Flammability Behavior of Fiber–Fiber Hybrid Fabrics and Composites

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ABSTRACT: The effect of heat flux levels on burning behavior and heat transmission properties of hybrid fabrics and composites has been investigated using cone calorimeter and heat transmission techniques. The hybrid fabric structures woven out of E-glass (warp) and polyether ether ketone (PEEK) (weft) and E-glass (warp) and polyester (weft) have been studied at high heat flux levels keeping in view the flame retardant requirements of structural composites. The performance of the glass–PEEK fabric even at high heat flux levels of 75 kW/m^2 was comparable with the performance of glass–polyester fabric evaluated at 50 kW/m^2. The results further demonstrate that glass–PEEK hybrid fabrics exhibit low peak heat release rate, low heat release rate, low heat of combustion, suggesting an excellent combination of materials and fall under the low-risk category and are comparable with the performance of carbon fiber-epoxy-based systems. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 122: 2295–2301, 2011

Key words: cone calorimeter; flammability; heat transmission; heat flux; PEEK

INTRODUCTION

The scope of flame retardant (FR) textiles is increasingly gaining importance because of the stringent requirements put forth by various agencies. The increasing use of FR materials in aviation industry has put lot of thrust on the scientific community to develop new polymer materials, FR chemicals, and fiber combinations to cater to a wide range of end use applications. During the last two decades, there has been a resurgence of interest in the development of FR systems for railways and aviation industries. Fire hazard is associated with a combination of factors including the material ignitability, the rate at which heat is released, the total amount of heat that is released, flame spread, smoke production, toxicity of the smoke, degradation products of the polymer, the calorific value of the polymer, and ability of the material to char. It has now been established that the property which most critically defines a fire is the heat release.

The conventional method of assessing flammability behavior of FR polymers has relied on simple test methods for rating the relative ignitability or spread of flame. The vertical flammability test (VFT) (BS 3119) considered as one of the most stringent test is used to evaluate FR characteristics (after flame, after glow, and char length) of textile fabrics. Similarly limiting oxygen index (LOI) and nitrous oxide index (NOI) are basically meant for plastics and not exactly recommended for textile fiber-based materials. The above characterization techniques does not provide any meaningful scientific data which can be used to elucidate the mechanisms of action of the FR chemicals, assess hazard potential and study flammability behavior of fabrics or composites under simulated conditions especially at very high heat flux levels (temperature in the range of 500–900°C). Standard laboratory methods use different conditions for testing protective fabrics and composites. Various protocols require heat flux levels in the range of 20–84 kW/m^2 and call for different mixtures of radiant and convective energy and use many configurations to expose the sample to the heat source.[1]

Many methods are available to measure the heat release from materials involved in fire. However, methods based on cone calorimeter, which uses

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oxygen consumption principle is one of the important techniques that covers most of the aspects related to fire and can be effectively used to characterize and assess functional performance of fabrics and fiber-reinforced composites. The technique provides a meaningful data to design protective clothing and systems and lend more thoughtful interpretation and extrapolation, which is not possible in the conventional techniques. Further, the technique also takes into consideration external heat flux (550–900°C), simulating the burning sample under immense heat, which is not available in conventional FR evaluation techniques such as LOI and VFT.

Although significant contributions have been made by various researchers[2–11] toward evaluations of fire retardant polymer systems, woven fabrics, foam-fabric combinations, work on flammability evaluations of comingled or cowoven hybrid fabrics and their fiber-reinforced composites used for aerospace applications are still scanty. Schartel et al.[2] has broadly categorized the heat flux levels experienced by aviation industry (35 kW/m^2), railways (50 kW/m^2), and navy (100 kW/m^2). Heat release rate (HRR) has been widely used by researchers to assess the hazard potential of fabrics and peak heat release rate (PHRR) is considered by a number of researchers for characterizing and differentiating fire resistant materials.[12–14] Scudamore[15] in his work on thermoset (glass with epoxy, phenol, and polyester) based composites observed decrease in thermally thin to thick effect with increase in heat flux levels. Thomson and Drysdale[16] measured the surface temperature at ignition in the presence of a pilot flame of six common thermoplastics using two different instruments over an irradiance range of 10 and 40 kW/m^2. Within experimental error, they found that the surface temperature at ignition was not significantly affected by changes in the level of external radiant flux above some minimum value.

There are numerous structural components used in airborne vehicles where the use of composites and high-performance FR fabrics would provide substantial advantages over metals and plastics with respect weight reduction, corrosion resistance, and design simplification. The tactical advantage with composites is the nonmagnetic character which is quite unique in its own and cannot be achieved in metal based composites. However, on the other hand, the organic-based resin systems also present an increased potential threat for fire hazard, which needs to be systematically assessed to study their fire behavior. This would immensely help to reduce the damage to the structural components and also save the precious lives of aircrew.

Glass–epoxy-based composites are widely used in aviation industry due to good impregnation and adhesion offered by epoxy-based systems. However, these systems suffer from inherent drawbacks of flammability associated with very high HRRs.[17] Various efforts concentrated on using FR additives in the epoxy formulation although provide some level of flame retardancy, still the systems have some level of limitation in meeting the multifunctional requirements expected from the aviation industry. The increasing pressure on aircraft structural composites manufacturer to meet stringent FR requirements has resulted in the scientific community to explore thermoplastic fiber–fiber-based composites, which offer satisfactory level of FR properties. One such combination of thermoplastic-based systems is the use of fiber–fiber-based glass–PEEK combination as both the reinforcement fiber and matrix are inherently FR and does not call for any FR additives. Further the effective FR systems generally used in epoxy matrix are halogen based, which are carcinogenic and cause increased health associated risks.

In the light of foregoing and differing from the above citations, which study the behavior of fabrics and or composites alone, an attempt has been made to study the flammability behavior of selected thermoplastic based glass–polyester (saturated) and glass–PEEK hybrid fabrics and their composites under various levels of radiative and convective heating at moderate and high heat flux levels (50 and 75 kW/m^2). The second objective is to study heat transmission (20–80 kW/m^2) properties of fabrics and composites. The above heat flux levels are chosen so as to simulate fire scenario in which the composite material is itself burning or in which it may be in close proximity to the burning material. The realization of flammability behavior from fabric to composites is also discussed. The primary focus here is on glass–PEEK based materials and the flammability behavior of selected glass–polyester fabric is carried out for the purpose of comparison only. Although a number of flame-related parameters were recorded in cone calorimeter, the discussion is restricted to few selected parameters which control the overall flammability behavior of a material.

METHODS

Materials

Fiber grade PEEK (Victrex 151 G, with a number average molecular weight of 20,000, polydispersity ratio of 2.0, T_g 143°C, melting point 343°C) was obtained from ICI, UK. The PEEK granules were dried in a hot air oven at 120°C for 4 h before spinning to obtain the PEEK yarn. A laboratory model extrusion machine (Bradford University, UK) working on cylinder-piston principle was used to obtain the continuous PEEK filaments through melt spinning technique. The extrusion temperature of the

TABLE I
Details of Constructional Particulars of Hybrid Fabrics

Parameters	S11	S13
Thread density, $n_1 \times n_2$ (per 2.5 cm)	48 × 32	48 × 32
Proportion of weft (%)	34.0 (PEEK)	31.0 (polyester)
Yarn linear density, $N_1 \times N_2$ (Tex)	136 × 122	136 × 122
Weave	2/2 basket	2/2 basket
Mass per unit area (g/m^2)	415	420

polymer was maintained at 370–380°C. The RAM pressure and speed was maintained at 7500–10,000 MPa and 7–10 mm/min, respectively. The filaments were doubled and twisted before subjecting it for weaving process.

The coweaving of extruded PEEK fiber (weft) with E-glass fiber (warp) and glass (warp) and polyester (weft) was carried out on a rapier loom (80 picks per minute speed) at Urja Products, Ahmedabad, India, to obtain the hybrid fabric in 2/2 basket weave configuration. The polyester fiber (multifilament) used was obtained from M/s Ankur polyesters, Saharanpur (molecular weight of 22,000, T_g 85°C, melting point of 262°C).

E-glass–PEEK (sample code, S11) and E-glass–polyester (sample code, S13) hybrid cowoven fabrics were designed to contain 34% polyester fiber and 31% PEEK respectively. Glass fiber was used in the warp direction, whereas PEEK (S11) and polyester (S13) was used in the weft direction. The details of above fabrics and their constructional particulars are given in Table I.

The hybrid fabrics were then converted into laminates using compression molding technique. The heating and consolidation process was optimized with respect to pressure (250–300 MPa), rate of heating (5°C per minute) and soaking time (30 min at 385°C). Glass–PEEK composites with different thickness viz., 3.3, 3.6, and 4.5 mm were fabricated by varying the number of layers of fabric viz., 15, 17, and 20, respectively.

Glass–polyester (S13) and glass–PEEK (S11) hybrid fabrics were subjected to cone calorimeter test and heat transmission tests (ISO 6942). Glass–PEEK composite (S17) was subjected to cone calorimeter test and heat transmission tests (ISO 9151). All the specimens in cone calorimeter were evaluated by directly exposing the samples to radiant heat flux levels without the use of grid.

Techniques

Preparation and evaluation of fabric sample in cone calorimeter

The flammability behavior of hybrid fabrics and composites were evaluated using Cone calorimeter from "Fire Testing Technology Limited" UK. The instrument works on "oxygen consumption" principle using radiant heat as per ASTM E 1354/ISO 5660. A preset heat flux of 50 kW/m^2 was used to evaluate glass–polyester hybrid fabrics. Heat flux of 50 and 75 kW/m^2 was used for glass–PEEK hybrid fabrics and their composites. The above heat flux was chosen to simulate conditions of severe fire exposure. Single fabric layer and composite samples of size 100 mm × 100 mm were exposed to a selected radiant flux and ignited by means of a spark ignition source. The fabric surface facing the heat flux source was secured firmly by stapling at all the four corners to ensure that the surface of the fabric exposed is free from any creases that may possibly aid in cracking of the fabric during testing. A minimum of three samples in each of the category was subjected to testing and the results reported are thus the average of three samples. A single layer of aluminum foil of 5-μm thickness with its shiny side toward the sample was used to wrap the sample from the underside covering the four edges of the combination sample leaving the top surface open. The sample so prepared was secured in the sample pan with a sufficient backing of ceramic wool blanket (60 kg/m^3 density) over the ceramic block so as to expose the sample at a distance of 25 mm from the radiating source. As the material is not expected to swell profusely, the optional wire grid was not used over the top of the combination sample. The retainer frame was placed around the above assembly so that only 88 cm^2 of the top surface of the combination sample was exposed to the radiating conical heater. A similar arrangement was used for the evaluation of composite samples.

Heat transmission: Fabrics and composites

Heat transmission properties of glass–PEEK and glass–polyester hybrid fabrics were measured by subjecting the samples to incident radiant heat flux of 20 and 40 kW/m^2 and the time required for increase in temperature to 24°C was recorded as per ISO 6942 (method "B"). A pretension of 2*N* was applied to the specimen. The sample size was 90 mm × 260 mm. The composites made out of the above hybrid fabric samples could not be tested as the equipment do not permit mounting of rigid samples. Performance level for radiant heat test as per ISO 11612:1998 was used to rank the material as per the details given below.

C1: HTI (24) in second: 8–30
C2: HTI (24) in second: 31–90
C3: HTI (24) in second: 91–150
C4: HTI (24) in second: min 151

TABLE II
Flammability Behavior of Hybrid Fabrics and Composites at Different Irradiation Levels

	Glass–polyester fabric (S13), 50 kW/m^2	Glass–PEEK fabric (S11), 50 kW/m^2, 75 kW/m^2		Glass–PEEK composite (S17), 75 kW/m^2
Ignition time (s)	20	Did not ignite	37	128
Flame out (s)	52	NA	68	441
Peak heat release rate (kW/m^2)	120.55	5.61	79.93	173.71
Average heat release rate (kW/m^2)	34.84	2.66	36.88	83.32
Total heat evolved (Mj/m^2)	2.1	0.2	2.5	26.2
Average effective heat of combustion (Mj/kg)	15.64	0.00	31.20	19.43
Average specific extinction area (m^2/kg)	437.45	0.00	454.29	575.28

Determination of heat transmission properties on exposure to flame was also carried out as per ISO 9151 wherein the glass–PEEK composite sample (11-cm circular specimen) is subjected to convective heat flux of 80 ± 5% kW/m^2 from the flame of a gas burner (propane) placed beneath it. The heat passing through the specimen is measured by means of a small copper calorimeter on top of and in contact with the specimen. The time in seconds, for the temperature in the calorimeter to increase (24 ± 0.2) is recorded. The mean result of three test samples is calculated as "heat transfer index" (HTI).

RESULTS AND DISCUSSION

Cone calorimeter: Ignition time

Ignition times for hybrid fabrics and composites are noted so as to get a measure of the resistance of materials to pilot ignition under radiative heating. The values of ignition time (t_{ig}) of the fabric samples and composites evaluated in cone calorimeter are given in Table II. It can be seen from the data that glass–PEEK hybrid fabric (S11) at a heat flux of 50 kW/m^2 did not ignite and recorded a time of 37 s at high heat flux level of 75 kW/m^2. On the other hand glass–polyester sample (S13) with more or less similar mass per unit area as that of glass–PEEK sample ignited within 20 s (50 kW/m^2) suggesting the vulnerability of polyester fiber. In very large fires, one might expect similar ignition-delay times even for composites, whereas in incipient fires one would expect ignition-delay times to depend on the chemical composition of the resin and configuration of the material. It is generally agreed that pyrolysis rate of the resin controls the rate at which the vapor concentration at the composite surface approaches its lower flammability limit. Taking cognizance of the fact that pyrolysis being a temperature dependent parameter, it appears reasonable to infer that ignition occurs only after the surface temperature of the resin (PEEK) has reached a critical level of the ignition temperature.[18] The glass–PEEK composite (S17) composed of 17 layers of hybrid fabrics (thickness of 4 mm) exhibited excellent ignition-delay times of 128 s.

Heat release (cone calorimeter)

HRR and PHRR have been widely used by researchers to assess the hazard potential of fabrics.[1,18–20] In this study discussion pertaining to PHRR is restricted as it represents more of a surface behavior and mainly influenced by specimen surface area, ignition time, care with which the sample has been prepared or mounted. The heat release characteristics of hybrid fabrics and composites are presented in Table II and Figures 1–3. A comparison of the glass–polyester (S13) and glass–PEEK (S11) fabrics shows that the latter exhibited average HRR and PHRR as low as 2.6 and 5.6 kW/m^2 (at heat flux of 50 kW/m^2), respectively, indicating excellent combination of glass and PEEK toward achieving FR properties. As expected, the HRR of glass–PEEK hybrid fabric increased with the increase in heat flux level (75 kW/m^2) (Fig. 1). The superior performance of glass–PEEK hybrid fabric compared with glass–polyester hybrid fabric suggests that chemical

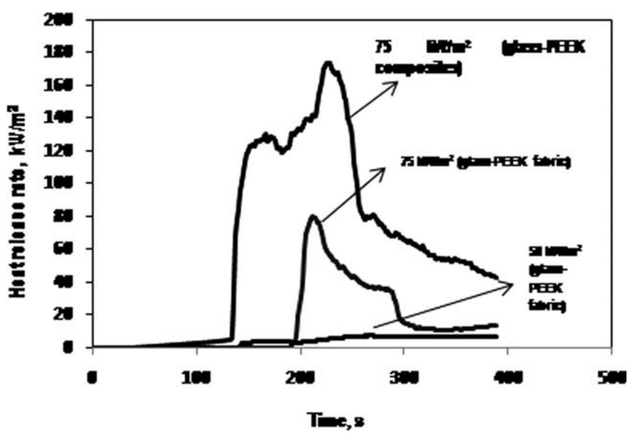

Figure 1 Heat release properties of hybrid fabrics and composites (glass–PEEK).

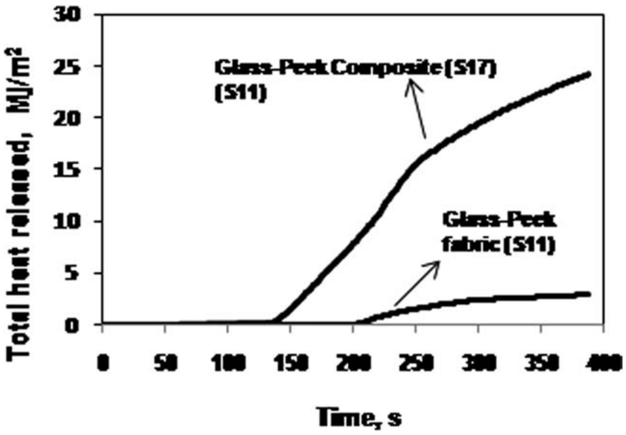

Figure 2 Total heat released: glass–PEEK fabric and composites at 75 kW/m^2.

composition and degradation products of the polymer plays an important role in controlling the overall flammability behavior including pyrolysis and susceptibility to oxidation. The FR properties of hybrid fabrics when converted into composites (glass–PEEK) were also investigated at high heat flux levels of 75 kW/m^2. The results of HRR and total heat released for selected fabrics (50 and 75 kW/m^2) and composites are shown in Figures 1 and 2. From HRR curves (Fig. 1), it is observed that the curve for composite show an initial sharp rise in heat release representing peak heat release that occurs at the time of ignition. The subsequent reduction in heat release is attributed to the thickness of the material wherein essentially the energy is utilized by different layers of the composite before undergoing degradation. The second peak observed (Fig. 1) is a result of an increase in the pyrolysis rate of the unburnt substrate caused by an increase in the bulk temperature of the substrate.[16] It may also

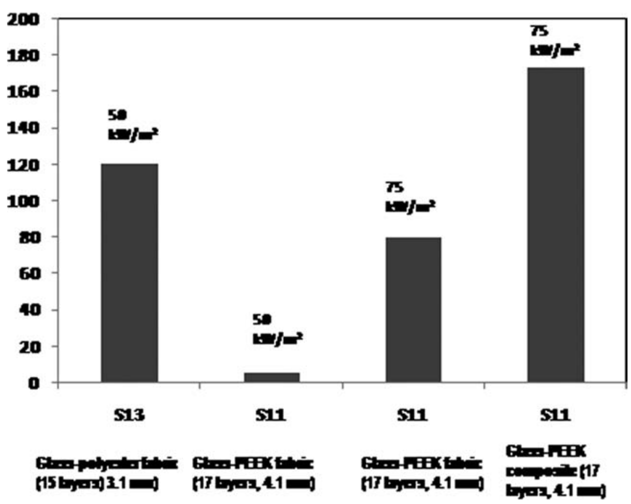

Figure 3 Comparative performance of PHRR of glass–polyester and glass–PEEK fabric and composites.

be expected that any carbonaceous network that forms after initial exposure may actually retard the process of heat release. This may be explained based on the fact that the char may actually act as a barrier to prevent flammable gases of the resin to combine with oxygen and thus reduce the radiant heat reflected back to the polymer surface. Further, the thickness of the sample provides a greater heat sink property resulting in a smaller volume of PEEK being raised to the decomposition temperature. In addition to above attributes, the surface to volume ratios are lowered when two layers are placed together which reduces the exothermic heat loss from the structure. It has been reported that the thin air layer between the fibers also serve as a heat transfer medium.[18] Further, it was also observed that the total heat evolved at 75 kW/m^2 was found to be less and do not exhibit synergistic behavior.

Glass–PEEK composites with average HRR values of 83.32 k/W/m^2, heat of combustion values as low as 19.43 Mj/kg (Table I) further indicate the rate at which the volatile flammable gases present in PEEK are released and its thermal stability to withstand heat flux levels almost at par with its counterpart glass fibers. This compatibility of the thermoplastic resin fibers suggests the suitability of glass–PEEK material for structural components in aerospace applications to encounter fire accidents. Toldy et al.[17] in their work on carbon fabric with phosphorus containing FR epoxy (4-mm thick at 50 kW/m^2) has reported ignition temperature of 43 s, peak HRR of 195 kW/m^2 and HRR of 115 kW/m^2) which are significantly high compared with equivalent systems reported in this study. Le Lay and Gutierrez[19] in their work on glass–polyester (unsaturated) equivalent systems even after incorporating halogen based FR in the matrix has reported HRR values in the range of 200 kW/m^2 (at 50–75 kW/m^2), which are significantly high compared with glass–PEEK systems studied in the present work. In this context, it is noteworthy to observe that "x" and "y" values (Tables III and IV) indicative of fire risk classification lie in the intermediate range (Tables III and IV). It is pertinent to note that glass–PEEK composites in the absence of FR additives do not suffer from the inherent drawbacks of reduction in inter laminar shear strength as is generally observed in glass–FR

TABLE III
Fire Risk Classification Based on the Arbitrary Value of x and y Parameters

Arbitrary value	Risk type (x)	Risk type (y)
0.1–1	Low	Very low
1.0–10	Intermediate	Low
10–100	High	Intermediate
100–1000	–	High

TABLE IV
"x" and "y" Values of Cone Calorimeter Test Results of Hybrid Fabrics and Composites

	x	Risk classification	y	Risk classification
Glass–polyester fabric (50 kw/m^2), S13	6.0	Low	2.1	Very low
Glass–PEEK fabric (50 kw/m^2) fabric, S11	0.0	Low	0.2	Very low
Glass–PEEK (75 kw/m^2) fabric, S11	2.2	Low	2.5	Very low
Glass–PEEK (75 kw/m^2) composite, S17	1.4	Low	26.2	Intermediate

containing epoxy systems. This has been confirmed by Toldy et al.[17] and is attributed to the polar character of the FR component, which does not fit to the sizing of the reinforcing fiber designed for the unmodified epoxy resin.

A number of researchers[21–23] have also reported about the contribution of reinforcement fibers in the composites toward overall flammability some of which are summarized below.

i. Reduces potential HRR of flammable gases present in PEEK (composites).
ii. Limits the optical smoke density and related parameters.
iii. Limits the weight loss of PEEK thereby preventing the decomposition of the polymeric material and effectively reduces the radiant heat directed back to the polymer surface.

Specific extinction area

Specific extinction area (SEA) is an important parameter in the overall assessment of flammability behavior as it considers parameters like opacity and amount of smoke produced. It is observed from Table II that the SEA values for glass–PEEK composites even at high heat flux of 75 kW/m^2 was significantly less (575 m^2/kg). A value in excess of 1200 m^2/kg has been reported for glass-brominated polyester thermoset systems.[19]

Heat transmission characteristics of hybrid fabrics and composites (glass–PEEK)

Table V shows the results of heat transfer rate of glass–PEEK and glass–polyester hybrid fabrics at low and medium intensity heat flux levels of 20 and 40 kW/m^2. As can be seen from the data, the observed time required for a 24°C rise in temperature at low intensity heat flux level was quite high ranging between 15.85 and 15.95 s. Both the samples (S11 and S13) with PEEK and polyester as resin fibers, respectively, exhibited similar behavior suggesting that the samples can be effectively used for low and medium intensity exposure threats. However, it was observed that glass–PEEK hybrid fabric except for some embrittlement maintained its structural integrity, whereas glass–polyester hybrid fabric showed prominent drawbacks such as break open behavior and shrinkage.

The HTI presented in Table VI further reinforces the potential applications of glass–PEEK materials. It can be seen from the data that the time required for rise in temperature for both 12 and 24°C increased with increase in thickness (number of layers) and is attributed to increasing number of layers and greater proportion of fibers available. The greater proportion of fibers although may facilitate conduction, the lack of air nor the available mechanical spaces in the composite do not facilitate heat transfer by conduction. As can be seen from the data in Table VI that the time required for 24°C rise in temperature was as high as 36.4 s (sample S20) resulting in a HTI of 9.2 s. The HTI of 9.2 s achieved meets the highest level of performance rating of B5 (ISO 11612). Further the above samples did not exhibit any shrinkage, hole formation, glowing, scorching, melting nor dripping further confirming the excellent combination of glass and PEEK fibers toward achieving FR properties. Earlier work at the National Bureau of standards showed that heat transfer through fabrics is generally higher when the incident heat flux is radiative only rather than a mixture of radiative heat flux and convective heat flux.[18] The data obtained from radiant heat exposure suggest that the exposed fiber (glass) does not have any major role to play and the combustion behavior of fabrics and composites essentially depends on the thermal stability of the constituent matrix in the fabric/composite. However, Morse and Becker[24,25] suggest that optical properties of fabrics also play an important role in deciding the flammability behavior especially in high-intense heat exposures.

The excellent FR properties of glass–PEEK hybrid fabrics and composites observed above is attributed

TABLE V
Heat Transmission Properties of Glass–PEEK and Glass–Polyester Fabrics at Low and Medium Heat Flux Density of 20 and 40 kW/m^2 (ISO 6942)

	Heat transmission (s)	
Sample	20 kW/m^2	40 kW/m^2
S11	15.95	10.0
S13	15.85	9.5

TABLE VI
Heat Transmission Properties of Glass–PEEK Composites at Incident Heat Flux Density of 80 KW/m^2 (ISO 9151)

Sample	Time (s) for temperature increase to 12°C HT1 (12)	Time (s) for temperature increase to 24°C HTI (24)	HTI (12)–HTI (24) (s)
S15	20.7	27.2	6.5
S17	21.6	29.0	7.2
S20	27.2	36.4	9.2

to a combination of various factors that include (i) thermal stability of the PEEK fibers and high LOI (of 33–35%). The absence of nitrogen, sulfur, and phosphorus in the PEEK structure contribute to extremely low levels of toxic or acid release as well. Although PEEK fiber releases carbon monoxide and carbon dioxide, the usual combustion products, does not release halogen based gases. The latter properties which are not generally observed in thermoset-based composites will play a major role in advanced composite applications toward meeting flammability standards.

CONCLUSIONS

The fire response of hybrid fabrics and composite materials aimed at structural components was characterized using cone calorimeter and heat transfer techniques and rated according to their performance in terms of fire risks and hazards. The following conclusions are drawn from the study.

- The results demonstrate that the glass–PEEK hybrid fabrics exhibit low peak HRR, low HRR, and low heat of combustion, suggesting the excellent combination of materials and fall under the low-risk category.
- At 50 kW/m^2, glass–polyester based hybrid fabric exhibited PHRR of 120 kW/m^2 and average HRR of 35 kW/m^2, which is significantly high compared with glass–PEEK based hybrid fabrics.
- The performance of the glass–PEEK fabric even at high heat flux levels of 75 kW/m^2 was comparable with the performance of glass–polyester fabric evaluated at 50 kW/m^2.
- The performance of the glass–PEEK based composites compared with glass–FR incorporated epoxy systems reported were much superior and exhibited average heat release of 83 kW/m^2. The absence of nitrogen, sulfur, and phosphorus in the PEEK structure and is attributed to low levels of toxic gases or acid release.
- The HTI of 9.2 s (glass–PEEK composites) achieved meets the highest level of performance rating of B5 (ISO 11612). Further, glass–PEEK based materials did not exhibit any shrinkage, hole formation, glowing, scorching, and melt dripping.

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