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Experimental Studies of Resin Systems for Ablative Thermal Protection System

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ABSTRACT

The present work was initiated to finalise resin for the development of thermal protection system (TPS) for the external surface of a polymeric composite rocket motor case made up of Carbon roving and Epoxy resin. The temperature on the outer surface of the composite case increases due to kinetic heating caused by aerodynamic drag and vehicle velocity. These rocket motor casings are functionally required only in the ascent phase of missile trajectory till motor action time and stage separation. Due to which the experienced heat flux is relatively less, and the temperature on the external composite case is in order of 250 °C - 300 °C depending on missile configuration and trajectory, unlike extreme thermal conditions on ablative nozzle liners exposed to rocket motor exhaust. The maximum allowable temperature in the present study for the Carbon-Epoxy case is 100 °C due to degradation in mechanical properties. The thermal protection system on the external surface will function as a heat-insulating layer based on the working mechanism of ablation. The resin of the thermal protection layer has a substantial impact on the manufacturing process and curing aspects, especially compatibility with the pre-cured carbon epoxy case layer. The generation of test results for thermal stability, cure characteristics and Tg for Epoxy resin has also been included in present studies as an additional objective that provides significant inputs for process development. The test results for Epoxy resin is also used as a basis for the finalisation of resin for the thermal protection layer for processing aspects apart from its basic thermal stability characteristics. The ablative thermal protection working mechanism is based on the ablation phenomenon. In the case of ablation, resin plays a vital role due to pyrolysis and other thermal characteristics. In the present experimental studies, the Phenolic resin and Silicone resin are considered as candidate resin materials for ablative thermal protection system based on available literature and in house experience. The main objective of the present studies is to evaluate thermal stability, char yield after final decomposition through DSC and TGA techniques for both resins as these are fundamental characteristics needed for the present specific application. The test results for specific grades (formulation) of phenolic and Silicone resins are generated and compared. In the present work, the experimental studies to evaluate glass transition temperature (Tg), thermal stability, and cure characteristics for Epoxy resin is also carried through DSC. The test results of specific grade Epoxy resin provides a basis to assess thermal margins for resins selected for ablative thermal protection system and inputs for process development and design requirements. The scope of the present studies is aimed to finalise the resin system for external thermal protection of composite rocket motor case based on thermal characteristics test results and other compatibility aspects with the structural layer.

Keywords: Polymeric resin; Composite rocket motor case; Ablative thermal protection system; Ablation; Aerodynamic drag; Carbon fiber; Phenolic resin; Silicone resin; Epoxy resin; Thermal gravimetric analysis; Differential scanning calorimetry

1. INTRODUCTION

Thermal protection systems are essential requisites for various aerospace applications like space crafts, launch vehicles, and missiles to protect the primary structure or subsystems from severe kinetic heating due to aerodynamic drags and high velocities[1]. Apart from kinetic heating, thermal protection is also needed for the interaction of flames/plumes from engines & motor exhaust with surrounding surfaces and subsystems. In the preliminary stages of design iterations and configuration studies of launch vehicles & missiles, the need for various thermal protection systems are also conceived. Design and development considerations and inputs are derived for these thermal protection systems based on the overall configuration of launch vehicles/ missiles and their mission objectives. There are various types of thermal protection schemes that are conceived and configured depending on mission objectives, trajectory, and application.

Launch vehicles and long-range missile systems configurations consist of various propulsion stages depending on mission objectives. These stages get separated one by one after the propellant of respective rocket motors gets consumed as per trajectory design, and motor case is spent. In the present scenario, the rocket motor casings are designed and made up of PAN based carbon roving, and epoxy resin for achieving a high-performance factor with minimum insert mass. However, when launch vehicles/missiles accelerate through atmosphere, the external surface temperatures on composite rocket motor case increases due to high velocities and aerodynamic drag.

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In such a case, thermal protection is essential as the rocket motor case is made from polymeric composite material. The ablative thermal protection system is a vulnerable option for such application as it is flexible and good feasibility of process development considering compatible with CE structural layer. The lightweight, integrity with motor case CE structural layer, manufacturing process parameters including curing aspects are prime fundamental requirements for the development of this thermal protection layer apart from thermal design inputs. The operational requirements of these rocket motor casings are only there in the ascent powered phase of missile trajectory till motor burn time, followed by its separation. Due to such specific application, the generated heat flux on external composite case is relatively less, and the temperature is in order of 250 °C - 300 °C depending on missile configuration & trajectory, unlike extreme thermal conditions on ablative nozzle liners exposed to rocket motor exhaust and re-entry conditions. In the present study, the maximum allowable temperature for the Carbon-Epoxy case is 100 °C due to degradation in mechanical properties. The thermal protection system on the external surface will function as a heat-insulating layer based on the working mechanism of ablation to limit the interface temperature within allowable limits. Ablative thermal protection functions are based on the ablation phenomenon[2-3] in which physicochemical transformations take the place of solid substances by heat transfer mode of convection or radiation heat flow[5]. In other words, energy is managed through material depletion and pyrolysis.

The working mechanism of Ablative thermal protection system[4-5] is briefly described as follows:

- Hot gases in the boundary layer heat the surface through convection.
- Surface get heated due to radiation heat from the boundary layer.
- Heat absorbed by surface is dissipated through conduction inside and radiation outside.
- Decomposition of matrix in the composite leads to gases formation, carbon residue layer of porous char starts forming on the exposed side.
- More decomposition of polymers occurs due to thermal front recession through the material
- The gases formed due to pyrolysis inside composite material are at a lower temperature than gases near the char surface. When these gases flow through char take away heat, and the temperature gets reduced.
- The surface at which char formation takes place reacts through the shock layer, and char is depleted, causing chemical erosion.
- Convection heat transfer to the surface is reduced due to the formation and movement of pyrolysis gases into the boundary layer as these gases make a denser medium in the vicinity.

The present work is carried out to finalise the resin for the ablative external thermal protection layer of composite rocket motor case based on the following considerations:

- Thermal Characteristics (Thermal stability, Char yield, and decomposition temperatures)
- Manufacturing process requirements (In-situ layup over CE structural layer, Curing characteristics and Tg of Epoxy resin).

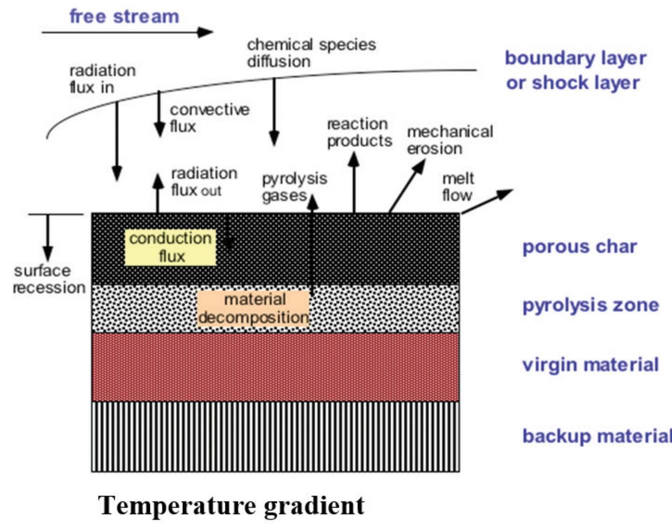

Figure 1. Ablative mechanism.

The scope of present work includes studies of thermal stability, char yield & decomposition temperatures of Phenolic and Silicone resins by TGA and DSC techniques. The thermal conductivities for Glass phenolic and Glass silicone composite are also tested considering present application. The results of thermal characteristics of Phenolic and Silicone resins are compared. The Cure characteristics and Glass transition temperature of Epoxy resin of structural layer are also evaluated through DSC to assess process aspects compatibility with TPS resin especially curing.

2. EXPERIMENTAL STUDIES AND METHODOLOGY

The selected materials & their properties, details of sample preparation and various experimental studies are briefly described in this section.

2.1 Materials for Ablative TPS

Ablative materials for TPS design are typically polymeric composites. The constituents will be physically distinct at the microscopic level and are non-homogeneous and anisotropic. The main two individual elements for polymeric composites are reinforcement and matrix[6-8].

- Main reinforcement are carbon and glass roving and fabric materials
- Matrix are mainly polymeric resins (Thermoplastic or Thermosetting) e.g. phenolic and silicone resins which undergoes pyrolysis when exposed to heat and weight loss takes place with the formation of char (porous carbon) behind.

The scope of present studies are limited to ablative TPS resin only considering specific application. In the present studies, Phenolic Resin and Silicone Resin are chosen for experimental evaluation as both are very good candidate materials for ablative TPS. In the case of Ablative TPS, resin characteristics[8] are very significant for ablation to meet functional requirements. The details and properties of Phenolic and Silicone resins are given as follows:

2.1.1 Phenolic Resin

Phenolic resins consist of a chain of polymers and these are synthesised by chemical reaction and polymerisation of phenolic monomers with aldehyde chemicals[9]. Phenolic resin is condensation polymerised in which formaldehyde is substituted on the phenol's aromatic ring. The various reaction conditions are chosen to obtain optimum dispersion of molecular weight and required fraction of residual monomer based on application[10]. In the present study, Resol Phenolic Resin is considered. The Specifications for Resole Phenolic resin (Make: IVP Ltd, Tarapur, India, Grade: Resole IVA REZ ISRO) which is considered in the present work are given as follows in Table 1.

Table 1. Specification of phenolic resin

Parameter/Property	Specification
Viscosity of resin at 30 °C	100 -150
Specific gravity t 30 °C	1.12 -1.16
Point of Trouble , ml (resin is diluted with AR grade of Ethyl alcohol to get specific gravity of diluted resin is 0.860)	8 -12 ml of H2O (per 10 ml diluted resin)
Solid resin content (% Weight)	60- 65
Volatile content (% Weight)	32 -38
Free phenol content (Max %) (Bromination method)	6
Free rormaldehyde (Max %) (Hydroxyl amine HCl method)	3

2.1.2 Silicone Resin

Silicone resins are synthesised of silicon-oxygen lattice having some fraction of the SiO4/2 or R-SiO3/2 elements, where R signifies any alkyl or aryl groups generally methyl or phenyl [11]. Silicone resins possess greater resistance to thermal degradation in comparison to organic resins which have their carbon-carbon as prime element[12]. The high thermal stability against thermal exposure of these resins are attributed to excellent bond strength between silicon and oxygen.

Further condensation occurs to form three-dimensional siloxane lattices as shown in Fig. 2.The silicone resins are highly versatile and exhibits excellent compatibility with many other polymers. These features facilitates for synthesis of silicone resins having flexible properties namely curing characteristics, mechanical & thermal properties and adhesion properties to suit specific applications. The most vital characteristic of silicone resins is its excellent thermal stability. These resins possess excellent thermal stability at 200 to 250^0C during continuous heat flux exposure and even up to 600^0C for shorter period of heat exposures. Their excellent oxidation resistance and superb mechanical properties make them ideally suited for critical application[13-14].

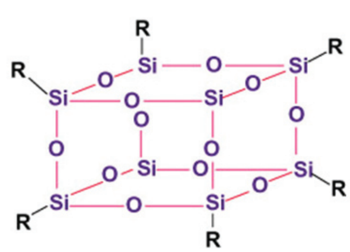

Figure 2. Silicone structure.

In the present work Silicone solid flake resin is chosen which contains 100 percent silicone and silanol functional resin. This resin for application is made as a solution with a 60% Toluene solution. Technical Specifications of Silicone flake Resin (Make: Dow Corning Corporation, USA, Grade RSN 0249) is given as follows in Table 2.

Table 2. Specifications of silicone resin

Parameter/ property	Specification
Silicone solid flake resin	
Non volatile content (% by weight)	98 (Minimum)
Specific gravity at 25 °C	1.33-1.43
Hydroxyl content , (mg KOH/g)	5.5-6.5
Melt Viscosity at 150 °C, cP	212-258
Balance Shelf life on receipt at ASL	24 months
As 60 % toluene solution	
Solid resin content (% by weight)	58-62
Volatile content (% by weight)	38-42
Specific gravity at 30 °C	1.05-1.15
Solution viscosity at 30 °C, cP	30-110

2.2 Techniques/ Test Methods for Resins Studies

The following two techniques are used for resins characterisation for functional requirements.

2.2.1 Thermal Gravimetric Analysis

Thermal gravimetric analysis (TGA) is a test method which involves recording of material specimen mass with respect to temperature or time under condition of specified temperature program with controlled ambient conditions. The TGA method works on the principle that whenever any material is heated its weight increases or decreases. The instrument used was make TA Instruments with Model Q-500.

2.2.2 Differential Scanning Calorimetry

A Differential scanning calorimetry (DSC), is a test method based on thermal analysis which monitors and records the change in heat capacity of a material by varying temperature. In this technique, a specimen of measured mass is subjected to heating or cooling and the change in heat capacity is recorded and correlated as variation in the heat flow. The change in heat flow gives a basis for identifying critical materials transformations like melting, glass transitions, phase changes, and curing/polymerisation. The instrument used was make TA instruments with model no Q200.

2.3 Samples Preparation

Samples for neat Phenolic resin to carry out TGA and DSC were prepared by taking two gram of resin into a plate and same was heated to 70 °C for duration of one hour to allow volatiles to get evaporated. The curing of resin was carried out in oven with following curing cycle: Increasing temperature to 90 °C with dwell period of one hour, increasing temperature to 120 °C and keep dwell period for two hours, increase temperature to 150 °C with dwell time of one hour and increase temperature to 170 °C with dwell time of two hour finally reduce he temperature to ambient. Similarly for Silicone resin also two gram of resin was taken into a plate and sample was heat to 90 °C for one hour to allow volatiles to get evaporated. Later resin sample curing was carried out in oven through curing cycle given as follows: Oven temperature increased to 105 °C with dwell time one hour, temperature increased to 120 °C with dwell time one hour , temperature increased to 140 °C with dwell time four hour followed by cooling to ambient. To carryout TGA and DSC runs, 10 mg cured specimens were taken for both Phenolic and Silicone Resins.

The samples for Glass Phenolic and glass Silicone composites thermal conductivity testing were prepared from their laminates. Ten glass fabric layers of 300x300 x0.35 mm size were cut for each laminate type and fabric layers were impregnated with phenolic and Silicone resins. The plies were laid as specified on metallic mould by manual lay-up process. After lay-up, the vacuum bagging were prepared and curing carried out in autoclave. The curing cycle followed for Glass Phenolic were at temperature 90 °C with soak time one hour with steps of 120 °C for soaking of two hours, increasing to 150 °C with soak time one hour and 170 °C with soak time two hour with autoclave pressure and vacuum of five bar and one torr respectively. Curing cycle followed for Glass Silicone was at 105 °C for 1h with further temperature steps of 120 °C with soaking of one hour, 140 °C with soaking of four hour with five bar pressure and one torr vacuum. Specimens of size 80mmx 80mm x 4mm were cut from respective laminates.

2.4 Experimental Evaluation of Resin Systems

The experimental works were conceived to test and evaluate the thermal stability and char yield due to pyrolysis of Phenolic and Silicone resins through TGA and DSC. The thermal conductivity for glass phenolic and glass silicone are also tested to generate data. The cure characteristics and glass transition temperature (Tg) of Epoxy resin in composite rocket motor case structural layer are also tested through DSC. This will generate basic characteristics of ablative TPS resin and Epoxy resin of the structural layer for thermal stability and curing parameters for design considerations, processing, and available thermal margins.

TGA for Phenolic Resin and Silicone were carried out. The weight of the samples are measured as a function of temperature under purging of nitrogen gas. The plots between weight as a percentage of initial weight and derivative weight percentage and temperature are obtained and shown in Figs. 3 and 4, respectively:

DSC for Phenolic and Silicone resins were carried out. Phenolic and Silicone Resin samples were taken and cured for specimens preparation to for DSC analysis and graphs between Heat flow vs temperature were obtained. The plots are given in Fig. 5.

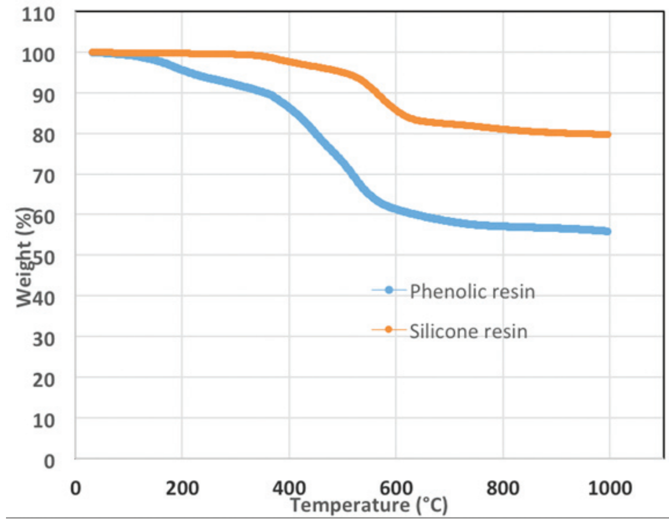

Figure 3. TGA curve for phenolic and silicone resin.

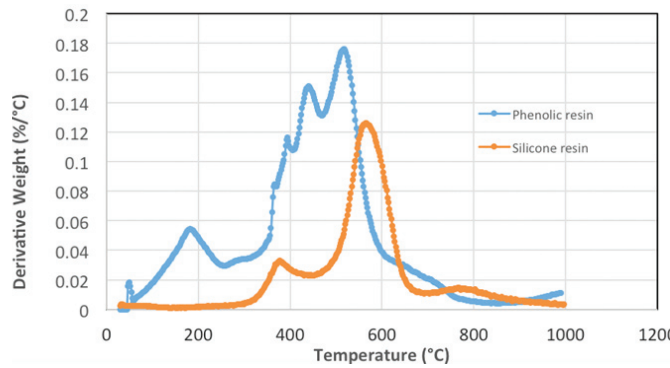

Figure 4. DTG curve for phenolic and silicone resin.

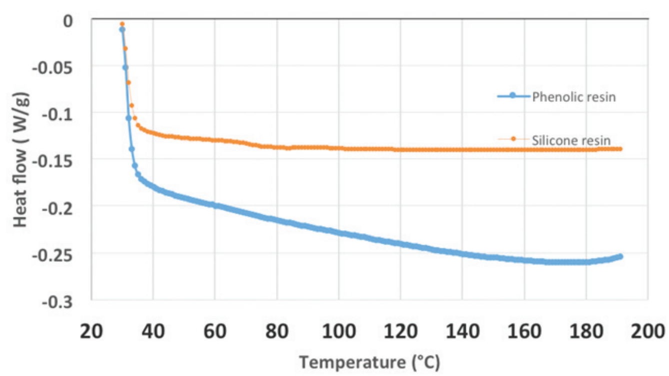

Figure 5. DSC plots for phenolic and silicone resin.

The evaluation of thermal conductivity of glass phenolic and glass silicone composite samples were carried out by test method as specified in ISO 22007-2. Two samples were taken for each composite having approximate size of 80mmx 80mm x 4mm for tests. The sensor was inserted between specimens and sensor fulfills two functional needs namely as heat source and thermometer. The sensor was subjected to electrical heating with recording of its temperature with time. The increase of sensor temperature will depend on thermal conductivity and diffusivity of the specimen material. Based on this principle, the thermal conductivity of both composites were calculated and results are as follows in Table 3.

Table 3. Thermal conductivity results

Temperature (°C)	Thermal conductivity of glass/ phenolic composite	Thermal conductivity of glass/ silicone composite
25	0.26	0.28
100	0.29	0.31
200	0.31	0.32
300	0.32	0.34

DSC was carried out for Epoxy resin to evaluate thermal stability and cure characteristics which provided basis for design and process considerations for TPS over CE structural layer of composite case. The DSC run was given to evaluate glass transition temperature (Tg) and cure characteristics and plots for same are given as follows in Figs. 6 and 7, respectively.

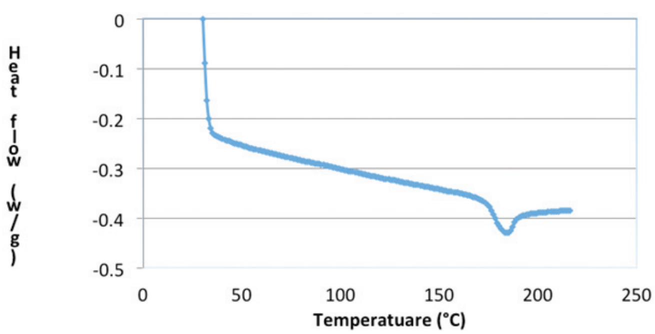

Figure 6. Epoxy glass transition temperature (Tg).

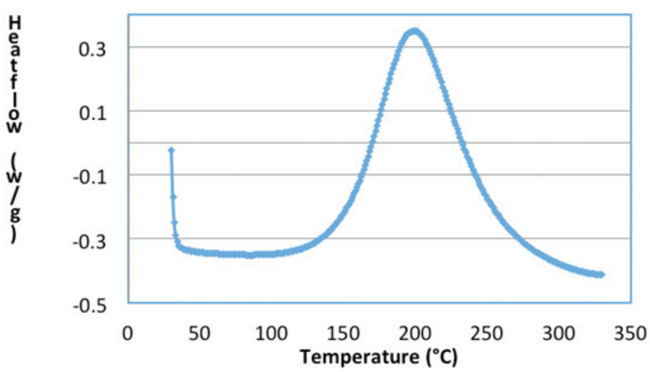

Figure 7. Epoxy cure characteristics.

3. RESULTS AND DISCUSSION

TGA for Phenolic resin and Silicone resin is carried out to evaluate thermal stability i.e resin initial decomposition temperature and char yield (residual weight after final decomposition) of resins. The thermal stability of Phenolic and Silicone resin are compared based on their initial decomposition temperature, temperature at 10% weight loss and residual weight char yield) of resin after final decomposition at 1000 °C. DTG curves indicate mass loss rate depending on an increase in temperature. DTG curve shows peak for differential weight loss for silicone resin is at higher temperature with lower magnitudes than Phenolic resin. These parameters are true basis for functional requirements of thermal protection system for composite case external surface.

- The Initial Decomposition temperature for Phenolic Resin is 190 °C and 10% weight loss is at 350 °C. The char yield i.e residual weight of resin after final decomposition is 55 %.
- The Initial Decomposition temperature for Silicon Resin is 370 °C and 10% weight loss is at 590 °C. The residual weight (char yield) of resin after final decomposition is 79.82 %.

In the DSC run, no exotherms are observed which confirms that specimens were fully cured. Thermal stability is much better for silicone resin in comparison to Phenolic resin based on above plots.

The Epoxy resin DSC test results for Glass transition temperature and cure characteristics provides basis to compare with TPS resin characteristics. These are required for thermal margins and to decide various processing aspects including curing parameters. The Initial Decomposition temperature for Epoxy Resin is 250 °C and 10% weight loss at 375 °C. The other cure characteristics results are as follows:

- Glass transition temperature (Tg) is 161.58 °C
- Cure initiation Temperature is 93 °C
- Cure Onset Temperature is 140 °C
- Cure Peak Temperature is 195 °C
- Cure Completion Temperature is 295 °C

Thermal Conductivity Test results for Glass Phenolic and Glass Silicone samples shows that both are almost same.

4. CONCLUSIONS

Resin thermal characteristics and properties play the most vital role for ablative Thermal protection systems performance. In this regard, TGA and DSC test results for Phenolic resin and Silicone resin are evaluated and compared. The TGA plots for Phenolic Resin and Silicone Resin are compared and summary of TGA characteristics for both Resins are given as follows in Table 4.

The test results of present studies on Phenolic and Silicone Resins determines that Silicone resin is better for ablative thermal protection application based on thermal stability characteristics namely initial decomposition temperature, temperature at ten percent weight loss, and residual weight (Char yield) after final decomposition. These parameters are directly related to TPS resin driven functional requirements. Thermal conductivity of Glass Phenolic and Glass Silicone composites were also measured and found similar.

The DSC studies on Epoxy resin are carried to assess the margins and generate temperature constraints as input

Table 4. TGA characteristics for resins

Resin	Initial Decomposition Temperature (°C)	Temperature at 10 % weight loss of sample (°C)	Char Yield after final decomposition (%)
Phenolic	190	350	55
Silicone	370	590	79

for the design of an ablative thermal protection system for a composite rocket motor case which is made up of Epoxy resin. Test results of DSC for Epoxy resin i.e glass transition temperature (Tg), Initial decomposition temperature, and temperature at ten percent weight loss also confirm availability of thermal stability margins for Silicone resin in ablative thermal protection system.

Experimental TGA and DSC studies for Phenolic Resin, Silicone, and Epoxy resin as a part of structural composite concludes Silicone Resin as a suitable one for ablative thermal protection layer for Carbon Epoxy structural layer. Present studies also provide design inputs, temperature constraints, and curing parameters that are useful for configuring thermal protection for CE composite rocket motor case.

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Contribution in the current study, he has given valuable suggestions and guidance for writing this paper.

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His contribution in this study is providing support for data acquisition and test equipment.