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**MATERIALS SELECTION OF SURFACE
COATINGS IN AN ADVANCED SIZE REDUCTION FACILITY**

John L. Briggs

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**Chemistry Research and Development
COMPONENT DEVELOPMENT**

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Chemistry Research and Development
Component Development

SUBJECT DESCRIPTORS

Coatings
Corrosion
Decontamination
Materials Selection

**ROCKWELL INTERNATIONAL
ENERGY SYSTEMS GROUP
ROCKY FLATS PLANT
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GOLDEN, COLORADO 80401**

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MATERIALS SELECTION OF SURFACE

COATINGS IN AN ADVANCED SIZE REDUCTION FACILITY

John L. Briggs and Arlene F. Younger

Abstract. A materials selection test program was conducted to characterize optimum interior surface coatings for an advanced size reduction facility. The equipment to be processed by this facility consists of stainless steel apparatus (e.g. glove boxes, piping, and tanks) used for the chemical recovery of plutonium. Test results showed that a primary requirement for a satisfactory coating is ease of decontamination. A closely related concern is the resistance of paint films to nitric acid - plutonium environments. A vinyl copolymer base paint was the only coating, of eight paints tested, with properties that permitted satisfactory decontamination of plutonium and also performed equal to or better than the other paints in the chemical resistance, radiation stability, and impact tests.

INTRODUCTION

A new chemical processing facility under construction at the Rocky Flats Plant will be operational by 1981. As the new facility is activated, decommissioning of the old facility will be initiated. To facilitate decommissioning, the construction of an Advanced Size Reduction Facility (ASRF) has been proposed. The primary function of the proposed facility would be to reduce the volume of discarded, stainless steel, chemical equipment generated by the decommissioning operations.

The corrosion laboratory of Component Development R&D has undertaken a literature survey and has conducted a materials selection test program to characterize optimum interior surface

coatings for the proposed size reduction facility.

Motivation for performing the studies is the substantial economic savings that may be realized. This savings would result from the possible use of coatings rather than 304L stainless steel, which has been suggested for the interior surfaces of the ASRF. The purpose of the work reported here was to identify suitable protective coatings for ASRF service. A comparative evaluation of coatings with stainless steel was not included in the scope of this study, although applications of the alloy are discussed.

ADVANCED SIZE REDUCTION FACILITY ENVIRONMENT

The chemical equipment to be processed by the ASRF will consist of stainless steel apparatus (e.g., glove boxes, piping, and tanks) used for the chemical recovery of plutonium. Preliminary site decontamination and cleaning of the equipment will be performed in the old facility; however, an undetermined amount of radioactive and chemical residues will be retained on the equipment as it is received at the ASRF. During the size reduction processes, the residues may become physically or chemically detached to form a corrosive, radioactive environment to which the structural surfaces of the facility will be exposed. The selection of suitable surface coatings for the facility, therefore, is dictated by the following environmentally induced prerequisites:

1. Ease of Decontamination - Interior surfaces of the facility structure should provide for ease of decon-

tamination. Decontamination of the surface structure will be routinely performed to avoid high accumulated radiation levels in the facility.

2. Chemical Resistance - Interior surfaces must be resistant to corrosive chemical fumes or liquids generated by the size reduction operations.
3. Radiation Stability - Surfaces should exhibit suitable radiation stability in the presence of plutonium compounds and americium-241, a decay product of plutonium.
4. Structural Strength - Surfaces should provide adequate resistance to impact by heavy equipment and tools that could be dropped on the floors or strike the walls during size reduction.

This report cites relevant literature and describes the results of laboratory tests performed to characterize optimum surface coatings for the floors, walls, and ceilings of the proposed ASRF.

PROTECTIVE COATINGS SELECTION

Coatings have been used by the nuclear industry in the past to protect facilities and equipment from radioactivity and corrosion. Lacking a standard method of selection, various empirical tests were employed to evaluate paints for particular operations.

The choice of protective coatings for nuclear industry often had to be made based on inadequate knowledge of the behavior of the coatings under conditions of intended use. This often resulted in coatings that were expensive to maintain and difficult to decontaminate.

A standard test was drafted by the American National Standards Institute (ANSI) whereby nuclear users could systematically test and evaluate paints. The test provides a basis for comparison and selection of protective coatings for the nuclear industry. This standard, ANSI N5.12-1974, provided guidance in setting up a coatings selection test program for the ASRF.¹

The ANSI standard pertains mainly to reactor-oriented facilities. Some modifications were necessary to include the specific needs of the Rocky Flats ASRF; these are described in the tests that follow.

COATING SAMPLES

Candidate paint samples for testing were obtained by contacting manufacturers. The eight paints tested are shown in Table 1. All of the examined paints discussed in this report were recommended by the manufacturers for application on both concrete and steel.

Sample size was limited because of the analytical equipment used to determine activity. For that reason, mild steel was selected as a substrate for the paint samples instead of concrete, the more probable substrate in the ASRF. An extended test program with painted concrete samples will be required pending acceptance of coatings for use in the ASRF.

Square sample coupons of mild steel (1.5 X 1.5 X 0.125 in.) were used for all ANSI tests. The coupons were grit-blasted to a white finish and cleaned with aqueous detergent. The sample coupons were submitted to the paint companies for coating to manufacturer's specifications - with three exceptions. Three sample coupons were coated to manufacturer's specifications by maintenance painters at Rocky Flats. One of the three coupons was vinyl copolymer; the other two were polyamide cured

epoxy from two different manufacturers.

PLUTONIUM DECONTAMINATION TESTS

The ANSI standard test adopted for the plutonium decontamination studies was developed principally for mixed fission product studies. This required revision of the test for use with plutonium. These revisions included initial contamination to an alpha activity of 3×10^5 dpm instead of contamination to a beta-gamma activity of 5×10^6 dpm, a contaminant solution normality of 2N instead of 8N, and no neutralization of the contaminant solution pH. The recommended pH of 4 would have precipitated the plutonium from solution. The 2N acid was used rather than 8N to prevent immediate blistering of the paint.

The samples were measured for total alpha activity using a flow proportional detector. The effectiveness of plutonium removal was reported as the decontamination factor (DF) and was calculated as follows:

$$DF = \frac{\text{Original activity}}{\text{Activity after treatment}}$$

The contamination/decontamination tests were performed as follows:

1. A 0.1-ml, 2N, nitric acid - plutonium solution providing 3.2×10^5 dpm was used to contaminate the 1.0-in.² coupons. The contaminant was pipetted to the center of the coupons and was allowed to air dry for 24 hr.
2. After drying and prior to decontamination, the samples were scanned (counted) with a flow proportional detector to establish an initial count. An average of 3.28×10^5 dpm was measured for all coupons.
3. The first decontamination was performed with 400 ml of distilled water in a 600-ml beaker. The coupons were suspended in the solution by a clamp at the bottom of the beaker, and the speed of the stirrer was adjusted so that the water vortex touched the stirring bar. After 10 min., the samples were removed, air dried and scanned for DF determination. Fresh water was used for each of the three coupons.
4. The same water-decontaminated sample was then decontaminated with 400 ml of an aqueous mixture of 0.4M oxalic acid, 0.05M sodium fluoride, and 0.30M hydrogen peroxide at 25 °C using the method previously described. Fresh acid was used for each sample. The DF for this cleanup cycle was then determined.
5. Using the same specimen, a third decontamination was performed using the acid mixture just described except that the acid temperature was elevated to 60 °C. Again, fresh acid was used for each sample. The DF was then measured for this cleaning, and the overall DF for the three decontaminations was calculated.

Results of the decontamination tests are reported in Table 2.

It is evident from Table 2 that the water wash provided no significant decontamination for any of the samples tested. In fact, higher radiation counts were measured for the paint samples after the water wash, which indicated some masking of radioactivity had occurred during the initial radiation measurements. The masking was probably caused by entrapment of the plutonium resulting from softening of the paints by the plutonium - nitric

acid contaminant solution. This softening was observed immediately after contamination with the nitric acid solution; it occurs as the plutonium - nitric acid solution permeates through pores in the paint surfaces. The observed blistering occurs because of gas formation as acid reacts with the steel substrate. The results for the control samples in Table 2 indicate that the first acid wash at 25 °C removes most of the surface contamination. As a result, low decontamination factors are obtained for the subsequent hot acid wash at 60 °C.

Low decontamination factors were measured for all of the paints except the vinyl-base paint, which exhibited overall decontamination factors two orders of magnitude higher than the other paints tested.

Other researchers obtained similar decontamination factors ($DF = 93$) for the vinyl-base paint on aluminum substrates.² The contaminant solution for those studies consisted of mixed fission products in 6N nitric acid with a gamma radiation dose rate of 10^6 rads.

GLOSS AND PROFILOMETRY TESTS

In correlation with the decontamination studies, measurements of surface roughness and gloss were taken for several selected surfaces. Surface roughness was measured by the stylus method with a profilometer. Gloss measurements were performed with a Photovolt Model 670 Glossmeter.

The results of the profilometry and gloss measurement tests are summarized in Table 3. Correlation of the profilometry and gloss measurements with decontamination factors of selected coated and uncoated samples are depicted graphically in Figures 1

and 2. Uncoated samples with less roughness and higher gloss values also show higher decontamination factors. Less correlation was apparent for the epoxy and vinyl paint coated samples. This is probably because of the previously mentioned nitric acid masking reaction.

CHEMICAL RESISTANCE

Glove boxes and equipment to be processed in the ASRF will be decontaminated and cleaned prior to removal from the production area. Some salts and other chemical residues may be retained on the equipment, which could produce severe corrosive environments, especially in the plasma arc remote cutting module.

Chemical resistance of the paints was therefore determined in acid, alkaline, and aqueous detergent environments. One acid environment test consisted of total immersion of the paint samples in a 1N nitric acid plus 0.1N hydrofluoric acid ($\text{HNO}_3\text{-HF}$) mixture. The immersion tests were conducted to National Association of Corrosion Engineers Standard Method TM-01-69 for 30 days at room temperature.³

The paints were also exposed in a glove-box acid-vapor environment (1N nitric acid plus 0.1N hydrofluoric acid) for 30 days at room temperature. The mixed acid solution was atomized by aspiration into the glove box at a rate of 1.4 $\mu\text{l/day}$. Airflow through the glove box was maintained at 8 cfm. A film of the acid vapor collected on paint sample surfaces in the glove-box environment. Subsequent evaporation of the acid films resulted in concentrating the acid mixture, which increased severity of the corrosion test. After terminating the nitric acid - hydrofluoric acid immersion and vapor tests, the sample surfaces were evaluated, as summarized in Table 4. The test samples also are

depicted photographically in Figures 3-6.

Modified phenolic paint samples displayed poor resistance to both acid immersion and vapor environments. The paint film peeled off and was nearly completely detached by the acid mixture.

Phenolic epoxy samples displayed blistering after the acid immersion test but only light discoloration in the acid vapor test. All paint samples showed some degradation in the HNO_3 -HF acid vapor environment except the vinyl copolymer samples, which displayed no visual defects in either acid environment.

The paint samples also were exposed by total immersion (NACE Method TM-01-69) in 1N sodium hydroxide and a proprietary decontamination liquid detergent, Huntington Mariko (alkali phosphate, silicate, sulfonate, polyoxyethylene alcohol, and fluorescein dye). No visual defects were observed on any of the paint samples in either test solution after 30 days of immersion at room temperature.

RADIATION STABILITY

The painted samples were tested for radiation stability by exposure to a cobalt-60 gamma source. Although alpha radiation will be present in the ASRF environment, a significant amount of gamma radiation also will be present. Gamma rather than alpha radiation was employed for the radiation stability studies because of the reproducible, controlled dosage rates available with the cobalt-60 gamma source. In addition, remote control features proposed for the ASRF probably will permit higher levels of gamma environment than would be allowed in non-automated size reduction operations.

After the gamma irradiation was terminated, a visual evaluation of the sample surfaces was performed. Each sample was examined for the following specific changes: (1) color variation, (2) checking, (3) chalking, (4) flaking, (5) blistering, (6) softening, and (7) cracking. Results of the radiation stability tests are summarized in Table 5.

Visual examination of samples exposed in the radiation stability tests showed some light yellow discoloration of the original, white, polyamide cured epoxy paint surfaces after one-hour exposure to gamma radiation. Some very light checking also was observed. Both visual discoloration and checking increased with time until the exposure was terminated.

A light discoloration was observed on the modified phenolic and the dull epoxy samples after 8 hours of exposure. The discoloration for both paints increased to a dark yellow with extended exposure time. Some checking of the modified phenolic also was detected after 10-days exposure and increased slightly with exposure time.

Discoloration was detected on the epoxy enamel and the light yellow polyamide cured epoxy samples after 26.5 hours of exposure; color intensity of both paints increased to a dark yellowish-brown for the duration of the test. The remainder of the samples showed light discoloration after 10.2 days of exposure, increasing slightly in intensity with time.

No other forms of degradation (e.g., chalking, blistering, and flaking) were detected for any of the paints tested.

IMPACT TESTS

Mild steel samples (16 X 2 X 0.125 in.) painted on both sides to the manufacturer's specifications were tested for

impact strength, as specified by ASTM D2794-69.⁴ The test equipment consisted of a vertically mounted steel tube with a lengthwise slot that guides a weight inside the tube. Cylindrical weights of 5, 10, and 15 pounds were fitted with pins to allow elevation and release of the weight. A 1.0-in.-diameter semi-spherical steel indenter was attached to the bottom of the weights for contacting the paint samples. The samples were positioned for test purposes at the bottom of the tube.

Both weight and height of the indenter were increased incrementally until impact failure by cracking occurred. Crack failure was confirmed with a Tinker & Raser Holiday Detector, which detects paint-crack defects by electrical continuity. Results of the impact tests are shown in Table 6.

Only one of the paint samples showed intrusion crack failures at less than 1080 in.-lb, the maximum limit of the impact tester. All of the paint samples showed extrusion failures at less than 22 in.-lbs.

Impact failures for the paints were characterized by brittle fracture cracking plus related loss of adhesion with one exception. The vinyl copolymer sample exhibited failure by plastic flow of the paint film. This was caused by the increased area of the indentation; i.e., no brittle fracture was noted and no adhesion loss was evident.

DISCUSSION

The limited number of paint samples examined by this study has not allowed a comprehensive selection of coatings for use in the ASRF. However, the systematic screening of candidate paints by ANSI Standard N5.12-1974 has

provided insight as to what chemical and physical characteristics a paint requires for satisfactory service in the ASRF.

Ease of decontamination has emerged as a primary requirement for a satisfactory protective coating. Closely related to this requirement is the resistance of paint films to nitric acid - plutonium solutions. The literature⁵ indicates that most paints are not resistant to nitric acid. Since an acid environment is very probable in the ASRF, it follows that a prerequisite qualification for any paint proposed for ASRF service would be resistance to nitric acid.

Decontamination factors determined for the paints and noted in Table 2 are related directly to the nitric acid - hydrofluoric acid resistance test results shown in Table 4. An immediate discoloration and softening of the paints was detected when the contaminant plutonium - nitrate acid solutions were pipetted on sample coupon surfaces. An exception was the vinyl copolymer sample, which was unaffected. That paint also showed superior ease in decontamination (DF ~ 100) compared to other paints tested.

A DF of 100 indicates 99% removal, i.e., the fraction of radioactivity removed and the DF are related as follows:

$$\text{Fraction remove} = 1 - \frac{1}{\text{DF}} \text{ (see Ref. 6)}$$

The vinyl base paint also exhibited excellent chemical resistance in the alkali immersion tests. This confirms data in the literature.^{7,8}

Although gloss and surface roughness^{9,10} have been reported to be related to ease of decontamination, no correlation was obtained for those properties with the two paints examined in Table 3.

Minimal degradation was detected for all paints tested for radiation stability. Extended exposure periods in the gamma cell could provide definitive acceptance criteria for the selection of candidate paints.

Results of impact tests showed susceptibility of the paints to impact cracking on a steel substrate. Further impact tests should be performed on paint and concrete systems that have been accepted for ease in decontamination and for nitric acid resistance.

The flammability of dry paint surfaces has not been addressed in this test program. No flammability test methods are offered by the ANSI standard; however, the burning characteristics of any paint considered for the ASRF would require evaluation. Another ANSI standard method specified ASTM-E84 for flammability testing.¹¹ This method could be adopted for ASRF acceptance criteria.

Other paints have been received for evaluation and will be tested for possible use in the ASRF. An initial method of screening paints for the ASRF, prior to extended evaluation testing, has been identified by this study. Preliminary tests of candidate paints to determine their ease of decontamination and their resistance to nitric acid can eliminate extensive evaluation of other submitted coatings.

CONCLUSIONS

The vinyl-base paint was the only paint tested that exhibited satisfactory ease in plutonium decontamination. The vinyl-copolymer coating also exhibited properties equal to or better than other paints in chemical resistance, radiation stability, and impact tests. In addition, it is reported that dried coatings of vinyl-

base paints do not support combustion.¹² It is concluded that vinyl-base paint is the only coating tested that meets the environmental requirements for walls and ceilings of the ASRF.

All 304L stainless steel control samples exhibited higher decontamination factors than any of the paints tested. This stainless alloy has exhibited an excellent service history at Rocky Flats and throughout the nuclear industry for the containment of radioactive and corrosive chemical environments in facilities similar to the ASRF. In addition, this alloy has been employed for covering all interior structures in one reported experimental-size reduction facility.¹³

It is concluded that 304L stainless steel would be more suitable than paint for floor coverings in the ASRF where the requirements are highest for ease of decontamination, for chemical resistance, and for impact strength. That alloy may also be required for partial wall surfaces in the ASRF remote cutting module where spatter from a hot plasma arc torch could contribute flammability hazards to a severe corrosive environment.

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TABLE 1. Test Paints

<u>Paint Base or Generic Type</u>	<u>Paint Thickness (mils)</u>
Vinyl Copolymer	7
Polyamide Cured Epoxy (White)	7
Polyamide Cured Epoxy (Light yellow)	11
Epoxy Enamel	11
Modified Phenolic	10
Phenolic Epoxy	12
High Build Gloss Epoxy	9
Dull Epoxy	11

TABLE 2. Decontamination Test Results

Sample Identification	Water Wash (DF) ^b	25 °C Acid Wash ^a (DF) ^b	60 °C Acid Wash ^a (DF) ^b	Overall (DF) ^b
CONTROLS				
304L S/S ^c (2B finish)	1.01	262	3.00	777
304L S/S (50 grit)	1.07	385	1.61	664
304 S/S (electropolished)	1.09	762	1.75	2999
Glass	1.11	1152	2.25	2010
Ceramic	0.99	182	3.83	797
Teflon	1.04	161	1.97	308
COATINGS				
Modified phenolic	0.95	1.09	1.04	1.07
Light yellow polyamide cured epoxy	1.11	1.15	1.03	1.29
Dull epoxy	1.01	1.01	1.04	1.06
High build gloss epoxy	0.99	1.10	1.16	1.27
Epoxy enamel	1.02	1.12	1.07	1.23
White polyamide cured epoxy	0.68	1.24	0.92	0.83
Vinyl copolymer	1.68	43.0	1.32	104.0

a. Acid Wash - 400 ml aqueous mixture of 0.4M oxalic acid, 0.05M sodium fluoride, and 0.30M hydrogen peroxide.

b. DF = decontamination factors = original activity divided by the activity after treatment. Results are averaged from triplicate samples.

c. S/S - stainless steel.

TABLE 3. Physical Test Measurements

	Reflectance Gloss Units ^a	Roughness Measurement, (microinch) ^b
304L S/S, ^c electropolished	490	7
Glass	150	0.2
304L S/S, 2B finish	150	5
304L, S/S, 50 grit	110	50
Porcelain enamel	74	6
Epoxy paint	28	15
Vinyl paint	6	70
Teflon	3	48

a. Gloss measurements with Photovolt Model 670 Glossmeter.

b. Measurement by stylus method with a profilometer.

c. S/S - stainless steel.

TABLE 4. Nitric Acid - Hydrofluoric Acid Paint Test Results

<u>Sample Identification</u>	<u>Evaluation</u>	
	<u>Acid Immersion*</u>	<u>Acid Vapor*</u>
Modified phenolic	Blistering, peeling, total separation	Yellow discoloration, blistering, peeling, total separation
White polyamide cured epoxy	No change	Yellow discoloration, blistering, peeling
Epoxy enamel	No change	Yellow discoloration,
Light yellow polyamide cured epoxy	No change	Blisters
High build gloss epoxy	No change	Blisters
Dull epoxy	No change	Light yellow discoloration
Vinyl copolymer	No change	No change
Phenolic epoxy	Light discoloration, blisters	Light discoloration

* 1N nitric acid plus 0.1N hydrofluoric acid at room temperature; 30-day test.

TABLE 5. Radiation Stability Test Results
(exposure time and radiation dose)

Sample Identification	Initial Color	65 min 5.2×10^5 rad	8.6 hrs 4.2×10^6 rad	26.5 hrs 1.2×10^7 rad	10.2 days 1.2×10^8 rad	32.3 days 3.8×10^8 rad	70.6 days 8.3×10^8 rad
Vinyl Copolymer*	White	NC	NC	NC	LDC	LDC	LDC Light gray
Polyamide cured epoxy*	White	DC,C	DC,C	DC,C	IDC,C	IDC,C	DC,C Dark yellow
Polyamide cured epoxy	Light yellow	NC	NC	LDC	IDC	IDC	DC Dark yellow
Epoxy enamel	White	NC	NC	LDC	IDC	IDC	DC Yellow brown
Modified phenolic	White	NC	LDC	IDC	IDC,C	IDC,C	C,DC Yellow brown
Phenolic epoxy	Gray	NC	NC	NC	DC	IDC	DC Green
Dull epoxy	White	NC	LDC	IDC	IDC	IDC	DC Yellow brown
High build gloss epoxy	White	NC	NC	NC	IDC	IDC	DC Light yellow

* Exposure times were reduced slightly in separate tests for these two paints to maintain equal dosage rates for all of the samples.

NOTE: DC = Discoloration

LDC = Light Discoloration

C = Checking

IDC = Increasing Discoloration

NC = No change

TABLE 6. Impact Resistance Test Results

<u>Paint Identification</u>	<u>Paint Thickness (mils)</u>	<u>Extrusion Impact Resistance (in.-lb)</u>	<u>Intrusion Impact Resistance (in.-lb)</u>
Epoxy enamel	14	9	590
High build gloss epoxy	9	12	>1080
Dull epoxy	9	22	>1080
Vinyl copolymer	8	18	>1080
White polyamide cured epoxy	12	12	>1080
Light yellow polyamide cured epoxy	7	12	>1080
Phenolic epoxy	12	12	>1080

FIGURE 1. Decontamination Factor/Gloss

FIGURE 2. Decontamination Factor and Surface Roughness

24435-5

LEGEND

- 305 - Modified phenolic
- A - Ameron polyamide cured epoxy
- KL - Epoxy enamel
- G - Glidden polyamide cured epoxy

FIGURE 3. Paint Samples After 30-Day Acid Vapor Test

24435-4

LEGEND

T - High build gloss epoxy
T' - Dull epoxy
A3 - Vinyl copolymer
7122 - Phenolic epoxy

FIGURE 4. Paint Samples After 30-Day Acid Vapor Test

24435-7

LEGEND

- 305 - Modified phenolic
- A - Ameron polyamide cured epoxy
- KL - Epoxy enamel
- G - Glidden polyamide cured epoxy

FIGURE 5. Paint Samples After 30-Day Acid Immersion Test

24435-6

LEGEND

T - High build gloss epoxy
T' - Dull epoxy
A3 - Vinyl copolymer
7122 - Phenolic epoxy

FIGURE 6. Paint Samples After 30-Day Acid Immersion Test