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DEVELOPMENT OF A SPECIAL PURPOSE SPACECRAFT COATING

Technical Report - Phase IV

Contract NAS 9-14403

Pennwalt Project 989147

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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 30 May 1978 through 30 May 1979. Mr. Calvin Schomburg of the Structures and Mechanics Division of the NASA L. B. Johnson Space Center was the Technical Monitor.

ABSTRACT

Coating formulations based on a fluorocarbon resin were evaluated for use on spacecraft exteriors. Formulations modified with an acrylic resin were found to have excellent offgassing properties; long term UV stability measurements are needed, however, before they can be recommended. A much less expensive process for increasing to solid content of the fluorocarbon latex was developed.

TABLE OF CONTENTS

I. INTRODUCTION

A. General

In the three earlier phases of contract NAS-914403, interior spacecraft coatings based on a fluorocarbon latex system were developed which could, without the use of a high temperature cure, pass all crew and maintenance safety requirements. The first two phases 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 off-gassing requirements. Flexibility, hardness, and abrasion resistance vary depending on the particular latex blend. In the third phase of the contract[3] several intumescent coating formulations, based on the same fluorocarbon latex system, were developed.

The exterior of a spacecraft is subjected to a number of harsh environmental hazards including low vacuum ($\sim 10^{-15}$ torr) and solar ultraviolet photons. These conditions could easily degrade all but the most stable coatings, with consequent condensation of the degradation products on the spacecraft windows and optics. Thus the choice of coating materials for this

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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.
3. Ibid., H. D. Gillman and Piero Nannelli, Technical Report - Phase III, April 1979.

application is very limited and the suitable coating systems which are based on organosilicon resins, are very expensive. Because of the high cost of these coatings NASA is seeking less expensive alternatives. A promising approach is based on the use of a fluorocarbon latex system because of its outstanding UV stability and offgassing properties. Investigation of such coatings was the main objective of Phase IV of this contract.

B. Spacecraft Thermal Control Coatings

Coatings for the exterior surface of spacecraft have been investigated for over a decade with primary emphasis on organosilicon systems. The preparation of these coatings is intricate and variations in their preparation are reported to have a significant effect on their properties. Interest in the fluorocarbon latex system for this application was first aroused when Martin Marietta Aerospace personnel investigated one of our formulations (#4334-112) for this purpose. They performed a 48 hour, 2.5 sun ultraviolet irradiation test making spectral reflectance measurements during the test and post test. The pretest solar absorbance values ranged from 0.15 to 0.26 and total reflectance did not change by more than 20% over the test period. This is a considerable achievement considering the coating was not formulated for this purpose. The composition of this formulation was as follows:

Components	Weight in Grams
Deionized Water	90.0
Propylene Glycol	11.0
Dapro DF-911	2.5
Tamol 731 (25%)	25.0
Triton CF-10	5.0
Dimethylaminoethanol	3.0
Rhoplex HA-4 (45% NVM*)	404.0
ZnO (St. Joe #17)	30.0
TiO_2 (DuPont R960)	270.0
Acrysol ASE-60 (28% NVM)	23.0
RC-9108** (54.1% NVM)	776.3

* NVM = non volatile materials
** RC-9108 = fluorocarbon terpolymer latex

C. Fluorocarbon Latex Coatings

The coating formulations developed earlier under this program (Phases I and II) were based on a fluorocarbon latex resin 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 used 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.[2] As in the first 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.

II. DEVELOPMENT OF FLUOROCARBON LATEX COATINGS FOR THE EXTERIOR OF SPACECRAFT

A. Concentration of Latex

The fluorocarbon latex used in these coatings is produced and stored as 20% solids which also contains 4% Pluronic F-108 (BASF Wyandotte Corp.) and 0.1% QP4400-Cellosize (Union Carbide). This latex is too dilute for coating formulations and it must be concentrated to ∿50% solids in order to produce an emulsion (RC-9108) suitable for the coating formulations. This concentration requires special care because of the tendency of the latex to coagulate and foam. Previous concentrating was accomplished using rotary evaporation at low heat (50°C) and mild vacuum conditions. Increasing the heat caused coagulation and foaming problems and increasing the vacuum caused the latex to foam uncontrollably. This process is laborious and time consuming on a laboratory level and would probably be costly on a production level. Because of the difficulties associated with the evaporative process we decided to evaluate creaming as another method of concentrating the original latex reaction product. In the creaming process a coalescing material is added to the latex to be concentrated causing the latex particles to agglomerate. Because these particles are now heavier they settle and the supernatent liquid can then be decanted off. Up to a point there is a direct correlation of the settling with the amount of coalescing agent added; however, too much coalescing agent will cause coagulation. The use of Union Carbide's QP4400-Cellosize, which is already present in low concentration in the original latex, was found to provide excellent settling characteristics. In order to concentrate the latex to ∿50% solids, 2.5% aqueous QP4400 solution is added until a 0.36% QP4400 concentration is reached in the latex (this included 0.1% already present).

B. Volatility of the Coating Components

The space environment is essentially a vacuum so that the volatility of the coating components is extremely important. The

volatility of the various components that are used or can be used with our coating systems was evaluated with a Thermogravimetric Analyzer (TGA) after being held at 0 to 5 torr for three days. The results, given in Table 1, show that the defoamers are the most volatile components of these coating systems.

C. Coating Development and Evaluation

Initially, two formulations were prepared which differed only on the acrylate resins used. For #4699-121-A, Rhoplex HA-4 (45% NVM) was used and #4699-121-B included Rhoplex HA-8 (45.5% NVM). The basic formulation is as follows:

36 g	Distilled Water
4.4 g	Propylene Glycol
0.5 g	Dapro DF 911
10.0 g	Tamol 731
2.0 g	Triton CF-10
1.2 g	Dimethylaminoethanol
161.6 g	Rhoplex Resin
12 g	ZnO (St. Joe's #17)
108 g	TiO_2 (DuPont's R960)

These materials were mixed and ball-milled overnight. The fluorocarbon latex was then combined with these and other ingredients, as follows:

83.93 g	above mixture
0.13 g	Dapro DF 911
2.3 g	ASE 60 (used only 1.8 g for 4699-121-B)
73.94 g	RC-9108 (56.8% NVM)
10 ml	Distilled Water

The main difference between Rhoplex HA-4 and Rhoplex HA-8 is that the latter resin imparted a much harder quality to the final coating.

These coating formulations were sent to White Sands Test Facility for Total Mass Loss (TML) and Volatile Condensable Materials (VCM) evaluations. The results, which appear in Table 2, were excellent. The VCM requirement of less than 0.1% was easily met even by the samples with the thickest coatings. Similar measurements were carried out at L. B. Johnson Space Center using the same procedures on thick (∿6 mil) film samples of these formulations which we prepared. Their VCM values were much higher (0.22% for 4699-121-A and 0.54% for 4699-121-B) than those measured at White Sands. Because of this discrepancy additional samples of the same formulations were sent to White Sands for additional testing. They obtained a VCM of 0.04% on the samples of both films in agreement with their earlier evaluations. Nevertheless, because of the high VCM values obtained at the LBJ Space Center we decided to carry out additional reformulation work to achieve lower offgassing values. The titanium dioxide (DuPont R960) was first dried at 170°C for about two hours in order to prevent it having any effect on the VCM (see Table 1). The defoamer DF 911 was replaced with the much less volatile Foamkill FBF. The new formulation was as follows:

18 g	Distilled Water
2.2 g	Propylene Glycol
0.23 g	Foamkill FBF
5 g	Tamol 731
1 g	Triton CF-10
0.6 g	Dimethylaminoethanol
80.3 g	Rhoplex HA-4
6 g	ZnO
54 g	TiO_2 (dried)

After it was mixed, this sample was ball-milled overnight and 84 g of it was combined with 134 g of RC-9108 (47.5% NVM) and 5 g of Cellosize QP15000. A thick coating (∿6 mils) was made from this formulation which was then removed from the aluminum backing

and sent to LBJ Space Center for evaluation. A VCM of 0.3% was measured. It was felt that lower values would be obtained if the film samples were thinner. We prepared thinner samples and sent them to the Space Center for evaluation. The results were not available at the time of this writing.

III. CONCLUSIONS AND RECOMMENDATIONS

The potential of an air drying fluorocarbon coating system for spacecraft exteriors has been amply demonstrated; however, more UV stability data are needed before coatings of this type can be used for that purpose. Because the cost of this coating system would be an order of magnitude lower than that of presently used coatings, we recommend that the UV stability studies be carried out.

TABLE 1. TGA Data on Coating Components[a]

Sample	State After 3 Days at 5 torr	% Wt. Loss Up To 250°F	Total % Wt. Loss After An Additional 2 hrs at [illegible]°F
Resins			
Rhoplex HA-8 (Rohm and Haas)	Solid	3	6
Rhoplex HA-4 (Rohm and Haas)	Solid	1.3	2.2
Rhoplex MC-2 (Rohm and Haas)	Solid	1.0	1.2
DER 331/732 Mixture (Dow)	Solid	10	(b)
RC-9108 (Pennwalt)	Solid	0.1	0.1
Thickening Agents			
Cellosize QP4400 (Union Carbide)	Solid	6.2	6.8
Cellosize QP15000 (Union Carbide)	Solid	7.7	11.2
Acrysol ASE 60 (Rohm and Haas)	Solid	2.5	2.9
Pigments, Extenders, Fillers, and Corrosion Inhibitors			
Titanium Dioxide-R960 (DuPont)	Solid	0	0.4
Zinc Oxide #17 (St. Joe's)	Solid	0	0
Calcium Carbonate (Thomson, Weinman and Co.)	Solid	0	0
ONCOR M-50 (National Lead)	Solid	0	0
MICA (English Mica)	Solid	0	0
Zinc Orthotitinate (Great Western Inorganics)	Solid	0.05	1.0
Defoamers			
Trokyd 999 (Troy Chem)	Liquid	10	(b)
Dapro DF 911 (Daniel Products)	Liquid	30	(b)
Dapro W-77 (Daniel Products)	Solid (c)	3	4
Dapro DF-944 (Daniel Products)	Liquid (c)	23	(b)
NOPCO NDW (NOPCO Division, Diamond Shamrock)	Liquid	32	(b)
Foamkill FBF (Crucible Chem. Co.)	Solid (c)	1.5	2.5
Colloid 600 (Colloids)	Liquid (c)	22	(b)
Colloid 677 (Colloids)	Liquid (c)	15	(b)
Balab 618 (Whitco)	Liquid (c)	5.5	21
Surfactants and Dispersing Agents			
Pluronic F108 (BASF)	Solid	0.8	1.5
Tamol 731 (Rohm and Haas)	Solid	4.0	6.5
Tamol 850 (Rohm and Haas)	Solid	4.0	6.3
Triton X-45 (Rohm and Haas)	Solid (c)	5	20
Triton CF-10 (Rohm and Haas)	Solid (c)	0.1	0.1
Triton X-405 (Rohm and Haas)	Solid (c)	0	0.5
IGEPAL-CO-630 (GAF)	Solid (c)	0	6
Shancosperse (Shanco Plastics & Chem.)	Solid (c)	5	5
Mildewcide			
Skane M-8 (Rohm and Haas)	Liquid	38	(b)

a. Measured by Mr. Calvin Schomburg at NASA L. B. Johnson Space Center.
b. Not measured because weight loss was already over 10%.
c. State not given by experimenter but it is most probably that given in this table.

TABLE 2. VCM (Volatile Condensable Materials) and TML (Total Mass Loss) Data for Fluorocarbon Latex Coatings[a]

Sample #	Number of Thin Coats[b]	TML[c]	VCM[c]
4699-121-A	2	0.75	0
	4	0.76	0
	6	0.73	0.04
	8	0.84	0.03
4699-121-B	2	0.58	0.05
	4	0.64	0.01
	6	0.64	<0.01
	8	0.71	<0.03

a. Measured at White Sands Test Facility using SP-R-0022A procedure. Sample Temperature = 125°C; Test Pressure = 1.4×10^{-6} Torr; Collector Plate Temperature = 25°C; Test Period = 24 hours.
b. A minimum of 24 hours room temperature curing time was obtained between coats.
c. Data represents average of two runs.