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The Behavior of Carbon Fiber-Epoxy Based Aircraft Composite Materials in Unmitigated Fires

Alexander L. Brown[†], Amanda B. Dodd[††], and Kenneth L. Erickson[†††]
Fire and Aerosol Sciences, Sandia National Laboratories[†], Albuquerque, NM 87185-1135
Thermal and Fluid Processes, Sandia National Laboratories[††], Albuquerque, NM 87185-0836
Thermal/Fluid Experimental Sciences, Sandia National Laboratories[†††], Albuquerque, NM 87185-0836

Corresponding author:
Alexander L. Brown
Sandia National Laboratories
P.O. Box 5800 MS 1135
Albuquerque, NM 87185
United States of America

Phone: (505)844-1008
Fax: (505) 845-3151
Email: albrown@sandia.gov

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ABSTRACT

New aircraft are being designed with increasing quantities of composite materials used in their construction. Different from the more traditional metals, composites have a higher propensity to burn. This presents a challenge to transportation safety analyses, as the aircraft structure now represents an additional fuel source involved in the fire scenario. Performance testing data for composites burning in a fire at the integral scales of an accident event are nearly non-existent. This report describes fire tests for relevant carbon fiber epoxy materials that were designed to explore the bulk decomposition behavior of said material in a severe fire. Together with TGA decomposition data, the material is found to decompose in three mostly distinctive and sequential phases, epoxy pyrolysis, char oxidation, and carbon fiber oxidation. Fires were moderate in their thermal intensity. Peak thermal intensities of around 220 kW/m^2 or 1100 ºC are achieved at very low air flow rates. The burn tests were remarkable in their duration, lasting 4-8 hours for 25-40 kg of combustible material.

Keywords: Composite fires, solid combustion, carbon oxidation, aircraft fires

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1. INTRODUCTION

Composite materials are increasingly being used in the design of aircraft. They offer comparable structural strength to traditional metals such as aluminum and titanium for a fraction of the weight. These are positive consequences to this transition, as a weight savings can affect a fuel savings. Fuel savings reduce emissions, increase transportation efficiency, and reduce operating costs. There are negative consequences as well. It is more costly to build aircraft with these materials. This cost is presumably offset by the fuel savings over the lifetime of the aircraft. Because composites are comparatively new, they have performance issues when compared to metal structures. Lifetime strength, maintenance, and reliability issues are important performance consideration, and have been moderately well studied. There are other metrics of performance, such as the focus of this study, which is the concern of the thermal environment resulting from an incident of a fire involving an aircraft with significant composite materials.

Aircraft fires are not an uncommon event. Airports have equipped and trained response personnel to help prevent loss of life and property. Response teams must be adequately trained and aware of safe and optimal methods to best perform their function, but not all fires occur in proximity to emergency fire fighters. There are remote events that may lead to aircraft fires that cannot be extinguished by a response team. Transportation safety can be improved when these environments are better understood.

Most aircraft composites consist of a binder and a fiber. These can vary widely in their characteristic behavior in a fire. Furthermore, it is important to understand the lay-up and configuration of the materials, and there are numerous common configurations. Aircraft parts are typically made from layers of either unidirectional fibers oriented in varying relative rotational directionality or woven fabrics of similarly varying orientations. Pre-cure, sheets are malleable and can be formed into a variety of shapes. Lay-up patterns are customized according to engineering requirements, and typically vary

throughout an aircraft. Various thicknesses are selected depending on design requirements. This results in a heterogeneous material that is often not easily characterized by traditional scalar properties applicable to homogeneous materials. The complexity of the part design methods challenges the ability to predict the outcome of a fire.

The behavior of composite materials in response to a fire has been studied previously by Tyson et al. [1], Fanucci [2], Brown et al. [3], Levchik and Weil [4], Mouritz [5], Jiang et al. [6], Quintiere et al. [7], Delfa et al. [8], Lopez de Santiago et al. [9], and Hubbard et al. [10]. This list does not include all of the research that has been done for composite materials, but highlights those authors who have studied carbon fiber epoxy composites, which are of interest in the current work. Jiang et al. [6], Levchik and Weil [4], and Fanucci, [2] focused on thermal and/or decomposition behavior of the epoxy resins and carbon fiber epoxy composites. Brown et al. [3] used the cone calorimeter to characterize ignitability and flammability of composite materials. Mouritz [5] developed a database of fire properties of polymer composite materials for both aircraft cabin and aircraft structural materials. By gathering information from the literature, Mouritz [5] reports the following fire properties as available: time-to–ignition, limiting oxygen index, peak heat release rate, average heat release rate, total heat release, flame spread rate, smoke, and combustion gases. Quintiere et al. [7] studied the behavior of a carbon fiber epoxy composite through the use of cone calorimeter data, microscale combustion calorimeter data, thermogravimetric analysis, differential scanning calorimetry, Ohio State University (OSU) fire calorimeter tests, and a flame spread apparatus. Delfa et al. [8] performed small scale testing to evaluate the structural failure time of composite materials in a fire under a structural load. Tyson et al. [1] performed an extensive study to examine the response of two different graphite epoxy composite materials. Their studies on 1 foot square, 0.5 inch thick samples were designed to simulate the wing design of a modern fighter aircraft. They found that composites exhibited a greater burn through resistance than aluminum, but after the flames were extinguished, smoldering combustion was observed. Subsequent studies of the smoldering combustion processes revealed two types of smoldering: epoxy-

smoldering (occurs in thick specimens around 400ºC and produces an exothermic reaction with its own supply of oxidizer) and carbon-fiber combustion (occurs after epoxy is consumed and after the temperature is "sufficiently high to sustain combustion of the individual fibers").

In a previous test series in which the authors participated where samples were exposed to 800ºC and 1000ºC radiative emitting panels, flaming combustion occurred for all the 1000ºC tests and for some of the 800ºC tests [10]. Peak heat flux to the samples was on the order of 30 kW/m^2. In these tests, there was no indication of carbon fiber consumption. However, under certain conditions, it is expected that sustainable oxidation of the carbon fibers will occur. Panels exhibited swelling and gases jetting parallel to the surface from the edges and more perpendicularly from gaps in the fiber matrix on the face. Previous work suggests up to 10 atm of gaseous pressure can be found in the fiber layers during composite decomposition [11].

Reports of large-scale testing of composite fires are scarce. This is likely because of the cost of materials and their relatively recent increase in application to aviation construction.

In the current study, a test enclosure was designed and constructed to evaluate the potential severity of a fire environment involving composite materials. The objective of this work was to achieve a thermally extreme environment where the composite materials were subjected to a controlled environment. In addition, the goal was to obtain decomposition of the carbon fibers, evaluate the duration of the decomposition, and determine the magnitude of the thermal intensity that is created during decomposition. The scale of these tests is by design sufficiently large such that there will be an expectation that the bulk material behavior may be similar to that of similar composite material in an aircraft accident. A series of experiments were conducted with a range of carbon fiber composite materials and some wood boards. The wood boards were used during the first few tests to study the test apparatus; however they also provide an interesting comparison to the composite material response. In the interest of brevity, this report contains only a subset of the experimental data results, but provides a

general discussion of some of the principal results and conclusions. To aid in the discussion of the results, TheroGravimetric Analysis (TGA) data will be presented in the methods section.

2. METHODS

2.1 ThermoGravimetric Analysis

ThermoGravimetric Analysis (TGA) can provide insight into the decomposition processes of composite materials. A detailed decomposition analyses using TGA (TA Instruments Model Q5000) was performed for a carbon fiber epoxy composite over a range of heating rates, isothermal temperature holds, and oxygen concentrations. Details of the measurement techniques can be found in Erickson [12] and Erickson and Oelfke [13]. The TGA experiments were performed with small, about 1 to 2 mg, samples that were heated in open 10-mm diameter platinum sample pans. The material used in the TGA experiments was Cytec 977-3, a carbon-fiber epoxy composite. The samples were cured to the manufacturer's specifications. The sample results reported here are for single layer of uni-directional 977-3, although both uni-directional and woven samples have been examined. The epoxy content is approximately 40% by mass of the matrix and the carbon fiber portion is approximately 60% of the matrix. Although this material is not the same as that tested in the large scale test, the authors believe that the general trends in behavior found in the analysis of the TGA data are applicable.

A small subset of the TGA experimental data is shown in Fig. 1. The response in both nitrogen and air at a heating rate of 20°C/min is shown. In both nitrogen and air, the epoxy begins to decompose slowly at approximately 280°C. Oxygen plays a role in the decomposition of the epoxy early in the decomposition process from approximately 400°C to 500°C. The interaction with oxygen enhances the char formation and delays the release of pyrolysis gases. This conclusion is in agreement with Rose *et al.* [14] and Levchik and Weil [4]. At temperatures starting at approximately 450°C, oxidation of the charred epoxy begins to occur slowly. Similar behavior is seen in the oxidation of polyurethane foams [13]. From 650°C to 700°C rapid oxidation of any of the remaining charred epoxy and oxidation of the

carbon fibers ensues. Data from a large number of experiments indicates that the charred epoxy forms around the carbon fibers. Literature on the combustion of graphitic carbon [15], [16] agrees with the high temperatures presented here that are needed to decompose the carbon fibers. Assuming that the charred epoxy coats the majority of the carbon fibers, the oxidation of the charred epoxy (which is characterized by a low activation energy decomposition process) must be complete before oxidation of the carbon fibers will occur. Differential Scanning Calorimetry (DSC) data provides information on the thermal capacitance and the degree of endothermicity and exothermicity of decomposition processes. It was found that the char oxidation and carbon fiber oxidation both result in a net exothermic process; however, the carbon fiber oxidation process is much more exothermic than the char oxidation process.

2.2 Intermediate-Scale Tests

Between 25-40 kg of composite materials were tested in a custom designed enclosure. A schematic of the enclosure is shown in Fig. 2. The enclosure was a 91 cm internal cube, with a 20 by 61 cm rectangular air inlet near the bottom on a side wall, and a 20 by 66 cm rectangular exhaust on the top. The interior walls were made from Unifrax Duraboard 3000 insulation board. The enclosure was constructed from steel mesh and tubing, with pins used to fasten the insulation to the housing. Two 8.8 kW pipe burners supplied propane for 2-5 minutes to ignite the composite materials. Air flow to the system was induced by a blower, and controlled with a power supply and with a damper valve in the 30 cm diameter supply duct before transitioning to the rectangular opening. Flow velocity was pre-characterized under cold-flow conditions with a TSI 8455-300 air velocity transducer. For these tests, the air inlet was from the west, and the air exhaust was on the east side of the removable top of the enclosure.

A bulk calorimeter was located on the floor in the middle of the enclosure. The calorimeter was constructed from 2.54 cm thick, 15.24 cm external diameter Inconel® 600 tubing. End plates were located 20 cm apart and were 2.54 cm thick. Four thermocouples were welded to the internal surface at

90º intervals, and alternated circumferentially between a 14 cm height above the base (pictured) and 6.4 cm height above the base. The internal portion was packed with Zircar 99 board insulation. The type K thermocouples provided data on the behavior of a solid body in response to the test environment. Type K thermocouples have a standard uncertainty of 0.75% of the reading in the range of 400-1300ºC. Composite materials were located around the calorimeter. A cross-section cut-away of the calorimeter is shown in Fig. 3.

Because the four thermocouple traces generally trended similarly, the average thermocouple temperature was used to extract the average heat flux to the calorimeter. The incident flux was calculated using a lumped capacitance model. The details of this methodology are found in a previous report [17].

Medtherm narrow-angle (5º) water-cooled, gas-purged, fast response thermopile radiometers (model P/N NVRW(ZnSe)-FTP-5.5-96-21248) were located near the top of the enclosure looking across the upper layer (labeled EAST and NORTH). An identical radiometer was located at the center of the top surface (labled TOP), oriented to look down at the calorimeter and crib. Radiometers were factory calibrated to the appropriate standards (ANSI/NCSL Z540-1 and ISO/IEC 17025). These were pre- and post-calibrated, and used to assess the radiation intensity in the exhaust layer at the top of the enclosure. The positions of the radiometers are identifiable as circular penetrations in Figure 2, with the north radiometer symmetrically opposite the hole in the south wall visible in the figure. These radiometers have an advertised uncertainty of +/- 3%, but are known to have higher uncertainties, as much as 30% when exposed to significant convection.

Post-test calibrations of the radiometers were performed after the entire test series was completed to provide confidence in the results. Even though the radiometers were aspirated and cooled, the performance decreased by 20% (north), 25% (east), and 40% (top) from the original calibration by the end of the test series. It is thought that the shift occurred in early tests (after the first two tests) and was not a gradual bias across the tests because of the similarity in trending in the output from the radiometers

from all experiments. These measured bias values have been used to correct the measured fluxes, and the corrected radiometer data are presented in subsequent sections.

Four composite tests were performed in this test series. Limited availability of material resulted in a decision to perform four distinctive tests with composite materials (no repeats). The first composite material test involved less uniformity, with a variety of miscellaneous pieces of varying sizes and thickness. The second composite material test involved more uniformity, with 9.5 mm thick panels arranged on two racks. The third test involved a regular crib of thin strips, about 2.4 mm thick, 25 mm wide, and 710 mm long. The fourth composite material test involved I-beams that were 76 mm square, 813 mm long and 2-3 mm thick constructed from Cytec 5208/T-300-12 fibernite. In this report, we give particular focus to the results of the I-beam test, involving 26.5 kg of pre-fractured carbon fiber epoxy I-beams. Figure 4 illustrates the test lay-up of the composite materials. Three lower layers and five upper layers were present in the test. Two of the other tests in the series have been described in a previous report [17].

All tests were conducted at the Sandia burnsite, about 2000 m elevation with ambient temperatures in the 0-10ºC range. Consequently, air density is approximately 15-20% lower than at sea level for the same temperature.

3. RESULTS

Some general observations were consistently found for all tests. All composite tests were characterized by 10-30 minutes of high intensity flaming combustion. This is believed to be dominated by gas phase combustion of the pyrolysis products from the epoxy. It is characterized by significant flaming and dark sooty emissions. Radiometers in the upper portion of the enclosure typically recorded fluxes over 100 kW/m^2 in this regime. Peak measured fluxes were approximately 220 kW/m^2. Subsequently, there is a significant decrease in the thermal flux as glowing combustion of the remaining solid material ensues. Comparatively low fluxes, around 40-100 kW/m^2, are found in this transition regime. From the local minima shortly after the end of flaming combustion, fluxes slowly increase and peak at about 60-90 minutes after the end of flaming combustion. Peak fluxes during glowing combustion are 60-160 kW/m^2. From that point, measured radiative fluxes slowly decrease until visible glowing diminishes and fluxes approach ambient levels. Peak calorimeter temperatures were typically 1000-1100ºC. Some thermal measurements from the I-beam test are shown in Fig. 5. The total duration for each test was in the range of 4-8 hours. The same general thermal trends were evident in the calorimeter and radiometer data.

A thermocouple in the exhaust flow was used to monitor the environment at that location. It also exhibited the same general trends: high temperatures during flaming, a sharp decrease, and a gradual increase and subsequent decrease. Figure 6 shows thermocouple readings in the exhaust for the four composite tests.

One of the tests, the I-beam test, is differentiated from the others because the air inflow was varied with time. The intent of the variations was to explore the conditions under which heat flux was maximized. The overall response of the radiometers and calorimeter due to changes in the airflow will be a balance between the net cooling effect of the airflow and the amount of available oxygen for consumption. There are two extreme theoretical conditions that are possible. If air flow velocity is

substantially high, a net cooling effect will cause extinguishment to occur. In the other extreme of a significantly low air flow regime, the system could become oxygen limited and reactions would extinguish. Figure 7 shows the velocity (top portion of the figure) and measured flux (bottom portion) from the three radiometers and calorimeter for the first 80 minutes of the experiment. The total experimental duration was approximately 240 minutes. As shown in Fig. 7, measured heat flux from the three radiometers and from the calorimeter did respond to the inflow conditions. Decreasing air flow in a stepwise manner from 10 to 25 minutes resulted in a corresponding increase in the heat flux to the calorimeter. Medtherm radiometer response was slightly decreasing over the same range. Near the calorimeter, in the lower region of the enclosure near the air inlet, the decreased airflow caused a decrease in the convective cooling effect on the calorimeter, resulting in a net increase in the thermal response of the calorimeter. In this location, we believe that the reduced effect of convective cooling was greater than the effect of limited oxygen availability; however, in the top portion of the enclosure, it is believed that the limited availability of oxygen is causing a net decrease in reactions and therefore net heat release, thus causing the net heat flux to the radiometers to decrease. Mostly similar but opposite trends are found as the duct velocity is increased from around 25 to 40 minutes. Upon the instantaneous velocity change, calorimeter fluxes decrease as air flow increases, and radiometer fluxes increase with increasing air flow. At the last incremental inflow step to about 80 cm/s, all flux measurements either decrease or the slope of the trend decreases. This suggests that the airflow at which maximum intensity occurs is at a fairly low inflow velocity, below 80 cm/s. This trend is partially replicated with the velocity increases at around 70 minutes. The Medtherm radiometers trend downward, while there is no discernable effect on the calorimeter.

Table 1 presents a summary of all tests in this series. Residual mass for the composite materials was found to be low, 10% or lower for all tests. Residual material was mostly partially oxidized fibrous layers, in the shape of the original material with evidence of swelling and degradation of structural strength. The total duration was somewhat subjective, as the precise point at which reactions cease is

difficult to ascertain. This was approximated during the tests through close visible examination of the residual materials for evidence of active glowing. The estimated pre-test surface area to volume ratio (SA/V) at the start of the tests is presented. The mean consumption rate is based on the initial mass and the total duration. For the composite tests where SA/V is reported, mean consumption increases with increasing initial surface area to volume ratio. This result is anticipated, as one would expect that oxidative reaction rates are dependent on the available surface area. The total duration of the test is influenced by the initial mass, as the I-beam test exhibited a lower mass and shorter duration than the other tests.

Table 1. A summary of various results from six tests.

Test	**Initial Mass**	**Residual Mass**	**Peak Flux**	**Flaming Duration**	**Total Duration**	**SA/V**	**Mean Consumption Rate**
	kg	*%*	*kW/m^2*	*min*	*min*	*cm^{-1}*	*g/s*
Wood Strips	40.8	-	220	-	90	2.4	7.56
Wood Panels	31.8	-	220	-	60	1.3	8.82
Composite Misc.	36.5	9.56	180	25	330	-	1.84
Composite Panels	38.5	2.59	175	30	420	2.0	1.53
Composite Strips	39.3	6.74	220	20	300	9.2	2.18
Composite I-beams	26.5	10.34	160	10	240	6.9	1.84

The wood tests burned much quicker than the composite tests, with peak fluxes slightly higher. They also exhibited flaming for a much longer duration, over most of the test. The wood also was observed to ignite much more readily.

4. DISCUSSION

Many aircraft fire scenarios take place on a runway with fire response crews in close proximity. Rapid mitigation response would be expected. Others take place in flight, in which case the ambient air flow velocities would be expected to be quite high. This work pertains more closely to a scenario where there is an unmitigated fire. This could occur in the event of a crash landing in a remote location.

However, a runway fire might be expected to behave similarly for the early duration until fire crews have time to respond to the incident.

By design, these tests are believed to be severe. The enclosure was insulated, providing thermal retention that would not be expected in an open environment. The composite pieces that were burnt had fairly high exposed surface area to volume ratio compared to what might be expected to be found on an intact aircraft. However, an aircraft crash will likely induce composite fracturing and expose fractured material surfaces to the fire. These tests did not involve large area panels, which may be found on some aircraft intact after a relatively soft crash landing, a consideration for future work.

TGA data suggest three phases of decomposition, as do the enclosure fire tests. These are interpreted sequentially as binder pyrolysis, binder char oxidation, and fiber oxidation. In the TGA data (Fig. 1), the pyrolysis phase is evident, as corresponding decomposition is found in air and nitrogen gas environments. After the pyrolysis phase, there is a shallow slope with additional mass loss in the nitrogen environment that is attributable to a char oxidation phase. The slope then steepens, suggestive of increased decomposition rate. This change in slope is attributed to the transition to fiber oxidation. In addition, there is an increase in heat generation as seen in DSC data. In the enclosure fire tests (see Fig. 5-7, Table 1), the binder pyrolysis took place from 10-30 minutes as flaming combustion consumed the pyrolysis vapors. Subsequently, an abrupt and significant drop in the heat flux is evidence of the transition to the char oxidation phase. The fraction of char oxidation is suspected to be minimized compared to the fiber oxidation as the measured heat fluxes in the enclosure environment increased to a peak at approximately 60-90 minutes after the end of the pyrolysis phase. Residual material was not found in the TGA air tests, whereas the 2-10% residual masses in the enclosure tests are believed to consist of un-reacted fiber and char due to local environment factors.

In surface oxidative reactions, the slowest physical mechanism restricting the reaction tends to be rate limiting. This is often expressed as a balance between transport and kinetic rates. A decomposition rate is transport, diffusion, or oxygen limited if the surface lacks oxygen to react. If there is plenty of

oxygen, it may be kinetically limited, and react faster with higher temperatures. In the I-beam tests where air velocity was varied, it is presumed that the increased velocity also increases oxygen availability. At the same time, it cools the materials near the air entrance by convection. The fact that mixed results were seen with changes in inflow velocity is suggestive of a transitional regime of reaction control. The magnitude at which this finding occurs is likely configuration dependent. Extrapolating to an actual accident scenario, one might expect the reactions to exhibit similar complexity. This suggests the need for kinetic and transport model fidelity if one is to accurately model this type of reaction.

The composite tests did not exhibit fluxes any higher than the wood tests. Peak fluxes from historical liquid hydrocarbon fuel fires have been found to be much higher [18]. Standard aircraft fuel loads can be consumed by an open fire in tens of minutes. Consequently, what we believe distinguishes fire environments involving carbon fiber epoxy materials is their duration. Up to 40 kg of material resulted in at least seven hours of continued reactions and heat generation. Additional material and additional types of materials might be expected to result in an even longer decomposition event. Because these tests are believed to be thermally severe compared to an accident scenario, we do not want to imply an expectation that an unmitigated scenario would result in findings of similar magnitude. We do expect, however, that these tests will be helpful in characterizing limitations on the potential environment from such an event for safety analyses.

5. CONCLUSIONS

Composite materials with carbon fibers and epoxy based binders decompose in an unmitigated fire through three mostly sequential phases: flaming pyrolysis of the epoxy, glowing oxidation of the char, and glowing oxidation of the fibers. The tests showed the highest intensity thermal environment in the flaming phase, and a fairly distinctive threshold between it and the next phases. The char and fiber combustion phases appear distinctive in TGA, but are more difficult to distinguish in the open fires due to these phases occurring simultaneously in different locations in the domain. However, the slower

decomposition rate and lower heat release rate of the char material found in the TGA and DSC data appears to coincide with a lower intensity combustion phase immediately following the flaming phase. Oxidative reactions appear to compete with convection heat transfer to limit the net reaction rate, resulting in peak heat flux at fairly low air flow rates. The duration of the decomposition reactions in these tests was remarkable, as long as 4-8 hours.

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Tables

Table 2. A summary of various results from six tests.

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Figures

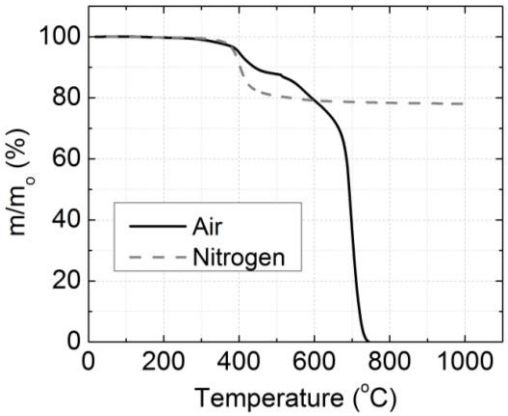

Figure 1. TGA experimental data for Cytec 977-3, a carbon fiber epoxy composite at 20°C/min in nitrogen and air.

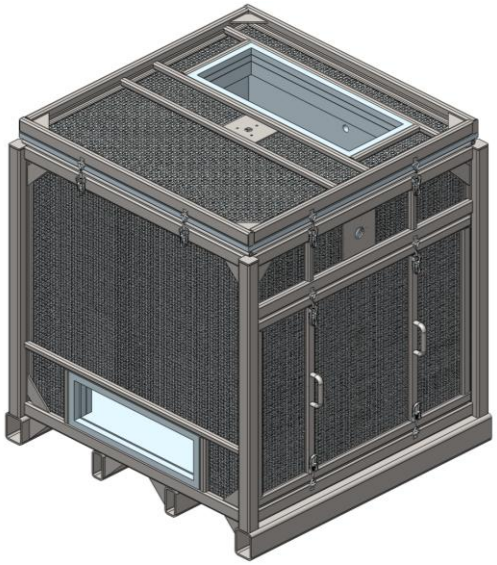

Figure 2. A schematic illustrating the test enclosure.

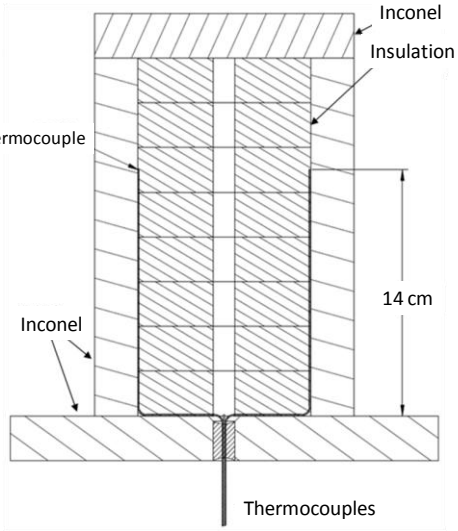

Figure 3. A cross-section cut-away drawing of the calorimeter.

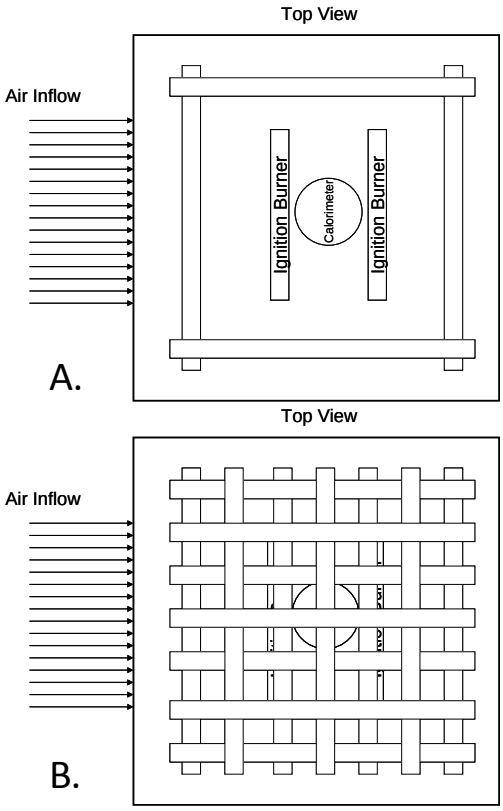

Figure 4. General layout concept for lower-layer (A) and upper-layer (B) I-beams

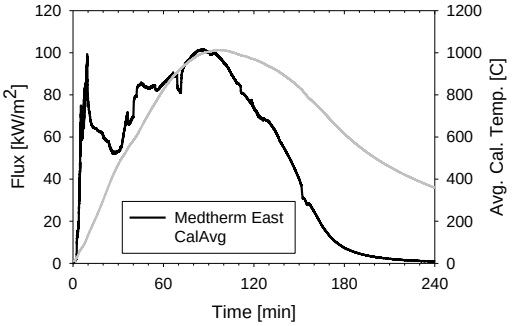

Figure 5. I-beam test; calorimeter and radiometer results.

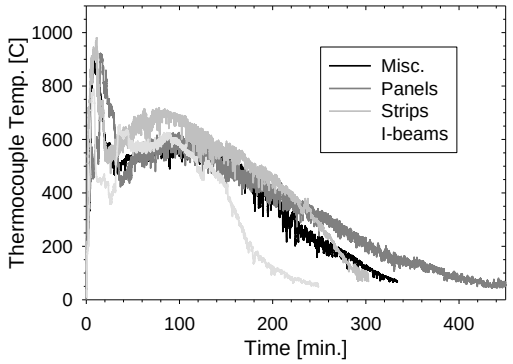

Figure 6. Exhaust thermocouple temperatures for the four tests.

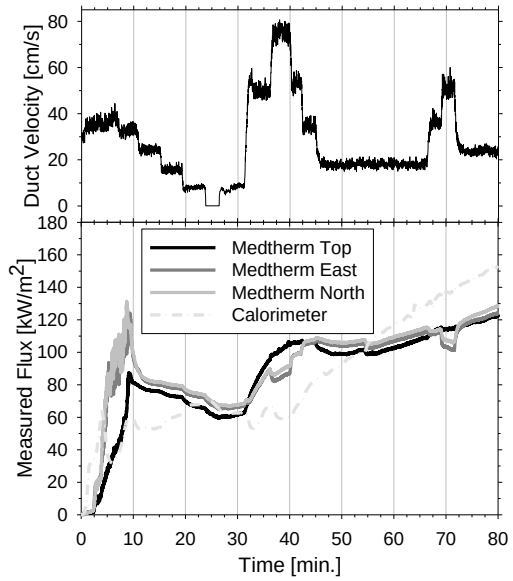

Figure 7. Velocity and measured flux from the I-beam test.

Figure Captions