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MARCH 1983

INTUMESCENT COATING DEVELOPMENT

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PREFACE

A lower weight thermal protection system was sought for the aft bulkhead of the shuttle external tank to replace the present ablative coating to provide increased payload. Work was performed under Contract Number NAS8-34544 by the University of Dayton Research Institute, Center for Basic and Applied Polymer Research, 300 College Park Avenue, Dayton, Ohio 45469. The work was administered under the direction of the George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, Alabama. Mr. Roger Harwell was the Project Monitor for this contract. The program was conducted during the time period July 1981 through July 1982.

This project was completed by Mr. Ival O. Salyer, Principal Investigator and Mr. Charles W. Griffen, Co-Principal Investigator. Important contributions to the work effort were made by Dr. Jack M. Butler, Dr. Richard P. Chartoff, Mr. Nicholas J. Olson, and Mr. Daniel E. Miller. Typing and compilation were ably performed by Mrs. Jeanne Drake.

SECTION 1
SUMMARY

A program has been completed at the University of Dayton Research Institute (UDRI) in which polyimide and phenolic intumescent coatings were evaluated as supplemental thermal insulation for the sprayed-on foam insulation (SOFI) on the aft bulkhead of the space shuttle external tank. The purpose of the intumescent coating was to provide additional thermal protection during lift-off in order to replace the ablative heat resistant layer with a lighter weight material for increased payload in the shuttle.

Exposure of 4.5" x 5" coated SOFI samples to the high level of radiant heat indicated excellent intumescence and thermal protection. During the thermal exposure, the intumescent coating and SOFI also expanded to effectively seal 1/8" wide cracks and 1/4" wedge cracks. However, in the same UDRI test, control samples of uncoated SOFI were charred only three-fourths of the way through. They did not develop the extensive cracking and charring which extended to the metal substrate, as observed on thermal exposure test samples at NASA.

To more nearly duplicate NASA test conditions, the size of the test sample was increased to 10" x 12". During thermal exposure, both uncoated and coated SOFI samples developed cracks after 25 seconds that continued to widen and char extensively. The coatings had intumesced between 5 and 10 seconds after the initial exposure but unexpectedly continued to burn after the radiant heat lamps were turned off. Testing of additional samples indicated entrapped solvent from the intumescent coatings as a probable cause of continued burning. Further research on solvent removal, a nonflammable solvent system, and designed thermal stress relief patterns are indicated research routes toward the successful solution to the overall problems.

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Due to insufficient time and funds the planned test of resistance to 165 dB vibration, as encountered during lift-off, was not evaluated. Three gallons of a solvent (acetone) solution preferred phenolic intumescent foam formulation are being delivered to NASA-Huntsville for their evaluation.

SECTION 2

INTRODUCTION

The modified polyisocyanurate foam insulation on the aft bulkhead of the shuttle external tank will not hold up for the necessary time (120 seconds) to the high radiant heat flux (8-12 BTU/ft^2·sec) and vibration (up to 165 dB) that is encountered during lift-off. Thus it cannot provide adequate thermal protection to the cryogenic fuel. To overcome this problem, NASA has installed underneath the polyisocyanurate foam, an additional thermal insulation system consisting of 0.4 inch thick, 18 lb/ft^3 ablative material (420 pounds). This heavy ablative material adds undesired extra weight to the shuttle system.

An effective lower weight intumescent thermal protection system was sought that could be applied directly to the top surface of the polyisocyanurate foam. In addition to reducing the weight, and providing thermal protection and vibration resistance, the intumescent coating should also seal over small cracks that may develop in the SOFI due to thermal contraction and expansion during fueling, or take-off, retain exterior durability for one year, and be readily applied.

SECTION 3

CONCLUSIONS

1. On small (4.5" x 5.0") samples of SOFI, polyimide and phenolic intumescent coatings provided excellent protection from exposure to the radiant heat flux of 8 BTU/ft^2·sec for the required two minutes.

2. During the thermal exposure test, the intumescent coating and SOFI expanded to close 1/4" wedge cracks and 1/8" straight cracks to effectively protect the lower layer of SOFI and subsequently the liquid hydrogen storage tank.

3. Regular 1/4" cracks (with straight edges rather than slanted, as in the wedge cracks) were not closed by the intumescent coating or expansion of SOFI during thermal exposure with resultant excessive charring and widening of the crack.

4. On 10" x 12" samples of SOFI, the polyimide and phenolic intumescent coatings, as applied and tested intumesced in 5 to 10 seconds after initial exposure, but did not prevent surface cracks from forming in the SOFI and the subsequent charring and burning of the SOFI along the cracks. This was attributed, in part at least, to the entrapped solvent from the intumescent coatings. Surface cracking occurred 25 seconds after initial exposure.

5. Photographs taken during the radiant heat tests, and separate oven heating tests (various temperatures) indicate that cracking of SOFI on exposure to 8 BTU/ft^2·sec radiant heat may occur in the following sequence:
   - Early in radiant heating, there is a rapid increase in the temperature and pressure of the Freon gas in the closed cell foam;
   - The increased temperature and pressure causes rapid volumetric expansion of the SOFI, cell wall rupture, and crack initiation; and

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- Once initiated the cracks enlarge and deepen during continued heating with attendant rapid rise in back side substrate temperature.

6. If the postulated mechanism is correct, a promising approach would be to investigate various thermal stress relieving mechanisms including specifically:

    - Make the top 1/2" of the SOFI insulation (for the aft bay area) of open celled polyisocyanurate foam. This should eliminate the rapid pressure increase in the early part of the heating cycle and delay the onset of the cell-rupturing expansion and cracking.

    - The SOFI might have to be somewhat thicker in this area (to provide the cryogenic insulation), but the weight penalty should be significantly less than for the heavy ablative undercoat now used.

    - The technology for making open celled rigid foams (dual, triple pneumatogen systems) is well known, but would have to be developed for this specific poly-isocyanurate foam system.

SECTION 4
RECOMMENDATIONS

1. It is recommended that additional exposure tests should be completed on samples with additional drying time to remove more solvent.

2. It is recommended that solvents with reduced flammability such as methyl formate or dimethyl carbonate should be evaluated to check the role of solvents in initiating and supporting combustion of the SOFI.

3. It is recommended that additional thermal exposure tests should be run on uncoated and coated SOFI samples with various crack configurations and spacing to investigate the ability of the SOFI to expand during initial exposure to seal the cracks but also to prevent additional cracks from forming during the continued thermal exposure.

4. Investigate the thermal stress relieving mechanisms such as making the top 1/2" of the polyisocyanurate foam in the aft bay area open celled.

5. It is recommended that additional methods of application of the intumescent coatings should be evaluated including spray application and multiple layers. The objective would be to reduce the amount of flammable solvent that is entrapped in the SOFI.

SECTION 5
TECHNICAL DISCUSSION

5.1 INTUMESCENT COATING FORMULATION

Two different types of coatings which are used to reduce
fire hazards are the nonflammable coating and the insulative
coating. The nonflammable coatings do not contribute fuel to
the fire or promote flame spread but they provide only slight
protection of the substrate. Consequently, these coatings are
used primarily on nonflammable substrate where the flammability
of the coating itself is the major concern.

The insulative coatings may contain insulative materials
such as asbestos or vermiculite but also include those coatings
which melt, bubble, and swell to form a thick, nonflammable,
multicellular insulative foam. These intumescent coatings
have been developed since 1938 (U.S. Patent 2,106,938).
Currently, many of these coatings contain an inorganic acid
which decomposes between 100° and 250°C resulting in a softened
resinous layer. Foam is generated by polyol esterification and
dehydration and by gases from the decomposition of the amines and
halogenated materials. As the temperature increases, the ester
gels and chars to form the thick, nonflammable, multicellular
insulative foam. For optimum foaming, the gases must be
released above the melting or softening temperature and before
the gelation temperature.

Polyimides are used primarily for their high thermal
resistance. They can be prepared by the reaction of aromatic
dianhydrides, such as pyromellitic dianhydride, with alcohols
to form a diacid-diester. Further reaction with a diamine
yields the diammonium salt which, upon heating, dehydrates to
form the polyammic acid. Further heating yields primarily the
polyimide and an alcohol which, depending on the reactants and
conditions, may act as a built-in blowing agent to produce an
insulative foam; Reference Figure 1.

Pyromellitic
Dianhydride   Diacid-Diester   Diamine

Diammonium salt   Polyammic salt   Polyimide
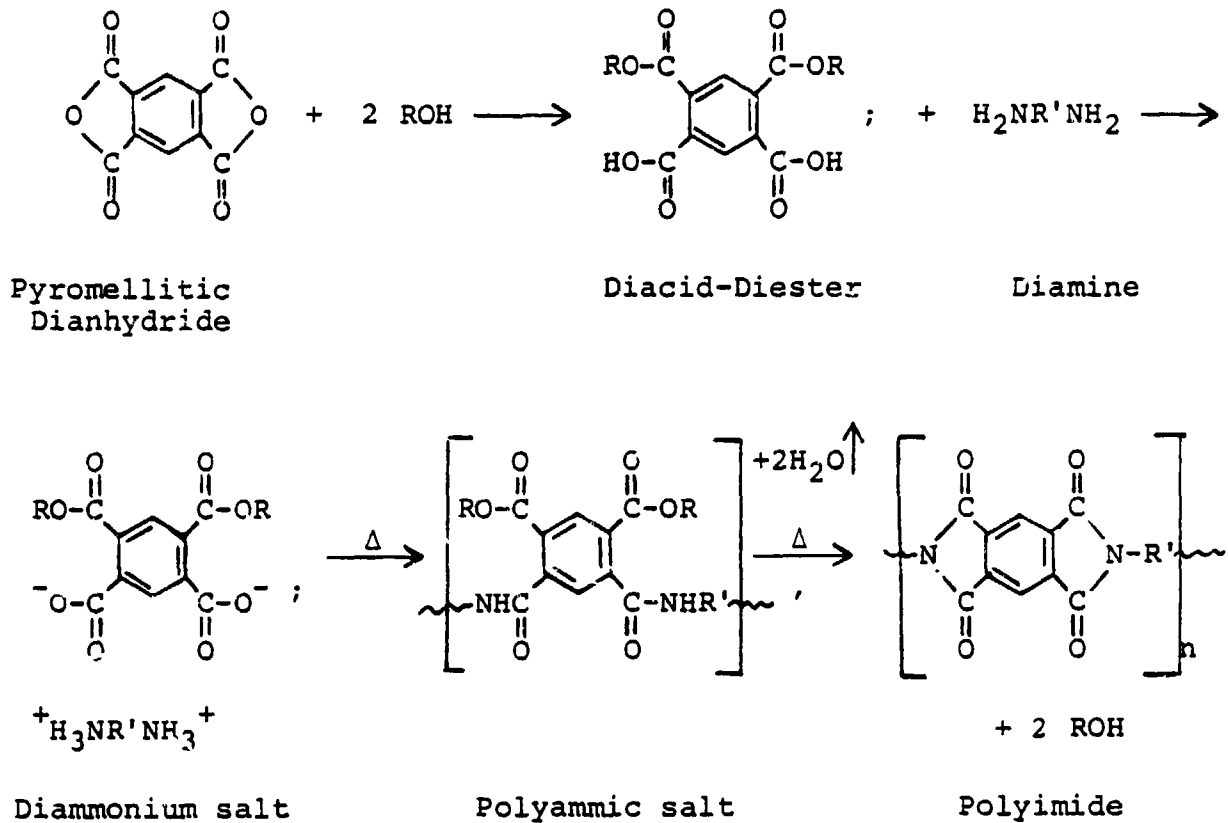
Figure 1. Polyimide Reaction.

Polyimide resin R7271 (Monsanto) has been used by itself
or with minor additions to produce preformed foam and intumescent
coatings for several high temperature applications. The commercial
resin is B-staged to form a fine, gold-colored powder. Using
carefully selected heating conditions, the polyammic acid melts
and upon further heating, releases alcohol as a built-in blowing
agent. The resultant very low density foam cures with additional
heat to form the low density, flexible, heat resistant polyimide
foam. Thermogravimetric analysis of the polyimide foam as shown
in Figure 2 illustrates the thermal resistance in comparison with
polyisocyanurate (SOFI) and polyurethane foams.

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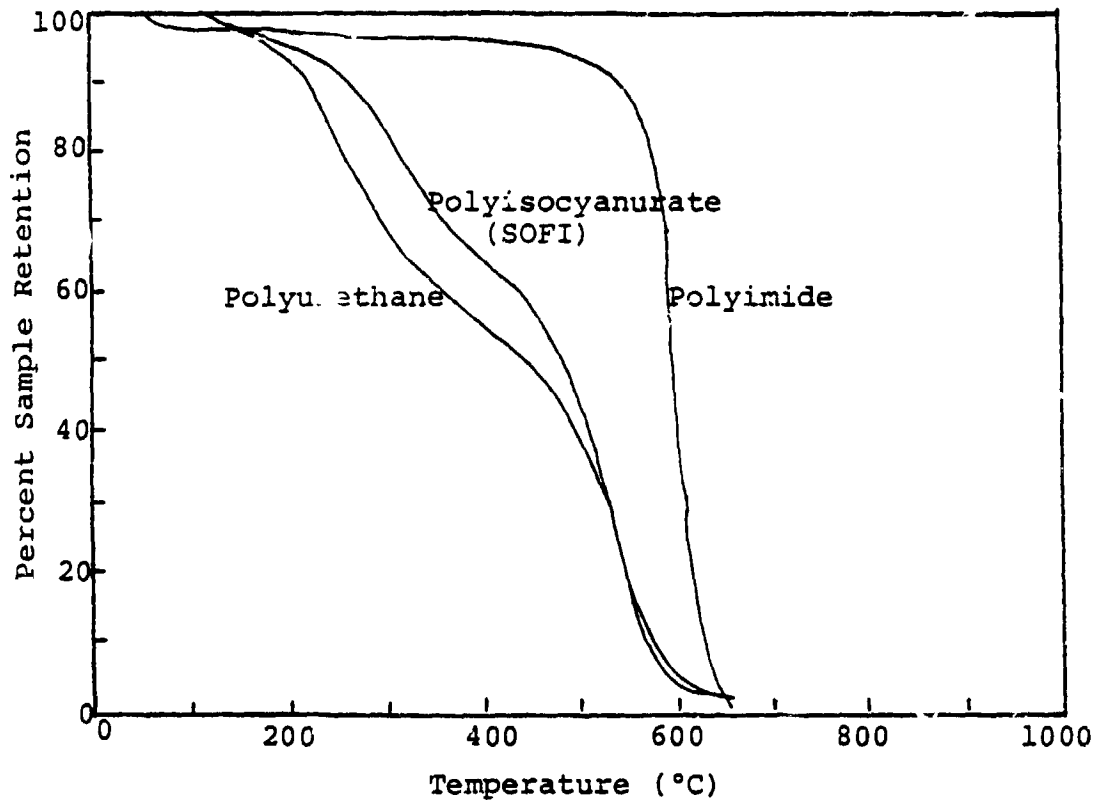
Figure 2. Thermogravimetric Analysis of Foams in Air
at 10°C/Minute.

The B-staged resin may also be combined with a solvent,
titanium dioxide, glass microballoons and glass fibers to form
a brushable intumescent coating, e.g., Table 1. Upon exposure
to fire or heat, the hard, dried coating will intumesce to form
a heat resistant, insulating foam. Thermogravimetric analysis
of the polyimide coating as shown in Figure 3 illustrates
the intumescence and cure at 100-200°C followed by thermal
degradation at 600°C.

Phenolic resins have also been used for many heat resistant
applications in many different forms. Phenol and formaldehyde
are reacted to provide low molecular weight ortho and para
isomers which, upon heating, crosslink principally through
methylene bridged phenolic units (see Figure 4). As intumescent

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TABLE 1

POLYIMIDE INTUMESCENT COATING

	Weight Percent
Polyimide resin R7271	62.76
Methanol	29.76
Titanium dioxide	1.65
Glass microballoons, A16	0.83
Glass fibers	5.00
	100.00
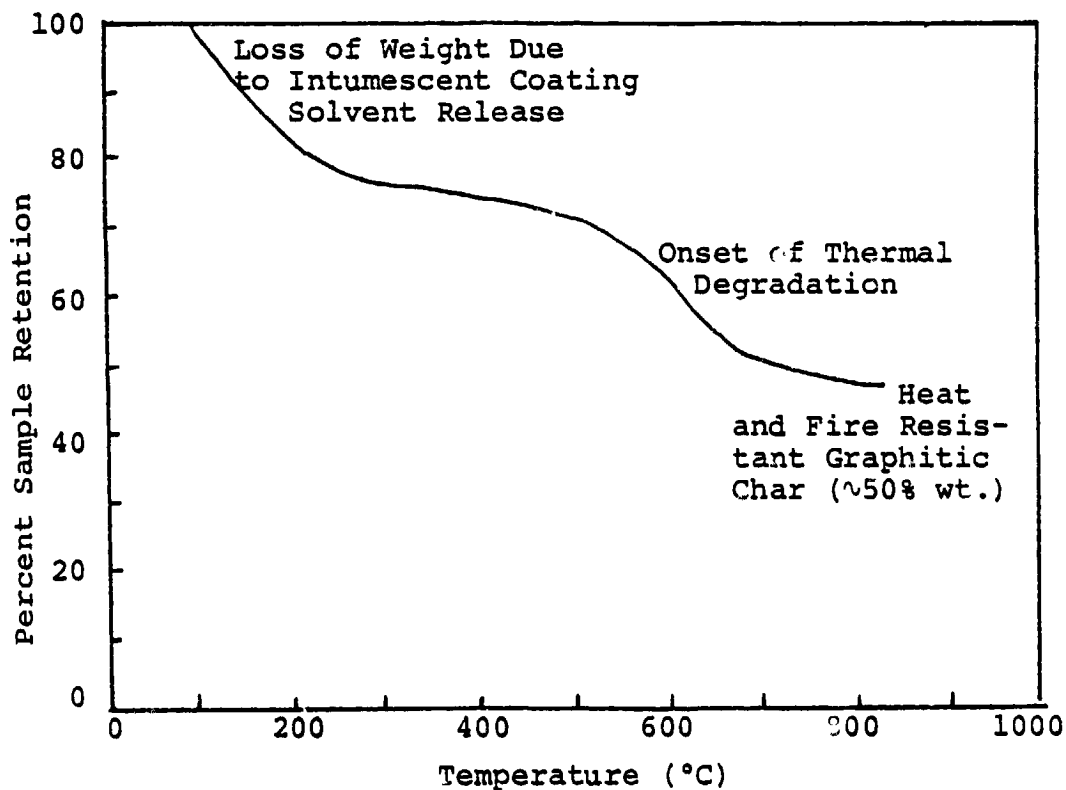
Figure 3. Thermogravimetric Analysis of Polyimide Intumescent Coating in Nitrogen at 20°C/Minute.
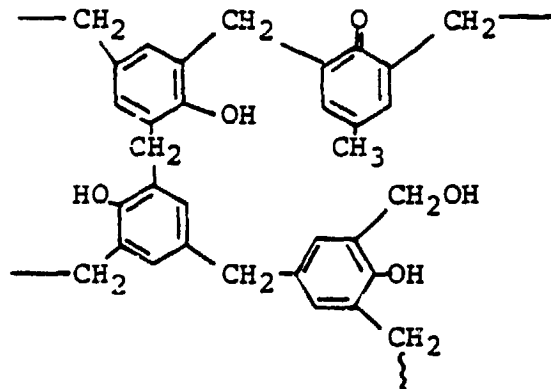
Figure 4. Crosslinked Phenolic Resin.

coatings, however, they require a separate blowing agent for increased foam volume and better uniformity. The basic phenolic intumescent coating used in this application is listed in Table 2.

TABLE 2
PHENOLIC INTUMESCENT COATING

	Weight Percent
Resinox R736 (Monsanto)	53.70
Acetone	38.26
Titanium dioxide	1.42
Glass microballoons, A16	0.71
Azo-bis-isobutyronitrile	5.37
Surfactant, L540	0.54
	100.00

This is a brushable coating which, when dried and subsequently exposed to fire or heat, will also form an intumescent, heat resistant, insulating foam.

5.2 THERMAL EXPOSURE TESTS

5.2.1 Preliminary Tests and Equipment

Preliminary thermal resistance tests were run with a Fisher laboratory burner using polyurethane foam coated with

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the phenolic and polyimide intumescent coatings. These results
indicated excellent thermal protection to the foam for over
two minutes except for the samples with precut cracks. These
tests were similar to some of the thermal exposure tests that
had been used in the earlier development of the intumescent
coatings. Factors affecting the thermal resistance tests
included the source of heat (e.g., a glass blowers torch),
the thermal conductivity of the substrate, the method of thermal
exposure, the position of the sample during the fire test, and
the type of intumescent coating. To minimize these variables,
subsequent thermal exposure tests on this project were
standardized using a radiant heat quartz lamp bank (see Figures
5 and 6) with a heat flux of 8 BTU/ft^2·sec for 120 seconds. This
equipment is also known as the Tri-Service Thermal Radiation
Test Facility and is used to simulate nuclear flash thermal
radiation. In addition to the quartz lamps, it also includes
a wind tunnel for aerodynamic loads, and a mechanical loading
device for tension and bending loads.

Some modifications in operations were required
since most of the previous testing with the quartz lamp bank had
used higher heat flux of 70 to 110 BTU/ft^2·sec but for shorter
periods of time, three to four seconds. The SOFI panels were
mounted vertically, approximately 12" from the lamps. A
thermocouple was mounted in the center of the back surface of
the sample to record the rise in temperature. Calibration
runs with a Hycal Asymptotic Calorimeter were made before each
series of thermal exposures to maintain the heat flux of
8 BTU/ft^2·sec. Initially, 30 preliminary runs were made varying
the distance, control voltage, and current. Five calibration
runs were then conducted, five minutes apart. The current of
81 amps gave a heat flux of 2.17 cal/cm^2·sec (8 BTU/ft^2·sec)
which was maintained by varying the control voltage. The plane
of the calorimeter and the surface of the sample at the
beginning of the tests were the same.
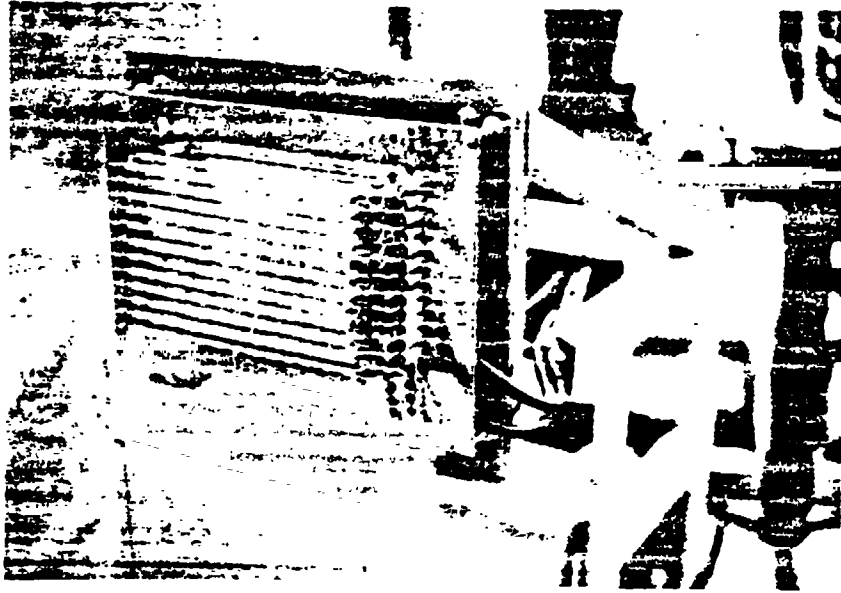
Figure 5.   Radiant Heat Quartz Lamp Bank.
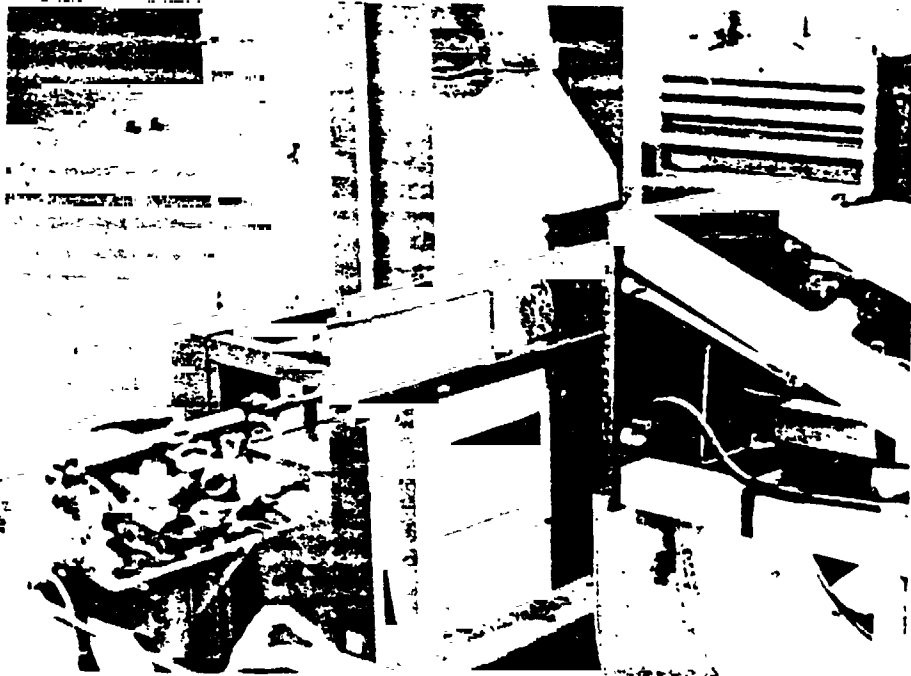
Figure 6.   Thermal Exposure Test (Quartz
            Lamp Bank at Right Side).

5.2.2   Regular Radiant Heat Thermal Exposure Tests

5.2.2.1 Samples 1-4 (Ref. Table 3)

The initial thermal exposure tests on the 1-1/4" thick SOFI were run using 4.5" x 5.0" panels to provide effective exposure from the approx. 5" x 6" radiation source.

The uncoated SOFI control samples formed a hard surfaced, charred layer with no surface cracks, and still had a 3/8" layer of uncharred foam remaining after the two minute exposure. These results would be repeated in subsequent tests and it presented problems in interpreting the exposure results of the coated samples. The three other SOFI samples had been coated with the polyimide coating. The results on these samples indicated additional thermal protection with 40% additional thickness of uncharred foam; there was no significant difference in protection provided by 13 to 19 mil coating thickness, there were no indications of cracks developing during exposure, the intumescent coating did not cover and protect 1/4" wide pre-cut cracks, and applying the coating to the sides of the foam did not significantly improve the thermal protection. Thermo-couples on the back surface were used on subsequent samples to record the temperature rise.

5.2.2.2 Samples 5-10 (Ref. Table 3), 4.5" x 5.0"

The uncoated control again showed good thermal protection with low back surface temperature rise and no surface cracks. With polyimide intumescent coatings, the SOFI expanded during thermal exposure to seal 1/8" cracks and 1/4" wedge cracks. Glass fibers in the polyimide coating provided additional wet strength to the intumescent coating during foaming and dry strength to the charred area. Aluminum foil as a surface laminate conducted more heat than reflected and although the foil melted, the net thermal protection was very good.

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5.2.2.3   Samples 11-16 (Ref. Table 3), 4.5" x 5.0"

The uncoated SOFI control continued to show very good thermal resistance with low back surface temperature rise and no surface cracks. The initial radiant heat tests on the phenolic coated samples were run with good results with the phenolic coating by itself and also in combination with the polyimide coating. A combination of glass fibers for added strength to the coating and increased titanium dioxide for increased reflectance resulted in excellent thermal resistance although the intumescent layer was not as high as with regular coatings.

5.2.2.4   Samples 17-35 (Ref. Table 3), 4.5" x 5.0" and 10" x 12"

Samples with precut 1/16" cracks provided very good thermal protection with the cracks closing during the exposure and no new cracks forming. However, 1/4" straight and wedge cracks continued to char extensively along the cracks even with increased coating weights.

Two commercial intumescent coatings, Athey 1401 and Athey 1403, provided very good thermal protection although they are not developed for exterior applications. When Athey 1401 was applied over the polyimide coating, dried and exposed, there was extensive charring to the substrate probably due to entrapped solvent. The results were very good with the Athey 1403 over the polyimide coating.

Included in this series were the first larger samples (10" x 12"). The results on these three coated samples compared favorably with the smaller versions with very good thermal protection except in the samples with precut cracks and the samples that developed wide cracks.

5.2.2.5   Samples 36-51 (Ref. Table 3), 4.5" x 5.0" and 10" x 12"

Additional small samples with the precut 1/8" cracks and the intumescent coatings still looked very good.

Reduced solvent and no blowing agent resulted in the only
small sample where 1/16" crack did not close and there was
extensive char along the crack. Carbon fiber cloth laminate
on the face with the phenolic coating provided very good thermal
protection but the cloth was not secure after the test was
completed. RTV silicone caulk in precut 1/4" cracks did not
protect the exposed edges.

In the larger 10" x 12" samples, the
charring and cracking were much more extensive than in the
4.5" x 5.0" samples. Even though the uncoated control samples
were charred and cracked, there were areas similar in size to
the smaller samples in which the charring did not extend to the
metal substrate. Dividing the larger uncoated SOFI samples in
four sections similar to the smaller sections and filling these
1/4" cracks with phenolic intumescent coating still resulted in
extensive charring although it was felt that this was due to
entrapped solvent from the coating. "Precuring" the SOFI in
an oven prior to the thermal exposure apparently did not
cure the foam enough to prevent cracks from forming during the
thermal exposure. Additional attempts with combinations
of laminates and precut cracks also did not prevent cracks
extending to the aluminum substrate accompanied by extensive
burning and charring of the SOFI.

Exposure to the elevated temperatures
for accelerated aging in the weatherometer resulted in surface
bubbles due to the entrapped solvent and also to decomposition
of some of the blowing agent.

Preliminary work on solvents with reduced
flammability due to their high oxygen content were encouraging;
these included methyl formate, $\overset{\text{O}}{\underset{\text{HC-O-CH}_3}{\parallel}}$, and dimethyl carbonate,

$\text{CH}_3\text{-O-}\overset{\text{O}}{\overset{\parallel}{\text{C}}}\text{-O-CH}_3$, both with 53% oxygen.

TABLE 3

SOFI THERMAL EXPOSURE TESTS
8 BTU/FT^2·SEC FOR 120 SECONDS
SAMPLE SIZE 1.25" x 4.5" x 5.0" UNLESS OTHERWISE INDICATED

Sample Descriptions And
Back Surface Temperature Plots

Results

1. Uncoated Control.

   Hard, charred crust on surface; SOFI did not crack or char completely to the aluminum substrate, top edges shrank, 3/8" thick uncharred layer.

2. Polyimide intumescent coating (PIC), 13 mils (approximately 62 lbs /700 ft^2).

   Better thermal resistance than control; charred surface was not as hard but the uncharred layer was 5/8" thick; no cracks.

3. PIC, 19 mils, sides also coated.

   Results similar to number 2, coated sides and thicker coating did not significantly improve the thermal resistance.

4. PIC, 18 mils, after coating dried, 1/4" wide crack cut in SOFI, extending to aluminum substrate

   Coated area provided good thermal resistance but 1/4" crack widened and charred heavily on both sides.

Cross-sectional view after thermal exposure. Results similar to number 1 with no cracks or charring to the metal substrate.

Time Minutes

Back Surface Temperature, °F
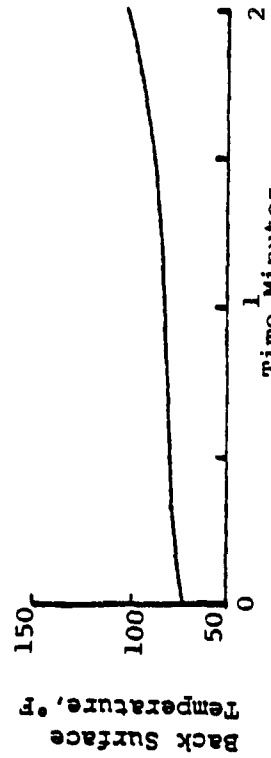
5. Control, uncoated, repeat of number 1.

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Good thermal resistance similar to number 2. Increased coating application did not significantly improve thermal resistance. (Additional charred layer was sheared off by fire test shutter at completion of test.)
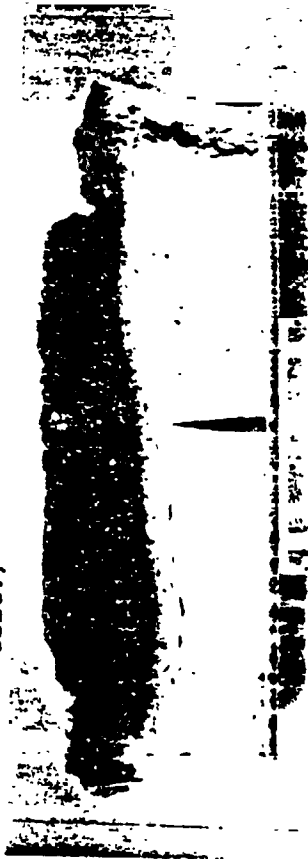
SOFI expanded to close 1/8" crack at top. Very good thermal protection with little charring of SOFI. (Additional charred layer was sheared off by fire test shutter at completion of test.)
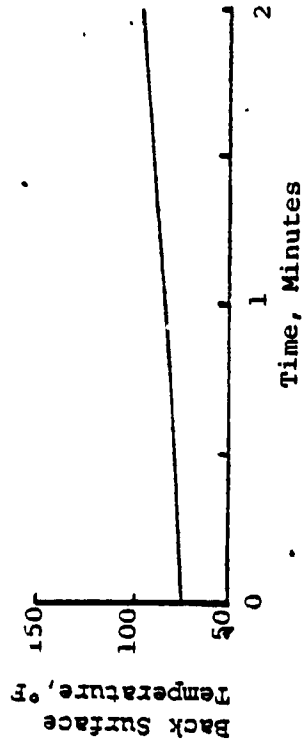
6. Polyimide intumescent coating (PIC) 24 mils.

Time, Minutes

Back Surface Temperature, °F
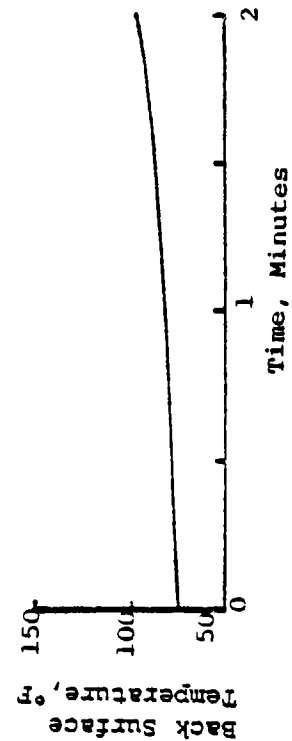
7. PIC, 40 mils, 1/8" crack cut after coating dried.

Time, Minutes

Back Surface Temperature, °F

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SOFI expanded to close the wedge crack. Very good thermal protection with little charring of SOFI; no cracks. (Additional charred layer was sheared off by fire test shutter at completion of test.)
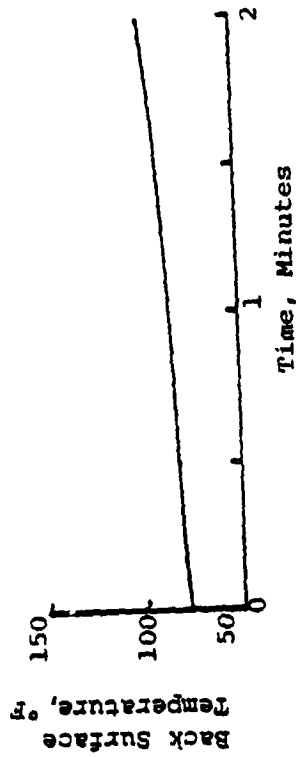
8. PIC 43 mils, 1/4" wedge crack cut after coating dried.

Slight improvement in strength of charred layer with little effect on intumescence (additional charred layer was sheared off by fire test shutter at completion of test.)
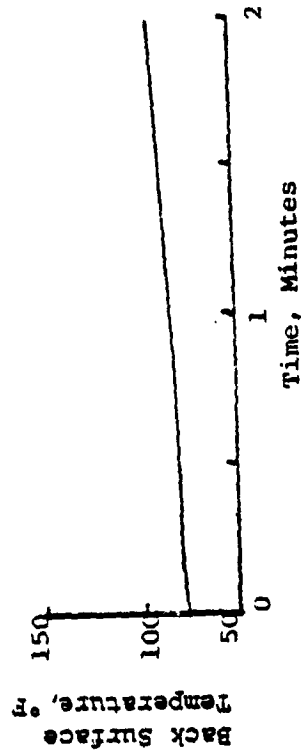
9. PIC, 40 mils, glass fibers in coating for added strength during exposure.

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Very good thermal protection but aluminum foil apparently absorbed and transmitted more heat than reflected.

Good thermal protection, no surface cracks, charred layer did not extend to aluminum substrate.

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Back Surface Temperature, °F

Time, Minutes
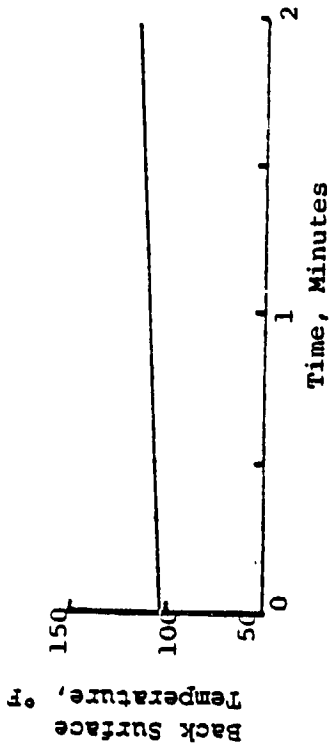
10. PIC, total 51 mils, aluminum foil laminate on surface.

Back Surface Temperature, °F

Time, Minutes

11. Control (uncoated).

20

Excellent thermal protection, thick uncharred SOFI layer, no cracks, no shrinkage of SOFI.
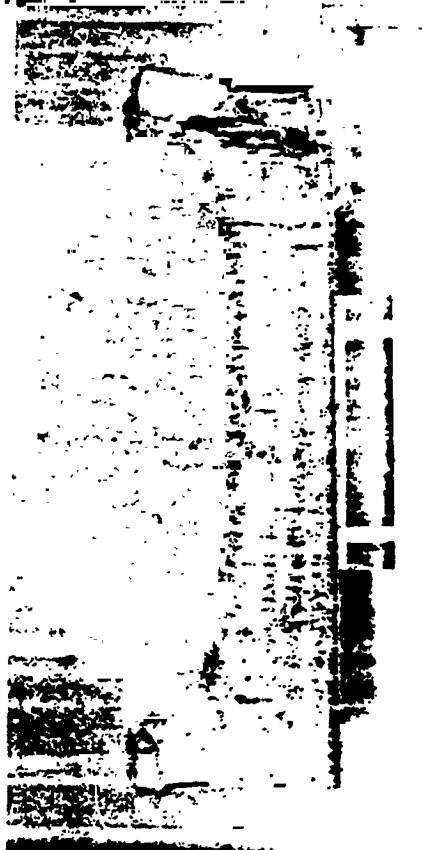
Phenolic coating also provided very good thermal insulation.
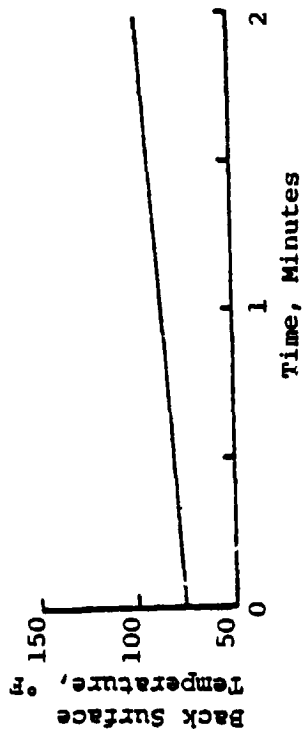
12. PIC, 48 mils.
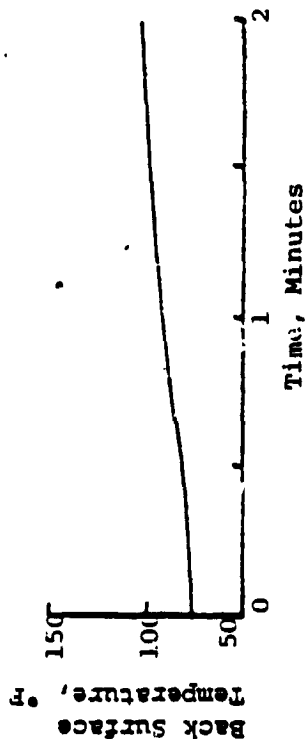
13. Phenolic intumescent coating (PHIC) 27 mils.

Intumescence was not as high as previous samples but thermal protection was excellent.
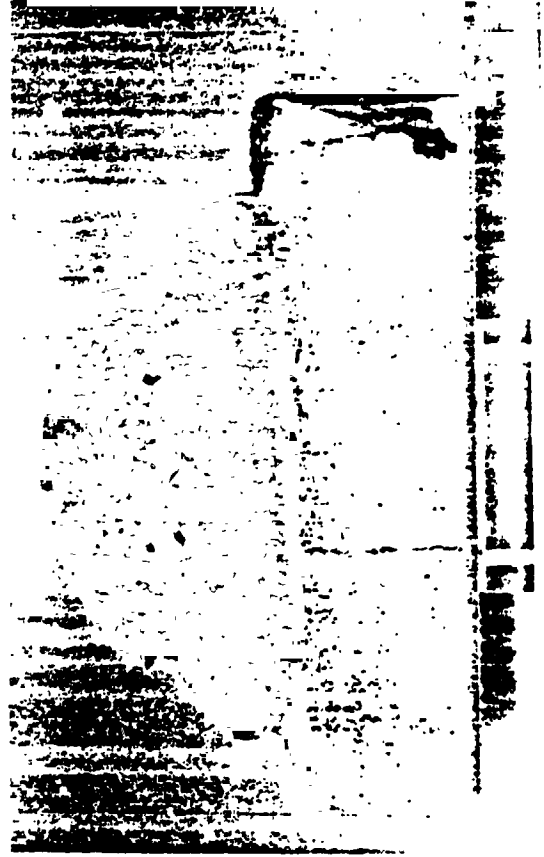
Excellent thermal protection with very little char of SOFI; limited edge shrinkage, no cracks.
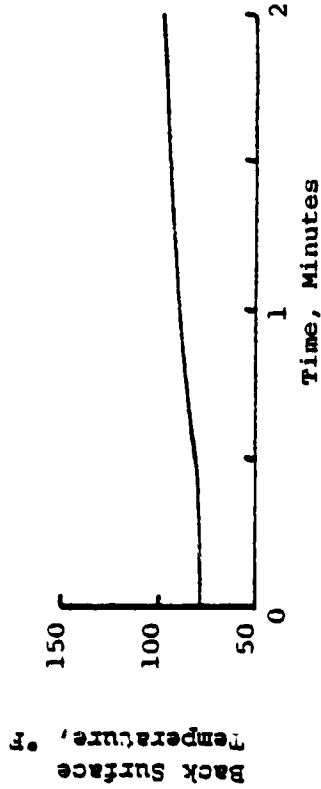
14. PIC with 5% glass fibers for added surface strength and increased TiO₂ for increased reflectance.
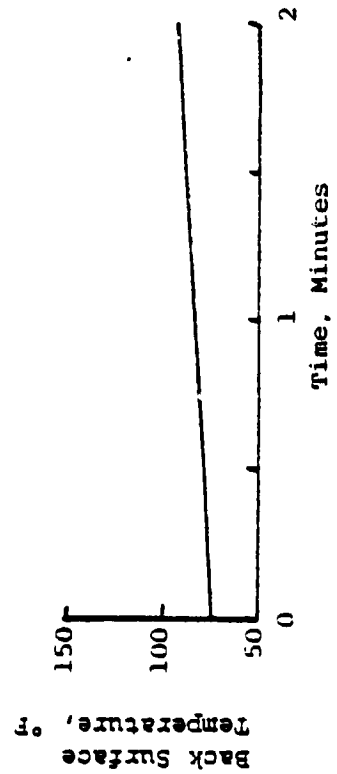
15. PHIC (19 mils) and PIC (34 mils) dual coating.

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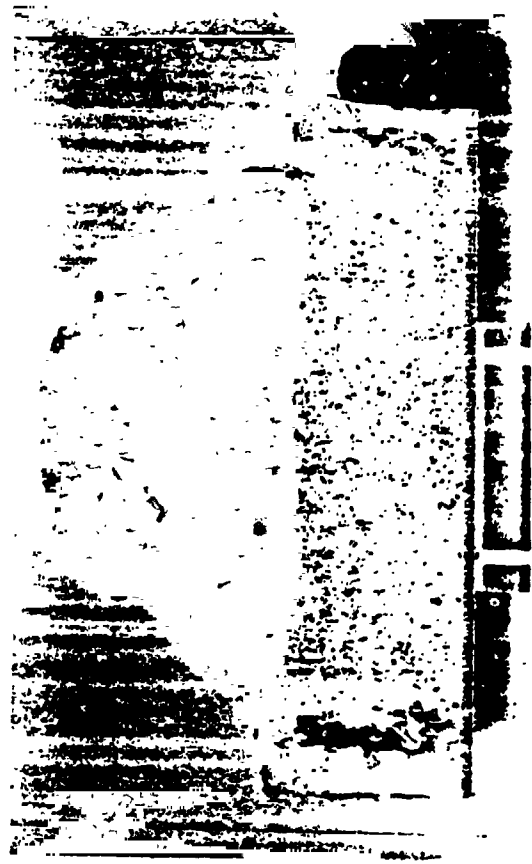
Excellent thermal protection with very little char of SOFI, no cracks in SOFI.
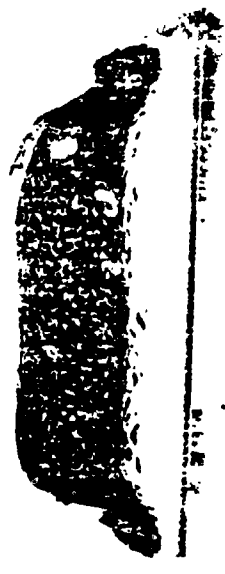
Similar to previous controls, i.e., hard, charred surface, not cracked or charred to aluminum substrate.

Data not available. Thermocouple fell off during test.

16. PHIC (35 mils) and PIC (12 mils) dual coating.
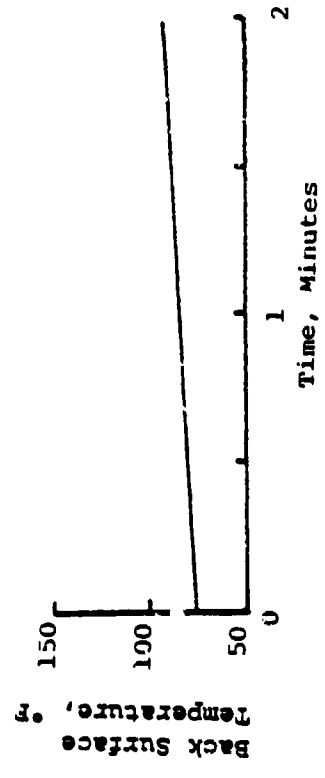
17. Control (uncoated).

Back Surface Temperature, °F

150

100

50

0

1

2

Time, Minutes

Cracks sealed readily, no additional cracks formed, 3/8" uncharred SOFI.

1/4" crack almost covered but not enough to prevent extensive charring and widening of the crack.

18. PIC, 17 mils; two 1/16" cracks after coating dried.

19. PIC, 22 mils, 1/4" crack.

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1/4" crack still not covered, extensive charring along crack.

Intumesced phenolic coating did not extend over 3/16" crack.
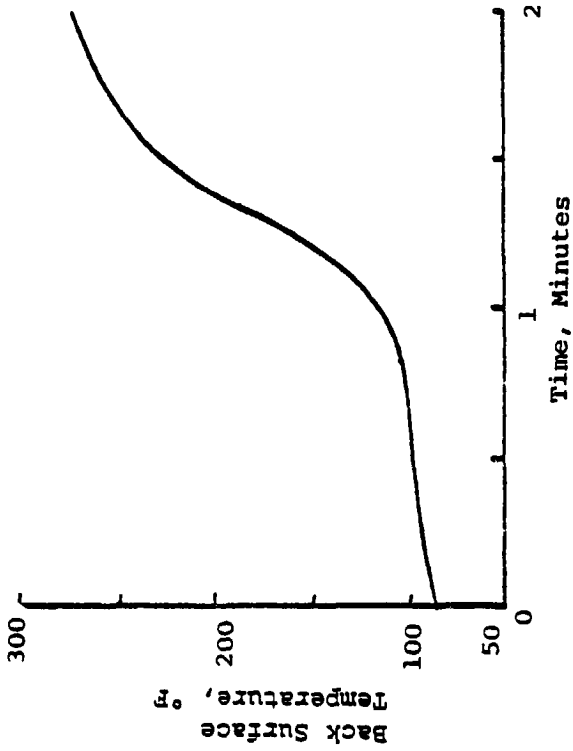
20. PIC, 29 mils, 1/4" crack (heavier coating.)

21. PHIC, 26 mils, 3/16" crack.

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Excellent thermal resistance with no cracks formed and very little charred SOFI.

Back Surface Temperature, °F

Time, Minutes

22. PHIC, 25 mils with $MgCl_2 \cdot 6H_2O$ for improved thermal protection.

Extensive charring along crack almost to metal substrate.

Back Surface Temperature, °F

Time, Minutes
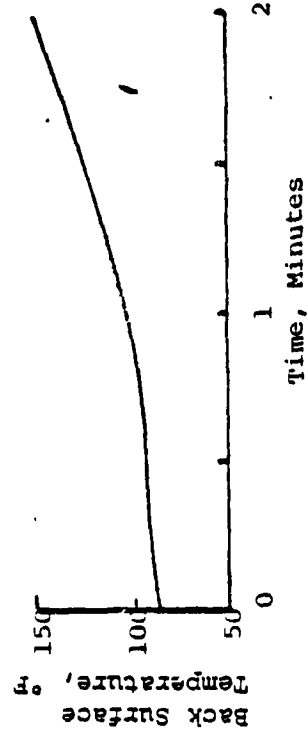
23. PIC, 14 mils, PHIC, 14 mils 1/4" wedge crack.

26

Good intumescence but TiO_2 was needed to reflect heat; the crack sealed but the charring extended almost to the metal substrate.

Thermal resistance was not as good without TiO_2; extensive charring along crack.
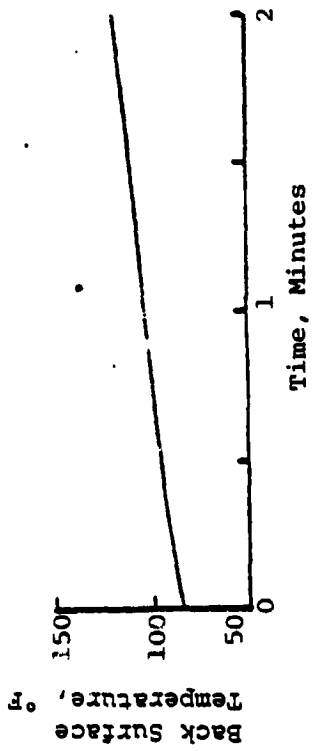
24. PIC, 22 mils, no TiO_2, 1/4" crack
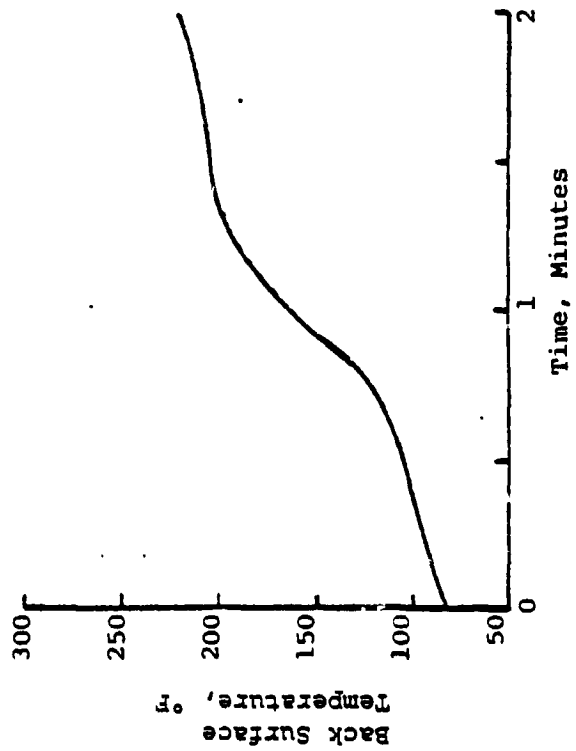
25. PIC, 23 mils, reduced TiO_2 and glass fibers; 1/4" crack.

1/16" cracks closed readily, good intumescence and good thermal resistance.

Good, uniform intumescence and thermal resistance.
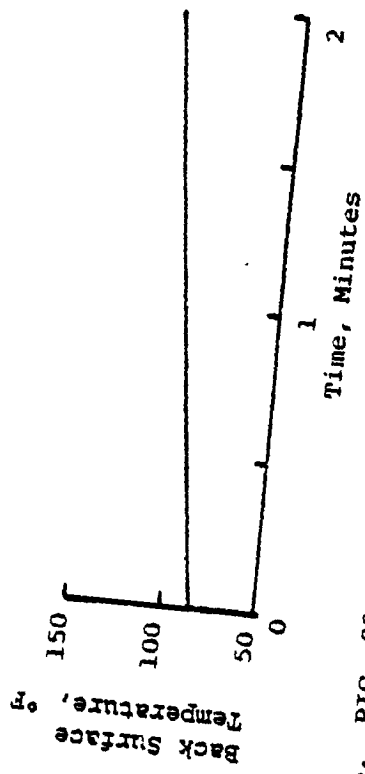
26. PIC, 22 mils, no glass fibers, two 1/16" cracks.
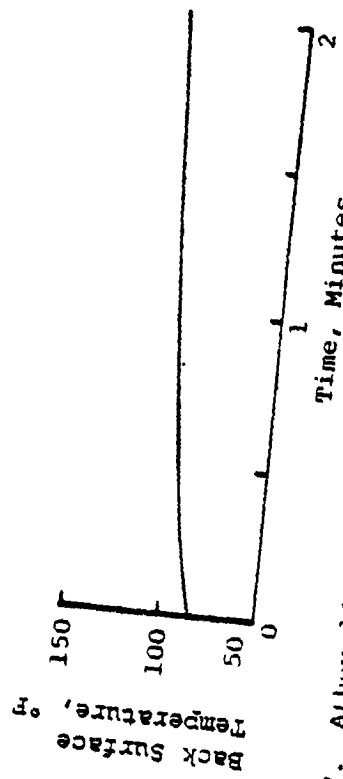
27. Atley 1401, 8 mils (commercial intumescent coating.)

28

Better intumescence with the increased coating thickness but coatings were not designed for exterior applications.
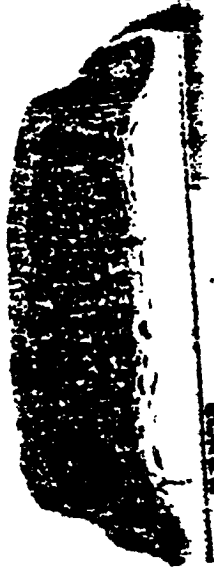
Good, uniform intumescence, results similar to number 27.

28. Athey 1401, 16 mils.

Back Surface Temperature, °F
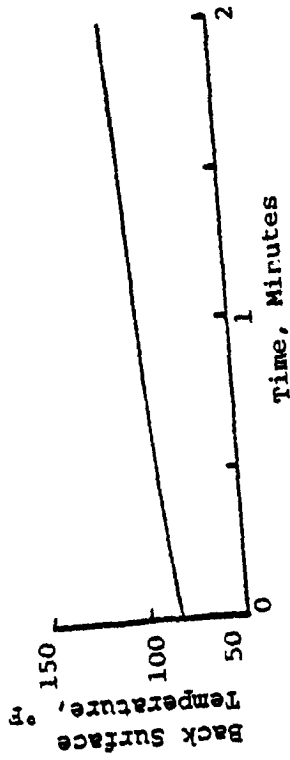
Time, Minutes
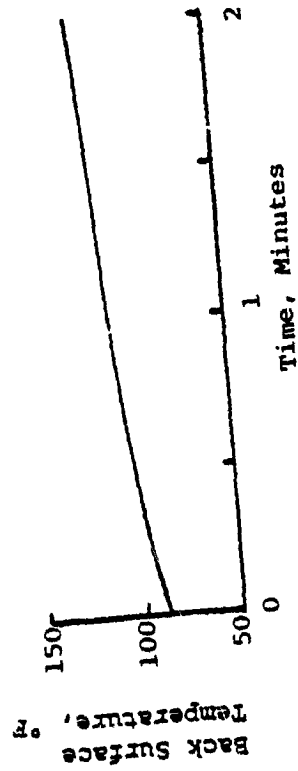
29. Athey 1403, 8 mils (commercial intumescent coating.)

Back Surface Temperature, °F

Time, Minutes

Better intumescence than number 29 but the coating was not designed for exterior application.

Very poor thermal protection with combination of coatings.
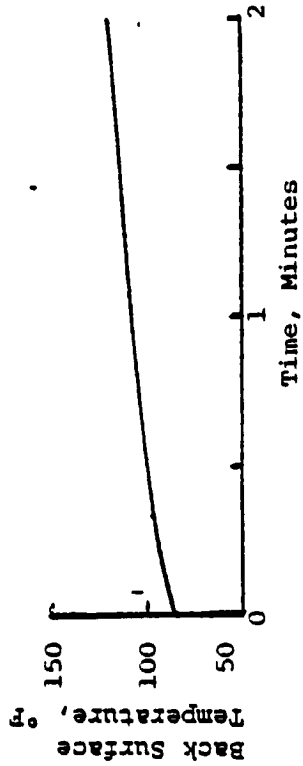
30. Athey 1403, 16 mils.
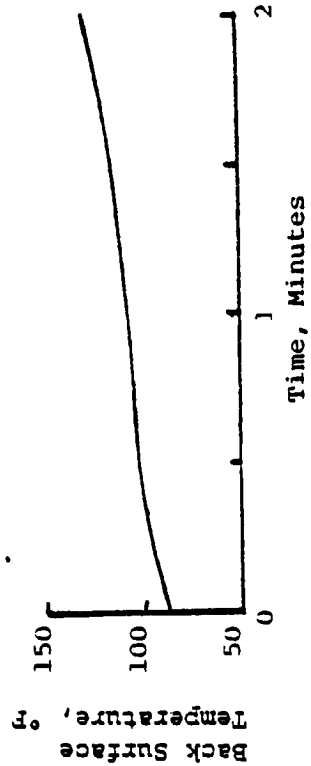
31. PIC, 23 mils plus Athey 1401, 8 mils.

30

Good uniform intumescence and thermal resistance.
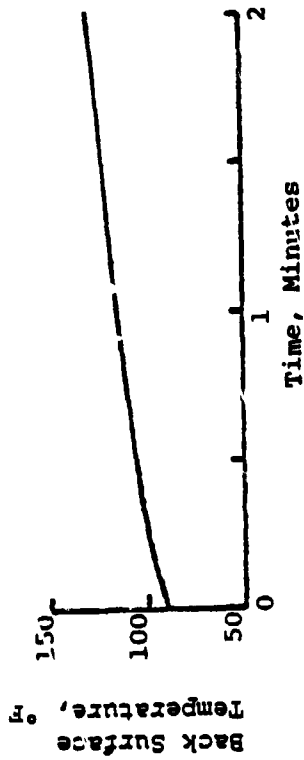
32. PIC, 23 mils plus Athey 1403, 8 mils.

(Graph: Back Surface Temperature, °F vs. Time, Minutes)

First tests of larger samples; very good thermal protection except where cracks occurred; charred areas extended to aluminum substrate along cracks; note low back surface temperature rise with thermocouple in center of sample.
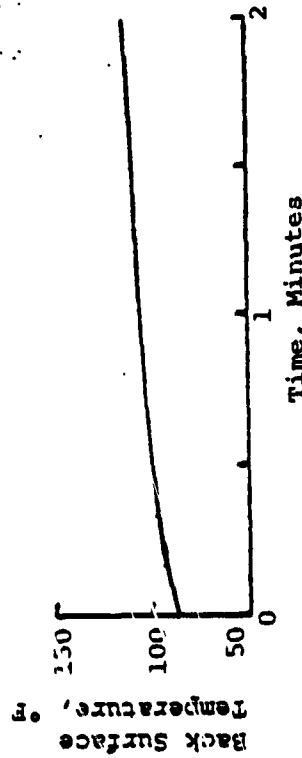
33. PIC, 24 mils, 10" x 12" sample.

(Graph: Back Surface Temperature, °F vs. Time, Minutes)

31

Very good thermal protection; no surface cracks developed with result that charred areas did not extend to aluminum substrate.

34. PIC, 14 mils plus PHIC, 13 mils, 10" x 12" sample.

Charring very extensive along cracks; back surface temperature didn't begin rapid rise until after one minute· some ~~~ ~~ st ~~ pr~~ ted
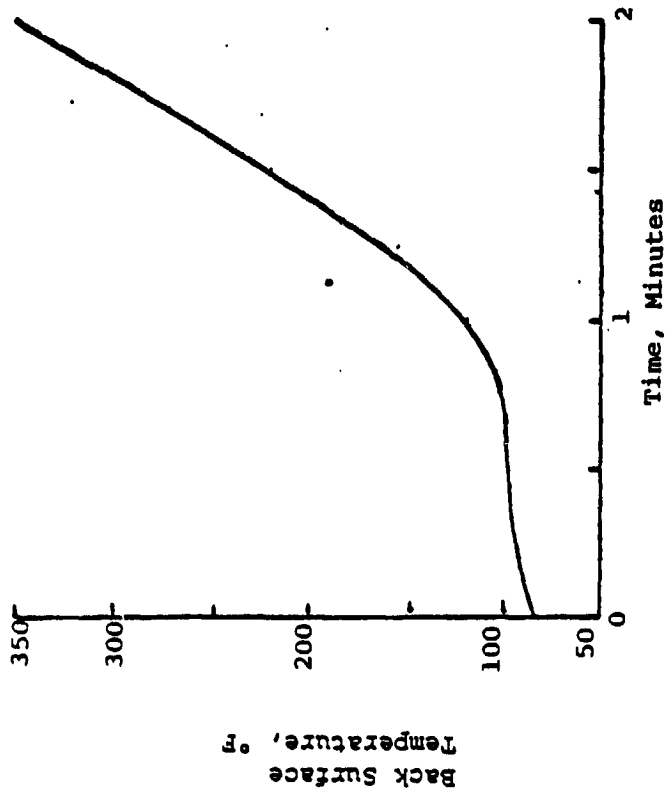
35. PIC, 22 mils, plus two perpendicular 1/4" cracks ~" x 1?" sample.

32

Cracks closed readily by expansion of SOFI, very good thermal protection.

Back Surface Temperature, °F

Time, Minutes
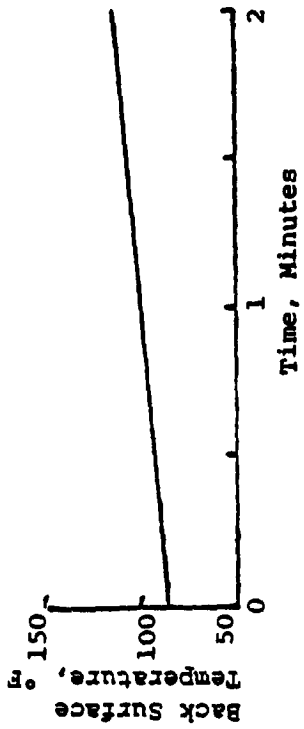
36. PHIC, two 1/8" cracks.

Cracks closed readily by expansion of SOFI, very good thermal protection.

Back Surface Temperature, °F

Time, Minutes

37. PIC, two 1/8" cracks.

33

Very good thermal protection; no cracks or charring extending to substrate.

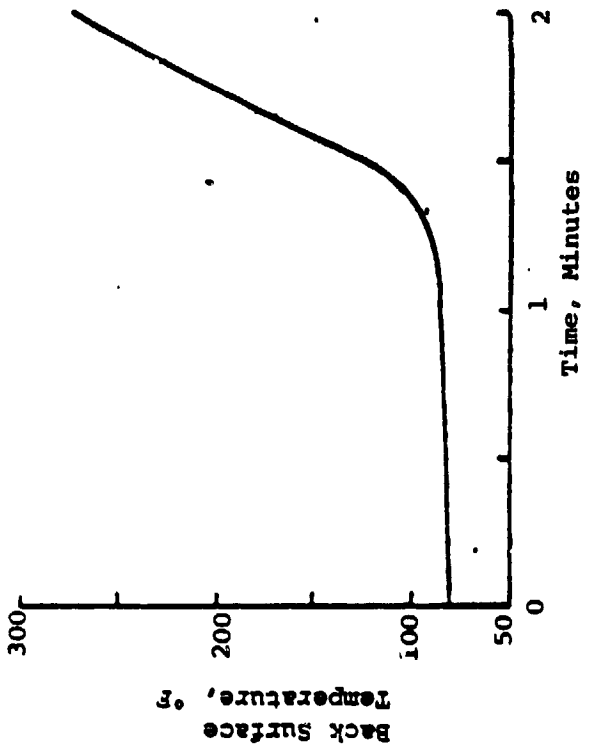Crack developed and extended to substrate with resulting char and high temperature rise, some areas were still protected.
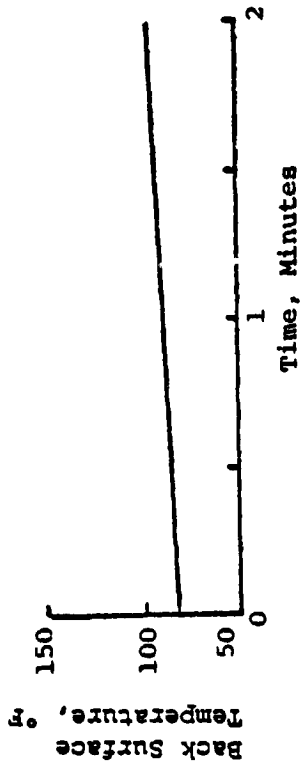
38. PIC, 10" x 12" sample.
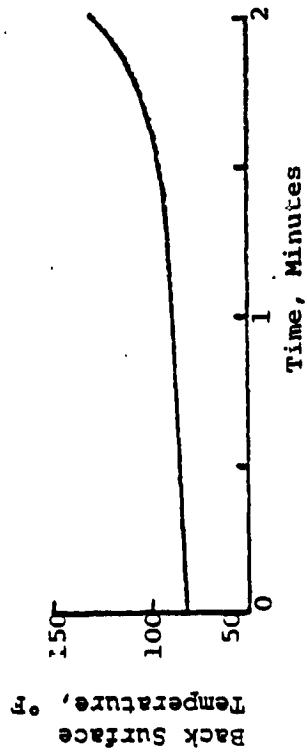
39. PHIC, 10" x 12" sample.

Extensive cracking and charring
although the areas without cracks
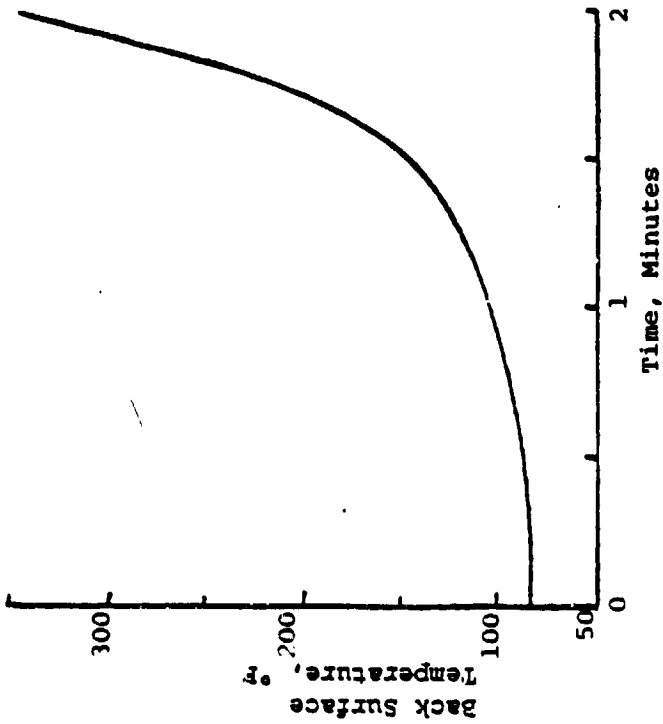were not completely charred.

40. Control (uncoated SOFI), 10" x 12" sample.

In some areas, the cracks closed; in
other areas, the cracks were much wider
with extensive char; some new cracks
developed with extensive char.
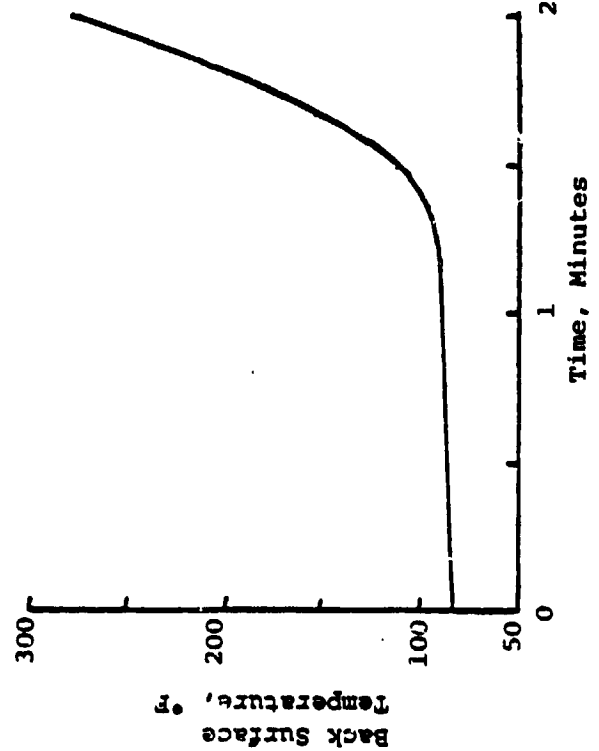
41. Uncoated SOFI, two 1/16" cracks, 10" x 12" sample.

35

Extensive char along cracks plus additional cracks developed with extensive char.

42. Uncoated SOFI, two 1/4" cracks, 10" x 12" samples.
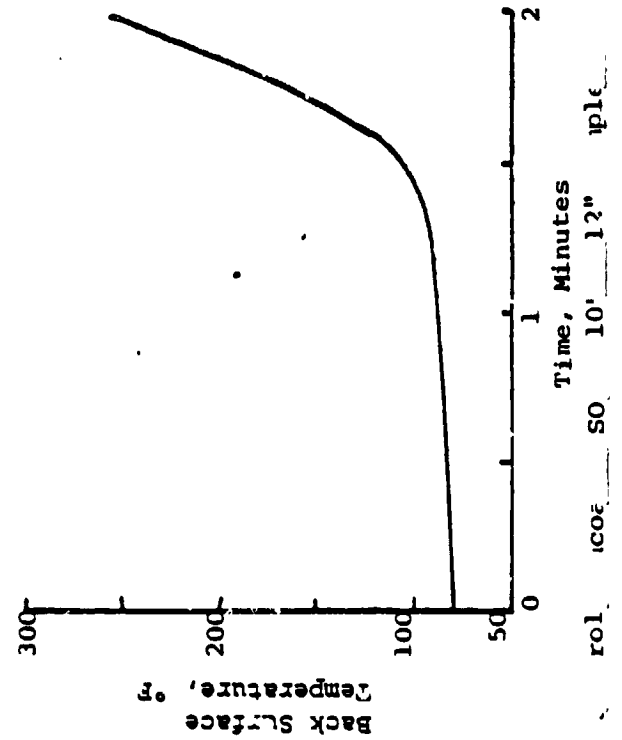
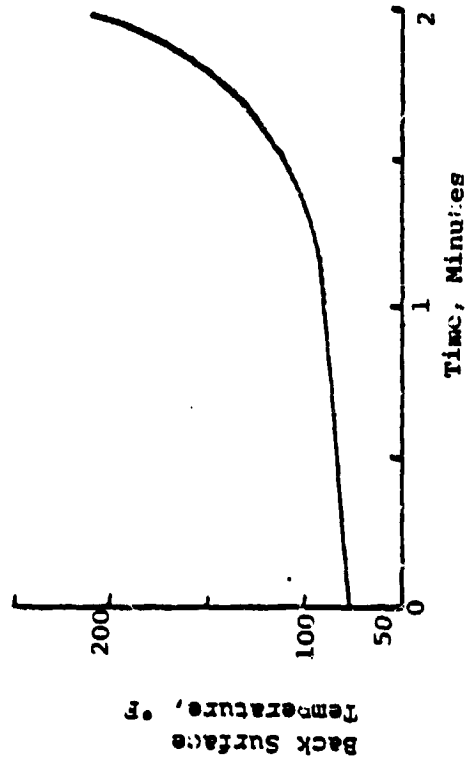Extensive char extended to substrate along cracks.

Back Surface Temperature, °F

Time, Minutes
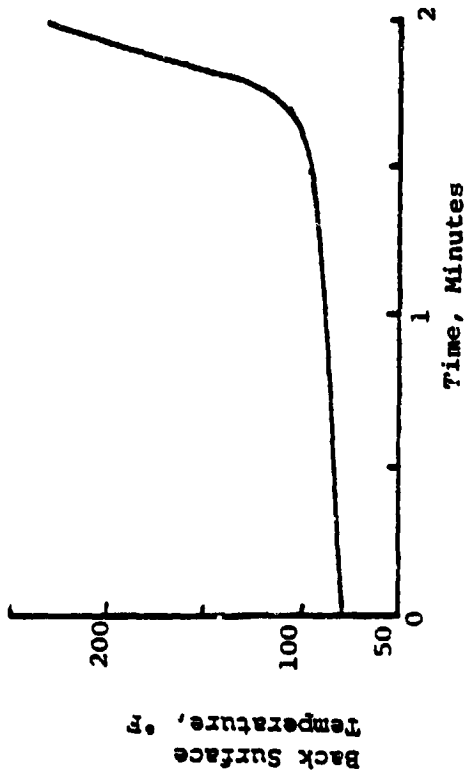
44. Two 3/16" x 1/2" deep cracks, plus one 1/8" crack full depth, cracks filled with PHIC, 10" x 12" sample.

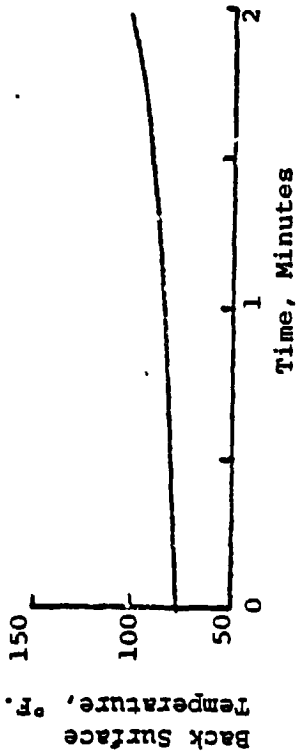Extensive char along cracks; shrinkage of SOFI was not significantly reduced.

Back Surface Temperature, °F

Time, Minutes

45. SOFI "precured" for four hours at 175°C; three 3/16" cracks filled with PHIC, 10" x 12" sample.

37

Appeared good during test but char under cloth was extensive.

46. PHIC, graphite and fiber glass cloth laminate, 10" x 12" sample, 1/8" crack filled with PHIC.

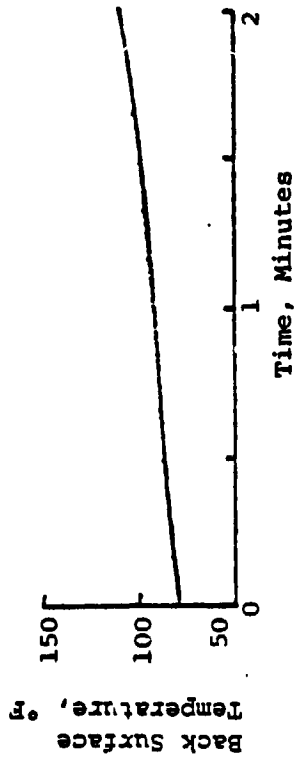Extensive char and loss of some pieces of charred SOFI.
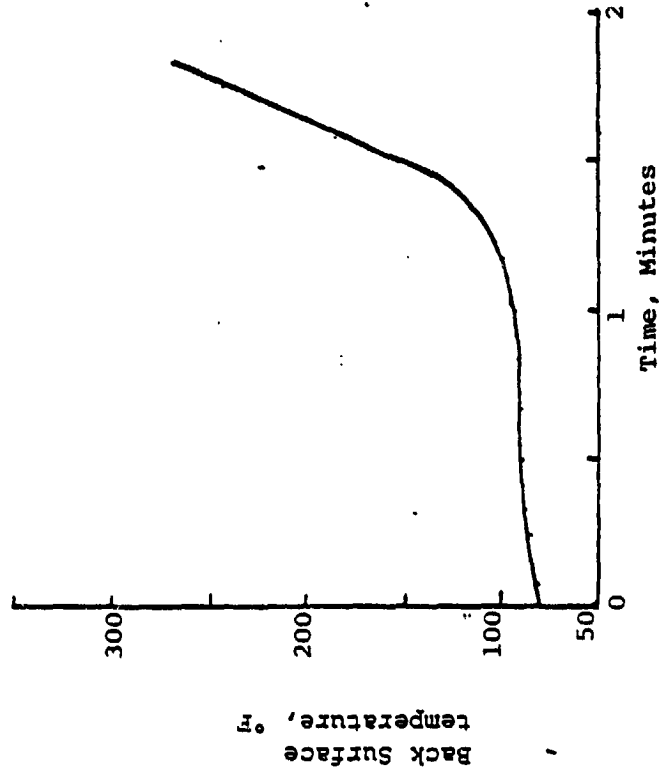
47. Three 3/16" cracks filled with PHIC, plus SOFI split orizontally and relaminated with PHIC, 10" x 12" sample.
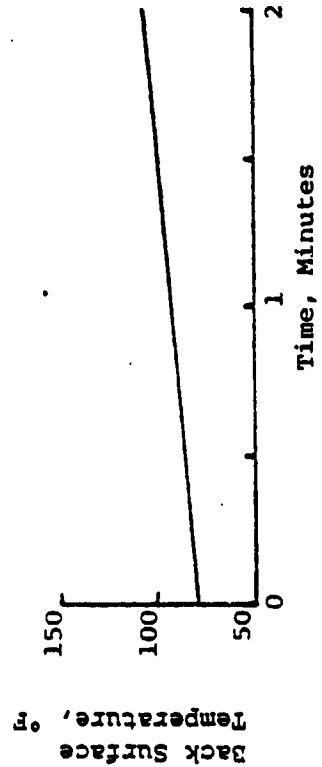
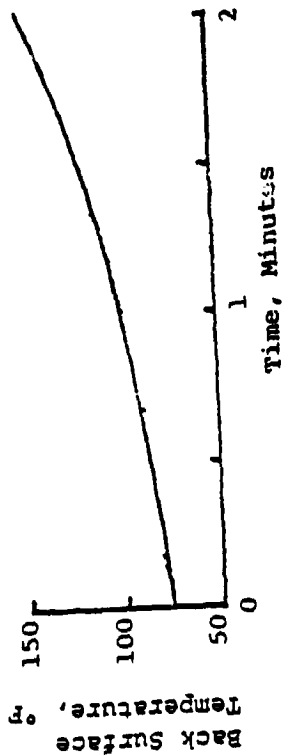Extensive char along crack; rest of area had very good protection.

Very good thermal protection but cloth was not secure after test.

48. PHIC, reduced solvent, no blowing agent, 3/16" crack.

49. PHIC, no blowing agent, carbon fiber cloth (Pluton).

39

Back Surface Temperature, °F

51. SOFI coated with two coats of aluminum paint 10" x 12" sample.

Very extensive burning, cracking, and charring of SOFI with loss of several pieces of SOFI during the thermal exposure.

Time, Minutes

Back Surface Temperature, °F

50. Two perpendicular 1/4" cracks filled with silicone RTV.

Cracks filled with charred RTV but extensive burning and charring occurred along cracks.

40