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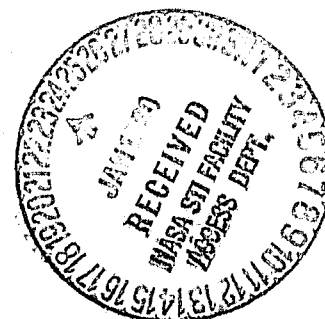
(NASA-CR-160441) DEVELOPMENT OF A SPECIAL
PURPOSE SPACECRAFT INTERIOR COATING, PHASE 3
(Pennwalt Corp.) 15 p HC A02/MF A01

N80-15199

CSCL 22B

G3/18

Unclas
46584



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DEVELOPMENT OF A SPECIAL
PURPOSE SPACECRAFT INTERIOR COATING

Technical Report - Phase III

Contract NAS 9-14403

Pennwalt Project 989147

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April 1979

Prepared for
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FOREWORD

The work described herein, which was conducted by the Pennwalt Corporation, was performed under NASA Contract NAS 9-14403 during the period from 18 April 1977 through 18 April 1978. Mr. Dale Sauers of the Structures and Mechanics Division of the NASA L. B. Johnson Space Center was the Technical Monitor.

ABSTRACT

A variety of intumescent coatings based on a fluoro-carbon latex resin modified with either an acrylic resin or an epoxy resin were prepared. Several intumescent systems were used for these studies including some based on ammonium polyphosphate and others based on sulfanilamide. The best coatings developed had a high concentration (60-70% by wt.) of intumescent additives and had to be applied thick (~ 100 mils) in order to have adequate intumescent/fire protection properties.

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I. INTRODUCTION

A. General

Under Contract NAS 9-14403 a program was initiated to develop a fluorocarbon latex coating system that can be applied to metal surfaces in a spacecraft and, without the use of a high temperature cure, can pass all crew and maintenance safety requirements. This effort has resulted in several coating systems that can be pigmented in a range of colors and are capable of meeting the objectives defined for this development program.^{1,2} Quantities of these paints have been supplied to various organizations, in the U. S. and abroad, for use in the Space Shuttle, satellites, ships, and other structures. These formulations consist of latex fluorocarbon polymers blended with either acrylic or epoxy resins or both. Typical compositions cure at room temperature within twenty-four hours, are self-extinguishing, do not contain toxic solvents, and can meet stringent offgassing requirements. Flexibility, hardness, and abrasion resistance vary depending on the particular latex blend.

Variations of the above coating systems are required for protecting substrates against fire. The trend in current and future space programs is toward the maximized use of off-the-shelf equipment. Typical commercially available equipment is not designed for flame resistance in the oxygen-rich environments that can exist in crew areas of spacecraft. A coating capable of rendering off-the-shelf equipment fire resistant, and thereby acceptable for space flight use, would be most cost effective.

1. "Development of a Special Purpose Spacecraft Interior Coating," E. J. Bartoszek and Piero Nannelli, Technical Report - Phase I, Contract NAS 9-14403, November 1975.

2. Ibid., E. J. Bartoszek, Alkis Christofas, and Piero Nannelli, Technical Report - Phase II, February 1977.

Coatings containing intumescent agents are currently available on the market. These coatings generally contain organic solvents, possess relatively poor physical and mechanical properties, and are vulnerable to humidity degradation. Accordingly a program was initiated under Contract NAS 9-14403 to develop fluorocarbon intumescent latex coatings that would possess none of the shortcomings of the commercially available intumescent paints.

B. Intumescent Coatings

Intumescent paint technology has produced many formulations that contain a number of different components. The components commonly used in commercial intumescent paints can, in general, be placed in one of four categories:

1. An inorganic acid, or a material yielding acid, at temperatures between 100 and 250°C. This acts as a catalyst to speed up the formation of a carbonaceous char. Ammonium polyphosphate is commonly used.
2. A polyhydric material, rich in carbon, to form the carbonaceous backbone of the intumescent char. Carbohydrates are usually used for this purpose, Pentaerythritol being one of the most common.
3. An organic amine or amide to act as a blowing agent. Melamine is often used for this purpose because of its low water solubility.
4. A halogenated material to stabilize the foam and render the vehicle non-flammable. Chlorinated waxes are used for this purpose.

A number of organic materials have also been reported to intumesce without the need for other special additives. For example, 4,4'-dinitrosulfanilide, ammonium 1,1-nitroaniline-

2-sulfonate and sulfanilamide are all proven intumescent agents.

C. Fluorocarbon Latex Coatings

The coating formulations developed earlier under this program (Phases I and II) were based on a fluorocarbon latex resin. This resin is a terpolymer composed of about 62% by weight of vinylidene fluoride, about 24% of tetrafluoroethylene and about 14% of hexafluoropropene. Designated RC-9108, it is a white solid capable of film formation at temperatures lower than most commercially available fluoropolymers.

Phase I of the program produced an optimum formulation based on a resin system formed by RC-9108 blended with Rhoplex HA-4 (Rohm and Haas Co.) acrylic latex in 70/30 weight ratio.¹ Pigmentation in different colors was possible. The resulting coatings dried to touch in about one hour and were fully dry in about twenty-four hours under normal room temperature and humidity conditions. They displayed good optical and mechanical properties including excellent bonding to metal, wood, and plastic substrates. In addition, they were found to be self-extinguishing when applied to non-flammable substrates and could meet the offgassing requirements specified by NASA for spacecraft application. However, improvements were needed in abrasion resistance and hardness.

The approach chosen in Phase II of the program consisted of trying to improve abrasion and hardness of the coatings by using harder room temperature cure acrylics, epoxies, or epoxy-acrylic combinations as modifiers for the RC-9108 latex. As in the previous phase, an intense screening effort was carried out. The most attractive combination of properties was obtained when the RC-9108 terpolymer latex was modified with an epoxy-acrylic emulsion system. This modifier consists of an epoxy blend (Dow Epoxy DER 331 and DER 732 in approximately 11/2

weight ratio) and an acrylic resin (Dow XD-7080) as a curing agent.²

II. DEVELOPMENT OF LATEX FLUOROCARBON INTUMESCENT COATINGS

A. Experimental

The initial effort toward the development of latex fluorocarbon intumescent coatings was concerned primarily with a relatively detailed evaluation of the compatibility and sensitivity of the latex fluorocarbon resin systems to selected intumescent additives. The bulk of the initial work was done with Phoscheck (ammonium polyphosphate), although some work was also done with ammonium phosphate, melamine phosphate, and sulfanilamide as the intumescent additive.

The fluorocarbon resin base (RC-9108) was found to be stable to all of the intumescent additives studied and did not seem to be affected by the way in which the additive is combined with it. On the other hand, the acrylic and epoxy resin modifiers, as well as the coating formulations containing them, were found to be sensitive and easily coagulated by mixing with the intumescent additives. Consequently, a study was carried out on methods for formulating stable mixtures with these additives.

The best process discovered for mixing the intumescent additives with the resins was to first disperse the additives in water with a dispersing agent such as Tamol 850 (Rohm and Haas Co.) or Shancospense (Shanco Plastics and Chemicals, Inc.) and add the resulting dispersion to the resin formulation. This process could be used for many different coating formulations and the resulting latices were generally stable with respect to coagulation.

Once a coating was prepared it was screened in-house for its intumescent properties by using a National Bureau of Standards Smoke Density Chamber (American Instrument Company). Sam-

ples were prepared by coating 3" x 3" x 3/8" plywood panels on one smooth sanded side. After drying, they were exposed for three minutes to resistance heating elements (2.5 watts/cm^2) on the front side of the sample. Additional heat was supplied by a gas fired flame impinging on the face of the sample. The extent of intumescence was measured by the depth of the char. The damage to the wooden substrate was estimated by its appearance.

In all of the formulations investigated a significant coating thickness (> 30 mils) was necessary before any intumescence was observed. In order to achieve thicker coatings it was necessary to use viscous formulations. On the other hand, applying a coating too thick resulted in poor adhesion and mud cracking. For this reason we prepared moderately viscous formulations which were applied in many coats in order to achieve the desired thickness. The results of some of our tests appear in Table I. It is clear from these results that it is possible to obtain latex fluorocarbon intumescent coatings with either sulfanilamide or a mixture based on ammonium polyphosphate (Phoschek). Two formulations that appeared best on the basis of our screening were evaluated further at NASA. These formulations, which are listed as samples #10 and #13 in Table I, were coated on a 1/4" plywood panel within which a temperature probe was embedded. The temperature differential between the front-side and backside of the coatings was then determined as a function of time (Figures 1 and 2). Both coatings had some protective qualities when thick; however, sample #10, which is based on ammonium polyphosphate and HA-8 acrylic resin, offered the best thermal protection.

III. CONCLUSIONS

It is possible to formulate intumescent coatings based on fluorocarbon latex resin systems. However, in order for these coatings to provide adequate protection against fire they must have a high concentration (60-70% by wt.) of intumescent agents

and must be applied thick (~ 100 mils). This is also typical of the commercial intumescent paints all of which, because of these restrictions, have relatively poor appearance and wear properties. Nevertheless, the exceptional UV stability of the fluorocarbon resins can make the intumescent coating systems described in this report particularly suitable for outdoor use.

Table I. Intumescent Properties of Various Formulations^a

Sample #	Intumescent Agent(s) (% Conc.)	Resin(s) (% Conc.)	Pigment (% Conc.)	Thickness of Coating (mils)	Extent of Intumescence and Protection of Wood
1	Sulfanilamide (47.4%)	Rhoplex HA-4 (8.2%) RC-9108 (24.6%)	TiO ₂ (26.2%)	30	Only traces of intumescence; wood barely protected
2	Sulfanilamide (47.1%) Delvet 65 ^b (9.4%)	Rhoplex HA-4 (9.4%) RC-9108 (4.0%)	TiO ₂ (30.2%)	30	Only traces of intumescence; wood protected only slightly better than Sample 1
3	Sulfanilamide (67%)	Epoxy ^c (7.4%) RC-9108 (17.8%)	TiO ₂ (6.7%)	100	Extensive intumescence; wood well protected
4	Phoscheck ^d (23.5%) Pentaerythritol (3.9%) Melamine (9.8%) Delvet 65 (4.9%)	Rhoplex HA-8 (22.3%) RC-9108 (27.8%)	TiO ₂ (7.8%)	35	Poor intumescence; wood slightly protected
5	Phoscheck (32.5%) Pentaerythritol (5.4%) Melamine (13.6%) Delvet 65 (6.8%)	Rhoplex HA-8 (30.8%)	TiO ₂ (10.8%)	40	Good intumescence; wood protected
6	Phoscheck (31.3%) Pentaerythritol (5.2%) Melamine (13.1%) Delvet 65 (6.5%)	Rhoplex HA-8 (14.9%) RC-9108 (18.5%)	TiO ₂ (10.4%)	50	Good intumescence; wood protected
7	Phoscheck (38.5%) Pentaerythritol (6.4%) Melamine (16.0%) Delvet 65 (8.0%)	Rhoplex HA-4 (18.3%)	TiO ₂ (12.8%)	50	Good intumescence; wood protected
8	Phoscheck (31.3%) Pentaerythritol (5.2%) Melamine (13.1%) Delvet 65 (6.5%)	Rhoplex HA-4 (14.9%) RC-9108 (18.5%)	TiO ₂ (10.4%)	35	Good intumescence
9	Phoscheck (36.2%) Pentaerythritol (6.0%) Melamine (15.1%) Delvet 65 (7.5%)	Rhoplex HA-8 (10.3%) RC-9108 (12.8%)	TiO ₂ (12.1%)	55-60	Excellent intumescence; 0.23" foam; wood well protected
10	Phoscheck (39.2%) Pentaerythritol (6.5%) Melamine (16.3%) Delvet 65 (8.2%)	Rhoplex HA-8 (7.4%) RC-9108 (9.3%)	TiO ₂ (13.1%)	55-80	Excellent intumescence; 0.31" foam; wood well protected
11	Phoscheck (40.7%) Pentaerythritol (6.8%) Melamine (16.9%) Delvet 65 (8.5%)	Rhoplex HA-8 (3.9%) RC-9108 (9.7%)	TiO ₂ (13.6%)	60-70	Excellent intumescence; 0.31" foam; wood well protected
12	Phoscheck (42.7%) Pentaerythritol (7.1%) Melamine (17.8%) Delvet 65 (8.9%)	Rhoplex HA-8 (4.1%) RC-9108 (5.1%)	TiO ₂ (14.2%)	60-70	Excellent intumescence; 0.38" foam; wood well protected
13	Sulfanilamide (50%)	Epoxy (12%) RC-9108 (24%)	TiO ₂ (12%)		Tested by NASA

a. Samples were coated on 3" x 3" x 3/8" plywood panels and exposed to 200°C plus a gas flame on coated face.

b. Delvet 65 (Diamond Shamrock) is an aqueous suspension of chlorinated wax (71% chlorine).

c. Epoxy is Dow XD7080, D.E.R. 331, and D.E.R. 732 formulated in the wt. ratio 25/18.5/3.4 as solids.

d. Phoscheck is Monsanto's ammonium polyphosphate.

Figure 1

Plot of differential temperature across coating vs.
time for coating #13. This coating contains

Sulfanilamide	50%
Epoxy (Dow's XD7080, D.E.R. 732, D.E.R. 331)	12%
RC-9108	24%
TiO ₂	12%

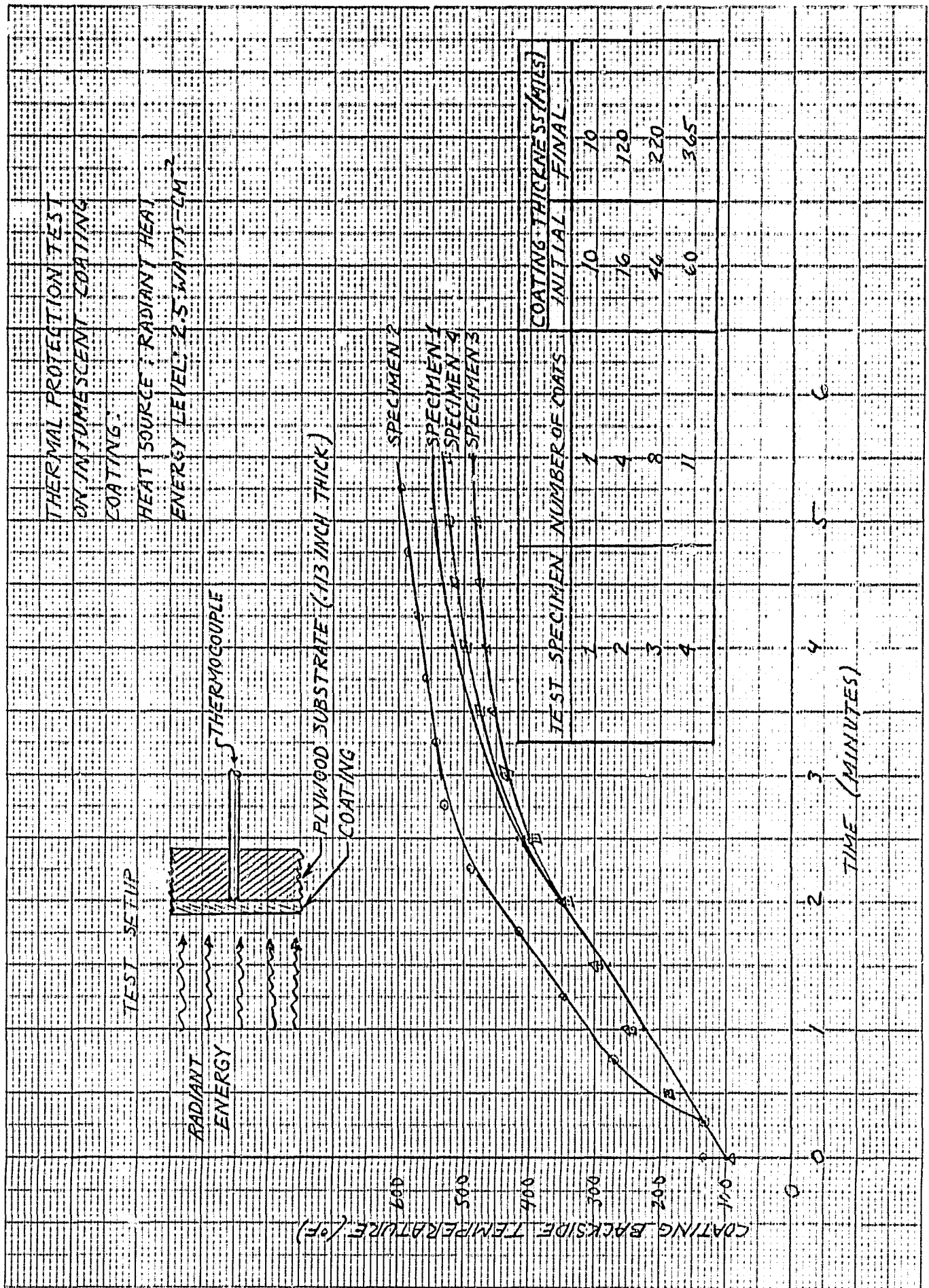


Figure 2

Plot of coating backside temperature vs. time for
coating #10. This coating contains

	<u>% Solids</u>
Phoscheck (ammonium polyphosphate)	39.2%
Pentaerythritol	6.5%
Melamine	16.3%
Delvet 65 (chlorinated wax)	8.2%
Rhoplex HA-8	7.4%
RC-9108	9.3%
TiO ₂	13.1%

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