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Effect of Ply Orientation on Thermal Erosion and Conductivity of Silica Phenolic Composites Under Oxy-Acetylene Flame

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Abstract

The thermal and erosion performance of fiber-reinforced composites is highly sensitive to fiber architecture, matrix chemistry, and processing techniques, factors that have shaped development of advanced thermal protection systems (TPS) over past two decades. Silica phenolic (SP) composites, in particular, are widely employed in aerospace and defense applications due to their flame resistance, char stability, and ability to withstand severe thermal shock. However, optimizing thermal conductivity while improving erosion resistance remains a key challenge in designing durable ablative materials. In this study, SP composites with varying ply orientations were fabricated using hand-lay-up technique to investigate the role of fiber-lay-up configuration on thermo-mechanical behavior. Four rosette lay-ups SP0, SP15, SP20, and SP25 were systematically evaluated through erosion and thermal conductivity testing. Erosion experiments revealed material loss rates of 0.0300, 0.0117, 0.0100, and 0.0067 mm s^{-1} *for SP0, SP15, SP20, and SP25, respectively, clearly demonstrating beneficial effect of angled ply orientation. Among all tested specimens, SP25 lay-up exhibited lowest erosion rate, indicating enhanced resistance to particle impact and surface degradation. Thermal conductivity analysis across a temperature range from ambient to 550°C further confirmed that SP25 configuration achieved superior thermal stability compared to other orientations. The combined results highlight that angled ply orientations, particularly at 25°, significantly improve both erosion resistance and thermal conductivity control, thereby reducing delamination tendencies and enhancing overall durability under extreme heat flux conditions. This study establishes that carefully optimized lay-up architectures can simultaneously meet conflicting demands of thermal insulation and structural integrity in ablative systems. Improvements in erosion resistance and thermal stability make SP25 configuration a promising.*

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Keywords: Ablative composites, erosion, rosette lay-up, silica phenolic prepreg, thermal conductivity

INTRODUCTION

Past two decades influence of fiber architecture, resin chemistry, and processing techniques on thermal properties of these composites are being examined for thermal conductivity and heat-flux resistance depend strongly on factors, such as fiber orientation, lay-up sequence, and interfacial bonding between fiber matrix. Demonstrated careful control of fiber orientation can substantially reduce effective thermal conductivity while mechanical performance required for thermal protection systems [1]. Industrial adoption of advanced composites is driven by their ability to deliver significant weight savings and to integrate multifunctional capabilities, such as corrosion resistance, thermal stability and fatigue endurance. Emphasized strategic design of fiber architectures and matrix chemistries, coupled with optimized manufacturing processes, enables composites to meet stringent requirements in environments that demand both structural efficiency and long-term durability [2]. Recent advancements in processing and fiber architecture have significantly improved performance of these materials. The innovations in resin formulations and surface treatments enhance fiber matrix interface, leading to improved thermal shock resistance and reduced susceptibility to delamination. Their review also underscored importance of optimizing lay-up configurations to achieve a balance between erosion resistance and thermal conductivity for next-generation defense applications [3]. The stability of silica fabrics at elevated temperatures depends strongly on microstructural characteristics, such as fiber morphology, surface chemistry, and degree of silica network polymerization. Their investigation highlighted how controlled processing and surface modification can limit microcracking by maintaining low thermal conductivity even beyond typical service temperatures. These insights underline importance of precise material selection and processing to ensure long-term performance in high-temperature environments [4]. Aerospace industry's shift toward carbon, glass, and silica-based fiber composites is motivated by need for materials that combine mechanical robustness with thermal stability and damage tolerance. Their review highlighted that optimized lay-up configurations and precision manufacturing techniques allow engineers to tailor thermal and mechanical properties to meet demanding conditions of flight and re-entry [5]. The evolution of composite technology in aerospace applications and highlighted key drivers, such as improved fuel efficiency, reduced emissions and enhanced structural integrity. Their review underscored that careful tailoring of fiber orientation, matrix chemistry and manufacturing processes allows engineers to optimize mechanical, thermal, and environmental resistance properties for specific mission profiles. They also noted that innovations in resin systems and automated lay-up techniques have accelerated adoption of advanced composites across both civil and defense aerospace platforms [6]. Advances in chemistry and processing of phenolic resins have expanded their utility in composite materials and engineered thermal barriers. They describe how modifications to resin formulations and curing strategies can tailor properties, such as thermal conductivity, moisture resistance and interfacial adhesion with various reinforcements. Such developments have strengthened the role of phenolic resins as key matrix materials in ablative composites, enabling reliable performance in aerospace thermal protection systems and other demanding environments [7]. Erosion behavior of composites is strongly influenced by factors, such as fiber orientation, matrix toughness, fiber matrix interfacial bonding and the size and velocity of impacting particles. It showed that optimizing these parameters can significantly reduce material loss rates and improve the service life of critical components. They further noted that experimental and modeling approaches are essential for correlating microstructural design with macroscopic erosion resistance [8]. The work highlighted how carefully designed thermal analysis experiments can reveal critical information about material behavior under thermal stress, inform processing parameters and guide the design of heat-resistant structures. Brown emphasized integration of multiple thermal analysis techniques provides a comprehensive understanding of a material's thermal response and its suitability for high-temperature environments [9]. Advanced fiber-reinforced phenolic composites have become indispensable in aerospace and defense applications where structures are subjected to extreme thermal and mechanical loading. Their high specific strength, low density, and excellent resistance to ablation and erosion make them ideal for thermal protection systems in re-entry vehicles, rocket motor insulation and high-temperature industrial equipment. Phenolic resins are valued for their inherent flame resistance, ability to form stable char layers, and chemical durability at elevated temperatures. Continuous developments in phenolic chemistry and processing have further improved interfacial bonding with reinforcing fibers and enhanced thermal-shock resistance, enabling long service life in demanding environments [10]. Reported that incorporating zirconia (ZrO_2) fabric reinforcement into a carbon phenolic matrix significantly enhanced these key properties. The theoretically estimated maximum back-wall temperatures (500 °C for C-Ph and 450 °C for Z-C-Ph) closely match the experimentally measured values of 516 ± 106 °C and 428 ± 70 °C, respectively. Under identical conditions, C-Ph shows a higher average erosion rate (0.020 ± 0.007 mm s^{-1}) compared with Z-C-Ph (0.016 ± 0.003 mm s^{-1}). Incorporation of ZrO_2 fabric yields an approximate 40% reduction in thermal conductivity and a 28% reduction in erosion rate relative to unmodified C-Ph composite [11]. Ablation properties of silica/phenolic composites can be significantly enhanced by incorporating ceramic fillers and optimizing fiber-matrix interface. Si_3N_4 content reached 36 phr, composite exhibited a flexural strength of 114.6 MPa at room temperature and 18.38 MPa after exposure to 1200 °C. Under a 1600 °C oxyacetylene ablation test for 30 s, corresponding linear ablation rate was 0.011 mm s^{-1}. By contrast, specimens without Si_3N_4 showed lower flexural strengths 105.4 MPa at room temperature and 10.35 MPa after 1200 °C treatment and a significantly higher linear ablation rate of 0.061 mm s^{-1} under identical conditions. These findings emphasize that microstructural tailoring through filler modification or improved interfacial bonding is an effective strategy for improving durability of phenolic-based ablators [12].

MATERIALS AND METHODS

Material

High-silica fabric and a resole-type phenolic resin were reinforcement and matrix materials, respectively. Their properties are summarized in Table 1 and fabrication of laminates / samples methodology in Figure 1.

Experimentation

To prepare plies at specific orientations, a mold-assisted lay-up method was employed. A plaster of Paris (POP) mold was fabricated to position plies at required angles (0°, 15°, 20°, and 25°). Prior to lay-up, POP mold was coated with a polymer-based release agent to ensure easy demolding of laminate after curing. Prepreg sheets were produced by impregnating silica fabric with phenolic resin and passing impregnated fabric through a heating zone to achieve a 'B-stage' (Pre-Peg laminate) semi-cure with desired properties. The manufacture of ablative nozzle liners began with pre-impregnation of silica fabric with phenolic resin, while carefully monitoring critical parameters, such as volatile content, resin content, and resin advancement index. The properties of phenolic resin at 40ºC for four days are presented in Table 2.

The prepreg was then cut to required shape using either POP mold or a metallic mandrel. Depending on component size, geometry, and process requirements, lay-up was performed by hand-lay-up or tape-winding techniques. The molded component underwent polymerization under pressure and vacuum in an autoclave or hydrocave and was subsequently machined to specified configuration. In this manufacturing process, tape lay-up or winding stage is critical, as more than 80% of final product's consolidation occurs during this step. Poor process control at this stage can result in defects, such as delamination. The quality of cured laminate was verified by ultrasonic through-transmission testing at 30 dB before further composite processing.

Table 1. Silica fabric properties

S No	Property silica fabric	Value
1	Thickness, (mm)	0.74 –0.94
2	Aerial Density (GSM), (g/m2)	578 -678
3	Specific Gravity	1.9-2.1
4	Weave Style	8 Harness
5	Breaking Strength, (Kg/Inch)	18 Minimum
6	Silica Content (% by Wt)	98 Minimum

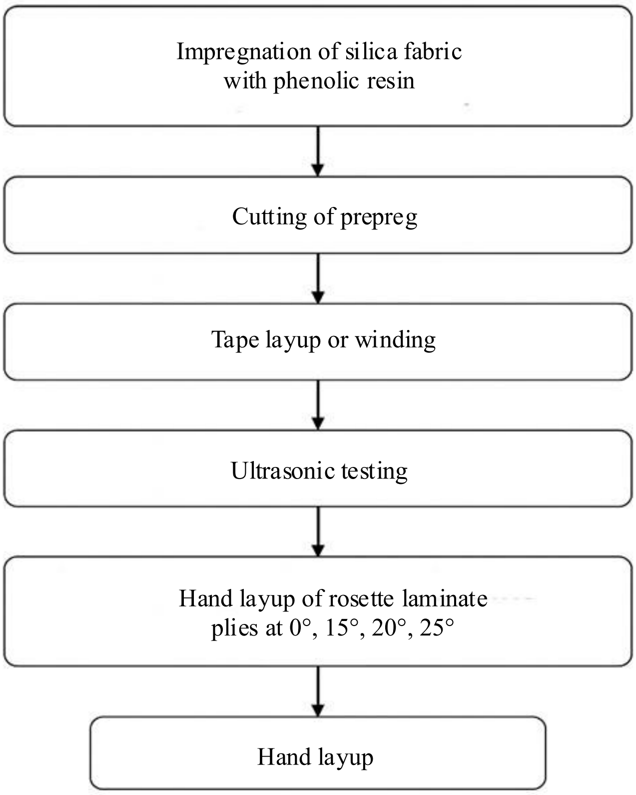

Figure 1. Methodology

Table 2. Phenolic resin properties.

S No	Property phenolic resin	Value
1	Color	Dark brown
2	Specific Gravity at 30ºC	1.14 -1.16
3	Solid Resin content	60 - 67 (%/Weight)
4	Viscosity of Resin at 30ºC	100-150 cPs
5	Volatile content	33 (%weight)

Table 3. Prepreg properties of rosette layup

S No	Parameter	Specified values
1	Resin content (wt. %)	35-40
2	The areal density of prepreg, GSM	450
3	Volatile content (wt. %)	<6
4	Thickness of prepreg (mm)	0.65
5	Resin	Phenolic
6	Chang’s index	20-28
7	The manufacturing process for laminate	Rosette hand lay-up
8	Stacking sequence	BD Fabric [0º,15º,20º,25º]

The prepreg was cut into straight tapes for hand layup of Rosette laminate plies at angles of 0º, 15º, 20º, and 25º are showed in Figure 2 corresponding to samples SP0, SP15, SP20, and SP25, respectively, to evaluate variations in thermal and erosion properties. while prepreg specifications are provided in Table 3 and laminates are fabricated as per Table 4.

Table 4. Density variation with change in orientation of sample.

Laminate orientation (angle)	Average density g/cc
0º	1.47
15º	1.56
20º	1.54
25º	1.55

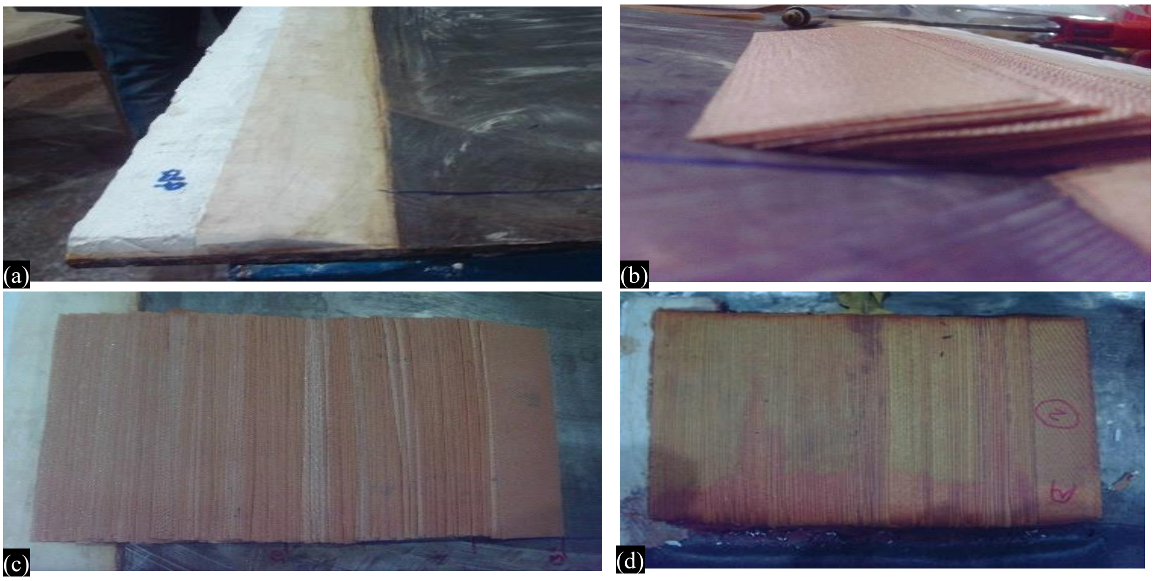

Figure 2. (a) Mold preparation by POP (b) Rosette layup (c) Green condition of laminate (d) Post autoclave cured

Table 5: Percentage of fiber and resin

S No.	Laminate angles	Fiber content % wt	Resin content % wt
1	O˚	68.88	31.12
2	15˚	68.57	31.43
3	20˚	66.54	33.44
4	25˚	65.36	34.64

CHARACTERIZATIONS

Ultrasonic Inspection

High-frequency sound waves were introduced into composite liners using transducer shown in Figure 3. A pulser/receiver generated ultrasonic pulses, and reflections from internal interfaces or defects, such as voids or delaminations, were recorded by same. This technique enabled detection of internal flaws without damaging liner.

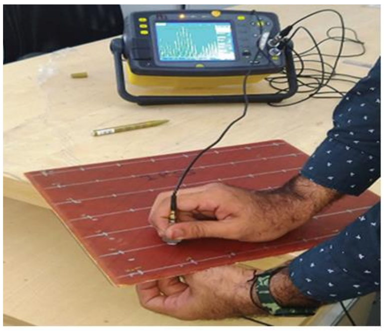

Figure 3. Ultrasonic inspection

To determine fiber and resin content in composite, laminate samples are subjected to a burnout method shown in Table 5. The samples were heated in a furnace in an oxidizing atmosphere, causing resin matrix to burn off and leaving only fibers. The fiber content percentage by weight was calculated using ratio of remaining fiber mass to initial mass. Differential Scanning Calorimetry was employed to assess degree of cure of laminate resin and to determine glass transition temperature (Tg). The sample was heated at a controlled rate while heat flow was recorded against a reference. Exothermic and endothermic peaks indicated curing reactions and glass transition temperature. Thermal diffusivity (α) was measured using Laser Flash Analysis. A short laser pulse heated one face of sample, and temperature rise on opposite face was monitored using an infrared detector. Thermal conductivity (k) was calculated from relation $k = \alpha \rho C_p$, where ρ is density and Cp is specific heat capacity. The thermal erosion resistance of liners was evaluated using an oxy-acetylene torch erosion test as per ASTM E-285–08 standard. A high-temperature oxy-acetylene flame was directed onto specimen for a fixed exposure time. The mass loss or thickness reduction was measured to quantify erosion resistance. A neutral oxy-acetylene flame was adjusted to deliver a steady heat flux of approximately 550 W cm^{-2}. Each specimen, cut to dimensions of 100 × 100 × 10 mm, was mounted in a refractory fixture so that its exposed face remained 50 mm from torch nozzle. The flame was applied continuously for 60 s, ensuring uniform exposure across surface. This controlled configuration reproduced intense thermal gradients and high convective heat transfer rates typical of service conditions, such as rocket nozzle liners or high-temperature combustion chambers. Immediately after exposure period specimens were allowed to cool to ambient temperature in still air, and mass loss, surface recession depth, and microstructural changes were recorded. These metrics provided a quantitative measure of thermal erosion resistance, enabling comparison of developed composite liners with conventional reference materials.

RESULTS AND DISCUSSIONS

Specific Heat of Composites

The specific heat values of composite material are key indicator of how SPC laminates respond to heat during service. A high specific heat means material can absorb a large amount of thermal energy with only a small increase in temperature. This ability is critical for maintaining structural integrity during thermal cycling and under peak heat loads especially in aerospace thermal-protection systems, which are exposed to extreme and rapidly changing temperatures. SP composites combine high-temperature stability of silica fibers with thermal and ablative properties of phenolic resins. The specific heat of these laminates depends on properties and proportions of their constituents as well as on composite's microstructure. The interaction between silica reinforcement and phenolic resin matrix determines laminate's overall thermal capacity shown in Figure 4.

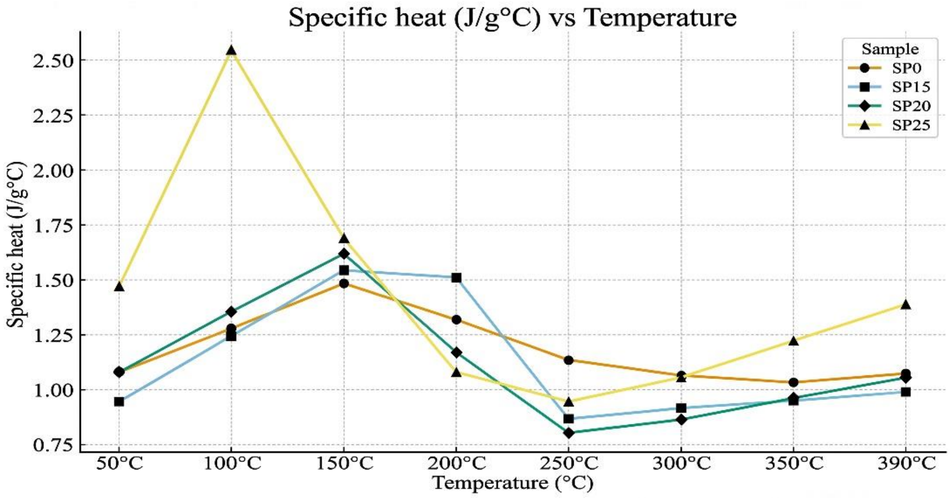

Figure 4. Specific heat Vs samples

Table 6. Micro focal X-rat system specification

Particulars	**Details**
X-ray energy	225 KV
Detector	Flat panel detector (Static 15FPS and Dynamic 30FPS)
Manipulator	5axies precision and anti-vibration
Maximum Payload	100 Kg
Soft wear for Sensualisation	VG Studio max 3.0
X –ray Source	One micro focus tube (3-4 µm) CT

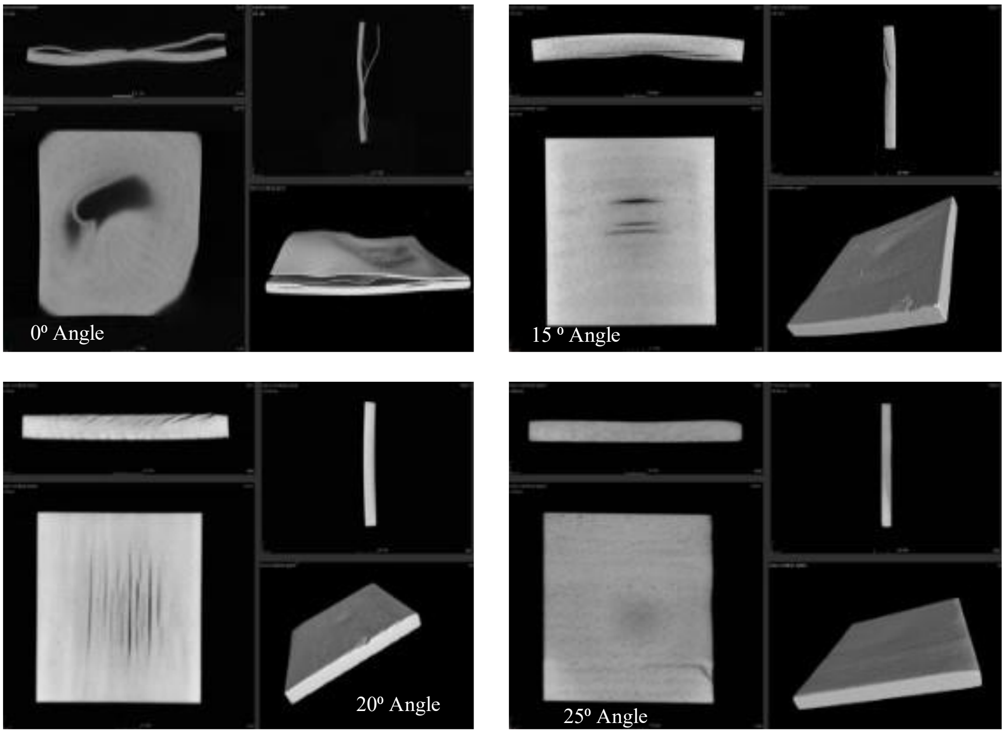

Figure5. Silica phenolic different angle laminate CT image.

Specific heat amount of heat required to raise temperature of a unit mass of material by one degree Celsius directly affects thermal performance of SPC laminates. Accurate knowledge of this property is, therefore, essential for predicting their behavior in high-temperature environments. Experimental results show specific heat of composite decreases as temperature increases.

As shown in Figure 5 and summarized in Table 6, X-ray micro-computed tomography (Micro-CT) was employed to investigate internal damage mechanisms, such as cracking, void formation, and delamination following thermal shock. This non-destructive technique enabled the three-dimensional visualization and evaluation of porosity evolution during the ablation and charring processes.

Thermal Conductivity of Composites

The thermal conductivity of SPC laminates is presented in Figure 6 variations in laminate orientation have a pronounced effect on thermal characteristics of these composites. Among tested samples, SP25 demonstrated superior performance compared to other laminates. Thermal diffusivity defined as ratio of thermal conductivity to product of specific heat capacity and density ($\alpha = k / (\rho\ c)$) indicates how quickly heat propagates through a material. In silica fabric phenolic prepreg laminates, this parameter is critical for evaluating material's ability to respond rapidly to thermal changes. A high thermal diffusivity signifies material can quickly adjust its temperature profile, which is essential in environments subject to dynamic thermal loads.

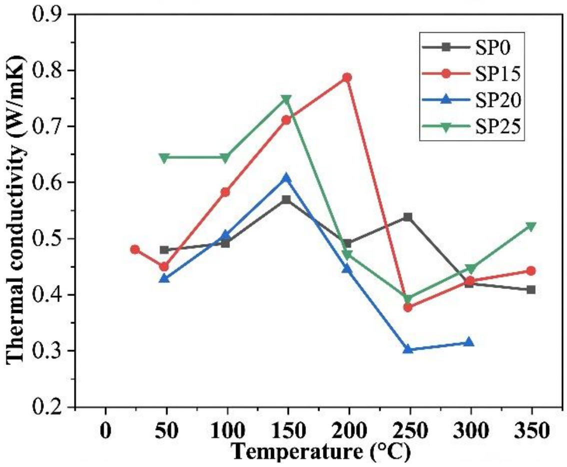

Figure 6. Thermal conductivity of composites

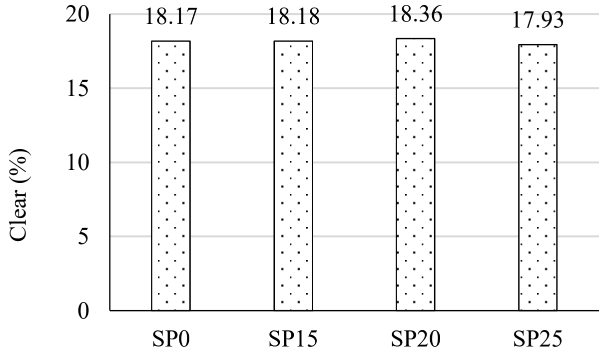

Figure 7. Material char of composite materials

Show that heat-transfer behavior of SP laminates is strongly dependent on both laminate orientation and temperature. Among all variants, the SP25 laminate maintains a relatively high conductivity across full temperature range and consistently outperforms other grades. SP15 exhibits a pronounced peak reaching nearly 0.8 W m^{-1} K^{-1} around 180 °C before dropping sharply at higher temperatures, while SP20 and SP0 display more moderate, stable trends. These results highlight that fiber architecture and resin distribution strongly influence the efficiency of heat conduction. The superior thermal performance of SP25 indicates that its microstructural configuration provides more effective heat-transfer pathways, making it particularly suitable for thermal-protection applications where rapid and stable heat dissipation is required.

Figure 7 reports material char (MC) for the four SP laminate variants. The measured char yields cluster tightly around 18%: SP0 = 18.17%, SP15 = 18.18%, SP20 = 18.36%, and SP25 = 17.93%. The mean MC is 18.16% with a population standard deviation of 0.15 percentage points and total spread across orientations is only 0.43 percentage points. This narrow dispersion indicates thermal residue formed after high-temperature exposure is essentially insensitive to laminate orientation. Orientation is consistent with mechanism of char formation in these systems, Minor differences slightly higher value for SP20 and slightly lower value for SP25 are small enough to fall within typical experimental variation and may reflect subtle changes in resin content, local cure state, or fiber–matrix interfacial area rather than a systematic orientation effect. This char provides an insulating barrier that slows heat penetration and stabilizes the underlying structure. Thus, orientation can be selected based on other performance criteria without compromising char-driven thermal stability. The thermal conductivity of the standard sample and angled laminate configurations was systematically investigated as a function of temperature. The SP25 specimen demonstrated comparatively lower thermal conductivity, indicating its potential suitability for thermal protection and ablation-related applications.

Erosion Test

The results of oxy-acetylene torch erosion test provides a controlled, high-heat environment for assessing thermal and ablative behavior of composite laminates when exposed to severe thermal stress. A constant heat flux of 550 W/cm² was applied for 60s, during which each laminate's surface was subjected to intense heating. The rate of material loss, surface recession, and any observable changes in structural integrity recorded to evaluate effect of laminate orientation on erosion resistance shown in Figure 8 and Figure 9. of erosion test data for different angled laminates

The measured erosion rates were 0.030 mm s^{-1} for SP0, 0.0117 mm s^{-1} for SP15, 0.010 mm s^{-1} for SP20, and 0.0067 mm s^{-1} for SP25. This data clearly show that laminate orientation strongly influences ability of material to dissipate heat and resist erosive attack. Among all configurations, SP25 exhibited lowest erosion rate, indicating superior thermal stability and a higher resistance to material degradation under high thermal loads.

These findings demonstrate that optimizing the orientation of SP laminates can significantly enhance their performance in demanding environments, such as aerospace thermal-protection systems. The superior erosion resistance of SP25 makes it particularly well-suited for applications that require reliable management of extreme thermal stresses.

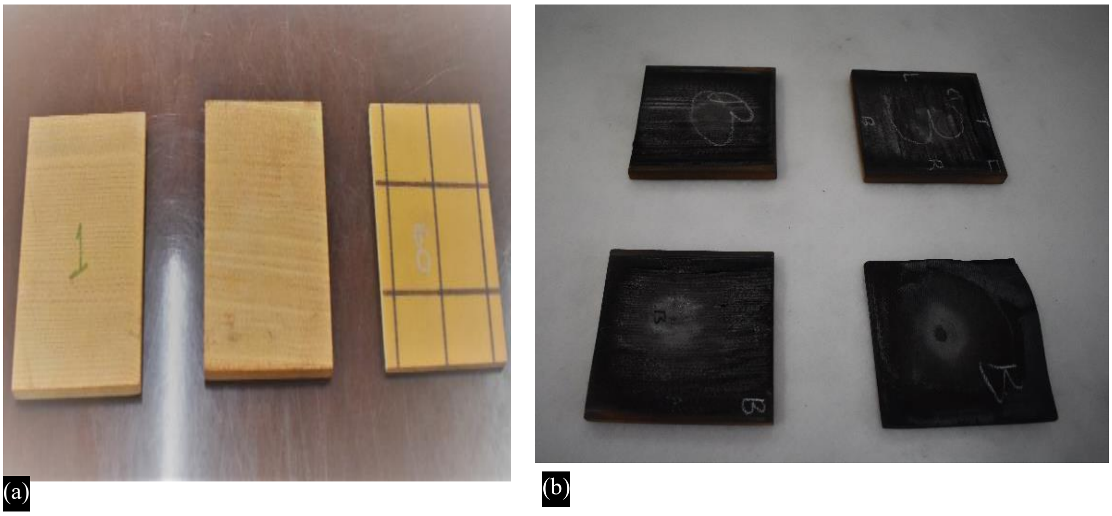

Figure 8. a) Pre Erosion test b) Post Erosion test

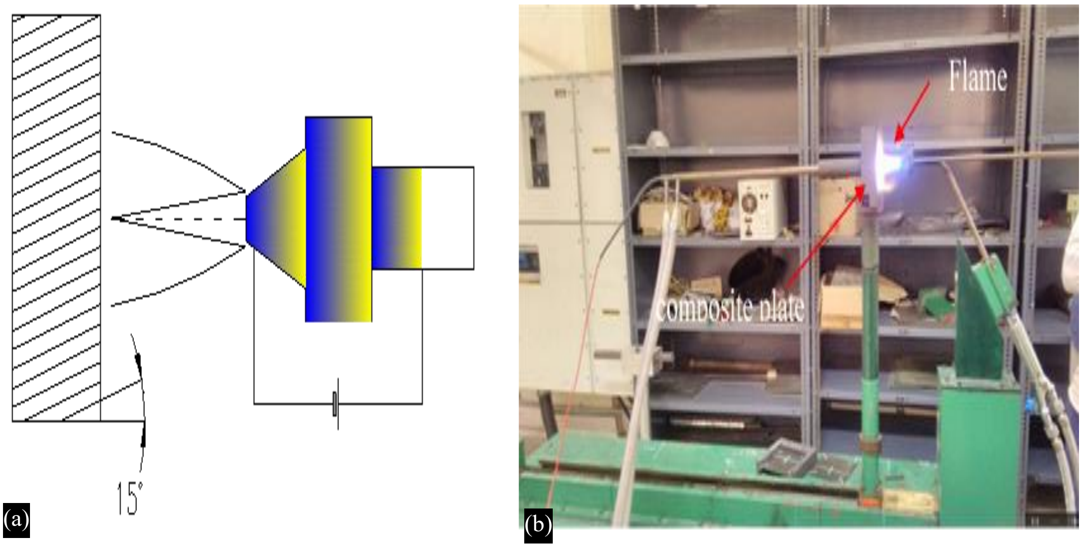

Figure 9. Erosion test - a) line diagram b) specimen with flame

Figure 10. Erosion test data for different angled laminates

From Figure 10 laminate SP0 suffered the greatest material loss with an erosion rate of 0.03 mm s^{-1} and a thickness reduction of about 1.8 mm, indicating severe matrix degradation and rapid ablation. As the fiber angle increased to 15°, 20° and 25°, both the erosion rate and the change in thickness progressively decreased, with SP25 exhibiting the lowest erosion rate of 0.0067 mm s^{-1} and only 0.4 mm thickness loss. This behavior is attributed to fact that fibers oriented away from direct flame path create a more tortuous heat conduction path and reduce rate of heat penetration into the laminate, thereby protecting resin fiber interface from intense pyrolysis. Consequently, laminates with higher off-axis fiber angles provide superior thermal protection and maintain their structural integrity better than unidirectional laminates, making them more suitable for aerospace and other high-temperature applications where resistance to thermal erosion is critical.

CONCLUSION

This investigation confirms that ply orientation plays a decisive role in enhancing both thermal and ablative performance of SPC. The experimental analysis shows that laminates with higher off-axis fiber angles, particularly the 25° rosette lay-up SP25, consistently exhibit lower erosion rates and superior thermal conductivity when subjected to severe oxy-acetylene flame exposure.

The SP25 laminate achieved lowest erosion rate of 0.0067 mm s^{-1} and maintained stable thermal characteristics over tested temperature range, outperforming other configurations. These improvements are attributed to tortuous heat conduction path and enhanced fiber matrix interaction created by angled plies, which reduce heat penetration and delay matrix pyrolysis.

The results clearly demonstrate that optimizing fiber orientation provides an effective, low-cost strategy for improving the durability and thermal resistance of SP composites, making them highly suitable for demanding aerospace thermal-protection applications, such as rocket nozzle liners and re-entry vehicle structures.

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