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ANALYSIS OF PARTICULATE AND CHEMICAL RESIDUE RESULTING FROM EXPOSURE TO BURNING AND ABRADING COMPOSITE MATERIALS

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■ **Air Force Materiel Command** ■ **United States Air Force** ■ **Tyndall Air Force Base, FL 32403-5323**

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14. ABSTRACT

Five tests involving cutting or burning a composite panel were undertaken in order to study the deposition of potentially hazardous particles and chemicals onto fabric samples that occurs during these processes. This was undertaken in order to understand the hazards that first responders may encounter when responding to incidents involving crashed or burning aircraft composed of composite materials. The composite panels tested were typical of material used in current aircraft. Panels were either burned by placing them in a pool of burning military specification jet fuel, or cut with a rescue saw typical of the equipment used by first responders. The fabric samples exposed to these events were materials commonly used on the exterior of protective equipment worn by Air Force firefighters. After exposing the fabric samples, half of the samples were vigorously washed with water to simulate cleaning and decontamination that firefighters might perform on their equipment. Sampling techniques were then employed to remove and study the particulates and chemical residue present on the fabric samples. Of particular interest were carbon fiber fragments, as these can potentially cause lung problems when inhaled, and polycyclic aromatic hydrocarbons (PAHs), as many of these chemicals are known or suspected carcinogens.

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1. SUMMARY

Five tests involving cutting or burning a composite panel were undertaken in order to study the deposition of potentially hazardous particles and chemicals onto fabric samples that occurs during these processes. This was undertaken in order to understand the hazards that first responders may encounter when responding to incidents involving crashed or burning aircraft composed of composite materials.

The composite panels tested were typical of material used in current aircraft. Panels were either burned by placing them in a pool of burning military specification jet fuel, or cut with a rescue saw typical of the equipment used by first responders. The fabric samples exposed to these events were materials commonly used on the exterior of protective equipment worn by Air Force firefighters. After exposing the fabric samples, half of the samples were vigorously washed with water to simulate cleaning and decontamination that firefighters might perform on their equipment. Sampling techniques were then employed to remove and study the particulates and chemical residue present on the fabric samples. Of particular interest were carbon fiber fragments, as these can potentially cause lung problems when inhaled, and polycyclic aromatic hydrocarbons (PAHs), as many of these chemicals are known or suspected carcinogens.

Fabric exposed to burning composite was found to have a significant quantity of soot particles deposited on the fabric surface. Washing the fabric with water removed approximately two-thirds of the soot particles from the fabric, and tended to preferentially remove the larger soot particles. No carbon fibers were located on fabric exposed to burning composites. Similarly, no PAHs were identified by chemical sampling techniques.

Fabric exposed during the cutting of composite panels was found to have a significant quantity of carbon fiber particles deposited on the fabric surface. Washing the fabric with water removed the approximately ninety-five percent of the carbon fibers from the fabric. No PAHs or other organic contaminant products were identified by the chemical sampling techniques.

2. INTRODUCTION

2.1. Composite Fire Hazards

Fires involving composite aerospace materials have been suspected as a source of unusual hazard. The hazards were originally suspected as a source of contamination and a danger to electronic equipment from fugitive reinforcing fibers released as the polymer matrix of the composite burned. National Aeronautics and Space Administration (NASA) initiated a study of reinforcing fiber release from graphite-epoxy composite, graphite-Kevlar, glass-graphite, and boron-graphite hybrid composites due to combinations of burning and impact. The results were inconclusive regarding threats to electronic equipment.[1] Follow-on experiments were performed that exposed operating electronic amplifiers to cut fibers and fibers from burning composite to gauge the dangers composite smoke and fugitive fibers presented toward electronics and avionics. The measured vulnerabilities to stereo amplifiers from cut virgin fiber and from fiber produced from burning graphite-epoxy composite were in close agreement.[2]

Composite aircraft accidents have generated reports of skin and respiratory irritation among firefighters and post-crash recovery personnel. A Royal Air Force (RAF) Harrier GR5, containing carbon/epoxy composite crashed in Denmark in 1991. The recovery team suffered eye and skin irritation and respiratory difficulties.[3] Firefighters responding to the 1997 crash of an F-117 reported nausea, headache, eye and skin irritation, and respiratory difficulties on exposure to the smoke from the aircraft.[4] The emissions from burning composite are believed to generate dangerous materials in the form of toxic vapors and gases, smoke particles and fugitive fibers.[5] A team (Courson *et al*.) from the Armstrong Laboratory Toxicology Division collected and analyzed smoke from burned composite. They measured smoke particle diameters and extracted, identified, and quantified a number of organic compounds, some of which were toxic or carcinogenic and some suspected of such threats. Their study did not identify or examine fugitive fibers.[6]

A large-scale observation of burned composites was conducted at the Air Force Research Laboratory's (AFRL) Tyndall Research Site, Tyndall Air Force Base (AFB), Florida. . This involved a team from the Air Force Institute for Environmental Safety and Occupational Health Risk Analysis, AFRL, the Mississippi Air National Guard, and the RAF to burn sections of composite wings, collect samples from the combustion solids and gases, and sample occupational threats encountered by post-fire recovery personnel. Both NIOSH-style occupational health sampling and EPA-style particulate collection equipment were adapted to sample smoke particulates in this study, and special emphasis was placed on particles in the 1-2.5 μm diameter range. Vapor-phase organic compounds were collected with solid-phase microextraction samplers (SPME) and qualitatively evaluated by gas chromatography/mass spectrometry (GC/MS).[7] Much of the collection equipment used for the combustion solids was designed with the concept that the particles would be roughly spherical in shape. Therefore, it is not known whether the sampling systems used in this study exhibited a bias against long particles.

AFRL conducted a study of effects on firefighter equipment and post-fire cleanup based on exposure of bunker suit fabrics to burned composite. Soiled coupons of bunker suit fabric, as would be used in close approach suits, were cleaned with water washing, brushing, vacuum cleaning, and using sticky lint rollers. Water washing and sticky rollers were found to be promising candidates to clean equipment exposed to particulate from burned composite.[8]

2.2. Contamination in an Operational Event

On 23 February 2008 a B-2 aircraft crashed on take-off from Andersen AFB, Guam. The airframe utilized large quantities of advanced composite material (ACM) and was largely consumed in the resulting fire. The Andersen fire department experienced difficulties in extinguishing the fire, having to request assistance from off-duty firefighters and firefighters from neighboring facilities. The fire required a larger-than-anticipated quantity of water and firefighting agent to extinguish and raised issues regarding appropriate firefighting techniques and agents for fires involving ACM.[10] Reports from Andersen AFB and the AFRL Advanced Composites Office indicate the fire department and aircraft maintenance squadron suspected contamination on their personal protective equipment (PPE) clothing and equipment. Lacking clear guidance on decontamination of the respirators and protective clothing used in ACM fires, all such equipment was reportedly discarded and replaced.

2.3. Equipment Contamination Studies

In response to the difficulties encountered in the 2008 B-2 crash, AFRL's Airbase Technology Division undertook a four-pronged study of (1) Damage Mitigation from Small Fires, (2) Extinguishing Burning Composites, (3) Penetrating and Overhauling Composites, and (4) Post Fire Decontamination of PPE and Equipment. Post Fire Decontamination of PPE and Equipment were studied by exposing coupons of fabric identical to that used in close approach bunker suits (silver suits) exposed to burned composite material. The contaminated coupons were cleaned with four trial techniques: brushing, vacuum cleaning, sticky roller pads, and water washing. Sticky roller pads and water washing produced cleaning results deemed acceptable, and water washing was recommended as a decontamination technique as it was expected to be more readily available than sticky pads.[8].

2.4. Scope

The current research undertook to extend the study of post-fire decontamination by studying particulates and organic chemicals associated with actual composite and jet fuel fires versus contact with burned composites.

The experiments reported herein intend to determine whether or not carbon fibers and PAH compounds are generated by a model ACM fire and deposited onto typical PPE materials, and to estimate the ease of removal of these materials by impromptu decontamination techniques. These experiments were intended to determine satisfactory decontamination procedures for PPE and other equipment.

3. METHODS, ASSUMPTIONS, AND PROCEDURES

A total of five test events were conducted as part of this test series. In four of these tests, samples of fabric from firefighter personal protective equipment (PPE) were exposed to smoke and soot from a burning composite panel. One additional test was conducted in which the fabric samples were exposed to dust and debris from a composite panel being cut with a rescue saw. All tests were performed at the Air Force Research Laboratory's Test Range I fire test facility located on Tyndall Air Force Base. Exposed fabric samples were then taken to an analytical laboratory facility also on Tyndall AFB for physical and chemical analysis of the particles and chemicals that were deposited on the fabric during the tests. Tests were conducted at intervals of a few days based on weather and other timing considerations. This allowed the test procedure in successive tests to be modified and improved based upon preliminary analytical results. The system of measurement used throughout this study was metric.

The composite panels used were rectangular samples of 20-ply IM7/977-3 that were cut to dimensions of 15 × 30 cm (6 × 12 in). This material is typical of composite materials currently used in composite aircraft.

The fabric samples used were samples of Newtex Z-Flex Silver P-202 that were cut into 2.5 × 2.5-cm (1 × 1-in) squares. This is a thin, woven fabric that has a layer of aluminum bonded to one face of the material. It is typical of the fabric used in the outer layer of PPE worn by Air Force firefighters (for example, see the firefighter visible in Figure 1). These 2.5-cm (1-in) fabric squares are referred to as test coupons for the remainder of this report.

3.1. Fabric Exposure Procedure

All tests were conducted inside a standard twenty-foot shipping container (Figure 1). The interior dimension of this container was 5.9 m long × 2.4 m wide × 2.4 m tall (19.3 × 7.8 × 7.8 ft). Testing in this confined space was intended to concentrate the particles and chemicals emitted by the composite material around the test coupons thereby enhancing the potential deposition of these materials onto the fabric.

Figure 1. Shipping Container used during the Test Series

3.1.1. Test 1

The first test involved burning a composite sample. A steel pan and holder was custom built for this procedure. It consisted of a square pan 30 × 30 cm (12 × 12 in) with 15 cm (6 in) tall sides. A metal frame was attached to the pan that held the composite panel above the pan, at an approximately 45-degree incline. The pan was placed at one end of the shipping container. The pan was then filled with 2 L of water, followed by 1 L of JP-8 jet fuel. One liter of jet fuel was used as it was estimated that this would result in the desired burning time of approximately 5 min. The water served to elevate the fuel to the proper level in the pan, as well as prevent the pan from warping or otherwise being damaged from the intense heat of the burning jet fuel. This apparatus, containing a composite panel and filled with water and fuel, is displayed in **Error! Reference source not found.**.

A total of 60 test coupon samples were used in Test 1. Two pieces of white poster-board measuring 20 × 40 cm (8 × 16 in) were used to mount the test coupons. Double sided tape was used to attach 30 test coupons to each piece of poster-board. The samples were arranged in a five × six grid, with approximately 1.3 cm (½ in) between each test coupon. For future identification and reference, the test coupons were numbered 1–60 by making a notation below each square on the poster board. One poster-board was placed on the floor of the shipping container approximately 1.5 m (5 ft) from the pan bearing the composite sample. The second poster-board was placed on the floor approximately 3 m (10 ft) from the pan. Figure 2 presents a photo of one of the poster-boards with attached test coupons used in Test 1.

Figure 2. Composite Panel Placed in the Sample Holder (left); Array of 30 Test Coupons Placed on the Floor of the Shipping Container (right)

After positioning the pan and the two poster-boards, the jet fuel was ignited using a propane torch, and the doors to the cargo container were shut. The fire from the burning fuel could be seen through cracks in the cargo container doors, and was observed to burn for approximately 5 min. The doors were kept shut for a further 10 min to give time for smoke and soot particles settle out onto the test coupons .

After opening the doors, each poster-board was cut in half, resulting in a total of 4 arrays of 15 test coupons each. One of the arrays that was originally placed 1.5 m from the composite panel, and one of the arrays that was originally placed 3 m from the composite sample were removed from the shipping container and subject to washing as follows. The two poster boards were first mounted vertically. A rubber garden hose with a typical spray attachment set to the widest spray setting was held at a distance of 0.9 m (3 ft) from the poster-boards. Water was then directed at each board for a total of 30 s, using a side to side motion to sweep water across the boards. This was intended to simulate the sort of impromptu cleaning or decontamination that firefighters might perform on the gear after responding to a firefighting incident. After washing, the two poster-boards were positioned horizontally on a table and allowed to air-dry for one-half hour.

Each of the 4 poster-boards, each containing an array of 15 test coupons, was then subject to sampling for particulates and chemical residue as described below. Within each array of 15 squares, 5 were subject to sampling for particulates, and 10 were subject so sampling for chemical residue. Thus a total of 20 test coupons were sampled for particulates and 40 test coupons were sampled for chemical residue. After sampling, the materials were transferred to the analytical laboratory for analysis as described in Sections 3.2 and 3.3.

American Clean Stat Tackymat “sticky pads” (product no. 183602WW-460) were used for particulate sampling. These are essentially very thin sheets of white plastic with strong tape-like adhesive on one side. The Tackymat sheets were cut into a large number of small squares measuring 1.3 × 1.3 cm. The sticky pad squares were then pressed firmly, sticky side down, onto the test coupons in order to transfer soot particles from the fabric onto the sticky pads. The sticky pad squares were then removed and pressed, sticky side down, onto standard microscope slides (Diagger frosted, precleaned microslides, catalog no. EF15975F). Two 1.3 × 1.3 cm sticky pad squares were used to sample different locations (roughly two opposing corners) of each 2.5 × 2.5-cm test coupon. Two sample blanks were also obtained by repeating the procedure (with two sticky pad squares) using a spare test coupon that had not been exposed to the burning composite. Thus a total of 42 microscope slides were generated for further analysis, as described in section 3.2.

Cotton cloth squares measuring 2.5 × 2.5 cm were used for chemical residue sampling. These cloth squares were first soaked in methylene chloride. The cloth square was then pressed firmly against the test coupon for approximately 15 s. It was intended that the methylene chloride would solvate the PAHs and other chemicals of interest, which would then be absorbed by the cotton square. The cloth square was removed and placed within a small amber vial for transfer to the analytical laboratory. A sample blank was also obtained by repeating the procedure using a spare test coupon that had not been exposed to the burning composite. Thus a total of 41 samples were obtained for further analysis, as described in Section 3.3.

Table 1 presents a summary of the test conditions, decontamination procedure, and sampling techniques applied to the 60 test coupons used in Test 1. Two additional test coupons were used for sample blanks, as described above.

Table 1. Summary of the Test Conditions Applied to the Fabric Sqaures used in Test 1

Number of Test coupons	Position in Shipping Container	Decon Procedure	Sampling Technique	Sample Media Obtained
5 test coupons	Positioned 1.5 m from fuel pan	Not washed	Sampled for particulates	10 microscope slides
10 test coupons			Sampled for chemical residue	10 cotton cloth samples
5 test coupons		Washed with water	Sampled for particulates	10 microscope slides
10 test coupons			Sampled for chemical residue	10 cotton cloth samples
5 test coupons	Positioned 3 m from fuel pan	Not washed	Sampled for particulates	10 microscope slides
10 test coupons			Sampled for chemical residue	10 cotton cloth samples
5 test coupons		Washed with water	Sampled for particulates	10 microscope slides
10 test coupons			Sampled for chemical residue	10 cotton cloth samples
1 test coupon (control)	Not placed in container or exposed to fire	Not washed	Sampled for particulates	2 microscope slides
1 test coupon (control)			Sampled for chemical residue	1 cotton cloth samples

3.1.2. Test 2

Preliminary results from the analysis of the particle and chemical residue samples generated in Test 1 indicated that only trace levels of PAHs and other chemicals of interest were detected. It was decided to repeat the test with a number of modifications intended to increase the amount of contaminants generated and increase the likelihood that they would accumulate on the test coupons. The following changes were made to the procedure followed in Test 1.

- The amount of JP-8 fuel was increased from 1 L to 2 Ls. This was intended to increase the damage and degradation of the composite panel and increase the amount of airborne contaminants available for deposition on the test coupons.
- The poster-boards bearing the test coupons were placed upon a 4-ft tall table rather than placed upon the floor of the shipping container (Figure 3). It was hypothesized that the more volatile PAHs and other chemicals were remaining airborne and had less tendency to adhere to an object at floor level. Elevating the test coupons was theorized to increase the potential for deposition.
- Cotton swabs (similar to Q-Tips) soaked in methylene chloride were used for chemical residue sampling rather than cotton cloth squares. It was theorized that PAHs and other compounds of interest were adhering to the test coupons, making them difficult to remove. The swabs were therefore rubbed aggressively over the surface of the test coupon in order to desorb any chemicals by a combination of physical agitation as well as

chemical solvation. The cotton bearing tip of the swab was then cut off, placed within a small amber vial, and taken to the analytical laboratory for further analysis.

Figure 3. Table used in Tests 2–5; in this Particular Test (Test 2), Arrays of 30 Test Coupons were Placed on Opposite Ends of the Table

3.1.3. Test 3

Preliminary results from the analysis of the particle and chemical residue samples generated in Test 2 indicated that deposition of smoke and soot particles was greatly increased by the changes made in Test 2. However, a problem was encountered with the chemical residue analysis. All samples, including the sample blank which was not exposed to the burning composite, had high levels of certain PAHs. It was theorized that these chemicals were present in the glue that was used to bind the cotton head to the wood shaft of the swab, and that the methylene chloride was leaching these chemicals out of the swab, contaminating the sample. Thus it was decided to perform Test 3 using the procedure followed in Test 2 with one change.

- Cotton balls soaked in methylene chloride were used for chemical residue sampling rather than cotton swabs. It was hoped that the cotton balls would not have the chemical contaminant found in the cotton swabs. The cotton balls were held with a pair of tweezers and rubbed aggressively over the surface of the test coupon in order to desorb any chemicals by a combination of physical agitation as well as chemical solvation. The cotton ball was then placed within a small amber vial, and taken to the analytical laboratory for further analysis.

3.1.4. Test 4

Test 4 involved cutting the a composite panel with a saw in order to generate particles rather than burning a composite panel as done in Tests 1–3. An approximately 3 ft tall metal pole was positioned vertically at one end of the shipping container, in the same location as the fuel pan previously used in Tests 1–3. A metal C-clamp was used to secure a composite panel to the top of the metal pole. A photo of the pole assembly with the composite panel is presented in the left panel of Figure 4. A Husqvarna K960 rescue saw equipped with a 30-cm diamond blade was used to cut the composite panel. A photo of the saw used is presented in the right panel of **Error! Reference source not found.**. This saw is typical of saws used by firefighters and first responders.

Figure 4. Composite Panel Clamped in a Support Stand (left); Husqvarna K960 Rescue Saw used to Cut Composite Panels (right)

A firefighter wearing protective clothing entered the shipping container and proceeded to make a series of cuts in the composite panel over the course of 5 min. A picture of this procedure is presented in **Error! Reference source not found.**. For safety reasons the door was left open during this procedure.

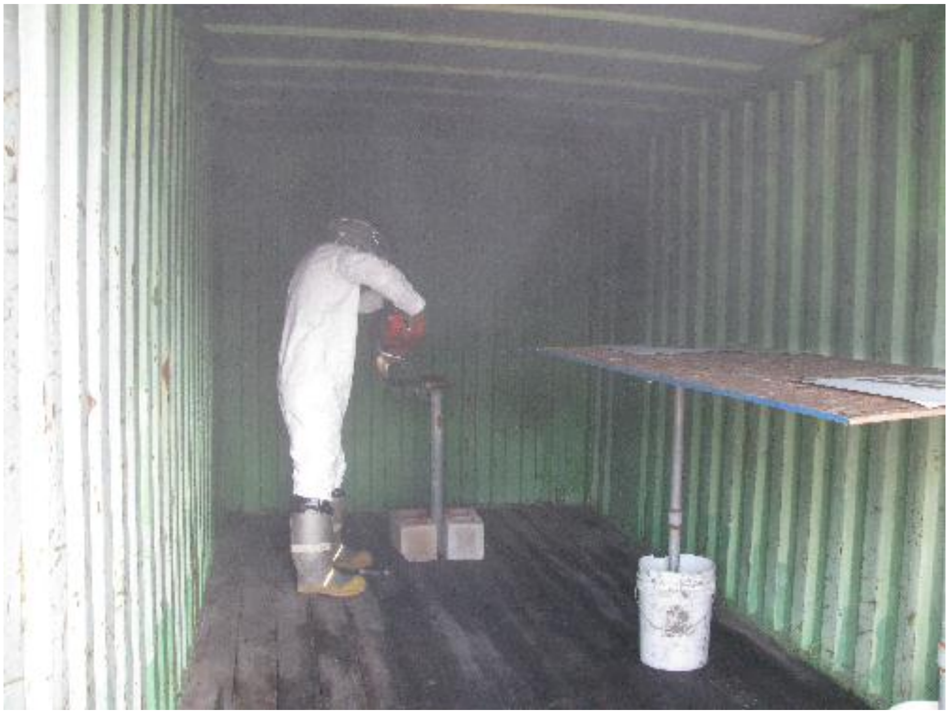


Figure 5. Composite Panel being Cut with a Rescue Saw; the Apparent Cloudiness in the Pictures is Due to the Presence of Airborne Particles Caused by the Cutting Process

The firefighter then left the shipping container and shut the doors. The doors were kept shut for a further 10 min to give airborne particles time to settle onto the test coupons. All procedures for first positioning the test coupons before cutting the composite panel, and then washing and sampling the test coupons for particles and chemical residue after cutting, were followed as described for Test 3.

3.1.5. Test 5

Preliminary results from the analysis of the particle and chemical residue samples generated in Test 3 indicated that only trace levels of PAHs and other chemicals of interest were detected. However, the use of cotton balls rather than cotton swabs did eliminate the PAH contamination that was observed when using the cotton swabs for chemical residue analysis. It was hypothesized that PAH compounds, if present at all, were simply being deposited in quantities too low to detect. One additional test was performed using the procedures developed for Test 3, with the following changes.

- One hundred test coupons were positioned, 50 on each sheet of poster-board paper, in ten 5 × 2 arrays of 10 test coupons. Test coupons within each array of 10 squares were placed adjacent to each other, making approximately continuous 5 × 25-cm (2 × 10-in) strips of fabric. See Figure 6 for a photograph of this arrangement. Both poster boards were placed adjacent to each other, approximately 2.1 m from the fuel pan in the shipping container.
- After the composite panel was burnt and the 10 min waiting period was over, a single cotton ball was rubbed aggressively over the surface of each array of 10 test coupons. This resulted in the cotton ball accumulating approximately 10 times as much residue as the previous procedure. It was hoped that this would enhance the detection of contaminants present in very low levels.
- No particulate sample collection was performed. It was deemed that samples obtained from previous Tests 1–4 were sufficient.

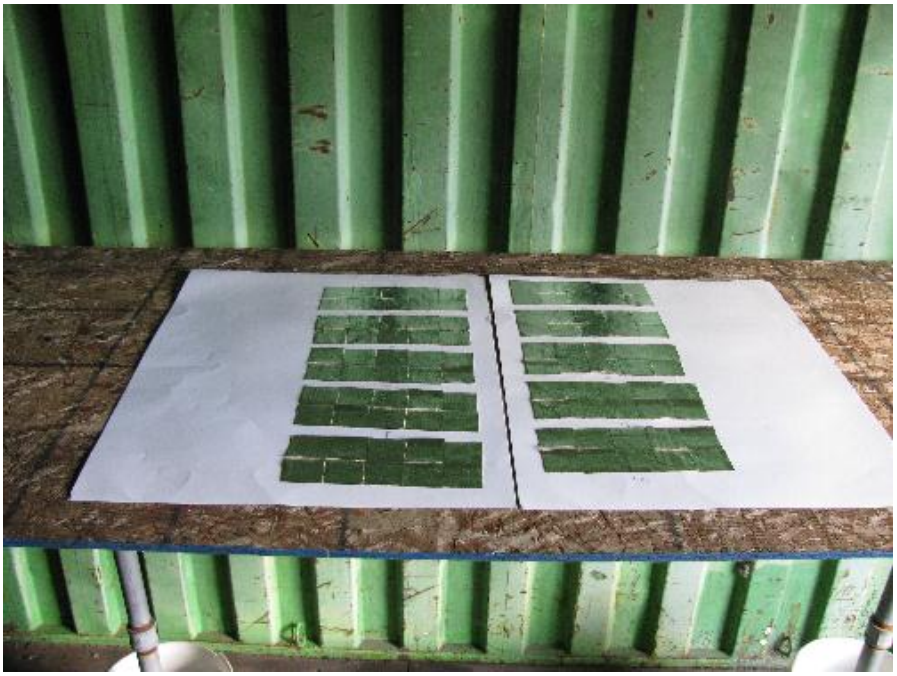


Figure 6. Ten Arrays of Ten Test Coupons used in Test 5

3.1.6. Summary of test procedures

Table 2 presents a summary of the test conditions and sampling techniques used in Tests 1–5.

Table 2. Summary of Test Procedures

Test Number	Description	Particle Sampling Method	Chemical Sampling Media
1	Burned composite panel in 1 L of JP-8	Sticky pads	2.5-cm cloth squares
2	Burned composite panel in 2 L of JP-8	Sticky pads	Cotton swabs
3	Burned composite panel in 2 L of JP-8	Sticky pads	Cotton balls
4	Cut composite panel with rescue saw	Sticky pads	Cotton balls
5	Burned composite panel in 2 L of JP-8	None collected	Cotton balls swiped across ten test coupons

3.2. Particle Analysis Procedure

A Southern Precision Instruments optical microscope was used to examine the mounted sticky pads obtained from test procedures 1–4 described above. A photograph of this microscope is presented in Figure 7. In addition to direct observation of the slides, photographs of material adhered on the sticky pads were taken using the microscope's digital camera (the red and silver box at the top of the camera in the photo) for further analysis using computer software, as described below. The digital microscope camera was a Moticam 2500, adapted to the microscope's auxiliary optical tube. Also note the flexible light tubing on either side of the microscope stage. These light sources were used to illuminate the surface of the slide, and the flexible light piping allowed the light sources to be positioned in search of uniform, even lighting of the slide. The image recognition software employed only functioned properly if the entire field of view in the image was brightly and evenly lit. Image capture was performed using the Motic 2.0 software suite operated on a laptop computer (Dell Latitude 610). Photographs were captured in JPEG format at a resolution of 1024 × 768 pixels.

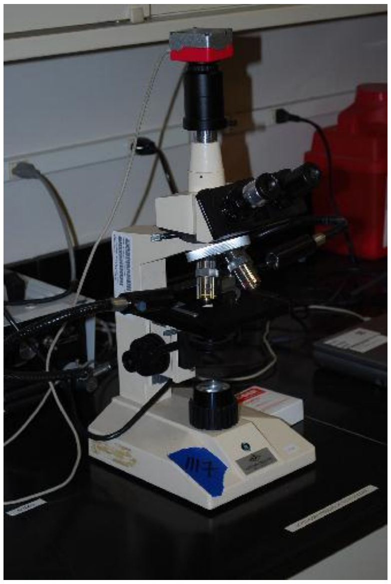


Figure 7. Microscope used during the Particle Analysis Procedure

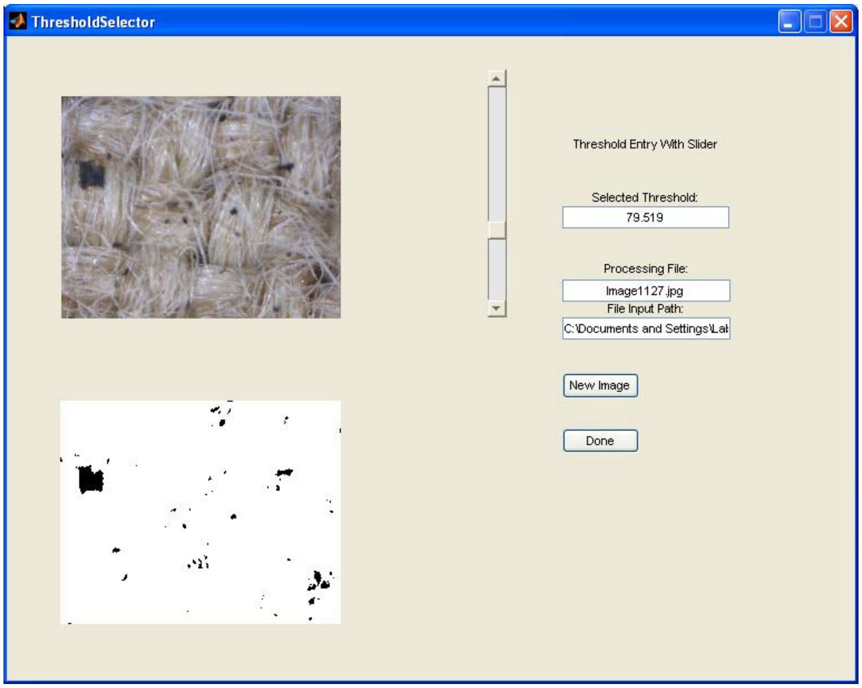
Each sticky pad was photographed in nine locations. Thus Tests 1–4, which produced 40 sticky pads each, generated 360 total photographs per test. The positions of the nine photographs were chosen according to the following scheme. Each sticky pad was conceptually divided into a grid of nine equal squares. One photo was taken in the approximate center of each square. Deviations from the center of each square region occurred for several reasons, including limitations in the microscopes focusing ability and light levels. Manual focusing knobs were used to focus the microscope and camera, and this required at least one object in the field of view, so the user was obliged to move the microscope stage until a typical particle view was obtained, hopefully with at least one particle to focus on.

In order to determine the dimensions of the field of view seen and photographed by the microscope a reference slide bearing a grid of lines with 0.1-mm spacing was placed in the microscope and photographed. The dimensions of the field of view of the microscope were then determined by dividing the digital photograph dimensions (1024 × 768 pixels) by the observed pixels per mm ratio seen in the reference slide photo. Using this procedure, the field of view was found to have dimensions of 0.655 mm × 0.523 mm, or a total area of 0.343 mm^2.

Software analysis of the microscope photos was performed using the Image Processing Toolbox module present in the MATLAB version 2008b programing suite. This software tool first converts the color photo into a greyscale image according to the following built in conversion formula:

$$GS_{(x,y)} = 0.2989\ R_{(x,y)} + 0.5870\ G_{(x,y)} + 0.1140\ B_{(x,y)}$$

Where $GS_{(x,y)}$ represents the grayscale value for the pixel at image coordinates x and y, $R_{(x,y)}$ represents the red value for the pixel, $G_{(x,y)}$ represents the green value of the pixel, and $B_{(x,y)}$ represents the blue value. The program then, via a user controllable threshold selector, converts the image into a black (binary 0) and white (binary 1) image. A picture of the user interface for this program is presented in Figure 8. In this way the program attempts to distinguish between a white or light colored background and darker foreground particles. The program then performs statistical analysis on the resulting black and white image. In particular, the program will count the number of black pixels to estimate the total surface area occupied by particles, and the program will count the number of discrete black domains to estimate the number of particles or particle clusters present.



Selected Threshold: 79.519

Processing File: Image1127.jpg

File Input Path: C:\Documents and Settings\La

Figure 8. Screenshot from the Matlab Program used for Particle Analysis

It was found that adequate lighting was critical to this process. When using the microscope's built-in light source, the center of the field of view tended to be brighter than areas towards the outer edges of the field of view, especially the four corners. This led to problems adjusting the greyscale threshold during particle analysis with the MATLAB tool. If the threshold was set relatively high, particles toward the center which were brightly illuminated were not detected. If the threshold was set relatively low then the shadowy regions toward the exterior and corners of the field of view were falsely identified as particles. The use of the built-in microscope light was also only possible using transmitted light. A clear sticky-pad medium was tried for transmitted light microscopy, but these revealed bubbles between the sticky pads and slides that were frequently mistaken as particulates. This issue was largely resolved by placing two additional light sources on opposing sides of the microscope stage and lighting the samples from above for reflected light microscopy. Careful adjustment of these light sources produced an image with bright and even lighting, resulting in good contrast between the white sticky pads and the dark soot particles and fibers.

Statistical analysis was then performed on the results of the MATLAB particle analysis as follows. For each test, the average and standard deviation of the total particle count and the percent coverage was calculated for each group of photos originating from identical test conditions (distance from fire and decontamination technique, etc.). In addition, the Cleanability Factor of the materials under the test conditions is determined. Cleanability is a measure of the ability to clean. The concept was obtained from standards for cleaning aircraft paint topcoats. The cleanability concept was originally applied to light reflecting from a painted surface, where paint soiled with carbon black particles was expected to reflect less light.[11] The concept was used in this study because of the similar nature of this study. For this report the cleanability *C* was calculated according to the following equation:

$$C = 100 * (A_{\mathrm{contaminated}} - A_{\mathrm{cleaned}}) / (A_{\mathrm{contaminated}} - A_{\mathrm{control}})$$

Where C (expressed as a percentage) represents the cleanability value for the material and cleaning technique, $A_{\mathrm{contaminated}}$ represents the average percent coverage of samples which were exposed to the burning composite and not cleaned, A_{cleaned} represents the average percent coverage of samples which were exposed to the burning composite and were cleaned, and A_{control} the average percent coverage of the control samples, which were not exposed to the burning composite.

3.3. Chemical Analysis Procedure

Tests 1–4 resulted in the collection of 40 pieces of media (cotton cloth squares, cotton swabs, or cotton balls), while Test 5 produced ten cotton balls. All sample media was analyzed according to the following procedure. Approximately 2 mL of methylene chlorine was added to the amber vial containing each piece of sample media in order to extract the PAHs and other methylene chloride soluble chemicals of interest. After soaking for a period of time, the methylene chlorine was removed and placed within a drying tube. The drying tube was placed in a Supelco drying manifold. Nitrogen gas was blown through the manifold to remove methylene chloride solvent until the volume was approximately 0.1 mL. The remaining liquid volume was then injected into a Thermo Trace-DSQ gas chromatograph-mass spectrometer (GCMS) system for analysis. The method extraction efficiency and level of detection was not determined for this overall procedure.

Thermo Excalibur software was used to acquire, display, and analyze data from the GC-MS injections. The Wiley Mass Spectral Library, 7th Edition, was used for qualitative mass spectral searches to identify unknown compounds discerned in the GC-MS data. This library included a number of compounds of interest including polycyclic aromatic compounds, volatile and semi-volatile organic compounds. To further aid in the identification of the components of the chemical residue samples an EPA 610 PAH mix kit was obtained from Sigma-Aldrich. This kit included reference samples of the 15 small PAH compounds listed in Table 3, many of which are known or suspected human carcinogens. Most compounds in this kit were present at concentrations of 100 μg/mL of PAH dissolved in methylene chloride. These reference standards were subject to GC/MS analysis in the Agilent System in order to obtain additional GC retention times and MS spectra to aid in the identification of the chemical residue samples collected during testing.

Table 3. List of the Chemical Compounds Present in the EPA 610 PAH Mix Kit

Acenaphthene	Dibenzo(a,h)anthracene
Anthracene	Fluoranthene
Benzo(a)anthracene	Fluorene
Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
Benzo(b)fluoranthene	Naphthalene
Benzo(ghi)perylene	Phenanthrene
Benzo(k)fluoranthene	Pyrene
Chrysene	

4. RESULTS AND DISCUSSION

Over the course of the five tests conducted, a total of 164 sticky pad slides and 175 chemical residue samples were collected, including sample blanks. These were subject to particle analysis and chemical analysis as described above.

4.1. Particle Analysis

A total of 1476 microscopy photos were taken of the 164 sticky pad slides used to collect transfer material during sampling. These were subject to particle analysis as described above in order to estimate the coverage ratio and particle density of contaminants originally deposited on the test coupons.

Unless otherwise stated, all photographs shown below are full-scale images taken by the digital microscope. As described above, these images have physical dimensions of 0.655 mm × 0.523 mm.

4.1.1. Control Samples

As part of each test, as control sample was collected from a test coupon that was not placed in the shipping container or exposed to the composite panel. These slides were found to be very clean, containing a small number of generally unidentifiable particles that were likely dust, lint, or other particles environmental origin. Figure 9 presents a photograph of a typical particle seen on the control slides.

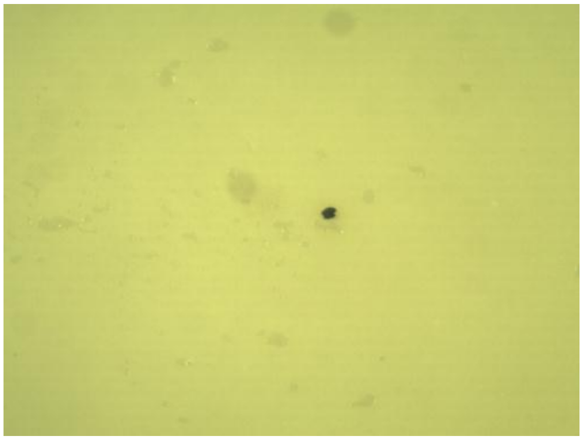


Figure 9. Example of an Unidentified Particle Observed on a Control Slide

Table 4 presents the results of particle analysis performed on all control samples collected. The average and standard deviation of the percent coverage and number of particles present in the field of view is shown. The average fraction of the field of view covered by particles was found to be approximately 0.03 percent. The average number of particles identified was approximately eight. This may seem high, considering that in most cases only a single obvious particle was present in the field of view. However, most photographs contained a few tiny, often one-pixel large, specks of material that were dark enough to be recognized and counted as particles by the Matlab software.

Table 4. Particle Counts and Fraction Coverage Determination for Control Test Coupons

Location	Condition	Percent Coverage (percent)		Particle Count	
		Average Value	STDEV	Average Value	STDEV
Control	Control	0.03	0.04	7.69	6.58

The relative scarcity of particles in the control slides, and also some of the washed slides, may actually result in an overestimation of particle coverage and count in some cases. In general, a photo was taken at the approximate center of each of the nine conceptual regions of each sticky pad. However, in order to properly focus the image seen in the microscope it is necessary to have at least one object in the field of view with well-defined borders and features. The sample blank slides, as well as some of the slides taken from test coupons that were subject to washing, had very few particles present. In these cases it was necessary to translate the slide until one or more objects came into the field of view so that the image could be properly focused. This likely lead to an overestimation of the coverage ratio and particle number calculated for these slides.

4.1.2. Test 1

Test 1 involved exposing test coupons to soot emitted from a burning composite panel Figure 10 presents two photographs of transfer material obtained from test coupons placed 1.5 m from the burning composite. The left slide is from a square that was not washed, while the right photograph is from a square that was washed. A number of irregular soot particles of various sizes are seen in the photograph of the unwashed slide. Most of these particles appear to be conglomerates of smaller soot particles. Significantly less contamination is seen in the washed slide.

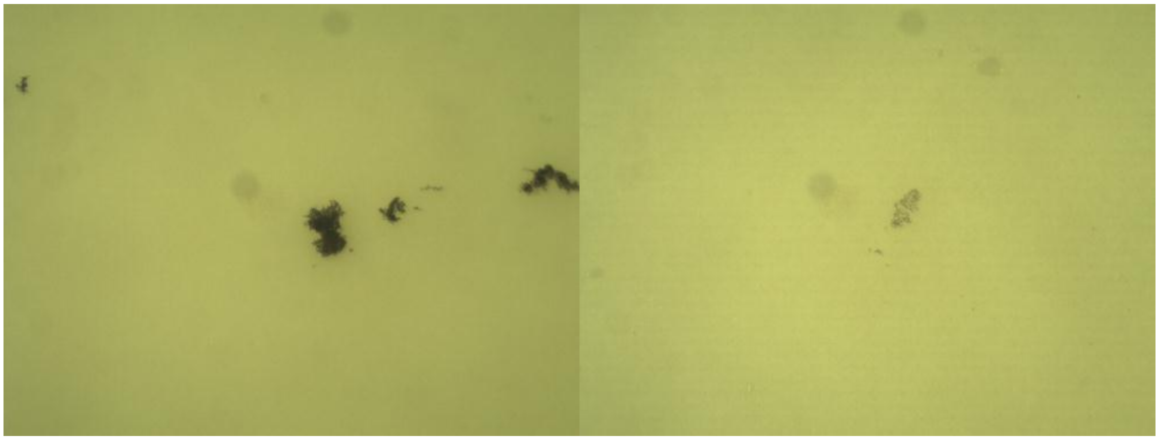


Figure 10. Sample Photographs of Soot Particles Observed in Test 1 Samples before Washing (left) and after Washing (right)

Soot particles up to approximately 0.1 mm in length were observed to occur in the transfer material obtained in this test. Most soot particles were significantly smaller. No rod-like particles were observed that appear similar to the carbon-fiber particles seen in Test 3 (see Section 4.1.4).

Table 5 presents the results of particle analysis performed on the photographs taken of Test 1 sample materials. The average and standard deviation of the percent coverage and number of particles present in the field of view, as well as the cleanability factor, is shown. The average percent coverage for unwashed samples was approximately 0.9 percent for samples originating 1.5 m from the burning composite panel, and approximately 0.8 percent from samples originating 3 m from the burning composite sample. Both are approximately three times the faction coverage seen in the control samples. The difference in proximity appears to have had only moderate effect on soot deposition. After washing, the fraction coverage drops to approximately 0.4 percent and 0.3 percent respectively. Rinsing the test coupons with water is also observed to increase the number of particles detected by the MATLAB tool. For example, the average number of particles recognized on samples originating 1.5 m from the fuel pan increase from about 30 to about 36 after washing. It may be the case that when many of the larger soot particles are being partially washed away they leave a number of smaller soot particles behind which increases the overall particle count. The cleanability factors for the test coupons 1.5 and 3 m from the burning composite were calculated to be approximately 63 percent and 68 percent. Thus, cleaning removed approximately two-thirds of the soot particles from the test material.

Table 5. Particle Counts and Fraction Coverage Determination for Test 1

Location	Condition	Percent Coverage (%)		Particle Count		Cleanability (%)
		Average Value	STDEV	Average Value	STDEV	
1.5 m from fire	-	0.94	0.90	29.53	40.01	63.23
	Rinsed	0.36	0.65	35.97	54.86	
3 m from fire	-	0.84	1.42	24.80	30.43	68.02
	Rinsed	0.29	0.36	38.95	38.59	

4.1.3. Test 2 and Test 3

Several changes to the test procedure were made during Test 2 in an attempt to increase the amount of material deposited on the test coupons. The test procedure and particle sampling technique performed in Test 3 were identical to the procedure conducted during in Test 2, only the chemical residue sampling technique differed. Therefor these two tests will be discussed together.

Figure 11 presents two photographs of transfer material obtained from test coupons placed 1.5 m from the burning composite during Test 2. The left slide is from a square that was not washed, while the right photograph is from a square that was washed. In general the soot particles were larger and more numerous than those observed in Test 1. Rinsing the squares with water appears to remove a large fraction of the contamination. However, numerous smaller particles still remain. Photographs taken of transfer material from Test 3 appear very similar to the photographs of the Test 2 transfer material.

Soot particles significantly larger than those observed in the Test 1 transfer material were noted in the Test 2 transfer material. In a few cases soot particles were longer than the field of view of

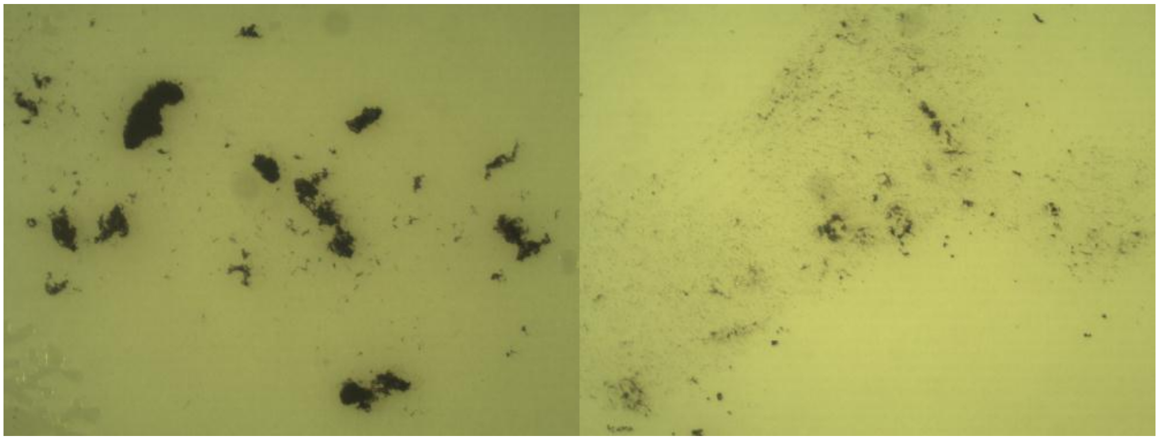


Figure 11. Sample Photographs of Soot Particles Observed in Test 2 Samples before Washing (left) and after Washing (right)

the microscope (0.655 mm). Most soot particles were significantly smaller. Very few rod-like particles (indicative of composite-fiber particles) were observed, and none of these objects were longer than approximately three times their diameter. The rod-like objects detected in Tests 2 and 3 may be soot-covered carbon fibers, or they may simply be clumps of soot that happen to have a rod-like shape.

Table 6 presents the results of particle analysis performed on the photographs taken of Test 3 transfer materials. The average and standard deviation of the percent coverage and number of particles present in the field of view, as well as the cleanability factor, are shown. The average percent coverage for unwashed samples was approximately 5.5 percent for samples originating 1.5 m from the burning composite panel, and approximately 4.6 percent from samples originating 3 m from the burning composite sample. Both are approximately six times the faction coverage seen in the Test 1 results. The difference in proximity appears to have had only moderate effect on soot deposition. After washing, the fraction coverage drops to approximately 1.9 percent and 1.5 percent respectively, again about six times the coverage seen in Test 1. Rinsing the test coupons with water is also observed to increase the number of particles detected by the MATLAB tool, as was observed in Test 1. For example, the average number of particles recognized on samples originating 1.5 m from the fuel pan increase from about 168 to about 234 after washing. The cleanability factor for the test coupons 1.5 and 3 m from the burning composite was calculated to be approximately 65 percent and 68 percent. Thus, cleaning removed approximately two-thirds of the soot particles from the test material.

Table 6. Particle Counts and Fraction Coverage Determination for Test 3.

Location	Condition	Percent Coverage (%)		Particle Count		Cleanability (%)
		Average Value	STDEV	Average Value	STDEV	
1.5 m from fire	-	5.46	4.78	167.51	151.46	65.04
	Rinsed	1.93	1.62	234.66	169.30	
3 m from fire	-	4.65	3.17	149.79	162.97	67.55
	Rinsed	1.53	1.32	217.02	145.02	

4.1.4. Test 4

Test 4 involved exposing test coupons to debris generated by cutting a composite panel with a rescue saw. Figure 12 presents two photographs of transfer material obtained from test coupons placed 1.5 m from the composite panel. The left photograph is from a square that was not washed, while the right photograph is from a square that was washed. The transfer material from the unwashed square contains a large number of carbon fibers. These fibers were approximately 0.005 mm in diameter and varied in length from a few times their diameter to greater than the width of the field of view (0.655 mm). Washing the test coupons appears to greatly reduce the quantity of graphite fibers. Little to no residue resembling the soot particles seen in Tests 1–3 is visible.

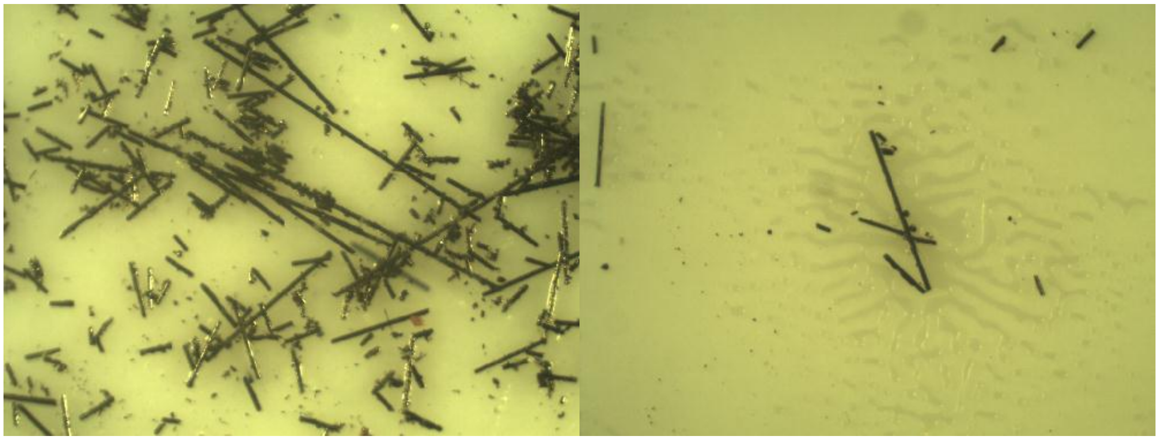


Figure 12. Sample Photographs of Soot Particles Observed in Test 4 Samples before Washing (left) and after Washing (right)

Even in relatively clean slides, fiber particles were often observed to cluster and overlap each other. The particle counting algorithm, which simply counts continuous domains of black pixels as a single particle, will count these multiple objects as a single particle. This is an inherent limitation of this particular software and could not be compensated for. This phenomenon can be observed in the two photographs presented in Figure 12. Overall, particle grouping was so frequent and significant in the slides studied that the reported particle count greatly underestimates the actual number of particles present in many cases and is of little value. However, the particle coverage ratio does not suffer from this effect and is still of significance.

Although the carbon fibers present in the composite panel generally appear black to the naked eye, they sometimes appeared quite reflective under the bright light sources used in the microscope. In general this occurred with fibers that had a particular orientation in the field of view. Figure 13 presents a photograph illustrating this effect. The long, nearly vertical fiber in this photo is seen to reflect much more light than the surrounding fibers with different orientations. Because of this, the majority of this fiber will not be resolved when threshold selector is set during particle analysis, leading to an underestimation of the actual coverage ratio in this slide. It was not possible to totally eliminate this effect by adjusting the position of the light sources around the microscope stage. However, with careful adjustment of the light sources only a small fraction of fibers exhibited strong reflection.

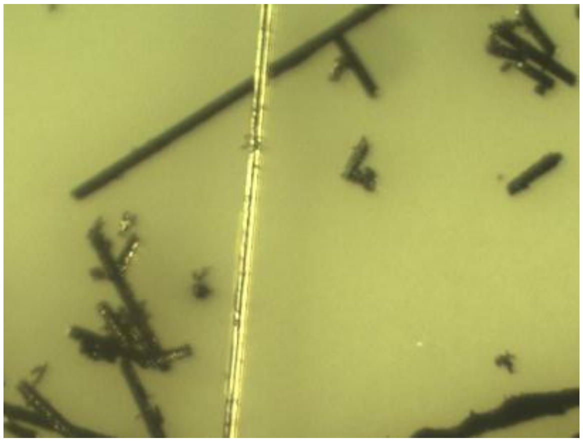


Figure 13. Photograph Displaying the Reflective Nature of the Carbon Fibers; the Nearly Vertical Fiber May Not be Recognized by the Particle Counting Software Due to Reflection

Table 7 presents the results of particle analysis performed on the photographs taken of Test 4 sample materials. The average and standard deviation of the percent coverage and number of particles present in the field of view, as well as the cleanability factor, is shown. The average percent coverage for unwashed samples was approximately 11.2 percent for samples originating 1.5 m from the burning composite panel, and approximately 10.3 percent from samples originating 3 m from the burning composite sample. Both are approximately 35 times the faction coverage seen in the control samples. The difference in proximity appears to have had only moderate effect on soot deposition. After washing, the fraction coverage drops to approximately 0.5 percent and 0.3 percent respectively, essentially the same fraction coverage as the control samples. Rinsing the test coupons with water greatly reduces the average number of particles detected by the Matlab tool. For example, the average number of particles recognized on samples originating 1.5 m from the fuel pan decreases from about 186 to about 95 after washing. This was opposite of the trend observed in the soot-contaminated test coupons in Tests 1–3. The cleanability factor for the test coupons 1.5 and 3 m from the cut composite was calculated to be approximately 96 percent and 97 percent. In both cases, cleaning removed the majority of fiber particles from the test material, although the percent coverage of contaminants on the cleaned test coupons was still approximately 10 times greater than the unexposed control samples.

Table 7. Particle Counts and Fraction Coverage Determination for Test 4.

Location	Condition	Percent Coverage (%)		Particle Count		Cleanability (%)
		Average Value	STDEV	Average Value	STDEV	
1.5 m from composite	-	11.16	3.70	185.56	61.57	95.77
	Rinsed	0.50	0.77	94.94	126.88	
3 m from composite	-	10.29	3.17	212.08	74.52	97.15
	Rinsed	0.32	0.47	44.10	50.97	

4.1.5. Test 5

No particle analysis was performed on the test coupons exposed during Test 5.

4.1.6. Additional Observations

4.1.6.1. Transfer of Aluminum Material

On a small number of slides, material was observed which was believed to be aluminum particles that adhered to the sticky pads strongly enough to be removed from the test coupons during the sampling process. Figure 14 presents a photograph from one slide containing a number of these particles. Although it might not be obvious in the picture below, under the microscope there was strong visual evidence that the particles were indeed aluminum material greatly resembling the coating present on the surface of the test coupons. When recognized, these patches of particles were not included in the field of view chosen for photography and therefore did not affect the particle analysis procedure. However it may be the case that in some cases aluminum material was not recognized and accidently included.

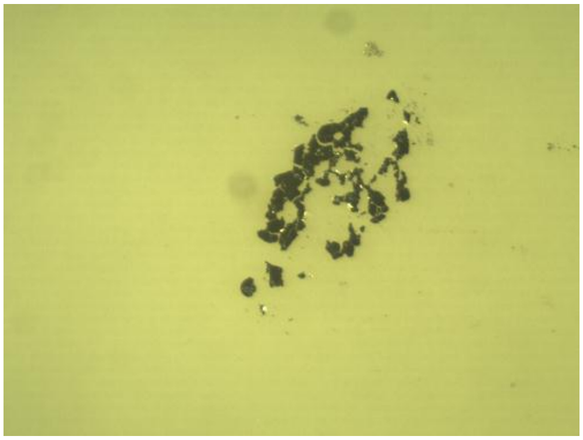


Figure 14. Example of Alumimum Material Removed from the Fabric Surface during the Transfer Process

4.1.6.2. Foreign Material

A few objects were noted that did not resemble the typical soot or fiber particles that made up the majority of the observed contaminants. These objects were generally string-like in appearance and may have been hair, plant material, or strings from clothing or other fabrics in the environment. However, we cannot conclusively say that they were not particles given off by the burning composite panel. Therefore these particles were photographed when they happened to occur in the chosen field of view and included in the particle analysis performed on these photos. Whatever their identity, these objects were generally small and observed in only a few photographs, making their effect on the statistical particle analysis minimal. Figure 15 presents a photograph of one of these objects.

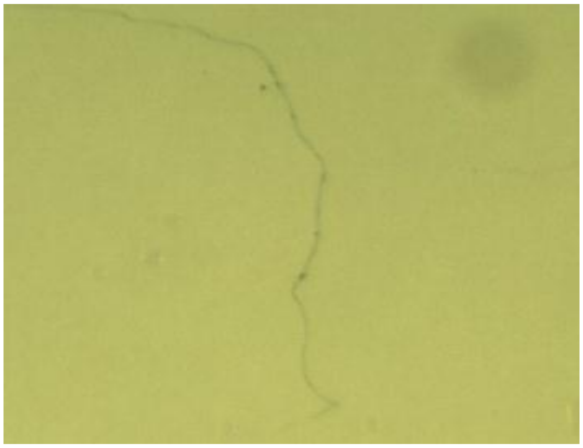


Figure 15. Example of a Possible Foreign Object Observed in a Small Number of Slides. This Object May Be a Hair, Thread, or Plant Material that was Captured during the Transfer Process

4.2. Chemical Analysis

Several methods were employed to extract chemical residue from the surface of the test coupons for chemical analysis.

- Pressing a cotton cloth soaked in methylene chloride against the test coupon (Test 1).
- Vigorous rubbing with a cotton swab soaked in methylene chloride (Test 2).
- Vigorous rubbing with a cotton ball soaked in methylene chloride (Test 3 and 4).
- Vigorous rubbing with a cotton ball soaked in methylene chloride across an array of 10 squares (Test 5).

As described in the procedure section, the cotton media was first soaked in methylene chloride in order to dissolve chemicals it might have absorbed. The resulting solution was filtered to remove particulates such as soot particles, and then concentrated by evaporation. In all cases, the resulting solution was a light orange to brown in color, indicating the presence of some solute. In the final case (Test 5) the solute concentration was so high the solution was dark brown in color and completely opaque.

Despite this, no PAHs (or other chemicals of interest) could be detected by GC/MS analysis of the chemical residue samples collected during Test 1 or 3–5. Some PAHs compounds were detected in the Test 2 chemical residue samples, but the same chemicals were also present in the control sample (a clean cotton swab that was not exposed to smoke and soot residue), suggesting that these chemicals were contaminants present in the cotton swab itself.

5. CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

Burning composite panels in proximity to the test coupons resulted in the deposition of soot particles on the test coupons, the observed coverage being dependent on amount of fuel/composite burned and proximity of test coupons to the burning composite. Maximum coverage of approximately 5.5 percent occurred on test coupons located 1.5 m from the burning composite. A baseline test, consisting of burning fuel in the test pan with no composite panel present was not performed, so it cannot be determined what fraction of this soot originated from the jet fuel and what fraction originated from the burning composite panel itself. Cleaning the test coupons with water reduced the observed coverage by approximately two-thirds in all cases. Very few if any composite fibers were observed on the exposed test coupons during these fire tests.

Cutting the composite panels with a rescue saw in proximity to the test coupons resulted in the deposition of carbon fibers on the test coupons, the observed coverage being dependent on the proximity of test coupons to the composite panel being cut. Maximum coverage of approximately 11 percent occurred on test coupons located 1.5 m from the burning composite. Fibers were observed to have a nearly uniform diameter of approximately 0.005 mm, and ranged in length from approximately 0.01 mm to in excess of 0.6 mm. Cleaning the test coupons with water reduced the observed coverage by approximately 95 percent in both cases. Very few if any soot-like fibers were observed on the exposed test coupons during these cutting tests.

Cutting and mechanical manipulations appear to add significantly to the particulate load from burned composites, and the particulate threats from cutting and manipulating composites appear to differ in their nature from the particulates produced in the fire. Physiologic tests were not performed as part of this study, so it cannot be confirmed that the rod-shaped particulates liberated from the sawed composite could produce the skin and respiratory irritation reported by aircraft recovery teams working historical crashes without respiratory or skin protection. The rod-shaped particulates observed from Test 4 remain suspicious as potential irritants.

No polycyclic aromatic hydrocarbon compounds were detected in any of the chemical residue samples taken during any of the five tests, other than suspected sample contamination that occurred in Test 2. The lack of identifiable chemicals may be due to several causes.

- No PAH compounds may have been emitted by the composite panel (or the burning jet fuel). This seems possible in the case of Test 4 in which the panel was simply cut with a saw, but unlikely in the other tests involving burning the composite panel.
- PAH compounds may have been generated but remained airborne, or have had little tendency to deposit onto the test coupons. This is plausible, as we did not sample the air in the shipping container for analysis.
- PAH compounds may have been deposited on the test coupons, but were not transferred into the methylene chloride soaked cotton media during the sampling process.
- The sampling and analysis techniques used may simply not have been sensitive enough to detect organic compounds present on the soot particles and other particulates.

PAH and other organic compounds from incomplete combustion are commonly encountered in combustion situations. Their absence in this study are likely not to indicate an absence of organic chemical threats associated with composite fires but rather inadequacies in the organic chemical collection and analysis techniques used.

5.2. Recommendations

Water washing can still be recommended as an effective technique for removing combustion particulates from firefighting and recovery equipment.

Respiratory and skin protection should continue to be employed during composite firefighting and recovery operations.

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LIST OF SYMBOLS, ABBREVIATIONS, AND ACRONYMS

ACM	advanced composite material
AFB	Air Force Base
AFRL	Air Force Research Laboratory
ALI	acute lung injury
cm	centimeter(s)
EPA	Environmental Protection Agency
ft	foot; feet
GC	gas chromatograph
in	inch(es)
JP-8	jet propellant number 8
m	meter(s)
min	minute(s)
mL	milliliter(s)
mm	millimeter(s)
MS	mass spectrometer
NASA	National Aeronautics and Space Administration
NIOSH	National Institute for Occupational Safety and Health
PAH	polycyclic aromatic hydrocarbon
PAN	polyacrylonitrile
PPE	personal protective equipment
RAF	Royal Air Force
s	second(s)
SPME	solid phase micro-extraction
μg	microgram