



## **Coating Technologies for Insensitive Munitions**

**by Pauline Smith**

**ARL-TR-3952**

**September 2006**

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**by Pauline Smith**

**Weapons and Materials Research Directorate, ARL**

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## 1. Testing Overview

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Currently, the United States Army is evaluating coatings for use on ammunition containers. One of the required coatings that allow packaged 60-mm mortar cartridges to pass fast cook-off requirements in accordance with military standard (MIL STD)-2105C is an Intumescent coating system<sup>1</sup> for fire and heat protection. However, several tests related to robustness of the Intumescent coating system have shown poor flexibility, impact and marginal moisture resistance. The Intumescent coating is marketed by No-Fire Technologies, Inc., and consists of a water-based A-18NV, according to military performance (MIL-PRF)-24596. The Intumescent coating system is generally applied over a forest green coating (base coat) used on the containers.

Material testing is the process used to evaluate materials for characteristics that include but are not limited to durability and overall strength. For this project, materials were put through arduous tests to see how long the system would endure before beginning to fail. Also, before being used in the field, munition containers with the coating on the outside were exposed to a large-scale fire, and the cook-off time is recorded.

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## 2. Objective

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The objective of this project was to improve paint delamination by an evaluation of the current packaging and platform coatings and to improve thermal protection and potential blast performance.

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## 3. Experimental

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Initial screening of coatings is being performed through a series of small-scale fire tests with the use of the American Society for Testing and Materials (ASTM) E1354 cone calorimeter. Data were taken to compare the fire performance as well as the insulating performance of the coating samples. Coating samples tested were labeled as follows: life shield (LS)-120 reinforced (RF),

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<sup>1</sup>An intumescent coating has all the properties of ordinary paint; furthermore, this coating will not sustain combustion. Consequently, it will not burn, thus providing a high degree of protection to the subsurface. Upon exposure to flame or heat, it immediately foams and swells, which contributes an effective insulation and heat shield for the subsurface.

LS-un-reinforced (URF)-60, 06-0215-43528-50-1, Pitt-Char XP<sup>2</sup> 1.8 mm thick, and Pitt-Char XP 2.65 mm thick. All tests were conducted at a heat flux of 100 kW/m<sup>2</sup>.

### 3.1 Standard ASTM E1354 Cone Calorimeter Tests

ASTM E1354 (*I*) provides a small-scale test procedure to measure the ignitability, heat release rate, mass loss rate, and combustion product generation rate of a material exposed to a specified irradiance level. During a test, a 100- by 100-mm sample is placed beneath the conically shaped heater that provides a uniform irradiance on the sample surface (see figure 1). The sample mass is constantly monitored with a load cell, and the effluent from the sample is collected in the exhaust hood above the heater. In the duct down stream from the hood, the flow rate, smoke obscuration, and oxygen (O<sub>2</sub>), carbon dioxide (CO<sub>2</sub>), and carbon monoxide (CO) concentrations are continuously measured.

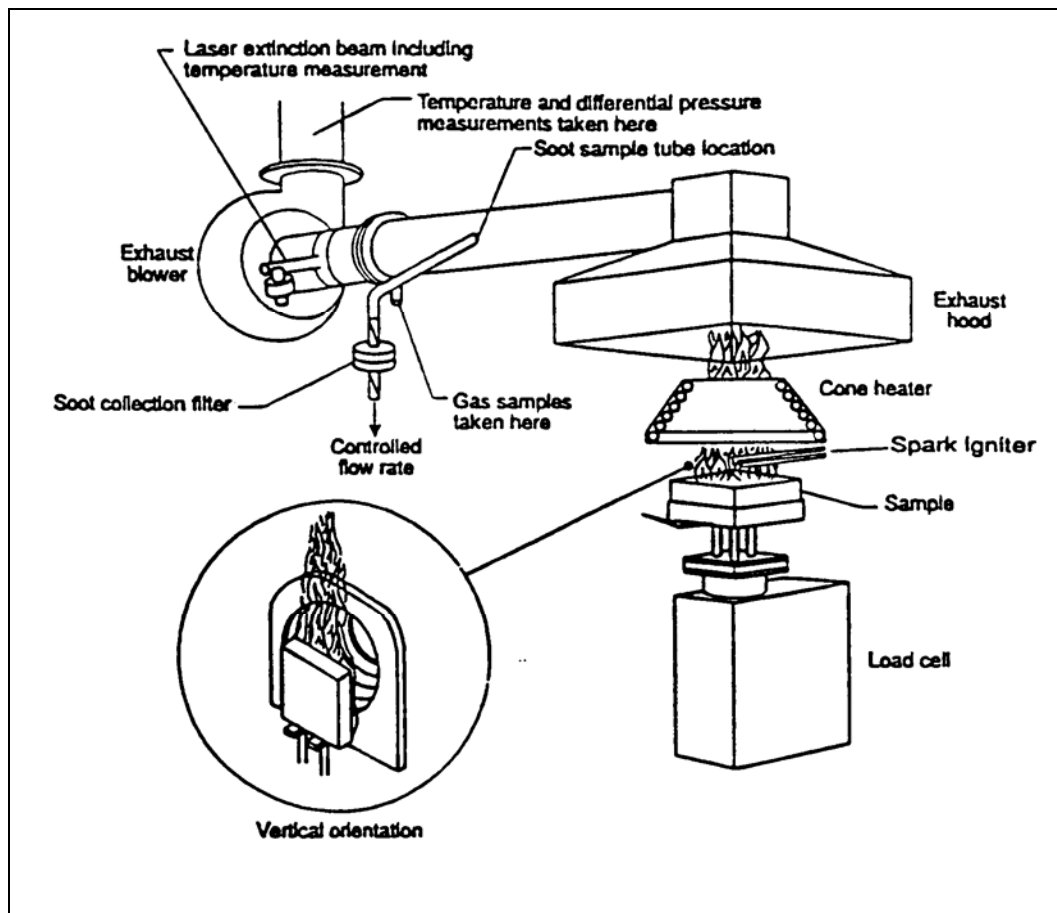


Figure 1. ASTM E1354 cone calorimeter test apparatus.

A spark igniter 12.5 mm above the sample surface is used to initiate the burning of any combustible gas mixture produced by the sample. Once the sample ignites, the burning of the sample

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<sup>2</sup>XP, which is not an acronym, is a registered trademark of PPG Industry.

causes a reduction in the oxygen concentration within the effluent collected by the hood. This reduction in oxygen concentration has been shown to correlate with the heat release rate of the material, 13.1 MJ per kilogram of O<sub>2</sub> consumed. This is known as the oxygen consumption principle. With this principle, we determine the heat release rate per unit area of the sample with time using measurements made in the duct.

The ASTM E1354 standard requires the following data to be reported for each material tested:

- Time to ignition(s),
- Peak heat release rate (kW/m<sup>2</sup>),
- Heat release rates averaged over various time periods, starting with the time of ignition (kW/m<sup>2</sup>),
- Effective heat of combustion (MJ/kg),
- Mass loss rate per unit area (kg/s m<sup>2</sup>),
- Specimen mass loss (percent),
- Average smoke specific extinction area (m<sup>2</sup>/kg). (Smoke production from a material has the rational units of square meters, representing the extinction cross section of the smoke. This is normalized by the amount of specimen mass loss (kg),
- Average CO and CO<sub>2</sub> production yields (kg/kg).

In addition to the standard measurements, the temperature on the unexposed side of the sample was measured at the middle of the sample with a glass braid, bare bead type K thermocouple. Each test was video recorded, and digital photographs were taken of the material before, during, and after fire testing.

### **3.2 Test Samples**

Test samples were 100 mm by 100 mm and were comprised of a 1-mm-thick aluminum plate with a polyurea coating on one side. A description of each sample is provided in table 1, and photographs of each sample are shown in figure 2. We determined coating thickness by averaging 16 thickness measurements around the edge of a sample with calipers. We determined the mass of the coating per unit area by subtracting the sample weight from the weight of the aluminum plate.

Table 1. Description of polyurea coatings on one side of a 1-mm-thick aluminum plate.

No.	Identification	Description	Average Coating Thickness (mm)	Coating Mass Per Unit Area ( $\text{kg/m}^2$ )
1	LS120 RF	Glossy black with silver specks, smooth coating	3.56	4.02
2	LS-URF-60	Glossy grey with silver specks, smooth coating	1.89	2.22
3	06-0215-43528-50-1	Glossy black smooth coating	2.81	2.90
4	Pitt-Char XP 1.80 mm	Grey dull finish with rough, uneven coating	1.59	2.25
5	Pitt-Char XP 2.65 mm	Grey dull finish with rough, uneven coating	2.31	2.93

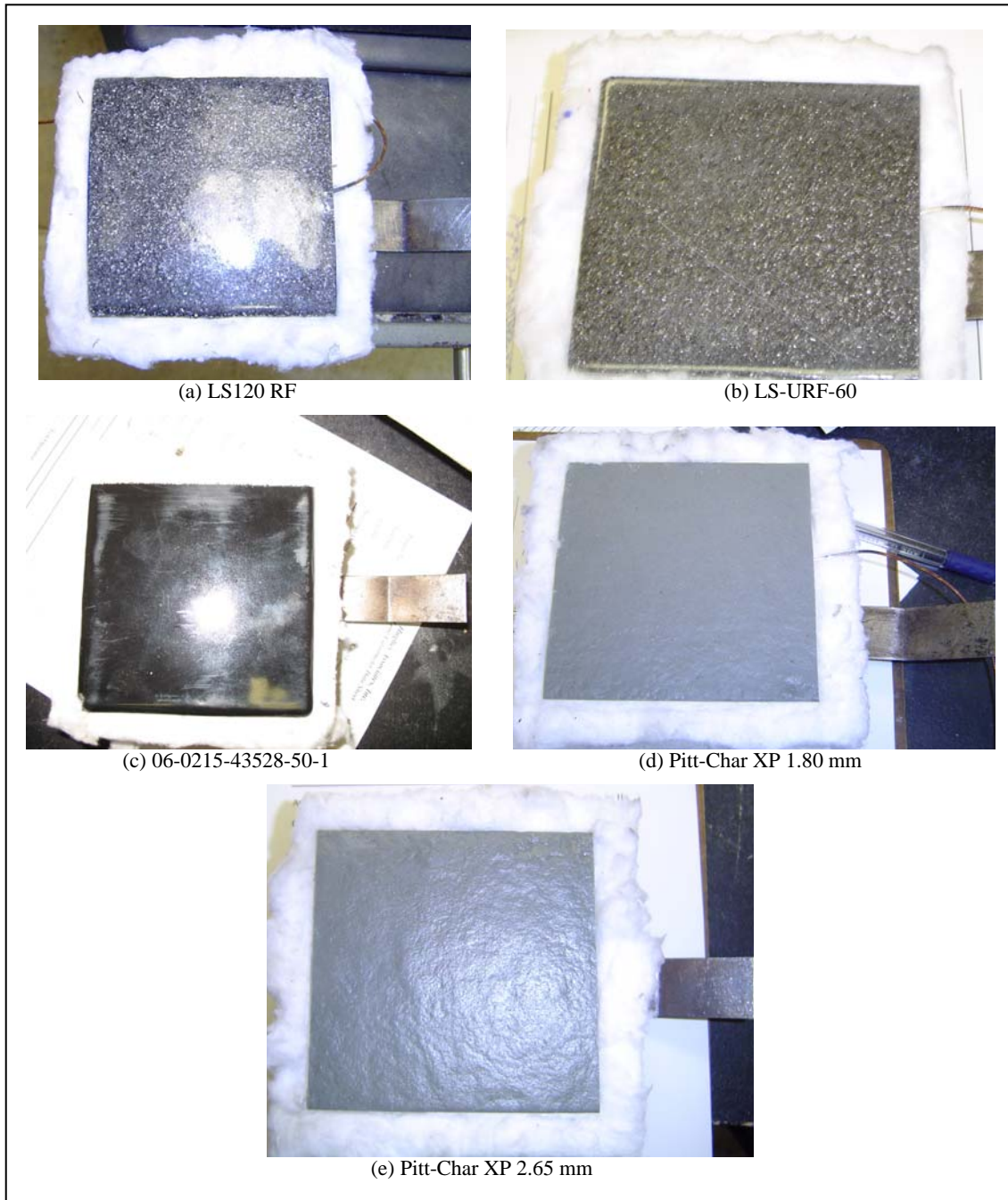


Figure 2. Pictures of coating samples tested.

### 3.3 Test Conditions

All tests were conducted in the horizontal configuration with an exposure heat flux of approximately  $100 \text{ kW/m}^2$ . Samples 1 through 3 were tested in duplicate, while the Pitt-Char XP samples (samples 4 and 5) were each tested once at the exposure heat flux.

One of the more important measurements in these tests was the temperature for the unexposed side of the aluminum plate. Sample holders recommended in ASTM E1354 are constructed of steel. These steel sample holders may serve as a path to conduct heat prematurely to the unexposed side of the sample. To reduce conduction effects on the unexposed side, samples were tested on a ceramic insulation holder, made with  $96\text{-kg/m}^3$  density ceramic insulation. The ceramic insulation was 125 mm by 125 mm, 25 mm thick with a 100- by 100-mm, ~3-mm-deep cut-out in the center of the insulation. The sample was placed into the cut-out so that the sample surface was flush with the ceramic insulation surface. The thermocouple measuring the unexposed temperature of the sample was placed between the sample and ceramic insulation with the thermocouple bead at the middle of the sample. The insulation was placed on top of a standard test tray filled with 25-mm-thick ceramic insulation to allow for easy placement of the sample underneath the heater.

Additionally, samples were tested 37.5 mm below the heater. In accordance with the standard, samples are typically tested 25 mm below the heater. However, these samples were expected to expand, which would have increased the heat flux incidental on the surface of the charred area. From (2), the heat flux from the heater will increase by approximately 10% with a 25-mm increase in height. Therefore, samples were moved down 12.5 mm to 37.5 mm below the heater in an attempt to compensate for the increase in heat flux as the sample expands during the test.

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## 4. Test Results

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The fire performance of the different coatings was compared based on the ignitability, heat release rate, and heat of combustion of the coatings. Pictures of each sample burning during the test are provided in figure 3.

A summary of the cone calorimeter test results is provided in table 2. All coatings were measured to ignite within the first 15 seconds of the exposure with the Pitt-Char samples that had the most resistance to ignition. Coating 06-0215-43528-50-1 performed significantly worse than the other coatings with heat release rates more than three times higher than those of the other coatings. The Pitt-Char XP coatings had the best fire performance, with the lowest heat of combustion and the lowest average and peak heat release rates. Figure 4 shows heat release rate curves from a single test on each coating. As indicated from the data in the summary table, the 06-0215-43528-50-1 coating was inferior to the other coatings, with an average heat release rate of  $509 \text{ kW/m}^2$  during

testing. The other coatings had average heat release rates similar to one another (119-154 kW/m<sup>2</sup>), but the LS120 RF coating burned nearly twice as long as other coatings.

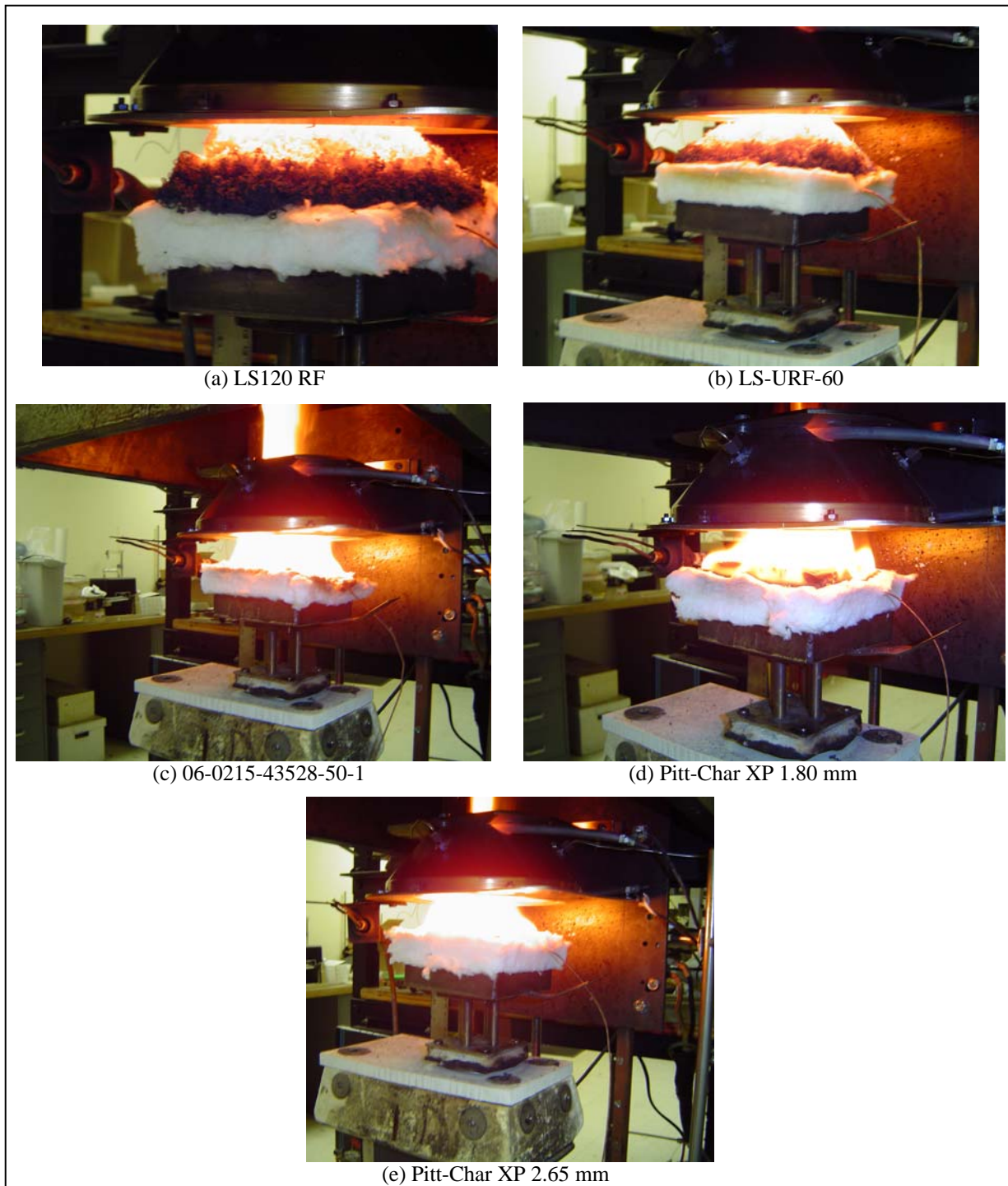


Figure 3. Pictures of coating samples during the cone test.

The temperature on the unexposed side of the aluminum plate measured during the cone tests is provided in figure 5. Overall, the LS120 RF coating was the most insulating while the 06-0215-

43528-50-1 coating provided the least protection to temperature rise on the unexposed side. The critical unexposed side temperature threshold for the testing was 350 °C. Table 3 summarizes the times required to reach the 350 °C threshold. As indicated from the temperature data in figure 5, the LS120 RF provided the best protection and on average, did not exceed the 350 °C threshold until 450 seconds (7.5 minutes). The LS-URF-60 was the second best with a time to exceed the critical temperature of 235 seconds (4 minutes) followed by the Pitt-Char XP samples.

Samples were inspected after the test to evaluate the state of the Intumescent char. Pictures of the samples after the test are provided in figure 6, and table 3 shows the char thickness measured with a ruler after the test. LS120 RF had a light, friable char that was 75 mm high in the center of the sample. The final char thickness was partly attributable to the reinforcement on the underside coating, shown in figure 7, bowing upward during the latter part of the test. The LS-URF-60 also had a light, friable char that was 35 mm in the center of the sample, but there was no reinforcement on the underside of the coating. The 06-0215-43528-50-1 coating had only a small amount of residual char (approximately 6 mm high) remaining after the test. The Pitt-Char XP samples had a rigid block of char remaining after the exposure. The Pitt-Char XP 1.8-mm char layer was 10 mm thick, and the Pitt-Char XP 2.65-mm char layer was 13 mm thick.

Table 2. Summary of cone calorimeter test results on the coatings.

Specimen ID	Irradiance [kW/m <sup>2</sup> ]	Initial Mass [g]	Percent Wt. Loss [%]	Time to Ignition [s]	Burn Duration [s]	Test Avg. Eff. HOC [MJ/kg]	Total Heat Released [MJ/m <sup>2</sup> ]	Test Avg. HRR [kW/m <sup>2</sup> ]	Peak HRR [kW/m <sup>2</sup> ]	Time of Peak HRR [s]	Test Avg. Smoke SEA [m <sup>2</sup> /kg]	Test Avg. CO <sub>2</sub> Yield [kg/kg]	Test Avg. CO Yield [kg/kg]
1-LS120 RF	100	40.7	86.0	1	684	28.8	100.7	147	242	12	340	2.03	0.034
2-LS120 RF	100	39.7	83.6	1	609	29.5	97.8	160	272	12	361	1.98	0.033
<b>Average</b>	<b>100</b>	<b>40.2</b>	<b>84.8</b>	<b>1</b>	<b>647</b>	<b>29.2</b>	<b>99.3</b>	<b>154</b>	<b>257</b>	<b>12</b>	<b>351</b>	<b>2.00</b>	<b>0.033</b>
1-LS-URF-60	100	22.9	86.0	4	330	26.7	52.5	158	272	11	609	1.88	0.032
2-LS-URF-60	100	21.5	85.6	3	344	27.0	49.7	143	240	10	432	1.90	0.035
<b>Average</b>	<b>100</b>	<b>22.2</b>	<b>85.8</b>	<b>4</b>	<b>337</b>	<b>26.9</b>	<b>51.1</b>	<b>151</b>	<b>256</b>	<b>11</b>	<b>521</b>	<b>1.89</b>	<b>0.034</b>
1-06-0215-43528-50-1	100	29.6	98.6	1	144	24.4	71.4	491	953	25	710	1.66	0.091
2-06-0215-43528-50-1	100	28.3	99.6	1	125	23.6	66.6	527	940	29	691	1.61	0.088
<b>Average</b>	<b>100</b>	<b>29.0</b>	<b>99.1</b>	<b>1</b>	<b>135</b>	<b>24.0</b>	<b>69.0</b>	<b>509</b>	<b>947</b>	<b>27</b>	<b>701</b>	<b>1.64</b>	<b>0.090</b>
1-Pitt Char XP1.8mm	100	22.5	66.7	12	201	20.6	30.9	151	248	15	717	1.36	0.028
<b>Average</b>	<b>100</b>	<b>22.5</b>	<b>66.7</b>	<b>12</b>	<b>201</b>	<b>20.6</b>	<b>30.9</b>	<b>151</b>	<b>248</b>	<b>15</b>	<b>717</b>	<b>1.36</b>	<b>0.028</b>
1-Pitt Char XP2.65mm	100	29.3	68.9	11	339	20.0	40.5	118	230	15	644	1.50	0.027
<b>Average</b>	<b>100</b>	<b>29.3</b>	<b>68.9</b>	<b>11</b>	<b>339</b>	<b>20.0</b>	<b>40.5</b>	<b>118</b>	<b>230</b>	<b>15</b>	<b>644</b>	<b>1.50</b>	<b>0.027</b>

Note: Initial mass and percent weight loss were based on the mass of the coating only.

HOC=Heat of Combustion

HRR=Heat Release Rate

SEA=Specific Extinction Area

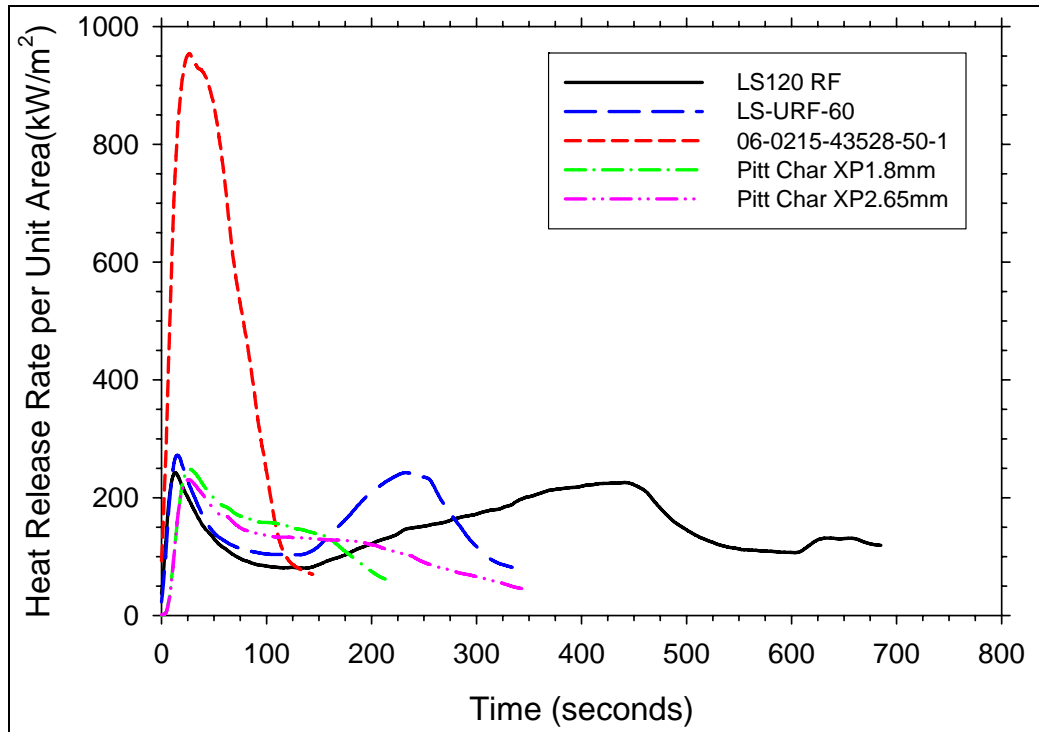


Figure 4. Heat release rates of the tested coatings.

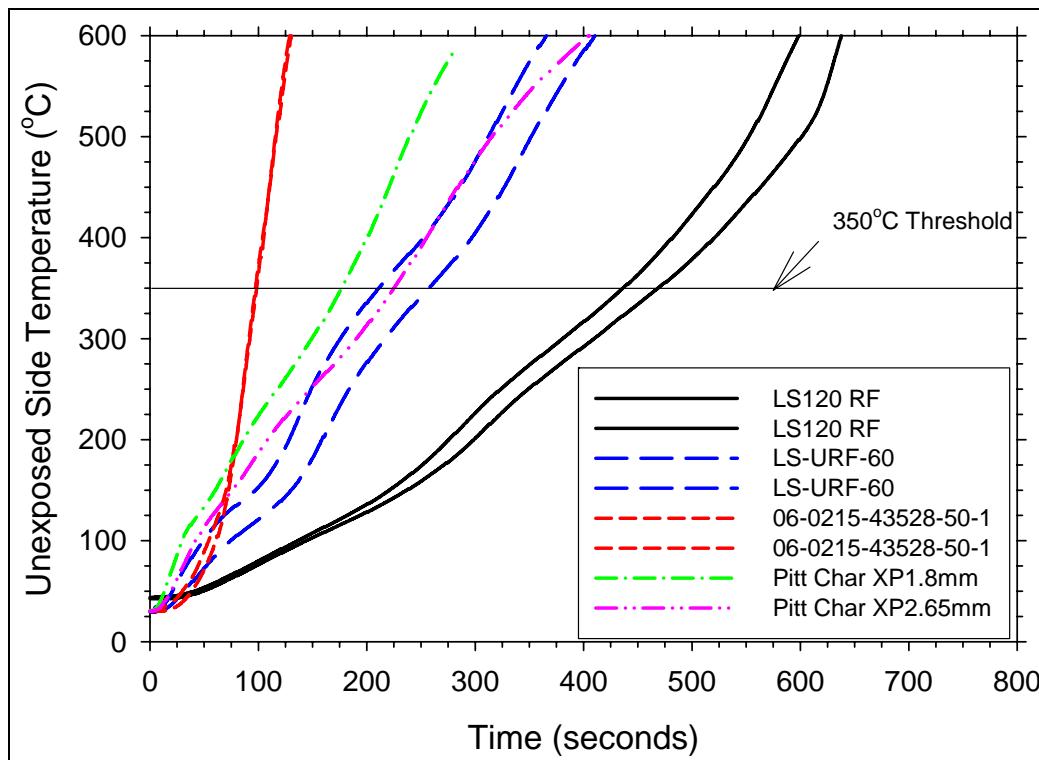


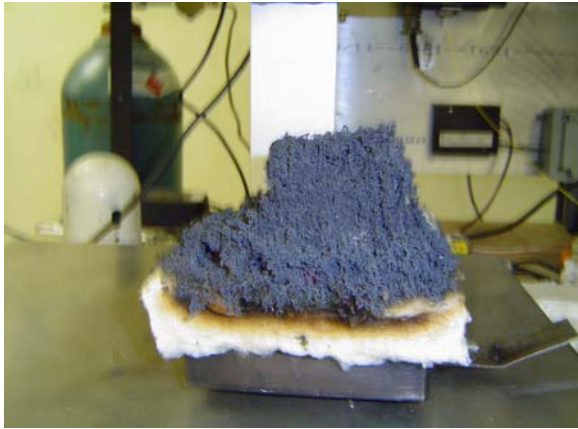
Figure 5. Unexposed side temperature for different coating samples.

Table 3. Time to exceed 350 °C temperature threshold.

<b>Specimen ID</b>	<b>Irradiance (kW/m<sup>2</sup>)</b>	<b>Time to Exceed 350 °C (sec)</b>
1-LS120 RF	100	469
2-LS120 RF	100	437
<b>Average</b>	<b>100</b>	<b>453</b>
1-LS-URF-60	100	212
2-LS-URF-60	100	258
<b>Average</b>	<b>100</b>	<b>235</b>
1-06-0215-43528-50-1	100	98
2-06-0215-43528-50-1	100	99
<b>Average</b>	<b>100</b>	<b>99</b>
1-Pitt-Char XP1.8 mm	100	177
<b>Average</b>	<b>100</b>	<b>177</b>
1-Pitt-Char XP2.65 mm	100	226
<b>Average</b>	<b>100</b>	<b>226</b>

Table 4. Coating intumescent char thickness measured after test.

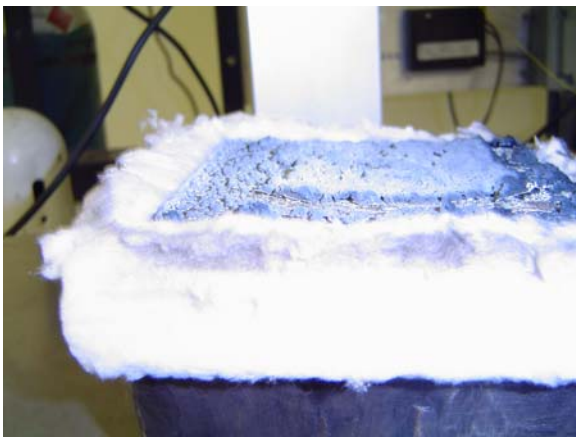
<b>Identification</b>	<b>Char Thickness (mm)</b>
LS120 RF	75
LS-URF-60	35
06-0215-43538-50-1	6
Pitt-Char XP1.8 mm	10
Pitt-Char XP2.65 mm	13



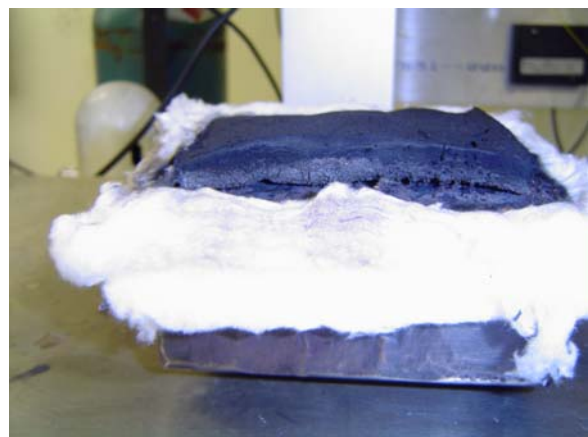
(a) LS120 RF



(b) LS-URF-60



(c) 06-0215-43528-50-1



(d) Pitt-Char XP 1.80 mm



(e) Pitt-Char XP 2.65 mm

Figure 6. Pictures of coating after testing.



Figure 7. LS120 RF char underside.

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## 5. Conclusions

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Cone calorimeter tests were conducted on five coating samples in the horizontal orientation with an exposure heat flux of  $100 \text{ kW/m}^2$ . Data were taken to compare the insulating and fire performance of the coating samples. Plots of these data for the different coatings are provided in figures 8 and 9. For comparison purposes, results from a cone calorimeter test with 7-mm-thick pine are also provided in the figures. The 7-mm-thick pine has a mass per unit area of  $3.2 \text{ kg/m}^2$ , which is similar to the average mass per unit of the tested coatings. The coating labeled 06-0215-43528-50-1 performed the worst from fire and insulating perspectives. The other coatings had similar heat release rates and ignitability performance, with the Pitt-Char XP samples having the best performance. The LS120 RF had the best insulating performance, followed by the LS-URF-60, with times to exceeding  $350^\circ\text{C}$  of 453 and 235 seconds, respectively. However, both of these coatings formed a light, friable char, and it is uncertain how they will perform when exposed to a large-scale pool fire where the higher velocity environment may degrade the char more readily. The insulating performance of the Pitt-Char XP coatings was only slightly less than the LS-URF-60, with the Pitt-Char XP 1.8-mm and 2.65-mm times exceeding  $350^\circ\text{C}$  of 177 and 226 seconds, respectively. In addition, the Pitt-Char XP formed a more rigid char which is likely to be more resilient to conditions during a large-scale pool fire.

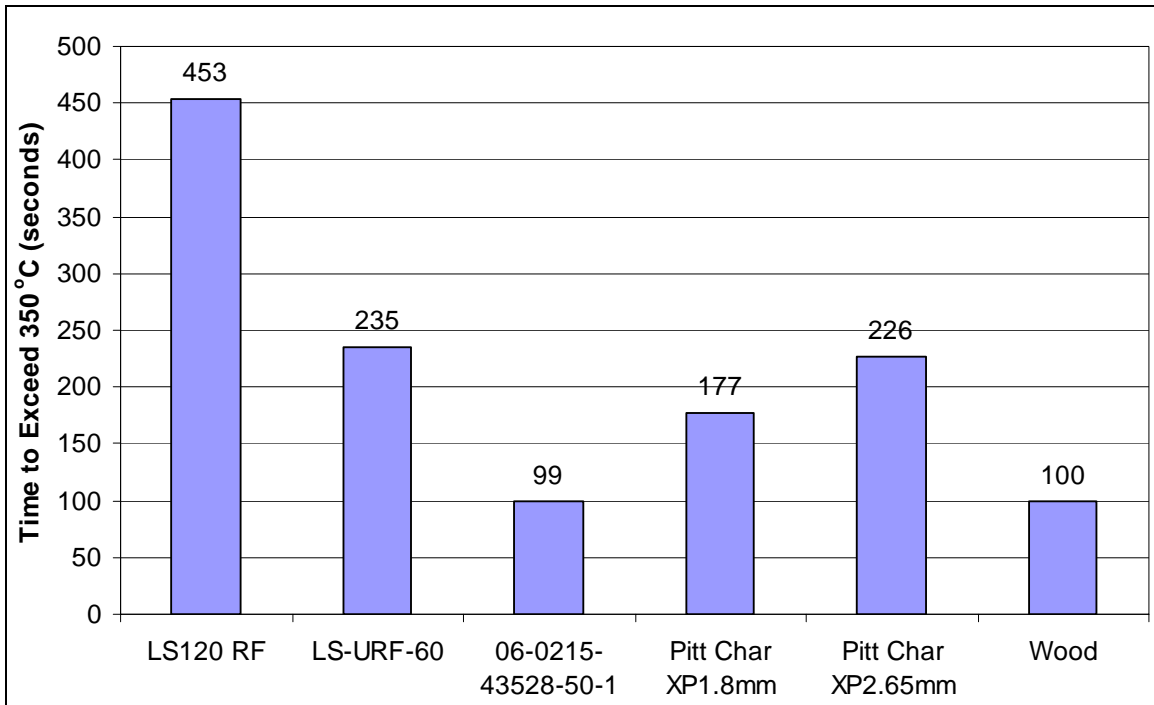


Figure 8. Comparison of time to exceed 350 °C on the unexposed side.

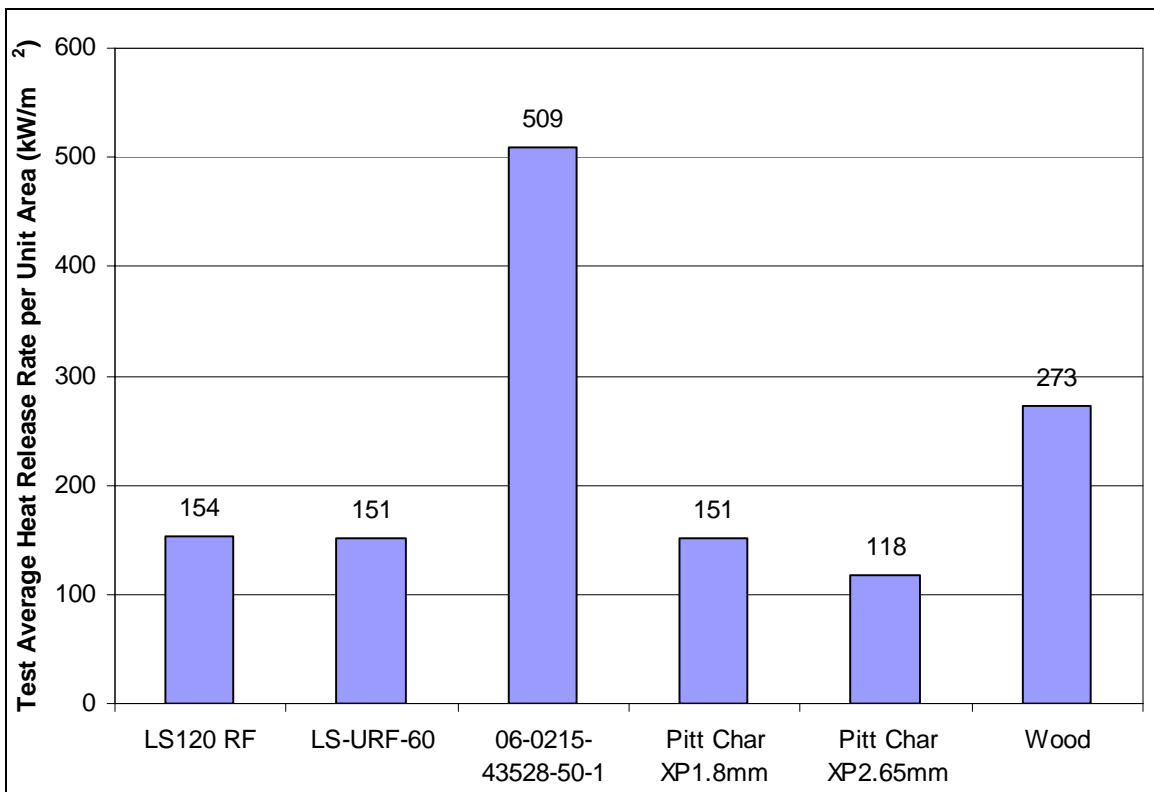


Figure 9. Comparison of test average heat release rates.

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## 6. Material Testing

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Depending on the material and application, there are several techniques and instruments designed for quality control testing. Material testing is the process of evaluating materials for characteristics that include but are not limited to durability and overall strength. We accomplish this by placing them through arduous tests to see how long the system endures before failing. In essence, any coating is expected to reliably withstand wears and tears of its target application.

The following coatings were tested as replacements for the containers:

1. Alkyd-based Firetex M78 Intumescent (IN) coating,
2. Water-based IN coating, epoxy primer (EP)/fire-resistant system (FS)/IN,
3. LS120 RF, LS-URF-60, and LS-URF-140 were tested at 60, 120, and 140 mils; this system is an un-reinforced 100% polyurea system. It is an aromatic polyurea with smoke suppression elements, combined with an aliphatic poly-aspartic with graphite particles.
4. Polyurea is a sprayable, rapid cure, two-component coating that provides physical properties that can enhance chip resistance, fire resistance, and blast mitigation.
5. Pitt-Char XP is a two-component 100% solid, flexible, epoxy Intumescent coating.

LS-URF-, Pitt-Char, and the polyurea are normally applied at much higher thicknesses because they are both 100% solid systems. LS-URF- was supplied at 60, 120, and 140 mils. Pitt-Char is designed to withstand much higher temperatures and for much longer times at its normal film thickness of 200 mils.

The polyurea is also normally applied at 60 to 80 mils, and its insulating capability depends on film thickness.

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## 7. Ballistic Testing

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Ballistic testing was conducted with a standard 0.22-caliber fragment-simulating projectile (FSP) weighing 1.1 grams. For velocities below 2300 ft/sec, the projectiles are fired from a 90-inch-long gas gun connected to a high-speed solenoid valve leading to a helium gas cylinder. Before firing, a pressure was selected; the gun was fired by manual closure of an electrical circuit that opened the solenoid valve. The projectile velocities were determined by a pair of printed silver grid paper screens in front of the specimen and connected to an electronic chronograph for time-of-flight measurements as shown in figure 10.

For all samples, the coated side was the strike face. The coatings increased the ballistic limit of the substrate.



Figure 10. Ballistic testing equipment.

## 7.1 Results

Of the six coatings tested, the Pitt-Char XP and the black, polyurea coating increased the ballistic limit about 20% for the same increase in areal weight (35%). The thicker film of the Pitt-Char coating added 15% more areal weight and increased the performance about 7%. This is to be expected. Ballistic performance should increase as areal weight increases. As the coating weight becomes a smaller part of the total system weight, the increase seen in performance will decrease. Included are data for polycarbonate for a similar thickness but half the areal weight to help us conceptualize the level of protection the samples are providing. The results are listed in table 5 and depicted in figures 11 through 24.

Table 5. Ballistic testing results of panels.

Construction	Thickness (in.)	Percent Increase	Weight (lb)	Area (ft <sup>2</sup> )	Areal Density (lb/ft <sup>2</sup> )	Percent Increase	V <sub>50</sub>	Percent Increase
Steel Control	0.032		0.436	0.334	1.31		651	
06-0265-4 3528-50-1	0.157	490	0.599	0.334	1.8	37	789	21
1.95 mm Pitt-Char XP	0.123	384	0.588	0.334	1.77	35	794	22
2.8 mm Pitt-Char XP	0.139	434	0.65	0.334	1.95	49	837	29
EP/ES/Primer Intum+TC	0.041	167	0.388	0.334	1.16		623	
No Fire Intumescent	0.08065	252	0.558	0.334	1.673	28	732	12
M78 /53039-1	0.07305	228	0.558	0.334	1.673	28	747	15
M78/ 53039-1	0.0941	294	0.558	0.334	1.67	28		
Polycarbonate	0.125				0.81		714	

## **7.2 Accelerated Corrosion Resistance**

The panels were subjected to accelerated corrosion exposure, salt fog ASTM B-117 (7) and Cyclic Corrosion exposure based on General Motors (GM) Standard Test 9540P (8). Salt spray resistance is based on procedures described in ASTM B117. This test is widely used by the paint industry as a quality control test and is not necessarily indicative of long-term performance of the coating.

GM 9540P is an accelerated cyclic corrosion test that was developed by the automotive industry to more accurately replicate long-term outdoor performance of coatings than the conventional salt fog test. Panels were evaluated with ASTM D 1654 (9) and ASTM D 714 (10).

## **7.3 Results**

The following samples were terminated after one week of ASTM-B-117 exposure.

- No fire Intumescent coating delaminated or failed miserably after one week in the chamber.
- M78 – top coated with MIL-DTL-53039, showed softening of the film.
- EP/epoxy solvent (ES)/primer IN/top coat (TC) showed 90% of wrinkling of the coating; however, the coating recovered after a couple of days.

The following samples were terminated after six weeks of GM 9540P exposure.

- No fire Intumescent coating failed with cracking along the scribed areas.
- M78 – top coated with MIL-DTL-53039, showed popping of the film along the scribed areas.
- EP/ES/primer IN/TC showed of wrinkling of the coating.

The black polyurea coating -06-0265-4–3528-50-1 exposed after 66 cycles in GM 9540P is being tested until failure. The panels have passed 1,500 hours of salt fog exposure and will continue through failure.



Figure 11. Back of panel-no fire intumescent.



Figure 12. Coated side-no fire intumescent.

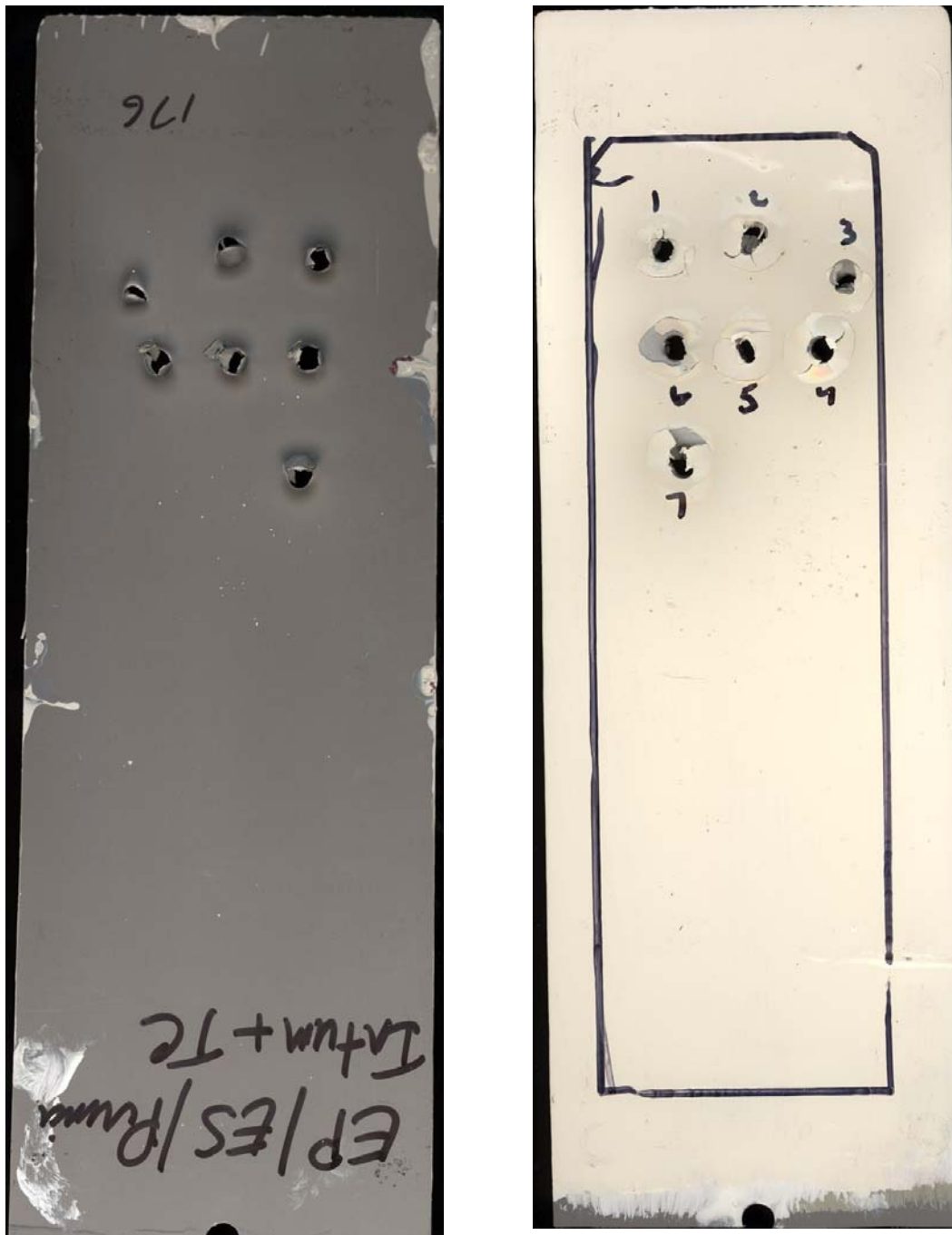


Figure 13. Back of panel and coated side -EP/ES/primer Intumescent+TC.

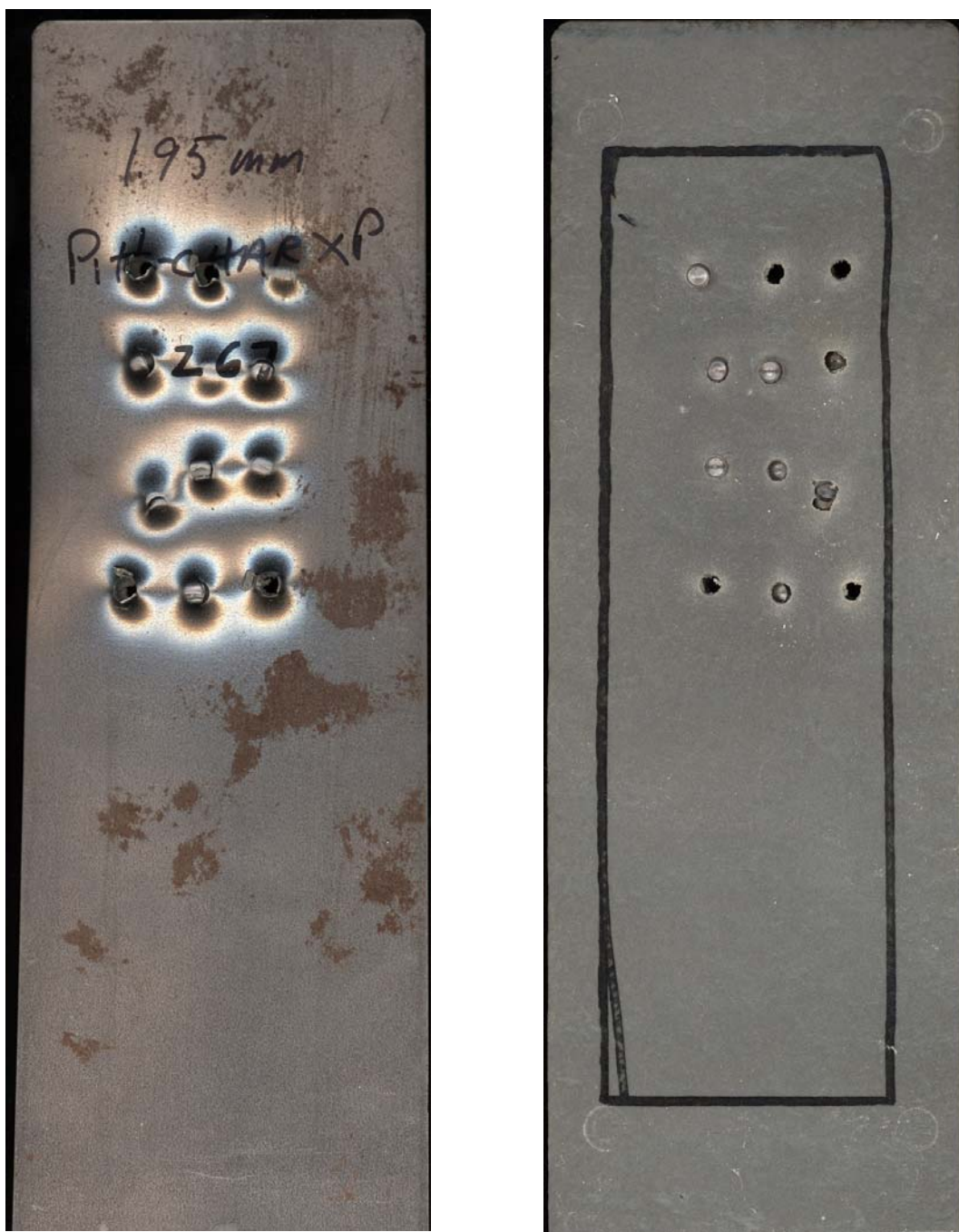


Figure 14. Back of panel and coated side 1.95 mm Pitt-Char XP.

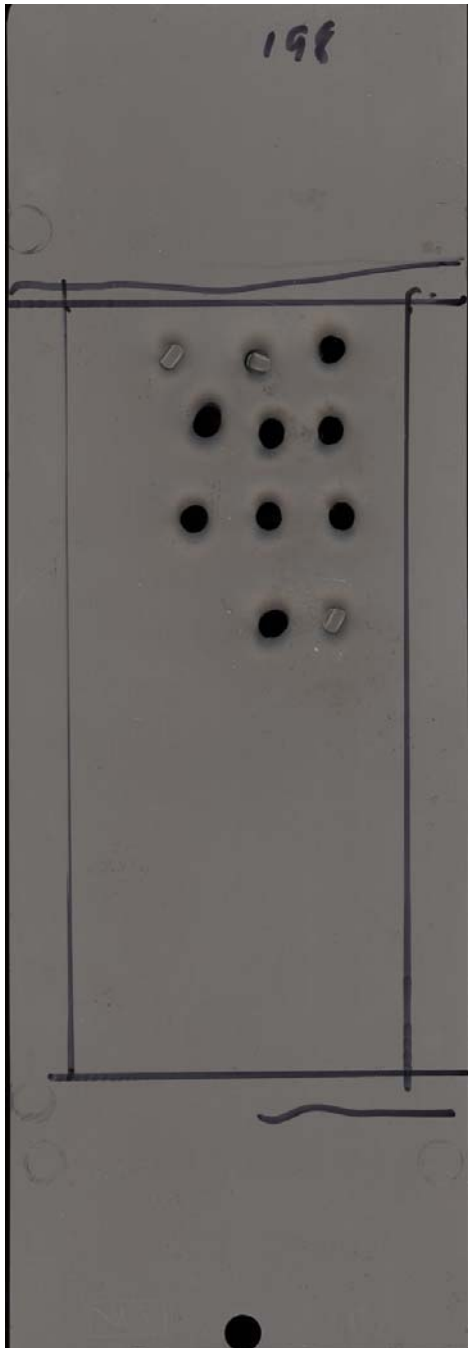


Figure 15. Bare panel-no coating.

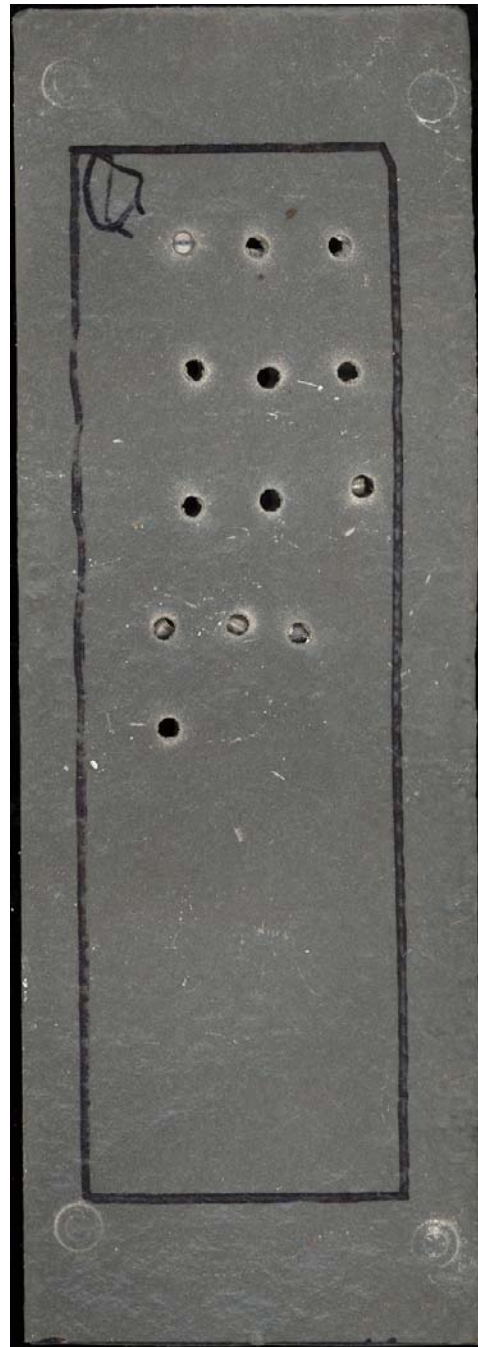


Figure 16. Coated side-2.8 mm Pitt-Char XP.



Figure 17. Back of panel and coated side M78 – top coated with MIL-DTL-53039.

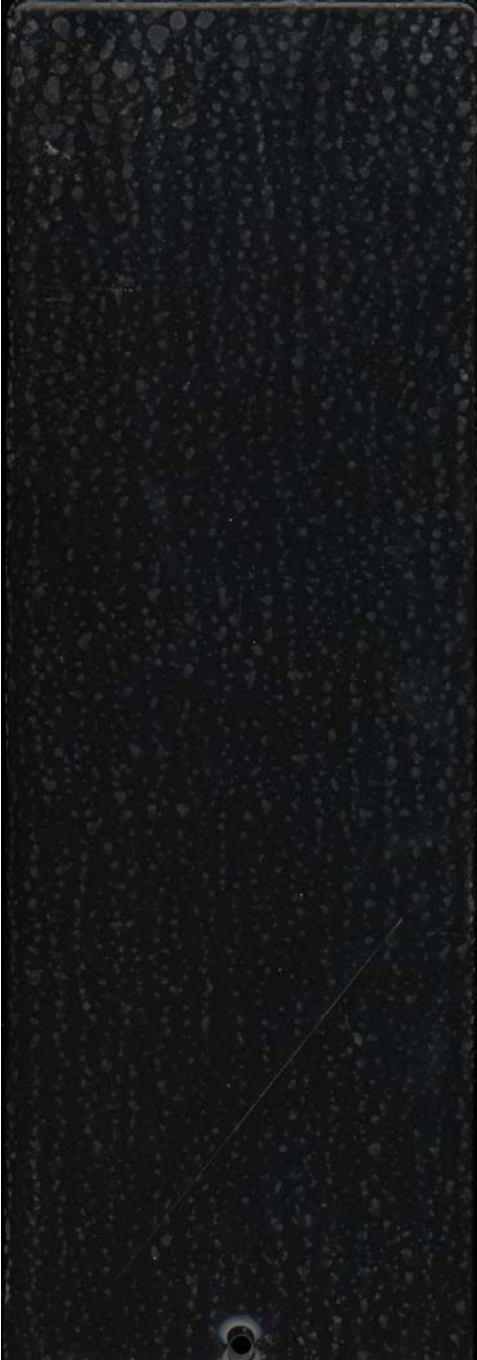


Figure 18. B-117 results 06-0265-4/3528-50-1.



Figure 19. GM-9540 results 06-0265-4/3528-50-1.



Figure 20. GM-9540 results M78 – top coated with MIL-DTL-53039.

#### **7.4 Impact Resistance**

The standard test for resistance to deformation (impact) was performed with a Gardner height impact tester that consists of a vertical guide tube and a cylindrical weight that is dropped on a punch resting on the test panel. Impact resistance can be described as a paint property that

quantitatively characterizes the adhesion and flexibility of a coating with respect to a rapid impact event. The impact-resistance test, based on ASTM D 2794 (5) was performed on all coatings with 100- to 160-lb-per-inch weights. Pitt-Char XP (1.78 mm) performed worse than the other coatings. Results are listed in table 6.

Table 6. Impact resistance results.

	<b>Direct Impact</b>	<b>Indirect Impact</b>	<b>Weight (lb/inch)</b>
LS-URF-60	pass	moderate	100
	pass	severe	120
	pass	severe	140
	pass	severe	160
LS-120-RF	pass	slight	100
	pass	slight	120
	pass	moderate	140
	pass	severe	160
LS-URF-60	pass	moderate	100
	pass	severe	120
	pass	severe	140
	pass	severe	160
Pitt-Char XP (1.78mm)	pass	severe	100
	pass	severe	120
	pass	severe	140
	pass	severe	160
Polyurea (Black)	pass	pass	100
	pass	pass	120
	pass	pass	140
	pass	pass	160



Figure 21. LS-120-RF.



Figure 22. LS-URF-60.



Figure 23. LS-URF-60.



Figure 24. Pitt-Char XP.



Figure 25. Polyurea (black) - 06-0265-4/ 3528-50-1.

## 7.5 Humidity

Panels were exposed to high humidity (95%) and high temperature (71 °C) for a total of 8 weeks. After 8 weeks of exposure, there were no changes in the black polyurea (06-0265-4–3528-50-1) coating system. It maintained good flexibility and retained its appearance.



Figure 26. Polyurea (black) - 06-0265-4/ 3528-50-1.

The control Intumescent and the water-based Intumescent coating EP/FS/IN showed softening and poor flexibility.

## **7.6 Plans**

The following combination of coatings will be tested as previous panels.

1. Polyurea over Pitt-Char XP,
2. Pitt-Char XP over Multiprime 97-680 primer, with Pitthane urethane TC,
3. Pitt-Char XP with Pitthane TC, without primer, and
4. LS-URF-140.

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## 8. References

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1. American Society for Testing and Materials. *Standard Test Method for Heat and Visible Smoke Release Rate for Materials and Products Using an Oxygen Consumption Calorimeter*; ASTM E1354. *1997 Annual Book of ASTM Standards*, Vol. 04.01: Building Seals and Sealants; Fire Standards; Dimension Stone, Philadelphia, PA, pp. 649-666, 1997.
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