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CARBON FIBER EPOXY HAZARDS FROM FIRE ENVIRONMENT TESTING

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ABSTRACT

Composites are increasingly used in the design of high performance systems because they can deliver high strength for low weight. Sandia National Labs is concerned with the thermal environment that is a consequence of carbon fiber epoxy materials involved in a fire. This interest relates to the need to understand the thermal environment of Sandia engineered systems that are deployed by the US armed services. Modern aircraft have increasing quantities of carbon fiber epoxy material in their construction. A series of tests has been performed during which the fire environment from burning composites was evaluated. A range of composite mass was employed, up to a few hundred kilograms. Even though the primary objective of the test series was to measure the thermal environment, ancillary instrumentation was fielded and secondary objectives were addressed to measure fire products that represent health hazards. Significant particulate yields were found in the various fire tests, mostly composed of what is classically termed soot. Sampling in the range of peak respiratory hazard did not suggest a significant amount of fiber in that size range.

INTRODUCTION

Carbon fiber epoxy composites are strong, light-weight materials that are finding increasing application in the design of aircraft. As more aircraft are constructed with these materials, they become a more relevant material to aviation fires. These materials generally displace aluminum as a structural and skin material. The behavior of aluminum and carbon fiber epoxy in a fire is significantly different. Aluminum normally melts and pools on the ground, and does not burn. The epoxy in carbon fiber epoxy materials will pyrolyze, contributing to the flaming. Residual material, or char, and the carbon fibers will react through a surface oxidation reaction, further contributing to the heat generated by the fire. Comparative work illustrating differences between aluminum and carbon fiber epoxy materials in the context of aviation fires has been done by Lopez et al [1].

Aviation carbon fiber epoxy fires are expected to typically involve aviation fuels, as the materials themselves are not prone to self-sustained ignition under normal environments. By quantity, jet fuel is the most abundant source of fuel used as an accelerant. But lubricating and hydraulic oils are also present, and other energetic sources may exist like rocket propellants, batteries, and other potential cargo. Real fires could involve a wide range of permutations of existing fuels.

Mouritz and Gibson [2] describe in Chapter 12 the current understanding of health hazards of composites in fires. The chapter leads with a description of several incidents of health problems observed after investigation and control teams responded to composite aircraft fires. The incidents were perplexing, especially since the responders were equipped with personal protective equipment (PPE). The type of health concerns were varied, and difficult to attribute. The exact cause of the issues remains uncertain, however the existence of fibers and toxicity of the products of combustion comprised two of the main suspected culprit sources. Hazards have been acknowledged, and the US Air Force has created a PPE standard for responders to fires involving composite materials. This is found in TO 00-80C-1, the aircraft recovery manual [3]. Respirators with P-100 cartridges, full body Tyvek suits, and leather/nitrile gloves are prescribed. Sandia prescribed similar guidelines. The FAA has also examined the hazards associated with

composites in general, and issued a detailed report on specific hazards [4,5]. A review of hazards (soot, fibers, char, and gases) has also recently been published, along with a computational analysis of the transport of fibers in the human respiratory airway [6,7].

The fundamental characteristics of carbon fiber epoxies are suggestive of the potential hazards. Good information on the chemistry of epoxies is found in May (1998) [8], one source for much of the background information detailed in this paragraph. Epoxy is a hardened polymer that forms when an epoxy resin and a hardener are mixed and reacted. Most domestic epoxies are cured at room temperature. Conversely, most aviation grade epoxies are thermoset, requiring a fixed heating profile to complete the hardening process. The most common epoxy resins are diglycidyl esthers of bisphenol A, or DGEBA. These have a fundamental monomeric repeating unit of $[C_{18}H_{20}O_3]_n$, and the monomer contains a single hydroxyl unit and two aromatic rings. The presence of aromatic rings is significant, because fuels with aromatic structures are generally expected to exhibit increased soot formation. The hardening compounds used in epoxy formation are typically treated as proprietary, and contribute to the uniqueness of the formulation. Amine groups (NH or NH_2) are functionally why the hardeners form polymeric reactions with the epoxy resins. A characteristic of thermoset epoxies is that the hardener contains aromatic ring structures. The presence of the aromatic rings in the hardener is also expected to contribute to the sooting tendencies of the material. In a fire, the amine groups in the hardener decompose to form other nitrogenated compounds. Fuels with significant quantities of nitrogen can be expected to produce toxins such as NOx as well as HCN. Other proprietary additions might exist in the epoxy matrix. Reduction in flammability is often a goal of these additions, in which case nanoparticles or halogens might be added at low concentrations. These generally reduce flammability by either increasing the energy required to release volatiles, or by augmenting the char formation pathway at the expense of the volatile release pathway. The carbon fibers are generally on the order of 5 μm diameter, and very long by comparison. They are strands primarily composed of carbon. Most are fabricated by a complex pyrolysis based process using polyacrylonitrile as a source material. These fibers are not a health hazard alone, but can be an irritant in contact with human tissue (i.e. skin, eyes, respiratory tract). They are too large to be a respiratory hazard at moderate levels because the human body naturally filters large particles. However, if they were to have an aerodynamic diameter in the 1-10 μm range, they might be respirable and hazardous. Such particles could be formed mechanically during an impact, or thermally through partial oxidation.

Based on descriptions above, the burn products believed to be the most hazardous for common carbon fiber epoxy materials involve the following:

1. Toxic gases including HCN, NOx, and CO
2. Polycyclic aromatic hydrocarbon (PAH) particulates, or soot
3. Partially decomposed fibers in the respirable range (1-10 μm)

Mouritz and Gibson [2] suggest that accident investigations have generally focused on the fibers as the source for the reported health issues.

Work with composite materials in fires over the past several years has been ongoing at Sandia National Labs [9-16]. The work has involved testing ranging from very small scale source materials to very large scale. The primary objective of the testing at Sandia has been focused on gaining understanding relating to the affect the carbon fiber epoxy has on the thermal environment. Unlike aluminum aircraft parts, the carbon fiber epoxy will participate exothermically in the fire. In the process of performing the tests, it was recognized that there were ulterior objectives that could be easily met for minimal extra effort. Not wanting to waste the opportunity, the tests were leveraged to explore secondary objectives. Because of the above described concerns and general lack of knowledge, it was thought that the products of these analyses would be of interest to the fire community.

This report details the ancillary (secondary objective) results from several composite fire tests with the purpose of providing quantitative data on the potential emission hazards from

carbon fiber epoxy fires. This report focuses on the PAH and fiber emissions. The details of the test configurations and diagnostic instrumentation will first be described to provide adequate context. Then the quantitative data extracted from the tests will be detailed in the results section. Had these objectives been primary to the goals of the project, a different approach would have been taken to collect more relevant data. The data obtained are believed to be significant and relevant, and will perhaps justify or help focus future efforts that are primarily aimed at studying the health effects of carbon fiber epoxy fires products.

METHODS

Results are taken from two test series, two tests within the test series each. The first test series involved between 25-40 kg of carbon fiber epoxy material. The material was placed in an insulated enclosure with a fan used to control the air flow. The carbon fiber epoxy was ignited with a gas burner, which was only on for a few minutes until the flames from the solid were clearly self-sustaining the fire. The other test series involved much larger quantities of carbon fiber epoxy material, and a much larger configuration. A mock B-2 bomb bay was constructed, and the top and bottom surfaces were fitted with composite panels. A 2-m diameter fuel pan was placed upwind of the center of the assembly in the cross-wind test facility (XTF), a 7.62 m (25 foot) square cross-section wind tunnel for fire tests. Table 1 summarizes details of each test. More detail can be found in the corresponding references.

Table 1. Parameters from the carbon fiber epoxy burn cases that were instrumented for soot and particulate emissions

Series	Test #	Accelerant	Composite Mass (kg)	Air Speed (mph)	Epoxy Material	Reference
Enclosure	5	Propane	38.5	Variable	Hercules 3501-6	[9,15]
Enclosure	6	Propane	39.4	Variable	Hexcel 3501-6	[9,15]
Mock B-2	A	JP-8	206	8	ACG/Umeco MTM45-1 Hercules 8551-7A	[16]
Mock B-2	B	JP-8	179	5	ACG/Umeco MTM45-1 Hercules 8551-7A	[16]

ENCLOSURE TESTS

In what is being termed the enclosure fire tests, between 25-40 kg of material was placed in a 1 meter internal cube. The cube was insulated, with a controlled air inlet at the bottom and an exhaust at the top. Inside it was instrumented with heat flux gauges, thermocouples and a calorimeter. Other instrumentation existed external to the enclosure. The enclosure design is illustrated in Figure 1. Gas burners were used to ignite the carbon fiber composite epoxy material, only for the 1-5 minutes of the test. Other than mass variations, the most significant variations from test to test were the lay-out and shape of the composites and some minor air flow variations. Major findings include the following:

1. The tests were extraordinary in duration. The flaming combustion lasted 10-30 minutes, but the glowing reactions extended the total test time to 5-8 hours.
2. Burn-out was higher than expected. Between 90-98% of the initial carbon fiber epoxy mass was consumed.
3. The rate at which air was introduced had a minor effect on the rate of consumption. Peak consumption occurred at a fairly low rate of air flow.

4. Heat flux measured from the test exhibited two peaks. The first occurred during flaming combustion. Heat fluxes dropped, and rose again during peak glowing combustion.
5. Peak heat flux was around 220 kW/m^2. This is fairly high, but typical of heat fluxes found during pool fire burns.

Because the test was in an insulated box, the thermal loss was limited. It is not clear whether it would be possible to sustain the same peak heat fluxes and mass loss in an open fire burn.

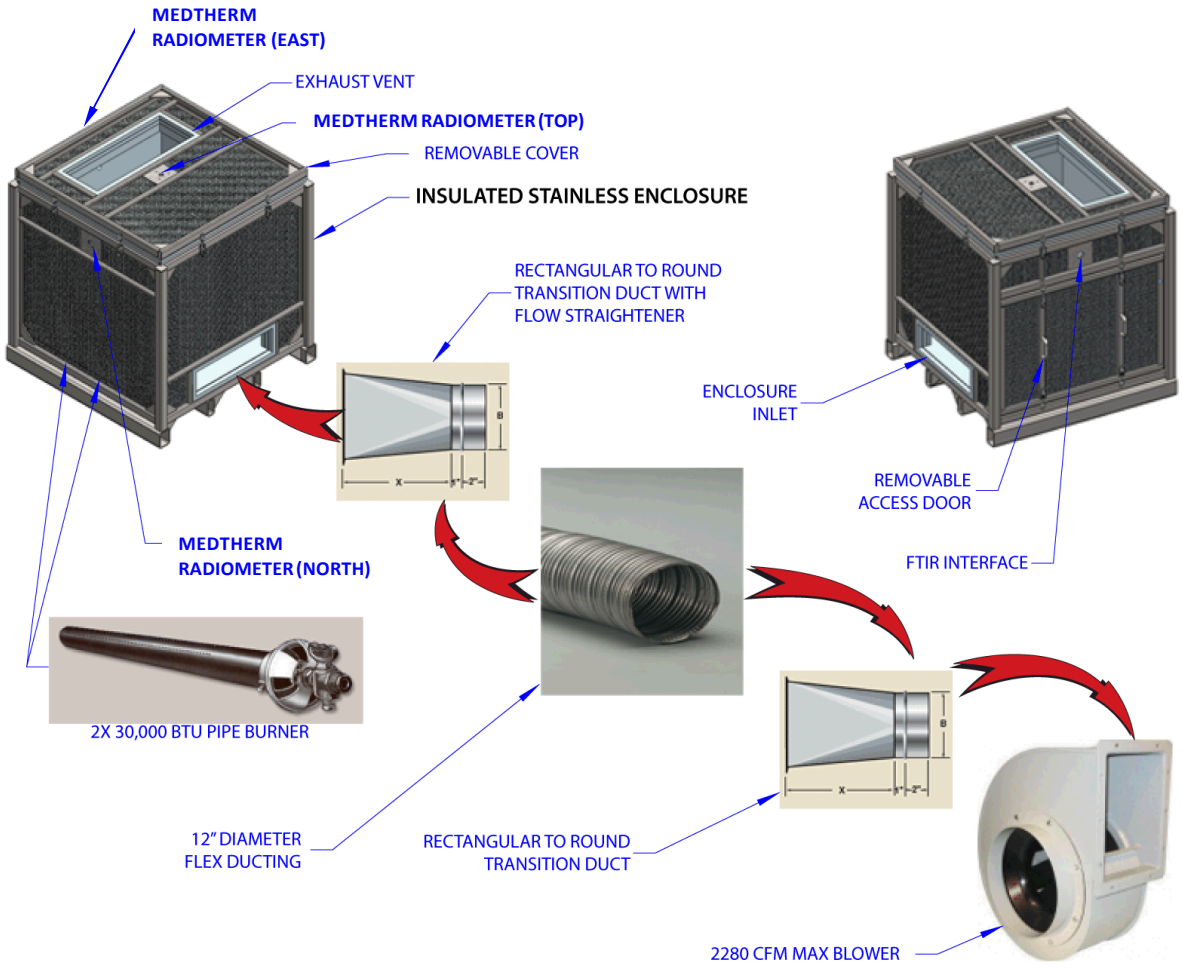

Figure 1. A schematic illustrating the test enclosure.

MOCK B-2 FIRE TESTS

This test series involved a 2-meter diameter pan of jet fuel on the ground below a structure that was designed to simulate the geometry and construction of a B-2 bomb bay. The bay area consisted of two cubes, with the major cubic dimension being 2.44 meters. A triangular section on the top is used to represent the complexity of the actual aircraft in that area. The cavity is roughly to scale in height and width, but 2/3 scale in length. The assembly was raised on six legs such that the bottom surface of the mock fuselage was 1.83 meters above the floor. The top and bottom surfaces were made from carbon fiber epoxy, surfaces which were changed

out for each test. Calorimeters and heat flux gauges (green and red in Figure 2 respectively) were located throughout the enclosure and served to quantify the heat fluxes and fire environment in the enclosure during the test. The legs were water cooled, and the metal side-walls were insulated on the outside. Primary variables differentiating the two tests were the distribution and quantity of composite material, the wind speed imposed in the cross-wind test facility (XTF) wind tunnel during the test, the location of the fuel pan, and whether the lower bomb bay doors were open or not. Primary findings include:

1. The low-wind case (Test B) produced very high heat fluxes >300kW/m^2. This was believed to be caused by the partial collapse of the composite into the fuel pan, and the subsequent vortical interaction between the wind, panel, and fuel.
2. Panels were consumed to varying degrees, primarily related to their proximity to the fuel fire. Up to about 50% of the original composite mass was recovered post-test.
3. This configuration did not exhibit significant glowing combustion once the fuel fire was extinguished. The low-wind scenario exhibited glowing decay from within the enclosure for about 5 minutes after the fuel pan was drained, but no indications suggest that significant reactions were able to sustain beyond that point.

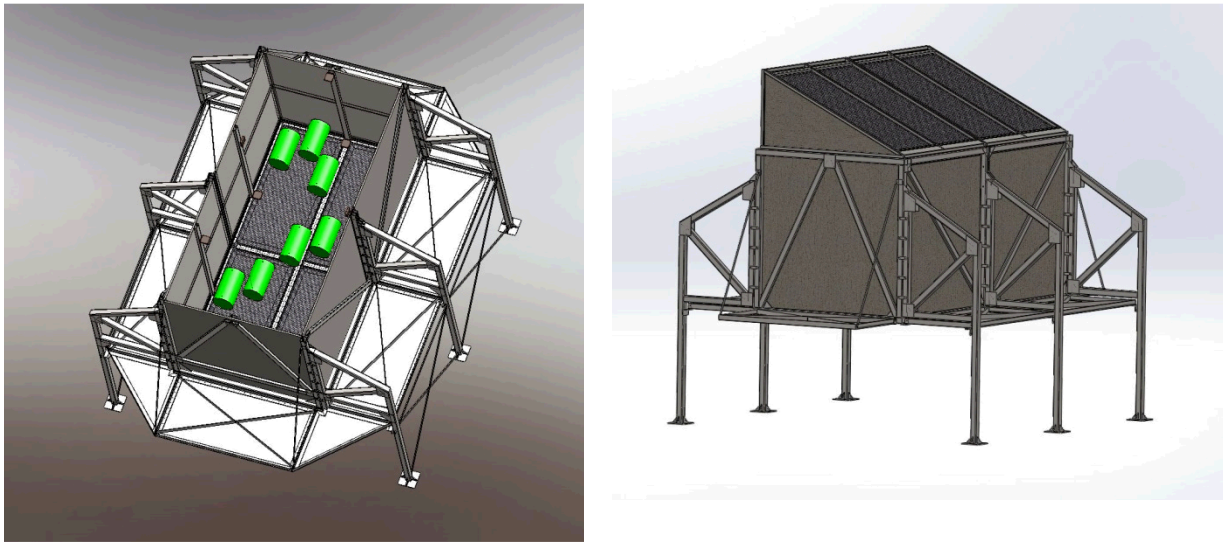

Figure 2 Two views of the mock aircraft design.

SOOT DIAGNOSTICS

Several methods were employed to measure sooty emissions from the burns. In the enclosure test series, a pneumatically actuated, aspirated collection grid was located above the exhaust. During peak smoke emission (flaming combustion), the sampling system was actuated, exposing the collection grid through the baffles and into the smoky fire. Samples were otherwise maintained in an inert environment, cooled by a nitrogen flow. Particulate samples were recovered following the tests, and analyzed with a transmission electron microscope (TEM). A figure depicting the sampling instrument is found in Figure 3 (from Jensen et al., 2005 [18]).

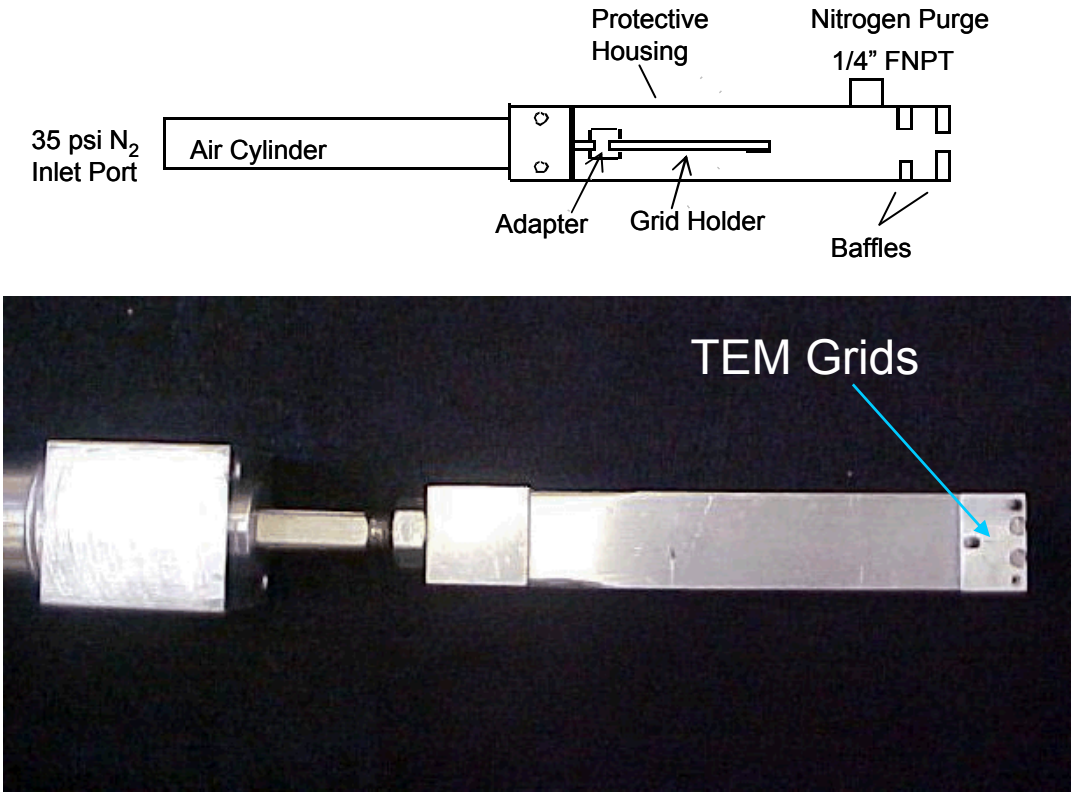

Figure 3. A schematic of the thermophoretic sampler for collecting soot from the flame, and photograph of the grid holder attached to the pneumatic cylinder (from [14]).

Two methods were employed for measuring soot from the mock B-2 tests. A cascade impactor was used to sample the product gases down-stream of the fire. Cascade impactors use a vacuum pump to draw effluent through a cascading series of collection filters. Aerodynamic separation results in a series of filter papers with deposits ranging by aerodynamic size. The filter papers can be pre- and post-weighed to quantify the mass of particulate matter in a given size range. A figure depicting the instrument is found in Figure 4. This instrument was located 3.66 m above the ground at 2.29 m from the north wall that constitutes the side of the XTF facility (about half-way to the center of the facility). The instrument was located about 18 m downwind from the last inlet screen to the facility, just up-wind of the make-up air grating, or 9 m downwind of the mock fuselage down-wind legs.

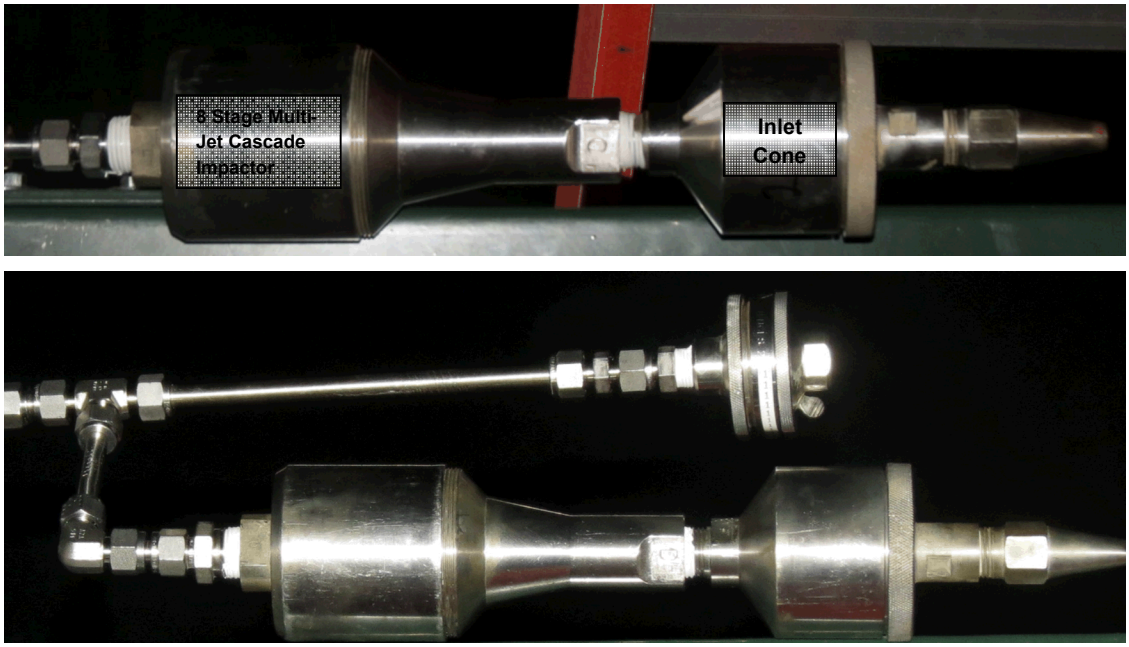

Figure 4. Photographs of Anderson Mark III Cascade Impactor used to measure aerodynamic particle size distribution

The second method used was more relevant to exposure hazards. After the test had concluded, air flow was left on for at least an evening to allow the structure and surroundings to cool. Technical support staff with appropriate PPE entered the test cell to clean the post-test environment. One of the staff wore a personal breathing zone sampling system that continuously sampled the exposure environment. Collected material was then tested according to OSHA ID-196 for carbon black content. The 2005 American Council of governmental Industrial Hygiene (ACGIH) 8-hour threshold limit values (TLV) for carbon black is 3.5 mg/m^3. The 2005 American Council of Governmental Industrial Hygiene (ACGIH) Threshold Limit Values (TLV) for Chemical Substances and Physical Agents and Biological Exposure Indices (BEIs) is the DOE's regulatory occupational exposure limit (OEL) as identified in regulatory requirement 10 CFR 851.

FIBER DIAGNOSTICS

Similar to the above, the aspirated collection grid and TEM analysis that was used to sample soot in the enclosure test series might have been expected to collect fibers. None were observed. This test series sampled during peak flaming combustion, which is probably too early to sample significant fibers if they are ejected as part of the burn process. Fibers were observed to decompose later in the test, but fibers in the respirable range are not possible to observe with the naked eye except in high concentrations.

Also similar to the above, the staff member who cleaned the mock B-2 post-test environment was outfitted with a pump and filter to sample for fiber testing. The NIOSH 7400 (OCM) synthetic vitreous fibers continuous glass fibers sampling method was followed. The 2005 American Council of governmental Industrial Hygiene (ACGIH) 8-hour threshold limit values (TLV) for synthetic vitreous fibers continuous glass fibers is 1 fiber per cubic centimeter for an 8-hour period.

RESULTS AND DISCUSSION

SOOT FROM ENCLOSURE TESTS

The soot that was extracted from the fire testing in the 1 meter cubic enclosure is believed to be due almost exclusively to the unburnt products of epoxy pyrolysis based on their time of extraction. Both of these images (Figure 5 and Figure 6) were taken with identical scale, 0.2 μm. The most significant aspect of these two that was noticed in the review of the images is the difference in size of the incipient spheres that make up the soot agglomerate. The particles from test 5 are about half the size of those from test 6. This is probably due to the comparative enhancement of one of two competing processes: the soot oxidation, and the surface growth of soot. It is generally understood that incipient spheres (starting at about 10 nm) will agglomerate, but will undergo further interactions (surface growth or oxidation) that change the size. Spheres will agglomerate to form the complex structures in the images. Soot agglomerates are typically found in the micron size range. These agglomerates fit that category. The samples were taken during peak flaming at the same height above the outlet. Residence times are thought to be similar. The differences are probably due to variations in the transient history of the particles in terms of temperature, and species concentrations. The differences between Hexcel and Hercules epoxy (same company, bought out by another) and the differences in the lay-up of the composites are suspected to be secondary to this finding.

Nothing exceptional is noted with respect to the shape, size, or nature of the soot from the burning of epoxy. Extensive analysis of soot from JP-8 pool fires has been performed with the same instrumentation in the past, and many of the images found in Jensen et al., 2005 [18] can be compared to these. Imaging of the soot from epoxy combustion suggests it is comparable to soot from JP-8, or other fuels.

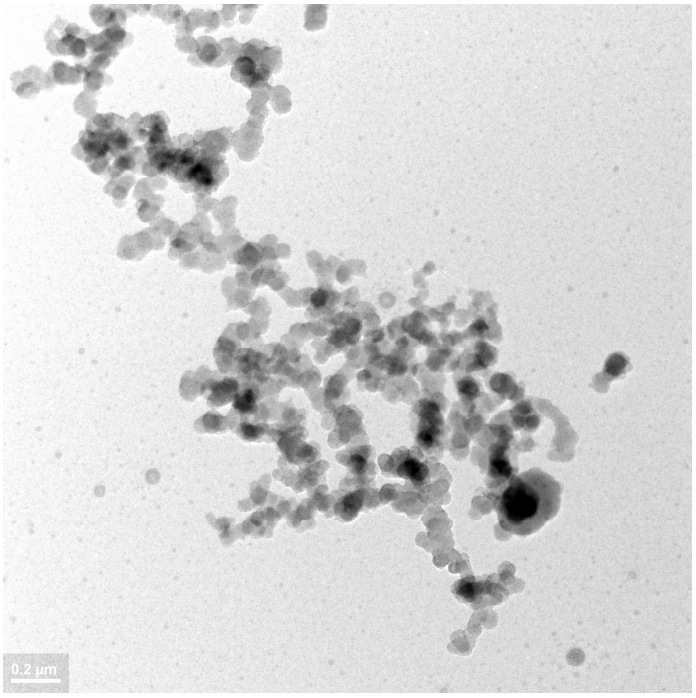

Figure 5. Test 5 TEM image of a soot agglomerate.

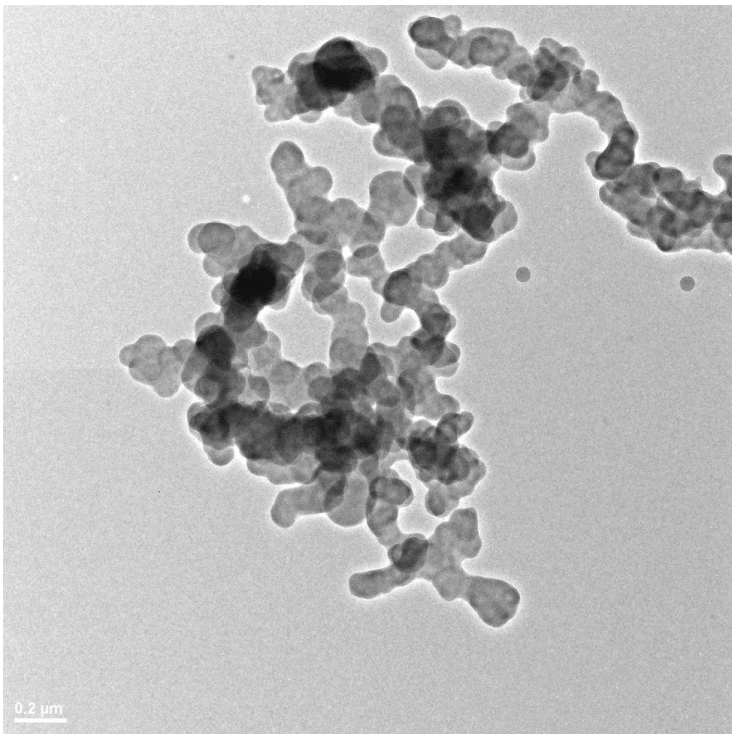

Figure 6. Test 6 TEM image of a soot agglomerate.

SOOT FROM MOCK B-2 TESTS

Personal breathing zone sampling during the clean-up was taken during Test B from this test series. Total carbon black was sampled during a standard cleaning shift. The total carbon black 8 hour time-weighted average (TWA) was 0.478, mg/m^3, whereas the regulatory limit is 3.5 mg/m^3. This suggests that the standard for carbon black was not exceeded for the duration of the test clean-up.

Test B from this test series was also instrumented with the cascade impactor. Particles were collected over a period of 11 minutes starting 10 minutes into the test. The duration was determined during the test, and the sampling was concluded when the flow rate was reduced by 10% from the starting value. Thus the flow rate varied between 72-65 lpm during the test. A total of 760 liters were sampled, and 38 mg were recovered from within the impactor. It is believed that the primary source for the soot is the JP-8 fuel, however the soot from the burning composite material is also believed to be a contributing factor. A similar set-up was located at half the elevation, but the filter paper did not collect significant product at that elevation. The particle distributions were based on custom collection size ranges, and are therefore binned according to the irregularly spaced bins corresponding to the instrument. Figure 7 shows the particle measurements based on the mass. In this and the next figure, the 20 µm bin is truncated for improved visibility of the lower-limit bins. It actually extends to 100 µm+, and was determined by sampling a total weight filter and subtracting the difference from that sampled in the impactor (63 mg versus 38 mg respectively). On a pure mass basis, the 20-100 µm range constituted nearly 40% of the mass. However, if the mass fraction data are normalized by the particle size range in µm, the distribution in Figure 8 shows that the peak of the mass distribution is closer to 0.5 µm. The distribution in Figure 8 suggests a relatively smooth distribution, much like expected from this class of data. The mean soot concentration based on the above data at the point of collection

was 83 mg/m^3, significantly higher than the 0.478 mg/m^3 measured in the post-test clean-up exposure.

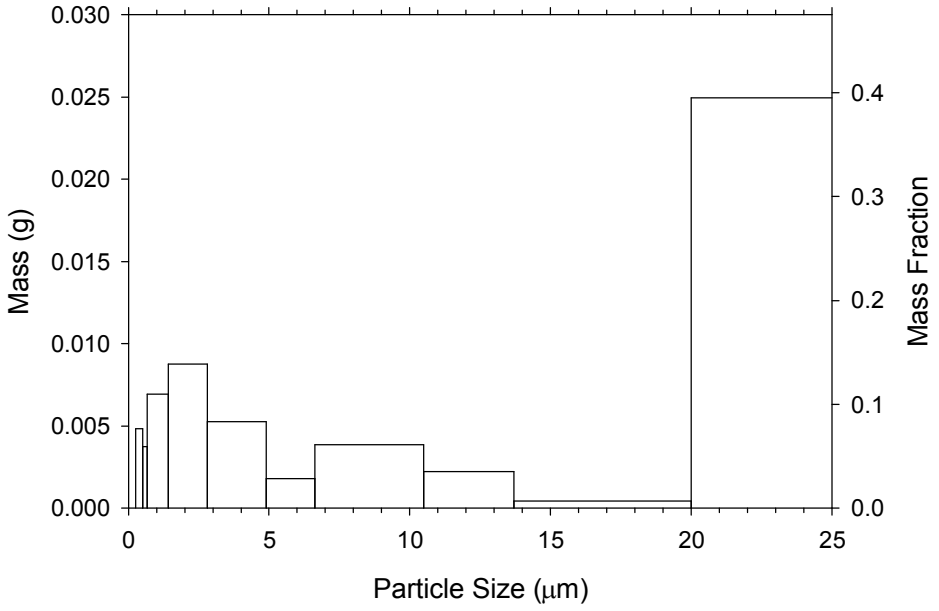

Figure 7. Mass of particles from the cascade impactor.

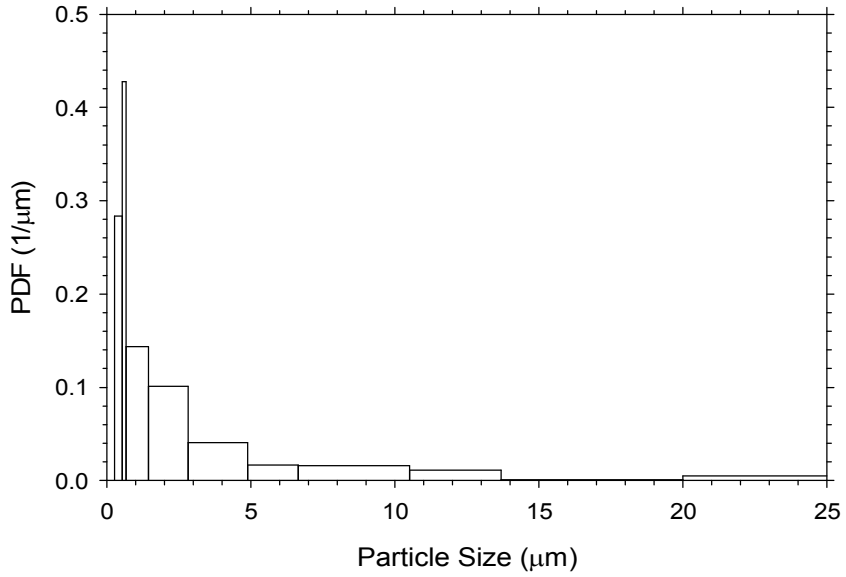

Figure 8. PDF of the particles collected in the cascade impactor.

The chronology of the sampling in this test compared to the previous test in the insulated enclosure is significantly different. The enclosure test sampled particulates during the peak epoxy flaming time. Sampling for the mock B-2 test occurred at 10 minutes, well after peak epoxy flaming on the lower panels. There was no adequate view of the top panels, as a smoke layer obscured the top of the assembly. It is anticipated that they burned later than the lower panels, but it is not possible to present precise details on this aspect of the burn. The soot collected from the cascade impactor is expected to be mostly from the jet fuel. The soot collected

during clean-up is probably based on residue in the facility, which likely includes contributions from the epoxy as well as the jet fuel.

FIBERS FROM MOCK B-2 TESTS

Personal breathing zone fiber sampling and analysis produced the data found in Table 2. Measured fiber count is significantly below the 8-hour time-weighted average OEL. This sampling was unfiltered, so it suggests that the respirators in the particular case of this test series might not have been a requirement to maintain worker safety. This does not constitute a recommendation. It suggests that for this particular test series that was designed as a mock aviation fire that the quantity of fibers measured after the burn did not constitute a significant hazard. Even though this test was quite large with significant quantities of carbon fiber epoxy material, it was still considerably smaller in length scales and in source material mass than may be found in an actual accident. It would be expected that a larger fire might yield increased fiber emissions, and represent an increased hazard. More data would be necessary to relax the current PPE recommendations.

Table 2 Fiber measurements from the mock- B-2 test clean-up

Test	Measured Fibers [Fibers/cm^3]	Occupational Exposure Limit [Fibers/ cm^3]
A	0.07967	1.0
B	0.07502	1.0

These fiber counts may be compared with similar sampling by Mahar [19], in which fiber counts were as high as 0.56 fibers/cm^3. Closer examination of the fibers in that work did not suggest that they were of carbon in origin. Other samples resulted in nearly undetectable quantities of fiber.

One potential reason fiber counts are significantly lower than the OELs is that the clean-up procedures were designed to reduce the hazard. The crew first vacuumed over the entire area with a HEPA vacuum. Then they took moist towel wipes and wiped down exposed surfaces. After this, they handled the panels by weighing them and bagging them for disposal. The extent to which these procedures reduced the exposures compared to the exposure had they not been used is not known.

GENERAL DISCUSSION

Because these data were secondary to the tests, they are not as complete as would be expected were this the primary focus of the effort. Despite the data being sparse and insufficiently complete to make recommendations on the safety protocol and hazards associated with this type of fire, the data still contribute to the general knowledge on material hazards. The mock B-2 fire tests were relatively unique in the quantity of composite involved in the burn. Other than accident scenarios, no other testing at the same scale has been found in the open literature. These data are thought to be significant in that regard.

The fiber yield was not found to be sufficiently high to be of significant concern. This may be partially due to the vacuuming and wet wiping done during clean-up. This was surprising in light of the description of concerns presented in this regard in Mouritz and Gibson [2], although it is not expected that the experiences described therein included such mitigation measures. Some of the health issues detailed could be attributed to sources other than the fibers, like toxic gas inhalation. Additionally, an aircraft fire involves more than just carbon fiber epoxy materials. It is possible that the presence of another material contributed to the health effects described. Since the exposures reported to have caused issues for firefighting responders were not quantified, it is

impossible to make any conclusions this way other than to raise it as an issue. If one were to consider relaxing the PPE requirements for response to composite aircraft fires, it would be important to consider the full range of materials that could contribute to the hazards. Even if carbon fiber epoxy composites are shown to not be a significant hazard to the level that the response teams require PPE to existing standards, it is important to qualify to the other material found on the aircraft as well.

There is need for additional information on the hazards of fire products from carbon fiber epoxy materials because these have been identified as potential concerns in aviation crashes. In particular, it would be helpful to have predictive models that could provide soot yield or soot source terms for burning epoxies. And it would be helpful to have more complete data on the formation of respirable fibers formed from carbon fiber oxidation. Mouritz [6] suggests that information on the toxicity of char materials is nearly non-existent. Additional data on toxic gas yield from the burning of these materials would also be helpful.

This paper does not present the data collected on the gaseous emissions from the composite burns. The FTIR used in the insulated enclosure test series did not resolve minor species. The most relevant aspect of those data was the CO yields, which were not particularly surprising. CO is released during flaming and glowing combustion, and generally follows what is believed to be the reaction rate in terms of magnitude. More information on this feature of the test can be found in Brown et al., 2011, 2013 [9,15]. Mouritz [6] presents a good review of gas product toxicity, and can also be looked at for additional guidance.

SUMMARY AND CONCLUSIONS

Secondary instrumentation was fielded for two test series where significant quantities of carbon fiber epoxy composite materials were burned. Soot from the epoxy burning appears much like soot from liquid hydrocarbon fuel under TEM imaging. A cascade impactor extracted soot particle distributions, suggesting the most likely particles are found in the 0.1-1 μm range. Fiber and carbon sampling taken from a member of the clean-up crew suggested that the post-test hazard was not high enough to exceed standard exposure thresholds. These results add to the existing body of work on the health hazards of carbon fiber epoxy materials when they are a significant component of a fire.

FUTURE WORK

As additional sampling is done during the fire test and clean-up for experiments and accidents involving carbon fiber epoxy aviation composites, additional knowledge about the safety of the various hazardous products of these materials will be known. Data presented in this study suggests the potential for relaxing the existing PPE standards. It is advisable to conduct further sampling to better gauge the range of yields that can be obtained to make sure that the current findings hold under a wider range of conditions. Post-test hazards in these tests were not seen to be particularly serious, however emissions from the active plume were above respiratory limits.

ACKNOWLEDGMENTS

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