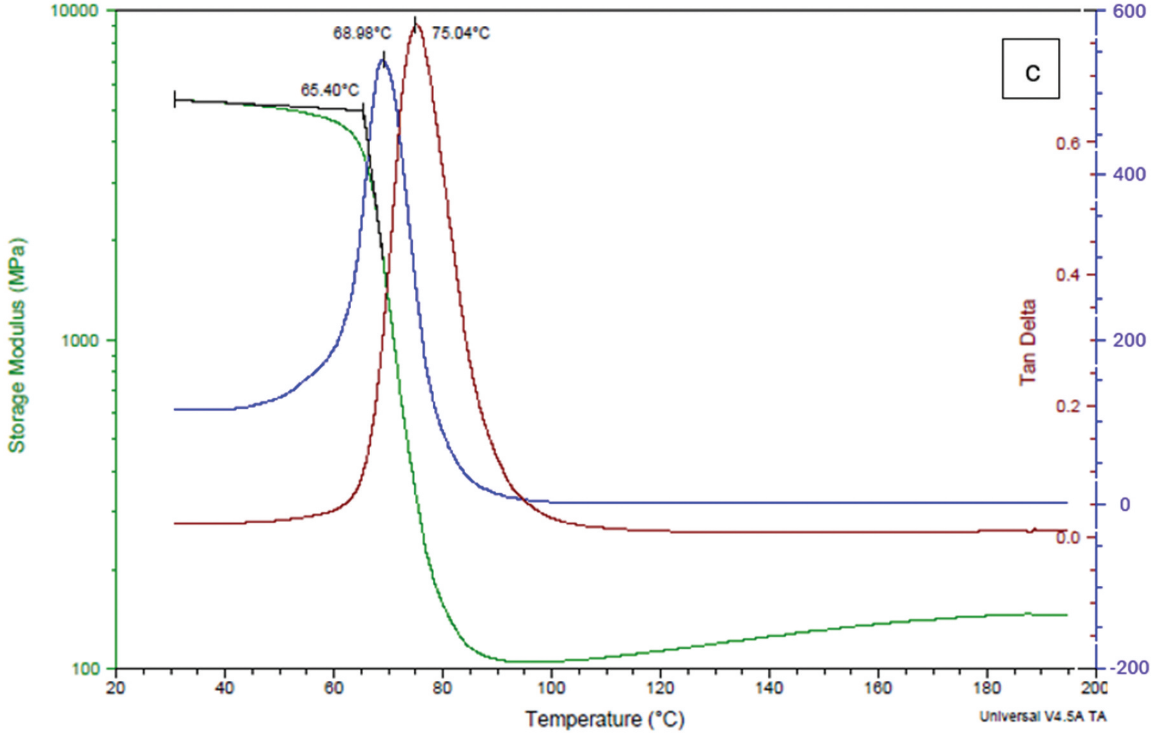


Figure 13. DMA result Curves; (a) CARALL; (b) CARALL + SiC; (c) CFFRAL; (d) CFFRAL + SiC.

particles in carbon fiber composites was also noted in the study by (Kumaresan et al. 2012). The findings are supported by (Diharjo et al. 2024) study where addition of SP fillers to CPF/polyester composites significantly enhances both loss modulus and storage modulus, indicating improved energy dissipation and stiffness due to better matrix–filler interaction. As the filler content increases, the composite exhibits higher rigidity and toughness, particularly in the glassy region, though excessive filler may lead to agglomeration and reduced performance at elevated temperatures.



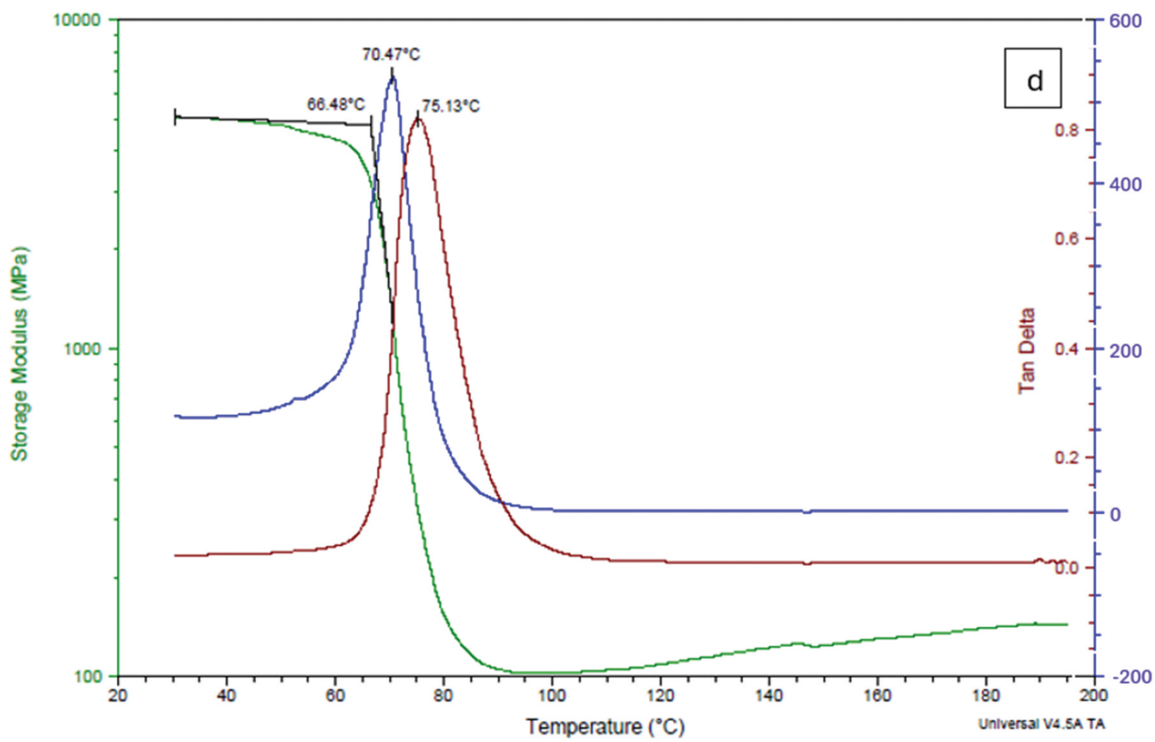


Figure 13. (Continued).

Table 9. Overall DMA test results of each laminate.

	Storage Modulus, *E*(GPa)					
Laminates	30°C	60°C	90°C	120°C	T_g(°C)	Tan δ_{max}
CARALL	3.63	3.18	0.076	0.084	75.15	0.85
CARALL + SiC	10.23	6.49	0.164	0.162	73.57	0.81
CFFRAL	5.37	4.05	0.107	0.114	75.04	0.78
CFFRAL + SiC	5.64	4.60	0.105	0.109	75.13	0.822

Table 10. Comparison between CARALL with CFFRAL and CFFRAL + SiC.

				Percentage of Difference (%)	
Criteria	CARALL	CFFRAL	CFFRAL + SiC	CFFRAL	CFFRAL + SiC
Carbon Fibre Content (Layers)	8	5	5	−37.50	−37.50
Fireproof	Yes	Yes	Yes	–	–
Thermal Conductivity (W/mK)	0.587	0.595	0.635	+1.36	+8.17
Weight Loss in TGA (%)	49.28	51.48	56.81	+2.20	+7.53
Onset Temperature (^{0}C)	349.71	344.78	345.51	−1.41	−1.20
Peak Temperature (^{0}C)	385.99	376.92	374.87	−2.35	−2.88
Glass Transition Temperature (^{0}C)	75.15	75.04	75.13	−0.15	−0.03

*(+) = increment compared to CARALL, (–) = decrement compared to CARALL.

The glass transition temperatures (Tg) recorded for all samples were relatively similar, ranging from 73°C to 75°C, indicating that all laminates transition from a glassy to a rubbery state within a comparable temperature range. This suggests that SiC reinforcement has the potential to enhance thermal performance characteristics under different operating conditions. However, the addition of SiC in CFFRAL resulted in an increase in the maximum tan delta. This indicates that the presence of SiC nanoparticles in the polymer matrix may enhance the damping characteristics of the laminates. The overall DMA results suggest that while all laminates undergo a drop in stiffness as temperature rises, the SiC-reinforced laminates exhibit a relatively slower decline in stiffness. This behavior indicates that silicon carbide reinforcement helps mitigate the loss of stiffness at higher temperatures, providing improved thermal stability to the laminates.

Table 10 summarizes the fire resistance and thermal properties of CFFRAL and CFFRAL + SiC by comparing them with CARALL. Although the proportion of flax fibers in the hybrid laminates is relatively small, their inclusion contributes to sustainability by reducing reliance on fully synthetic fibers and offering partial biodegradability. While recyclability of FMLs remains complex due to the mixed-material nature, the use of plant fibers introduces a pathway for more environmentally conscious disposal or thermal recovery. For instance, biochar can be produced from flax fiber components via pyrolysis, yielding a carbon-rich byproduct with potential environmental and agricultural applications. These marginal gains align with circular economy principles and support the broader aim of reducing the composite's lifecycle environmental impact.

Field Emission Scanning Electron Microscopy (FESEM) on burnt laminates

Due to the intense heat exposure during ISO 2685 fire testing, the CARALL and CARALL + SiC laminates underwent severe delamination, leaving behind mostly burnt carbon fiber mats with little to no residual polymer. As a result, surface morphology studies will focus on the exposed fibers to assess thermal degradation features. In contrast, CFFRAL and CFFRAL + SiC retained significant portions of the charred matrix, making them suitable for direct FESEM analysis of the polymer-char interface. This approach enables a comparative understanding of the fire degradation behavior across all laminate types, despite differences in post-burn structural integrity.

The FESEM micrographs in Figure 14 reveal significant differences in the thermal degradation and surface morphology of the burnt carbon fiber mats from CARALL and CARALL + SiC laminates after exposure to 1100°C under ISO 2685 fire conditions. The CARALL samples (Figures 14a,b) show extensive resin burnout, fiber fraying, and surface erosion, indicating poor retention of the matrix and compromised structural integrity. In contrast, CARALL + SiC samples (Figures 14c,d) display denser fiber packing, smoother surfaces, and visible ceramic residues, suggesting that the incorporation of 1 wt% SiC nanoparticles enhances thermal protection by improving char formation and limiting fiber damage. This is consistent with the TGA results, where CARALL + SiC had lower weight loss (47.6%) compared to CARALL (49.3%), indicating better thermal stability. Moreover, the improved fiber–matrix interface observed in the SiC-reinforced samples supports the DMA findings, where CARALL + SiC exhibited the highest storage modulus (10.23 GPa at 30 °C), reflecting enhanced stiffness and load-bearing capability. However, the same SiC inclusion also increased the thermal conductivity (by 6.75%), which resulted in higher back-face temperatures during fire testing, demonstrating a trade-off between mechanical performance and thermal insulation. The FESEM analysis corroborates that SiC reinforcement improves

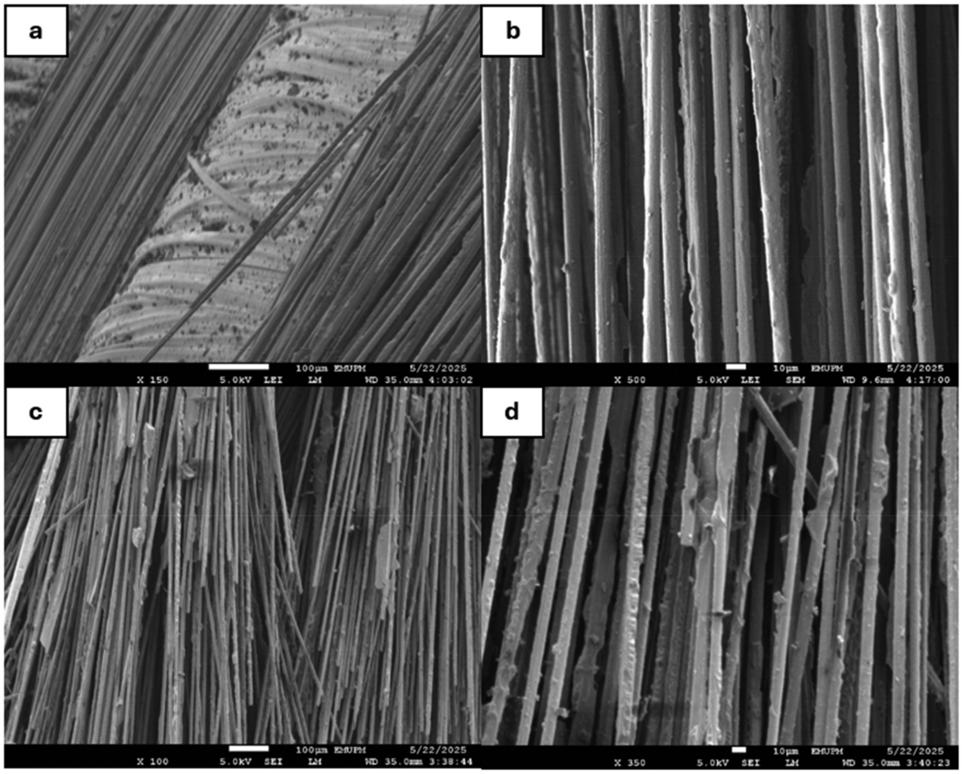


Figure 14. Surface morphology of burnt carbon fibre mats (a) CARALL (×150 magnification, 100 µm scale); (b) CARALL (×500 magnification, 10 µm scale); (c) CARALL + SiC (×100 magnification, 100 µm scale); (d) CARALL + SiC (×350 magnification, 10 µm scale).

structural integrity and thermal resistance but must be carefully balanced to avoid compromising fire-retardant performance.

The cross-sectional FESEM images in Figure 15 provide detailed morphological evidence of the thermal degradation behavior of the CFFRAL laminate after fire exposure. These images support the findings from the TGA, DMA, and fire testing by highlighting char development, microstructural failure, and polymer degradation pathways. In Figure 15a, the cross-section reveals a clear boundary between the flax char residue and the intact carbon fiber bundles. The char layer, resulting from thermally decomposed flax fibers, is crucial as it acts as a thermal barrier, inhibiting further combustion and contributing to fire retardancy. This is consistent with TGA data, which shows a residual mass of 48.5% for CFFRAL, suggesting that the lignocellulosic structure of flax aids in char formation, thereby enhancing post-combustion structural cohesion. Figure 15b (×1500) presents a granular, fragmented char morphology, indicative of the breakdown of the epoxy matrix and partial oxidation of the flax content. These particles may include carbonized fiber fragments and polymer residue. Although effective at forming char, the lack of ceramic reinforcement like SiC limits the compactness and thermal shielding efficiency of the residue.

Figure 15c reveals extensive microcracks and a foamed matrix structure, a typical outcome of volatile gas evolution during epoxy pyrolysis. These microcracks weaken the laminate's integrity and reduce its mechanical performance at elevated temperatures. The reduction in storage modulus observed in DMA (~5.37 GPa at 30°C for CFFRAL compared to 10.23 GPa for CARALL + SiC) corresponds to such microstructural deterioration, confirming that matrix degradation compromises stiffness. Lastly, Figure 15d highlights the presence of multiple voids within the polymer matrix. These voids are formed due to trapped gases escaping during thermal decomposition and incomplete resin curing post fire exposure. They contribute to increased heat transmission, reflected in the higher thermal conductivity (0.595 W/m·K) of CFFRAL compared to CARALL (0.587 W/m·K), as voids reduce the material's insulation capacity.

The cross-sectional FESEM images of the burnt CFFRAL + SiC laminate in Figure 16 reveal distinct microstructural features resulting from high-temperature exposure, highlighting the influence of silicon

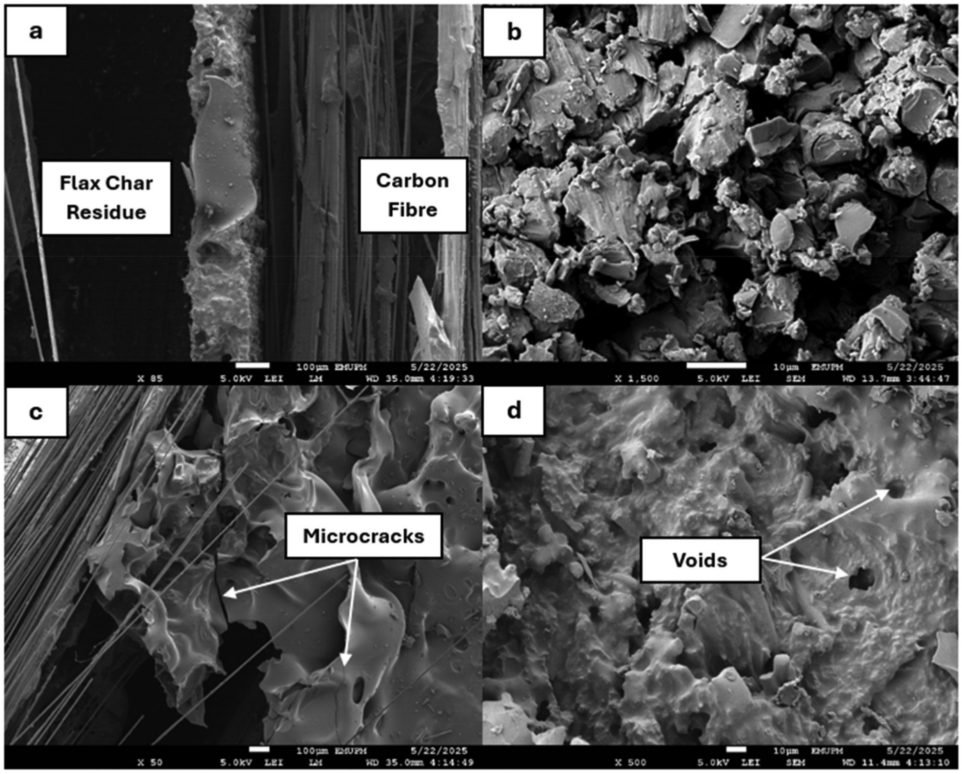


Figure 15. Cross section morphology of burnt CFFRAL specimen (a) boundary between the flax char residue and carbon fibre; (b) fragmented char morphology; (c) burnt polymer matrix structure; (d) void presence on burnt polymer matrix.

carbide (SiC) nanoparticles on thermal degradation, char formation, and residue morphology. In Figure 16a (×250), a well-defined laminated structure with stratified carbon fiber layers is visible, overlaid by a dense and compact ceramic-like char. This compact char indicates that flax fibers contributed to forming a thermally stable barrier, slowing down heat and mass transfer during combustion. This observation is consistent with the fire test results, where CFFRAL + SiC withstood flame exposure without burn-through and produced slightly higher back-face temperatures (375.4 °C) due to the enhanced thermal conductivity imparted by SiC (0.635 W/m·K). Figure 16b (×5000) shows carbon fibers partially embedded with SiC residues, confirming the persistence of SiC particles post-combustion and their interaction with the matrix. These residues likely reinforce the char structure, contributing to higher stiffness and modulus, as supported by the DMA data (5.64 GPa at 30°C for CFFRAL + SiC, higher than 5.37 GPa for CFFRAL alone).

Figure 16c (×50) exhibits a bubbled and foamed matrix, characteristic of epoxy pyrolysis, but appears less porous compared to CFFRAL without SiC (Figure 15d), suggesting better matrix cohesion due to the presence of SiC. However, the TGA results indicate a greater overall weight loss (56.8%), implying that while SiC enhances thermal conduction and structural integrity, it may accelerate polymer decomposition, particularly in the presence of plant-based fibers like flax, which are thermally less stable. Finally, Figure 16d (×1000) shows a granular, ceramic-rich surface, densely populated with char residue and SiC particulates. These remnants not only act as thermal conductors but also serve as physical barriers to volatile evolution and oxygen diffusion, thereby modifying the burn characteristics. Despite the high thermal conductivity, the composite remained fireproof, indicating an effective balance between structural degradation and flame resistance.

The FESEM analyses across CARALL, CFFRAL, and their SiC-reinforced counterparts (CARALL + SiC and CFFRAL + SiC) reveal distinct differences in post-fire degradation behavior, primarily driven by material composition and the inclusion of SiC nanoparticles. CARALL and CARALL + SiC exhibited significant resin burnout and delamination, leaving behind bare carbon fiber mats with little matrix residue. However, CARALL + SiC showed smoother fiber surfaces and visible ceramic deposits, indicating better thermal resistance and matrix retention due to SiC reinforcement. In contrast, CFFRAL and CFFRAL + SiC

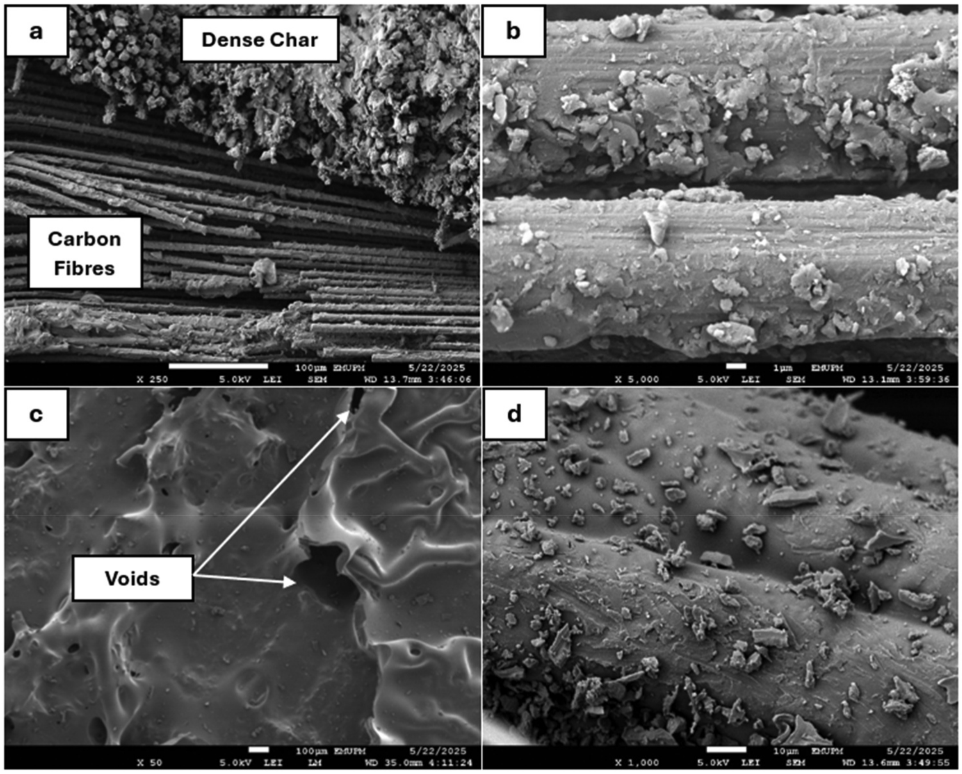


Figure 16. Cross section morphology of burnt CFFRAL + SiC specimen (a) dense char-carbon fibre interface; (b) carbon fibres surface with char residue; (c) foamed polymer matrix structure; (d) char residue on polymer matrix surface.

retained substantial portions of charred matrix post-burn, enabling detailed observation of fiber–matrix interactions. CFFRAL showed widespread voids and microcracks, a result of flax decomposition and epoxy pyrolysis, which correlated with higher thermal conductivity and lower stiffness in DMA. The addition of SiC in CFFRAL + SiC improved char compactness, reduced matrix porosity, and enhanced fiber bonding, as seen in denser residue layering and embedded SiC particulates, aligning with higher stiffness (5.64 GPa) but also greater thermal conductivity (0.635 W/m·K) and weight loss (56.8%). A key similarity among all laminates is their ability to withstand the ISO 2685 fire test without burn-through, confirming their fireproof classification. Overall, while SiC consistently improves structural integrity and stiffness, it introduces a trade-off by increasing heat transfer, particularly in hybrids containing thermally sensitive flax fibers. The formation of biochar from flax fiber decomposition during combustion contributes to sustainability by offering a carbon-rich residue that can be repurposed for environmental applications such as soil enhancement or carbon sequestration (Andrew et al. 2024; Mulla et al. 2025). This aligns with circular economy principles by enabling partial recovery and reuse of plant-based composite components post-service life.

Conclusion

In conclusion, the study demonstrated that all tested laminates are fireproof, successfully enduring extreme conditions of 1100°C for 15 minutes as per ISO 2685 standards. Among these, hybrid laminates with flax fibers exhibited slightly reduced fire-retardant properties compared to all-carbon fiber laminates; however, the differences were minimal, highlighting flax fibers as a viable, sustainable alternative for high-temperature applications. The inclusion of SiC nanoparticles showed mixed effects. In CARALL laminates, SiC improved thermal stability by reducing weight loss and enhancing heat absorption. However, in CFFRAL laminates, SiC increased thermal conductivity and weight loss, suggesting a less effective interaction with flax fibers under thermal stress. Thermal analysis (TGA) indicated that CFFRAL laminates degraded slightly faster and lost approximately 5% more weight than CARALL. In mechanical performance

(DMA), laminates with SiC nanoparticles exhibited improved stiffness across temperatures, with CARALL + SiC achieving the highest storage modulus. Despite this, all laminates showed similar glass transition temperatures (~75°C) and damping characteristics, ensuring consistent thermal behavior. Overall, the findings validate the potential of flax fibers to partially replace carbon fibers in hybrid laminates for aerospace applications, balancing sustainability and performance. However, careful optimization of SiC nanoparticle content is crucial to mitigate its adverse impact on fire-retardant properties while maximizing mechanical and thermal benefits. Future research should focus on alternative fillers and coatings to further enhance the multifunctionality of these hybrid composites.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

Author contributions:

Conceptualization, TSB, SS, NM, and YTS; methodology, TSB, and YTS; formal analysis, TSB, and YTS; investigation, TSB, and YTS; data curation, TSB, and YTS; writing – original draft preparation, TSB; writing – review and editing, SS, NM and SYN; visualization, TSB, and YTS; supervision, SS, NM, and SYN; project administration, SS, SYN, and NM. All authors have read and agreed to the published version of the manuscript. All authors have accepted responsibility for the entire content of this manuscript and approved its submission. In contrast, the authors assisted in important tasks, such as manuscript preparation, clerical assistance, and technical assistance.

Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article.

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