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TECHNICAL DIVISION
900 First Avenue
King of Prussia, Pa. 19406

DEVELOPMENT OF A SPECIAL PURPOSE SPACECRAFT INTERIOR COATING

Technical Report - Phase II

Contract NAS 9-14403

E. J. Bartoszek
Alkis Christofas
Piero Nannelli

Pennwalt Corporation
Technological Center
King of Prussia, Pa. 19406

prepared for
National Aeronautics and Space Administration
L. B. Johnson Space Center
Structures and Mechanics Division
Houston, Texas 77058

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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 13 November 1975 through 31 May 1976. Mr. Dale Sauers of the Structures and Mechanics Division of the NASA L. B. Johnson Space Center was the Technical Monitor.

ABSTRACT

Further work on the air-drying latex fluorocarbon coating systems developed in the previous phase of this program has produced coatings with improved hardness and abrasion resistance.

Numerous acrylic and epoxy modifiers for the fluorocarbon latex resin base were investigated. Optimum coatings were developed by modifying the fluorocarbon latex with an epoxy-acrylic resin system. In addition, a number of other formulations, containing hard acrylics as modifiers, displayed attractive properties and potential for further improvements.

The preferred formulations dried to touch in about one hour and were fully dried in about twenty-four hours under normal room temperature and humidity conditions. In addition to physical and mechanical properties either comparable or superior to those of commercial solvent-base polyurethane or polyester coatings, the preferred compositions could meet the flammability and offgassing requirements specified by NASA for the intended application.

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I. INTRODUCTION

The NASA manned spaceflight and test programs impose rigorous requirements on materials selection. Nonmetallic materials must pass severe flammability requirements, in some cases even in pure oxygen. Because closed loop oxygen recirculation systems are used, tight tolerances are imposed on the amount of toxic and non-toxic offgassing that can occur if the material is inadvertently overheated. Even the odor of the materials is measured. If a material is to be exposed to the vacuum of deep space, other tests are required to assure that volatile components will not be given off by the material. Such components could subsequently recondense on lenses of optical instruments or on thermal control surfaces. Of course, the materials must also possess outstanding physical/mechanical properties for high durability and functional performance.

Paint systems posed a particular problem for NASA. At the time this program at Pennwalt was initiated no paint system existed that could meet all of the NASA requirements without an elevated temperature cure. The requirement for baking was undesirable because of the increased costs, limitations on painting assembled components, and restrictions on in situ repairs. Accordingly, a project was initiated for a paint system that could meet all of the above requirements after a room temperature cure.

The first phase of this program produced a latex fluorocarbon coating system that was capable of meeting all NASA requirements except abrasion resistance and hardness.[1] These properties have been improved through additional work carried out in the second phase of the project, which is described in this report.

II. TECHNICAL APPROACH

An analysis of the various requirements summarized in the Introduction, combined with a study of the potential offered by a number of novel or improved coating systems, led to the selection of fluoropolymer coatings as preferred candidates for this development work. More specifically, attention was devoted to the development of a room temperature cure, water based, fluoropolymer coating system. A fluorocarbon resin considered particularly promising for room temperature cure was a terpolymer composed of about 62% by weight of vinylidene fluoride, about 24% of tetrafluoroethylene, and about 14% of hexafluoropropene. This terpolymer, a white solid with a film formation temperature lower than most fluoropolymers that are commercially available at the present time, was selected as the basic resin system of choice for this development work. Preliminary coating experiments with this resin in a solvent system showed very encouraging results. A latex system having the same fluoropolymer composition could conceivably produce coatings with the required flammability and offgassing characteristics in addition to excellent mechanical, optical, and weathering properties.

III. COATING DEVELOPMENT

The first step of the coating development consisted of efforts to produce a fluorocarbon terpolymer latex formulation, here designated as RC-9108, that would be stable at a 50% minimum solid content by weight. For this purpose, procedures had to be developed to stabilize the latex sufficiently to permit concentration. Addition of a surfactant and a protective colloid was found to be necessary to prevent coagulation of the latex during the concentration step. Subsequently, a suitable modifier for the fluorocarbon terpolymer had to be found in order to obtain formulations with adequate film forming characteristics at room temperature. The ability of the terpolymer to form alloys with acrylic resins suggested that an acrylic latex would be an ideal modifier for the system. Indeed, a relatively extensive screening study was carried out involving numerous RC-9108/acrylic latex combinations. This phase of the program eventually 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.[2] However, improvements were needed in abrasion resistance and hardness. It was with the goal of improving these characteristics that the second phase of this coating development was initiated.

The approach chosen in this phase of the program consisted of trying to improve the abrasion and hardness properties 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 made. The more significant coating experiments and relevant observations are summarized in Table I. Some interesting results were obtained by using harder acrylic latex resins that have recently become commercially available. However, 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. The latter is an experimental product designed for curing water-based epoxy coatings and consists of an aminohydrochloride salt of an acrylic resin. It is water-soluble and is very effective as both curing agent and emulsifier for epoxies in aqueous systems. One of its particularly attractive properties is that, in addition to emulsifying in water solvent-based epoxies, it does not react with them until water is removed from the system. In the case of coatings, the curing agent does not effectively react with the epoxy until the coating has been applied and allowed to dry, thus providing relatively long shelf life to an otherwise unstable formulation. The preferred RC-9108/epoxy-acrylic formulation (#3715-49-II) is initially a three component system, as illustrated by the following example:

Part A:	Dow XD-7080 (49% solids)	50 g
	Pigment	100 g
	Defoamer (DAPRO DF 911)	0.6 g
	Cyclohexanone	8 g
	Dionized water	75 g
Part B:	Dow Epoxy DER 331	18.5 g
	Dow Epoxy DER 732	3.4 g
	Methyl Cellosolve Acetate	6.1 g
Part C:	Fluorocarbon Terpolymer Latex RC-9108 (52.4% solids)	192.9 g

The paint resulting from mixing these components has a modifier content corresponding to about 31% of total resin content and is shelf-stable for up to six days at ambient temperature. It can be applied by brush, roller, or spray to metals such as aluminum, steel, and titanium and to wood. The coatings dry to touch in about one hour under ordinary ambient temperature and humidity conditions and are fully cured after about 24 hours. A faster cure can be achieved by heating at higher temperatures. As the next section indicates, the cured coatings have the mechanical and physical properties required by NASA for the intended application. Excellent corrosion protection of metals can be achieved when this fluorocarbon topcoat is used in combination with a latex primer system developed earlier in this program,[1] which is based on an acrylic latex (Rhoplex MU-2) containing various corrosion inhibitors and whose properties, including flammability and offgassing, have been found suitable for spacecraft use.

IV. EVALUATION

The coatings obtained with the preferred epoxy-acrylic modified fluorocarbon formulation, described in the previous section, were evaluated through a series of tests. These included measurements of a range of physical, mechanical, flammability, and offgassing properties in accordance with the test requirements specified by NASA. In some cases these coatings were tested side by side with high performance commercially available coatings such as polyurethane and polyester coatings. In addition, accelerated weathering tests have been initiated, and preliminary data indicate that coatings of this type are as weather-resistant as the conventional baked-on fluorocarbon industrial finish.

Table II lists the results of a number of mechanical and physical tests and related NASA requirements. Offgassing and flammability test data and requirements are shown in Table III.

V. CONCLUSIONS

It is apparent from the performance data that a fluorocarbon latex formulation of the type described here is an ideal coating system in those cases where low offgassing, fire resistant, air drying, hard, weatherable coatings are desired. In addition to application in space vehicles, coatings of this type can be most advantageous for certain industrial and military uses.

VI. REFERENCES

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. NASA Document NHB 8060.1A and SP-R-0022.

Table I

Selected Screening Formulations of RC-9108 Latex with Different Modifiers and Priming Systems[a,b]

#	Topcoat: RC-9108 Wt.% on Total Resin	Topcoat: Modifier Type	Topcoat: Modifier Wt.% on Total Resin	Topcoat: Coalescing Agent Type	Topcoat: Coalescing Agent Wt.%	Topcoat: Pigment Wt.% on Total Solids	Primer Type	Film Forming Observations: Room Temp. Drying	Film Forming Observations: Drying at Higher Temperatures
4487-112 (Used as Standard)	70	Acrylic Rhoplex HA4	30	none	—	30	Acrylic 4487-13	Excellent film appearance. Soft. High dirt pick-up.	No significant difference from ambient temperature drying on properties
3715-13-1	70	Acrylic Blend Rhoplex 73 Rhoplex HA4	 25.5 4.5	"	—	33	"	Threshold film forming temp. Very few minute hairline cracks.	Substantially harder than 4487-112 when dried at 150°F for 5 min. Excellent film.
3715-13-3	70	"	"	Butyl Cellosolve Acetate	10 on RH-73	30	"	Good film. Harder than 4487-112.	Excellent film when cured at 150°F. Good integrity.
4487-112	70	Acrylic Rhoplex HA4	30	none	—	30	3715-16 Water-based Epoxy Genepoxy-Versamid. Corrosion Inhibitors.	Hard to touch. Excellent adhesion.	
3715-20-1	80	Epoxy + Acrylic Genepoxy } Versamid Rhoplex HA4	 6.4 13.6	"	—	35	"	Few fine mudcracks. Excellent adhesion.	As with ambient temp. drying.
3715-20-1A	80	Epoxy Genepoxy } Versamid	20	"	—	33	none	Good, not tacky film.	Glossy, not tacky at 150°F.
3715-23	70	Epoxy Genepoxy } Versamid	30	"	—	35	"	Good, hard film. Good adhesion.	Glossy. Very good adhesion.
3715-24-1	78	Epoxy Genepoxy } Versamid	22	"	—	35	"	Good adhesion. Satisfactory hardness.	Very good adhesion. Satisfactory hardness.
3715-25-2	68.3	Epoxy + Acrylic Versamid } Genepoxy Rhoplex HA8	 12.6 19.1	"	—	35	"	Good film. Acceptable adhesion. Harder than 4487-112.	Excellent appearance. Medium hardness.
3715-26-1	70	Epoxy + Acrylic Versamid } Genepoxy Rhoplex HA8	 10 20			30	"	Acceptable film. Harder than 4487-112.	Excellent appearance. Medium hardness.
3715-35-1	70	Epoxy Apogen	30	"	—	30	"	Rather soft film. Poor compatibility.	
3715-35-2,3,4	70	Blends of above with Rhoplex HA8, AC73, and HA4	30	"	—	30	"	As above.	
3715-36-1 to 5	70	Epoxy Epirez blended with Rhoplex HA8, AC73 and HA6	30	"	—	30	"	Limited compatibility. Not tacky. Acceptable films.	
3715-46-2	70	Acrylic Blend AC75 Troykyd	 28 2	Butyl Cellosolve Acetate	10 on AC75	30	"	Good film of acceptable hardness.	Excellent film at 150°F.
3715-49-II	69	Acrylic-Epoxy Dow DER XD7080	31	Cyclohexanone Methyl Cellosolve Acetate	1.76 1.34 on total formula wt.	30	"	Hard film with good adhesion.	Excellent strong film at 150°F.
3715-51-2B	70	Acrylic Neocryl A-601	30	Butyl Cellosolve Acetate	15 on Neocryl	30	"	Acceptable film.	
3715-52-1	70	Acrylic Blend Carboset 514H + Troykyd XLBA-348	 20 10	none	—	30	"	Acceptable hard film.	

[a] A total of 41 acrylics, 9 epoxies, 18 acrylic combinations, and 12 acrylic-epoxy combinations were screened for compatibility and film forming properties with RC-[illegible].

[b] Paints were prepared from the most promising samples. Several combinations could not be tested further although found [illegible]

Table II

Mechanical and Physical Properties of the Preferred RC-9108/Epoxy-Acrylic Coatings (#3715-49-II)

	RC-9108/Epoxy-Acrylic Coatings (White)[a]	NASA Requirements and/or Test Data for Commercial Coatings
Indentation Hardness[b] (Knoop Number):	7.6	6-30
Abrasion Resistance[c] (Wear Index):	85	A Wear Index of 100 was recorded for Flecto polyurethane white finish under identical test conditions.
Adhesion[d]:	Passed	The coating should not separate from the test panel.
Elongation[e]:	Passed	The coated panels should bend 180° around a 1" diameter mandrel without cracking (equivalent to 3% elongation).
Perspiration Resistance[f]:	Not affected by human perspiration.	The coating should maintain a surface appearance equivalent to that of 3M Nextel 401-A10, white, by visual evaluation. No loss of adhesion should occur. Under identical test conditions the Nextel coatings showed softening and loss of adhesion.
Stain Resistance[g]:	Not affected by fluorinated oil (Krytox 143AC), vinegar (3% acetic acid), Freon 21, catsup, reconstituted orange juice, and 5% detergent solution. Mustard causes a discoloration, which disappears after several days.	The coatings should not soften, swell, blister, lose adhesion, or grossly discolor when subjected to the reagents listed.

Table II (Cont'd)

Mechanical and Physical Properties of the Preferred RC-9108/Epoxy-Acrylic Coatings (#3715-49-II)

	RC-9108/Epoxy-Acrylic Coatings (White)[a]	NASA Requirements and/or Test Data for Commercial Coatings
Corrosion Protection[h]:	No failure observed after three days of salt spray test.	The coating should protect an aluminum substrate generally as well as Flecto polyurethane white finish, as determined by visual observation. Under identical test conditions (and with the same primer) the Flecto coatings showed blistering after one day.

[a]Except for the corrosion test, the substrate was unprimed aluminum 60 mil thick. The coatings were applied with a wire draw bar in a thickness of 1.0±0.25 mils and were allowed to cure for at least 24 hours at room temperature before testing.

[b]ASTM D-1474, Method A.

[c]Federal Standard No. 141, Method 6192. CS-17 abrasion wheel, 500 g load, 500 cycles.

[d]Federal Standard No. 141, Method 6301.

[e]ASTM D-1737.

[f]ASTM D-2204.

[g]ASTM D-1308, Method b, spot test, open.

[h]ASTM B287-62, Acetic Acid-Salt Spray (Fog) Test, 2% salt concentration. The substrate was 60 mil aluminum treated with a 1 mil coat of acrylic latex primer containing lead silico-chromate as a corrosion inhibitor.[1]

Table III

Flammability and Offgassing Properties of the Preferred RC-9108/Epoxy-Acrylic Coatings (#3715-49-II)

	RC-9108/Epoxy-Acrylic Coatings (White)[a]	NASA Requirements
Flammability (Upward Propagation Test)[b]:		
3-mil Al Substrate, 76.2% N_2/23.8% O_2, 14.5 psia:	Self-Extinguishing	Self-Extinguishing
0.25" Stainless Steel Substrate, 100% O_2, 14.5 psia:	Non-Combustible	Self-Extinguishing
Flash Point (°F)[c]:	749	No flash point to 400
Fire Point (°F)[c]:	No fire point to 1000	No fire point to 450
Offgassing[d]:		
Total Organics (μg/g):	20	≤100
Carbon Monoxide (μg/g):	4	≤25
Odor[e]:	2.0	≤2.5
Total Weight Loss (%)[f]:	2.15	≤1[g]
Volatile Condensable Materials (%)[f]:	0.28	≤0.1[g]

[a] One coat of paint was applied by brush to the different substrates required by the test methods. The coatings were then cured 24 hours at 75±5°F and ambient pressure before testing.

[b] Test No. 1, NHB 8060.1.

[c] Test No. 3, NHB 8060.1, 76.2% N_2/23.8% O_2, 14.5 psia.

[d] Test No. 7, NHB 8060.1, Category 5(e)1, air composition, 11.8-12.0 psia.

[e] Test No. 6, NHB 8060.1, Category 5(b), air composition, 12.3 psia, no dilution.

[f] Tested in accordance with NASA Document SP-R-0022. Period: 24 hr. Final Pressure: 7.9×10^{-6} Torr. Sample Temp.: 125°C. Condenser Temp.: 25°C.

[g] For applications that require exposure to deep space.