Assessing the Flammability of Composite Materials*

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INTRODUCTION

COMPOSITE MATERIALS OFFER the potential for substantial weight savings in the structure of both surface ships and submarines. However, the organic nature of the binder resins in these materials implies that one would be replacing non-flammable materials (aluminum, steel) with materials that could possibly contribute to a fire. This points to a critical need for methods which allow reliable prediction of the extent of fire involvement which a given material may exhibit in a particular application. There are numerous aspects of this which must ultimately be considered; these range from the strength of the composite under a fire heat load to potential toxicity and corrosivity of the fire gases.

The focus of the present work is the extent to which an external ignition source (e.g., some nearby burning object) will cause fire spread on bulkhead (vertical wall) surfaces comprised of a composite material. Here, as with other aspects of the problem, one seeks the best small-scale measurements which will permit correct prediction of full-scale behavior. The ultimate full-scale problem of interest is that of fire

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spread in a compartment whose walls are composite materials. We have examined first, however, a single flat wall exposed to external radiation and an ignition source; the wall is not enclosed so oxygen vitiation is not a consideration.

The key aspects of flammability which are pertinent to this problem are ignitability, rate of heat release and flame spread rate (both concurrent and opposed-flow spread). One needs information on these over a range of heat fluxes to treat the problem in a general manner. The approach that is being followed in this study is an adaptation of that due to Quintiere [1]. The Cone Calorimeter is used in this study to characterize ignitability (ignition delay time as a function of heat flux) and rate of heat release; here this latter measurement is being made in a different manner than usual, as described below. The Lateral Ignition and Flame Spread Test (LIFT) device is being used to characterize opposed-flow flame spread behavior.

The main emphasis in this article is on upward flame spread. A simplified upward flame spread model due to Cleary and Quintiere [2] is being used to predict the conditions of occurrence and rate of upward spread on the wall surface. This model makes substantial assumptions in order to obtain these predictions in explicit algebraic form. The assumptions include the use of a constant heat flux from the flame and a constant average local rate of heat release from the material after ignition. A principal goal is to determine how well this model can predict upward spread behavior in an intermediate-scale test configuration.

This particular model uses inputs derived from the Cone Calorimeter data. Composite materials can pose special problems in the bench-scale measurement of heat release rate. The composites used here consist of several plies of resin-impregnated woven glass. The plies are bonded together by the resin. When a heat flux is imposed on one surface of such a composite, the resin between (as well as within) the plies degrades and the local inter-ply bond is weakened. As gases begin to be generated between plies, they force a delamination with the gases seeking the path of least resistance out of the composite. Depending on the particular resin/glass combination, this path may be out through the plies or it may be out of the sides of the sample (typically 10 cm square). An easy leakage path to the edges is not likely to be representative of a full-scale fire exposure. Gases which escape and burn at edges may not feed heat back to the zone in which they originated. The impact of preventing edge escape of the gases from a Cone Calorimeter sample was explored to a limited degree in this study.

EXPERIMENTAL DETAILS

Rate of Heat Release

The NIST Cone Calorimeter was used for rate of heat release measurements on several composite materials. Departures from normal usage procedures were made for two different sets of two materials each.

For the first set the effect of preventing edge escape of pyrolysis gases was explored. In this case the materials were two different polyester/woven roving glass composites 12–13 mm thick; one composite contained a halogen-based flame retardant. The behavior of these materials was compared at two incident heat fluxes, 30 and 60 kW/m². In one set of tests the composites were placed in the normal vertical-orientation sample holder which protects only about 3 mm of the outer periphery of the 10 cm square sample. In a second set of such experiments a special sample holder was used which effectively eliminated all escape of gases from the sample edges. To achieve this the sample was made larger (15 cm square) and the extra peripheral material (2.5 cm around the edge) was clamped in a special sample holder. The front face of this holder was water cooled to prevent it from heating up to the point where it might degrade the sample behind it. The peripheral 2.5 cm wide edge of the sample was insulated somewhat from the water-cooled face of the sample holder. The sample face area exposed to radiation from the Cone heater was the normal 10 cm square.

Post-test inspection showed that this arrangement was very effective in stopping delamination from spreading beyond the exposed portion of the sample face so that gases could not escape from the sample edges. Unfortunately, this special holder alters the rate of heat release behavior not only in the intended manner (preventing gases from escaping out the edges) but also by slowing the thermal response of the outer portions of the exposed sample face; the extra peripheral material around the sample edge is a heat sink for the exposed face material.

A procedure has been devised to correct the heat release data for this edge heat sink effect that is present with the edge clamping holder. However, the procedure is very tedious to implement since it requires running the same sample three times at any given incident heat flux and then multiplying one data set by the ratio of two others. This includes one test of an unaltered sample in the clamped edge holder followed by two tests with samples whose face is perforated (nearly full depth) by an array of very small holes to allow gas escape out the front of the sample; one of these is run in the normal sample holder and the other in the clamping holder. The ratio of the last two (normal over clamped) corrects the first for the heat sink effect of the extended sample edge.

The other departure from normal usage of the Cone Calorimeter did not incorporate the above special holder but rather operation at abnormally low incident heat fluxes using the standard vertical sample holder. This portion of the study employed different composites: one unretarded polyester/woven roving glass composite similar to that above plus an unretarded vinyl ester/woven roving glass composite.

The low incident heat fluxes were used in the Cone tests because the upward flame spread model mentioned above requires heat release data at the flux levels used in the upward spread tests. These levels are necessarily below the minimum flux for ignition of the material; fluxes at or above this level would yield instantaneous upward spread. In upward flame spread local ignition occurs in response to the combination of the external flux and the heat flux received from the flames anchored below the given locale. For turbulent flames this flame flux peaks at 20–30 kW/m^2 [1]. After ignition the flaming process supplies its own flux supplemented by that from the radiant panel. In the Cone tests this behavior was roughly simulated by igniting the material at a heat flux of 24–25 kW/m^2; this is well above the minimum flux for ignition of the two composites (13–15 kW/m^2). When the face of the sample was essentially fully involved in flaming (this could take anywhere from 25 to 45 seconds), a wire mesh screen was moved in front of the Cone heater to cut the heat flux to the desired low value, comparable to the external fluxes used in the upward flame spread tests.

This procedure is not a fully satisfactory simulation of what happens in actual upward flame spread, only a first approximation. In upward flame spread a point on the sample sees preheating for varying lengths of time depending on its height on the sample face. After it ignites, it also can get "support" (piloting plus a potentially sustaining heat flux) from the flames impinging from below. More effort is needed to explore the effects this may have on the local rate of heat release, as seen in Cone tests.

Lateral Flame Spread

The NIST LIFT apparatus provides data on lateral flame spread that can be converted to a generalized form useful in full-scale [1]. Here the LIFT device was used in the normal manner and also with a modified sample holder. The modified holder clamped the periphery of oversize samples in a manner identical to that used to prevent gas escape from the edges of the Cone Calorimeter samples. It was equally effective for this purpose.

Upward Flame Spread

These tests were conducted in the NIST Furniture Calorimeter using a new intermediate-scale radiant panel facility to provide the external heat flux. Figure 1 is a sketch of this set-up. The hood of the Furniture Calorimeter captures the entire fire plume; measurement of the flow rate and oxygen content of this plume allows calculation of the instantaneous rate of heat release. Because the fires here are smaller than the furniture fires for which this calorimeter was designed, the hood flow was lowered and the calibration fires (using a gas burner) were limited to 50–130 kW.

The test sample is 38 cm wide by 122 cm tall and is set into a flat vertical surface 1.8 m tall. An inert wall (aluminum) extends beyond the sample width and incorporates 5 cm fins, perpendicular to its surface, placed to either side of the sample; this proved to be necessary to prevent the fire plume from contracting severely and spreading up only one portion of the sample surface. The sample edges were not clamped. A gas flame igniter spans the width of the sample at its bottom edge. The sample face is irradiated by a pair of electrically powered panels arrayed so as to give a flux that is uniform within about 10%. To achieve this, the panels are necessarily substantially longer than the sample.

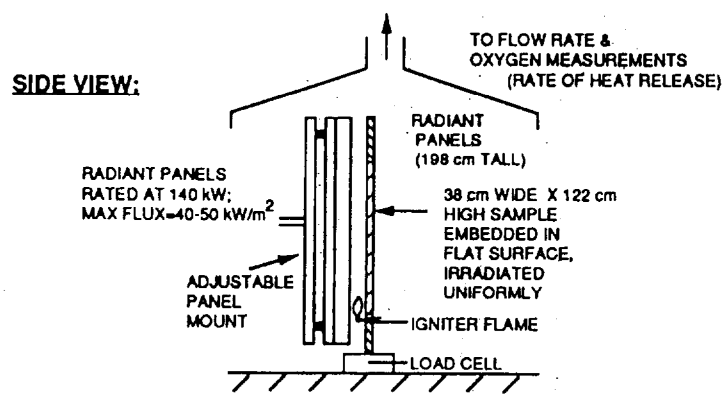

Figure 1. Sketch of radiant panel facility.

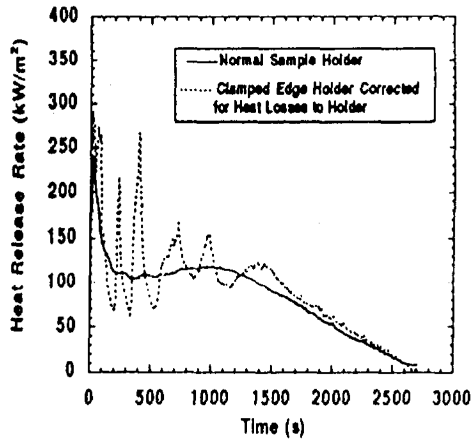

Figure 2. Effect of clamping sample edge on heat release behavior; polyester/ woven roving glass, 35 kW/m².

In a typical test, the irradiation and igniter exposure begin simultaneously as a shutter is removed from in front of the sample. The subsequent ignition and upward flame spread process are recorded on a video tape which also includes verbal commentary on the height of the flame tips and the attached flame front. The attached flame front is difficult to discern with many materials; here it is made more readily seen by virtue of the tendency for ignition to spread first to localized jets of gases emerging through the woven roving glass structure.

DISCUSSION OF RESULTS

Figure 2 shows the response of one composite material in the Cone Calorimeter to suppression of gas escape from the sample edges. The heat release curve for the sample with a clamped peripheral edge has been corrected for edge heat losses in the manner outlined above. The response here to edge clamping is rather extreme but other materials examined show the same tendency for a fairly smooth rate of heat release curve to be transformed into an erratic looking series of peaks. The peaks are probably associated with the delamination of successive plies within the composite structure. With each peak and valley in the curve the flames on the front of the sample can be seen to grow and shrink. The pattern of peak occurrence is not repeatable from one sample to the next of the same material.

The complex behavior in Figure 2 would be quite problematical if it had to be incorporated in detail into predictions of fire spread on large composite surfaces. First, as noted above, the process one must go through to get these data is tedious at best. Second, it would be difficult to input such complex data into a model. Third, as was just noted, the details of the behavior are not repeatable.

There are two flame spread modes where this complex behavior may be of concern: opposed flow spread (across a wall, down a wall, across an upward-facing horizontal surface) and concurrent spread (up a wall, across a ceiling). The data from the LIFT device are representative of the opposed-flow spread process. There was no discernible effect on the lateral flame spread rate over a polyester/glass composite of clamping the edges to prevent gases escaping there. The peak flux used in these tests was about 20 kW/m² and preheating times were varied. Evidently the gases from in depth can escape well behind the flame front in this spread mode and there is no interaction with the highly localized heat transfer processes at the front which dictate spread rate.

The potential impact of erratic local heat release behavior, like that in Figure 2, on concurrent flame spread is less obvious. Upward flame spread rate responds to the total rate of heat release behind the attached flame front [2,3]. This is what dictates the height of the flames which are heating the next elements of the material. The total rate of heat release per unit width of sample is given by the following expression:

$$\dot{Q}_{tot}(t) = \int_{o}^{t} \left(\frac{dh}{dt'}\right) RHR\,(t - t')dt'$$

Here $\dot{Q}_{tot}(t)$ is the total rate of heat release from the entire burning area at time t; h is the height of the attached flame front relative to the bottom of the sample or the burn-out front, if there is one; RHR is the rate of heat release per unit area of burning material. Both the attached flame front height, h, and the local rate of heat release, RHR, are time-varying functions and the above integral simply sums over the contributions from portions of material successively ignited by the upward moving flame front. The flame height above the attached flame front thus responds to a time and space average of the burning process behind it. This suggests that the easiest (and perhaps the best) way to deal with the highly complex behavior seen in Figure 2 is to average over time. Inspection of Figure 2 indicates that, as long as the averaging time is long compared to the peak widths, the average heat release rates for the two modes of sample holding are quite similar. At 1000 seconds, the averages differ by less than 15% which is probably better than the general reproducibility of Cone Calorimeter data for composite materials. Furthermore, if this is true, then there is no real benefit to be gained by going to the trouble of obtaining data with a clamped edge holder and correcting it for heat losses. Essentially the same model input is obtained from the averaged data produced with the normal sample holder.

The preceding argument is somewhat conditioned by the fact that the model of Cleary and Quintiere to be tested here only accepts a constant (averaged) rate of heat release. Other models are more general [3,4] in the form of the heat release curve they employ. An unsettled issue is the sensitivity of the predicted flame spread behavior to the form of the heat release rate function. In any event it is evident that if the flame spread process is not slow compared to the peak widths in Figure 2 and if this behavior carries over unchanged to full-scale flame spread, the spread process will likely be more erratic than the average behavior predicts.

Figure 3 shows a typical upward flame spread result, in this case at an external flux of 5 kW/m². The 6.5 kW igniter yields flame tips about 30 cm high; this size igniter flame was used throughout the present work. The flame spread process that results at this low external heat

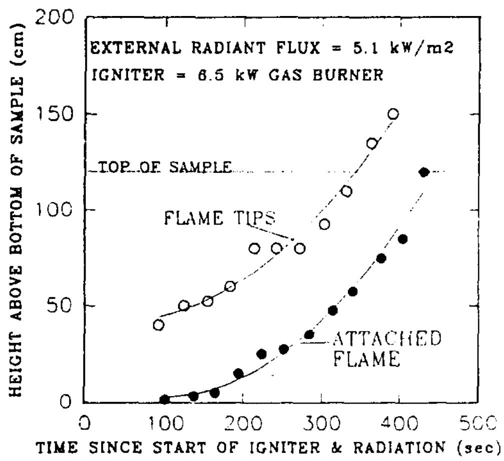

Figure 3. Upward flame spread on ortho polyester/woven roving glass

flux is clearly acceleratory, reaching the top of the sample 300 seconds after the bottom portion first ignites. In fact, flame spread on this unretarded polyester did not require any external heat flux but under such circumstances it took twice as long to reach the top of the sample. The other composite, based on a vinyl ester resin, yielded spread only 1/3 of the way up its surface in the absence of an external heat flux. (The radiant panels were present in these "zero flux" cases and slowly heated up via radiation from the sample; they may have ultimately re-radiated back to the sample as much as 2 kW/m^2.)

Figure 4 shows the measured and predicted pyrolysis (attached flame) heights for the two composites at an incident flux of 5 kW/m^2. Note that the time scales differ by about a factor of two for the two materials. Note also that two different tests are shown for each material. The data for the second test with the polyester composite are shown only up to a height of about 0.4 m. The flame spread behavior above this was highly erratic: the flame front moved very rapidly to the top of the sample then died back through most of the upper middle portion of the surface then spread laterally inward onto this area from the edges. The reason for this behavior is unknown but could be associated with an unusual delamination of the affected area. Aside from this unusual case, the upward spread behavior is only moderately noisy.

Figure 5 shows the measured and predicted overall rates of heat release which accompanied the spread processes in Figure 4. The heat release does tend to be more noisy than the spread process. It is probable that this reflects the kind of processes seen in the clamped edge heat release data of Figure 2.

The Cleary-Quintiere model predictions are also shown in Figures 4 and 5. The input heat release data were obtained at the indicated flux using the normal sample holder and were averaged over a time period equal to one-half of the experimentally observed upward spread times. (If one were doing these predictions *a priori* it would be necessary to guess the spread time and then iterate.)

One parameter in the model is the proportionality constant between the overall rate of heat release and the flame height. For turbulent flames there appears to be a universal relation between these two quantities [1] but the proportionality constant is somewhat uncertain in view of the difficulty in precisely defining flame height. Two values for this constant were used: that from the literature and a value 20% higher. The predictions are clearly quite sensitive to this constant but, in general, are comparable to the experimental results. One shortcoming is the fact that the model does not predict full length spread for the base value of the constant on either material; this is largely why it also

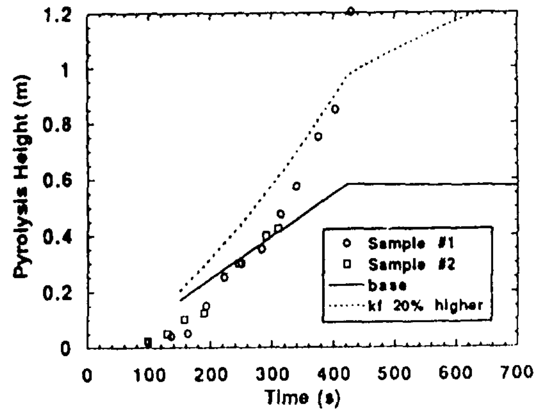

Figure 4a. Flame spread results for polyester/woven roving glass, radiant flux = 5.1 kW/m²; lines are prediction.

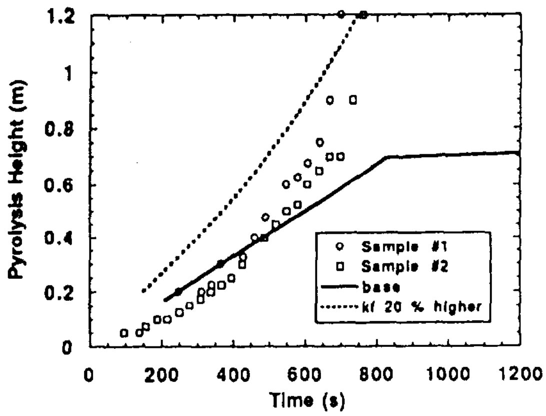

Figure 4b. Flame spread results for vinyl ester/woven roving glass, radiant flux = 5.1 kW/m²; lines are prediction.

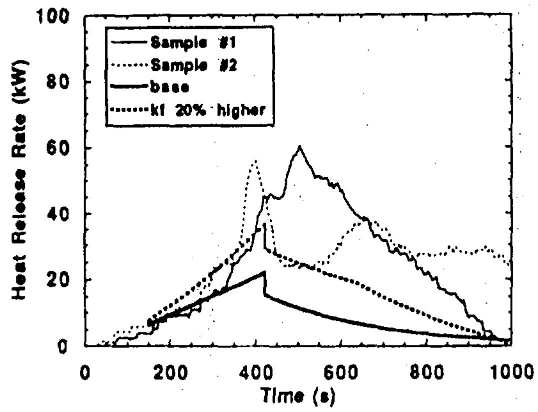

Figure 5a. Heat release rate results for polyester/woven roving glass, radiant flux = 5.1 kW/m²; smooth lines are prediction.

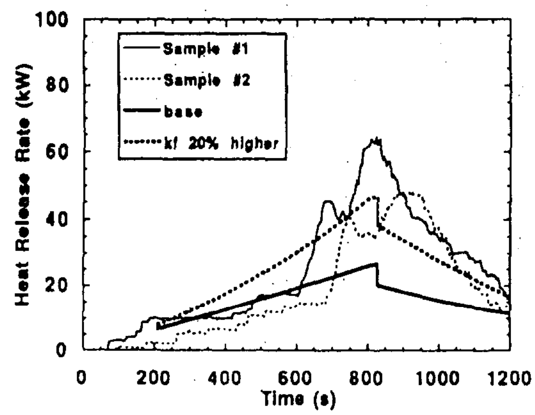

Figure 5b. Heat release rate results for vinyl ester/woven roving glass, radiant flux = 5.1 kW/m²; smooth lines are prediction.

substantially underestimates the peak rates of heat release for both materials when this base value is used. A second reason in the case of the polyester may be that sample burnout (well behind the spreading flame front) was not the decisive on/off process which the model assumes.

Predictions have also been made for the case of zero flux and for a flux of 12 kW/m². The model correctly predicts little spread at zero flux for the vinyl ester composite but it predicts only 60% spread on the polyester composite which actually exhibited full height spread. At 12 kW/m² only one test is available, for the polyester composite; the model somewhat overpredicts the speed of flame spread upward.

Overall it appears that the simplified flame spread model of Cleary and Quintiere combined with averaged heat release data obtained with the normal holder in the Cone Calorimeter is promising, at least for first estimates of composite flame spread behavior. Further study with other composites is needed, as well as further examination of the best technique for measuring the Cone heat release at low fluxes, before one can recommend this approach in fire safety design. Finally, it should be recognized that the delamination process that was so evident here in the bench-scale tests could be a source of erratic behavior in real-scale fires; seams and joints, in particular, may require separate fire tests.

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