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DEVELOPMENT OF TESTING PROCEDURES FOR PROTECTIVE
COATINGS TO BE USED IN NUCLEAR REACTOR
CONTAINMENT STRUCTURES

IDAHO NUCLEAR CORPORATION
NATIONAL REACTOR TESTING STATION
IDAHO FALLS, IDAHO

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by

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ABSTRACT

Tests were designed to examine protective coatings for characteristics important in applications to the interior surfaces of boiling and pressurized water reactor containment structures. The experimental verification of these tests also served to determine that there are coating systems available that contain all the desired physical and chemical characteristics.

SUMMARY

Some of the characteristics important in protective coatings that are to be applied to the interior surfaces of boiling and pressurized water reactor containment structures with emphasis on conditions present during and after a loss-of-coolant nuclear accident are:

- 1) Ease and reliability of application;
- 2) Repairability;
- 3) Resistance to gamma radiation;
- 4) Resistance to steam;
- 5) Chemical resistance to dilute base;
- 6) Non-sorption of boron compounds used as nuclear poisons;
- 7) Ability to seal minor imperfections in substrate;
- 8) Resistance to high pressure steam jet.

The first two requirements are not peculiar to the atomic energy industry. Tests were designed to examine coatings for these characteristics. The tests were verified by applying them to five coating systems shown earlier to have excellent resistance to radiation, chemicals, and pressurized steam. All of the projected tests, except the steam jet test, were successful in evaluating the coatings for the desired characteristics and in verifying that there are coating systems available containing all the desired characteristics.

With the equipment used the steam jet test did not raise the temperature of the coating, as would occur with a large steam jet. For an effective test it will be necessary to thermostat the coupons and/or have a higher capacity steam source.

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DEVELOPMENT OF TESTING PROCEDURES FOR PROTECTIVE COATINGS TO BE USED IN NUCLEAR REACTOR CONTAINMENT STRUCTURES

I. INTRODUCTION

In the event of a loss-of-coolant accident in a pressurized or boiling water reactor, any protective coating (paint) on the interior surfaces of the containment structure would be subjected to severe environmental conditions.

The purpose of the studies described here was to determine whether certain test procedures might be usable in a "Standard"^[a] for the evaluation of coatings for the above application, what levels of performance were possible for currently commercially available coatings, and whether or not the tests and performance levels measured were meaningful in terms of the desired application. Incidental to this primary purpose, valuable performance data were obtained on five protective coating systems^[b].

[a] Preparation of such a standard to be published ultimately by USA Standards Institute was begun in February, 1968.

[b] The successful performance of a protective coating depends on proper surface preparation, proper application and curing of all coats, and compatibility of the coats with each other and the substrate. Proper application for testing or use is dependent not alone on the quality of the protective coating itself, but also on the communication by the manufacturer of proper instructions for application to the designer and to the craftsman, as well as the experience and skill of the craftsman. In the studies reported here, several of these interrelated factors were involved. Where performance is less than perfect, careful analysis should be made to determine the source of the difficulty.

It should be emphasized that one coating is not to be considered generally superior because that coating happened to possess the particular characteristics of immediate interest. The experimental evaluations of protective coatings reported here were made to obtain information on the performance of these materials in the specific applications and environments described. Any conclusions as to the relative merits of the materials apply only to the specific conditions mentioned. The fact that some materials were observed to be superior or inferior to others in these tests does not imply any general findings of superiority or inferiority.

Water will be lost from the reactor in a loss-of-coolant accident as a jet of steam and water, and conditions in the containment structure following such an event will be characterized by the presence of fission products and radiation, and by steam-air atmospheres at elevated temperature and pressure in which water sprays containing chemical additives are operated to restore the structure to ambient conditions and scavenge fission products. These are relatively severe service conditions for a protective coating, and the tests required to evaluate coatings for this application are of necessity different from those used for more conventional applications. The radiochemical properties of these coatings (plate-out of fission products under accident conditions and decontamination) have already been evaluated^[1].

The five coating systems used in this work were selected because previous work^[1] showed them to have excellent resistance to pressurized steam, chemical reagents, and gamma radiation. The coating systems used to evaluate the proposed standard tests were:

- 1) Amercoat 66 - 1 coat Amercoat 66 Primer, 1 coat Amercoat 66 Surfacer, and 2 coats of Amercoat 66 Seal Gloss (white);
- 2) Phenoline 368 - 1 coat Phenoline 368 Primer, 2 coats of Phenoline 368 Finish, and 1 coat of Phenoline 368 Gloss (white);
- 3) Epoxy-Zinc Silicate System - 1 coat of a high quality chemically cured zinc silicate primer, and 3 coats of an amine cured epoxy finish coating^[a].
- 4) Tanclene - 1 coat Phenoline 368 Primer and 3 coats of Tanclene (white);
- 5) Phenoline 302 - 1 coat Phenoline 300 Orange Primer and 3 coats of Phenoline 302 (green).

Coatings tested were applied to carbon steel coupons except in the Containment Integrity Test where they were applied to stainless steel. The test coupon used in the latter test is shown in Figure 4; all other coupons were either 1-1/2 x 3/4 x 3/16 or 2 x 1-1/2 x 3/16 inch rectangles coated on all surfaces. All rust and scale were removed from the steel by sandblasting prior to fabricating coupons; the coupons were degreased in 1, 1, 1 trichloroethane and painted before blasted surfaces rusted. All coupons were painted by painters in the regular maintenance branch at the National Reactor Testing Station (NRTS), except where specifically noted.

[a] In the judgment of the authors, the poor performance of this coating system in several tests was due to improper curing of the primer prior to application of the finish coats. Variability in performance from this cause is a characteristic of some, if not all, zinc silicate primers; thus, these failures are judged to be associated more closely with the type of primer than the specific product. For this reason, the system is described here by generic type, rather than by product name.

During testing, the appearance in moderation of surface phenomena that did not greatly decrease a coating's ability to protect its substrate was not considered to constitute failure. Such phenomena would include color changes, chalking, and the appearance of a few small or medium sized unbroken blisters. Phenomena that produced particles, decreased a coating's ability to protect its substrate, or were the immediate precursor of either of these phenomena were judged to constitute failure. Such phenomena were: cracking, peeling, bubbling, loss of adhesion, excessive checking or alligatoring, delamination, excessive blistering, flaking, extreme tackiness, softening, disintegration, dissolution, and formation of large blisters.

II. EASE AND RELIABILITY OF APPLICATION

During extensive tests^[1,2] with a wide range of protective coatings, it had become obvious that some coating systems produced far more consistent results on test coupons than others. Where it existed, the inconsistency took the forms of:

- 1) Better performance from sample coupons prepared by the manufacturer than from those prepared by the NRTS shops.
- 2) Significant variation in performance between coupons prepared at different times by the same craftsman using the same equipment in the NRTS shops.

Consistent optimum performance from a coating is dependent on many factors including consistency of manufacture, adequate communication of instructions for all phases of application from the manufacturer to the craftsman, and careful adherence by the craftsman to these instructions. With so many sources of variability it was not possible to execute a program which would show the source of any inconsistency experienced, but it was possible to demonstrate with a relatively simple program that much variability could exist even among selected high quality coatings and that this variability was detectable by a simple testing program. In evaluating the results of this test, it should be noted that all five coatings gave excellent performance in earlier autoclave tests and that the autoclave test used here is a very severe one. Because of the very demanding and diverse conditions which will be present in a reactor containment structure during normal service and under accident conditions, it is felt that high performance under severe test conditions is a reasonable criterion in any pertinent evaluation test.

For the test, coupons were painted in three different paint shops with the standard coatings. The manufacturer's standard instructions for surface preparation, application, and curing were sent to each of the shops, with instructions that they be followed carefully. Test coupons (1-1/2 x 3/4 x 3/16 inch rectangles) were painted with the standard coating systems at the National Reactor Testing Station (NRTS) and the Oak Ridge National Laboratory (ORNL) and by a commercial paint contractor in Idaho Falls, Idaho. Painters at the two atomic energy sites have had considerable experience in applying coatings of the same generic types (epoxy and modified phenolic coatings) as are the five standard coatings. The Idaho Falls contractor had experience in applying one of the standard coating systems (a modified phenolic coating system) to a large structure.

The coatings were evaluated by exposure to pressurized steam at 30 psig (133°C) for five days. Coupons were tested in triplicate. In previous work^[1], it was found that coated coupons improperly prepared were seldom resistant to pressurized steam at 30 psig (133°C) for as

long as three days. All five standard coating systems would be satisfactorily resistant to pressurized steam at 30 psig (133°C) for three days if properly applied to carbon steel coupons^[1].

The majority of Amercoat 66 and Tanclene coupons painted by all three shops satisfactorily resisted the pressurized steam for as long as three days, the majority of the Phenoline 302 and 368 coupons painted at two of the three paint shops satisfactorily resisted steam for three days, and the majority of the Epoxy-Zinc Silicate System coupons painted by all three shops failed the steam test within three days (see Table I). Thus, the Amercoat 66 and Tanclene systems are the easiest to apply, and yield the most consistent results. Five days of autoclaving is probably too severe; three days of autoclaving may be more reasonable.

The results of this test agree with the overall experience that NRTS painters have had with these same coating systems. These painters have prepared 12 batches of Amercoat 66 coupons, nine batches of Phenoline 302, five of Phenoline 368, three of Tanclene, and one of the Epoxy-Zinc Silicate System. Based on resistance to the three-day pressurized steam test, NRTS painters have prepared no bad batches of Amercoat 66 or Tanclene coupons, one bad batch of Phenoline 368 coupons, and two bad batches of Phenoline 302 coupons. Test coupons painted with organic protective coatings by NRTS painters (including Amercoat 66 and Phenoline 368) over zinc silicate primers similar to the one used in the Epoxy-Zinc Silicate System have been very inconsistent in their behavior towards pressurized steam. Since the failures in these coating systems were of the type caused by loss of adhesion between zinc silicate primer and organic finish coating, the failures were probably caused by improper curing of the primer prior to application of the organic finish coating.

TABLE I

RESULTS FROM "EASE AND RELIABILITY OF APPLICATION" TEST

Coating	Painters	Deterioration After Autoclaving at 30 psig (133°C) for:														
		1 day			2 days			3 days			4 days			5 days		
		Coupon Number:														
		1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
Amercoat 66	ORNL	S[a]	S[a]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]
	NRTS	S[a]	S[a]	S[a]	S[a]	S[a]	S[c]	S[a]	S[c]	S[c]	S[a]	S[c]	S[c]	S[a]	S[c]	S[c]
	Idaho Falls	S[a]	S[a]	S[a]	S[a]	S[a]	S[c]	S[a]	S[a]	S[c]	S[a]	S[a]	S[c]	S[a]	S[a]	S[c]
Phenoline 368	ORNL	S[a]	S[a]	S[a]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
	NRTS	S[a]	S[a]	S[a]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]	S[c]
	Idaho Falls	U[d] [e]	U[d] [e]	U[d] [e]	--	--	--	--	--	--	--	--	--	--	--	--
Phenoline 302	ORNL	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	U[d]	S[a]	S[a]	--	U[d]	S[a]	--	--	S[a]
	NRTS	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]	S[a]
	Idaho Falls	U[d] [e]	U[d] [e]	S[a]	--	--	S[a]	--	--	S[a]	--	--	S[a]	--	--	S[a]
Tanclene	ORNL	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
	NRTS	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	U[e]	U[e]	U[e]
	Idaho Falls	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
Epoxy-Zinc Silicate System	ORNL	U[z]	S[a]	S[a]	--	U[g]	U[g]	--	--	--	--	--	--	--	--	--
	NRTS	U[f]	U[f]	U[f]	--	--	--	--	--	--	--	--	--	--	--	--
	Idaho Falls	U[f] [g]	U[f] [g]	U[f] [g]	--	--	--	--	--	--	--	--	--	--	--	--

S = Satisfactory performance
 U = Unsatisfactory performance

[a] = Unaffected
 [b] = Discoloration
 [c] = Unbroken blisters, # 4 in size or smaller, medium or few in density^[3]
 [d] = Cracking
 [e] = Peeling
 [f] = Excessive wrinkling
 [g] = Excessively large blisters, bubbles, and/or medium dense or dense blistering^[3]

III. REPAIRABILITY OF COATINGS

Small areas of the coated interior surfaces of a containment structure, as well as any other large structure, will need repairing from time to time. It is impractical to remove the coating from the entire surface, prepare the substrate, and repaint; rather, it is necessary to be able to repair the small areas that need repainting. The purpose of the test described in this section is to determine if an aged coating can be repaired without impairing the resistance of the coating to the most severe condition (presence of pressurized steam) that might exist in a containment structure. Detailed instructions for repair were requested directly from each of the manufacturers. These instructions were sent to the paint shop with the direction that they be followed exactly.

Carbon steel coupons (1-1/2 x 3/4 x 3/16 inch rectangles) coated with the standard coating systems were cured for greater than one year. Several of the coupons painted with each coating system were purposely damaged, others were damaged and repaired, while still others were allowed to remain intact. Coupons painted with coating systems were damaged by removing a 5/16-inch diameter area down to the substrate by drilling away the coating. A 5/16-inch metal drill with the point ground flat was used. Damaged coating systems were repaired by painting the 5/16-inch circular area with the entire coating system (primer and finish coats) already on the rest of the coupon following directions of the paint manufacturer for repair, making no effort to confine the new paint to the 5/16-inch circular area. The new paint (over the repair) was allowed to age for at least two weeks. Coupons of each coating system in various phases of damage and repair were autoclaved for five days at 30 psig (133°C). Coupons of each coating system that were autoclaved included:

- 1) one coupon coated with the aged coating system
- 2) three coupons coated with the aged coating system that had been damaged
- 3) three coupons coated with the aged coating system that had been damaged and repaired.

The results (see Table II) indicate a range of performance of the repaired coatings and some failures to give performances equivalent to the original application. While the five day autoclaving period may be unrealistically severe, it has served to display distinct differences in performance. Three days of autoclaving would probably be more reasonable than five days. Possibly the organic coatings could be aged more quickly by curing, at 60°C for several weeks.

TABLE II

RESULTS FROM "REPAIRABILITY OF COATINGS" TEST

Coating	Degree of Damage and Repair	Deterioration After Autoclaving at 30 psig (133°C) for:														
		1 day			2 days			3 days			4 days			5 days		
		Coupon Number:														
		1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
Amercoat 66	Original Undamaged Coupon	S[b]			S[b]			S[b]			S[b]			S[b]		
	Damaged Coupons	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
	Damaged and Repaired Coupons	S[b]	S[c]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
Phenoline 368	Original Undamaged Coupon	S[b]			S[b]			S[b]			S[b]			S[b]		
	Damaged Coupons	S[b]	S[b]	U[c]	S[b]	S[b]	U[c]	S[b]	S[b]	U[c]	S[b]	S[b]	U[c]	S[b]	S[b]	U[c]
	Damaged and Repaired Coupons	S[a]	S[a]	S[a]	S[a]	S[a]	U[c]	S[a]	S[a]	U[c]	S[a]	S[a]	U[c]	S[a]	S[a]	U[c]
Phenoline 302	Original Undamaged Coupon	S[b]			S[b]			S[b]			S[b]			S[b]		
	Damaged Coupons	S[a]	S[a]	S[a]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
	Damaged and Repaired Coupons	S[b]	S[a]	S[a]	S[b]	S[a]	S[a]	S[b]	S[a]	S[a]	U[b] [c]	S[b]	S[b]	U[b] [c]	S[b]	S[b]
Tanclene	Original Undamaged Coupon	S[a]			S[b]			S[b]			S[b]			S[b]		
	Damaged Coupons	S[a]	S[a]	S[a]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
	Damaged and Repaired Coupons	U[c]	S[a]	S[a]	U[c]	S[b]	S[b]	U[c]	S[b]	S[b]	U[c]	S[b]	S[b]	U[c]	S[b]	U[c]
Epoxy-Zirc Silicate System	Original Undamaged Coupon	S[b]			S[b]			S[b]			S[b]			S[b]		
	Damaged Coupons	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]	S[b]
	Damaged and Repaired Coupons	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]	U[d]

S = Satisfactory performance

U = Unsatisfactory performance

[a] = Unaffected

[b] = Unbroken blisters, #4 in size or smaller, medium or few in density^[3]

[c] = Cracking

[d] = Bubbling and wrinkling

IV. COMBINED EFFECT OF GAMMA IRRADIATION AND STEAM

Under loss-of-coolant accident conditions, protective coatings will be exposed to gamma radiation in the presence of steam, water, and air. Coatings to be applied in containment structures should be subjected to a uniform radiation-tolerance test procedure to determine if the coating is sufficiently resistant to high gamma radiation levels that might exist after a loss-of-coolant accident and to the low levels which might exist routinely. The test described in this section exposed the five different coating systems in the presence of steam because this condition is more severe than exposure in the presence of water or air. The steam was generated within the container containing the coated coupons. Because there is some uncertainty in the estimate of the radiation dose to a coating in a loss-of-coolant accident, coatings were exposed to a total gamma dose of 3×10^9 R but were examined after exposures of 1×10^9 , 2×10^9 , and 3×10^9 R. The maximum exposure dose in such a test should be about 1×10^9 R based on studies to determine the volatility of fission products during a loss-of-coolant accident carried out at the Idaho Chemical Processing Plant in the Contamination-Decontamination Experiment^[4].

The five coating systems on coupons $1\text{-}1/2 \times 3/4 \times 3/16$ inches were irradiated at a gamma dose rate of about 2×10^6 R/hr in steam at one atmosphere. Results are summarized in Table III. Four coating systems were satisfactorily resistant to a gamma dose of 1×10^9 R in the presence of steam at one atmosphere, and at least two of the systems were satisfactorily resistant to a gamma dose of 3×10^9 R in the presence of steam at one atmosphere.

TABLE III

PERFORMANCE OF COATINGS IRRADIATED AT 2×10^6 R/hr
IN THE PRESENCE OF STEAM

<u>Coating</u>	<u>Radiation Dose</u>	<u>Deterioration Noted</u>	<u>Performance Rating</u>
Amercoat 66	1×10^9 R	[a]	Satisfactory
	2×10^9 R	[a] [d]	Satisfactory
	3×10^9 R	[a] [d]	Satisfactory
Phenoline 368	1×10^9 R	[a] [b]	Satisfactory
	2×10^9 R	[a] [b] [d]	Satisfactory
	3×10^9 R	[a] [b] [d]	Satisfactory
Tanclene	1×10^9 R	[a] [e]	Satisfactory
	2×10^9 R	[a] [e]	Satisfactory
	3×10^9 R	[a] [f]	Questionable
Phenoline 302	1×10^9 R	None	Satisfactory
	2×10^9 R	None	Satisfactory
	3×10^9 R	[a] [c] [f]	Unsatisfactory
Epoxy-Zinc Silicate System	1×10^9 R	[g]	Unsatisfactory

[a] = Discoloration

[b] = Unbroken blisters, # 4 in size or smaller, medium or few in density [3]

[c] = Broken blisters

[d] = Surface webbed with fine brown lines

[e] = Faint alligatoring

[f] = Pronounced alligatoring

[g] = Bubbling, excessive blistering, and peeling

V. RESISTANCE TO STEAM AT ELEVATED TEMPERATURE AND PRESSURE

Coatings in reactor containment structures will be subjected to pressurized steam during and after a loss-of-coolant accident. These structures will generally contain engineered safety systems which might spray the inside of the building with solutions to reduce steam pressure more rapidly and pump solutions through the reactor to cool the reactor core. Numerous sets of design accident conditions can be predicted depending upon the number of engineered safety systems that operate and/or the efficiency at which the operating systems function. Assuming functional failure of about 50 percent of the core cooling, recirculation loop capacity, and containment cooling systems with the remaining systems operating on emergency power, the maximum steam pressure inside a pressurized water reactor (PWR) containment structure under accident conditions would be about 58 psig (141°C) and it would take weeks to decay to one psig (38°C); the maximum pressure in a boiling water reactor (BWR) containment structure would be about 42 psig (141°C) and it would again take weeks to decay to one psig (38°C)[5]. The preceding temperature-pressure relations occur when the structure contains one atmosphere of air pressure in addition to pressure from steam. The five standard coating systems were tested in pressurized steam-air mixtures simulating the most extreme conditions that would exist in either accident.

In the test, duplicate coupons painted with the five different coating systems were subjected to the steam pressure decay curve shown in Figure 1. The autoclave shown in Figure 2 was used to expose the coupons to pressurized steam. Pressure within the autoclave was controlled by varying the heat supplied.

Both Epoxy-Zinc Silicate System coupons failed due to bubbling, one of the Phenoline 302 coupons failed due to cracking, while both Tanclene coupons became discolored. All other coupons were unaffected. All observable deterioration occurred within the first seven hours of the test, during the high temperature portion of the cycle.

A simpler test that would also be effective in determining the resistance of a coating to the pressurized steam conditions that might exist inside a reactor structure during a loss-of-coolant accident would be to autoclave coatings for three days at 30 psig (133°C)[1].

Figure 1 Steam pressure decay curve for the pressurized steam test.

Figure 2 Autoclave for pressurized steam test.

VI. CHEMICAL RESISTANCE TO DILUTE BASE

In the event of a nuclear accident to a reactor with an emergency core cooling system and a containment structure pressure reduction system, the solution recirculating through the containment spray system will also have to be circulated through the reactor core. Under these conditions it is imperative that the protective coatings on the internal surfaces of the containment structure not degenerate during the spraying period. The presence of pieces of paint in the combined core cooling-spray system water could interfere with the flow of core cooling water. A test was designed to determine the resistance of each coating to sprays of dilute basic deluge solutions that might be used during a post-accident period.

One coupon of each of the five coating systems was continually deluged for one month at 65°C with 300 ml of a solution containing 0.37M sodium tetraborate, 0.13M sodium thiosulfate, and 0.20M sodium hydroxide^[a]. Test coupons used were 2 x 1-1/2 x 3/16 inch rectangles. The test equipment was an air lift and loop as shown in Figure 3. The loop contained a spray head, a device for holding the test coupon under the spray head, and a 40-mesh filter. During operation of the loop, most of the 300 ml of solution was always present in a reservoir. The solution deluging the test coupon was maintained at 65°C by applying heat to the solution in the reservoir from heating tapes wrapped around the exterior surfaces of the reservoir. The results of the test are shown in Table IV. The coating systems tested showed very little deterioration after being deluged with the dilute base for one month. The treatment did not remove solids of observable size from any of the coating systems tested.

[a] A number of different solutions containing nuclear poisons, bases, and reactants for iodine have been considered for use as core cooling and containment spray solutions. This mixture represents one possible combination of the many solutions being considered.

Figure 3 Air lift for deluging test coupons with dilute base.

TABLE IV

RESISTANCE OF COATINGS TO A DILUTE BASE DELUGE FOR ONE MONTH AT 65°C

<u>Coating</u>	<u>Deterioration Noted</u>	<u>Coating Solids 40 Mesh or Greater in Size Found in Deluge Solution</u>	<u>Percent of Total Coating Weight Lost From Speci- men During Treatment</u>	<u>Performance Rating</u>
Amercoat 66	[a] [b]	None	0.35	Satisfactory
Phenoline 368	[a]	None	7.2	Satisfactory
Phenoline 302	[a]	None	3.6	Satisfactory
Tanclene	[a]	None	0.88	Satisfactory
Epoxy-Zinc Silicate System	[b]	None	4.7	Satisfactory

[a] = Discoloration

[b] = Unbroken blisters, # 4 in size or smaller, medium or few in density^[3]

VII. SORPTION OF SODIUM BORATE (NUCLEAR POISON) BY COATINGS

In the event of a nuclear accident to a reactor with an emergency core cooling system and a containment pressure reduction spray system, the emergency core cooling solution containing borate will probably also have to be recirculated through the containment spray system. Since the borate is present to maintain the reactor core below critical conditions, sorption of borate by coated surfaces can not be tolerated. The purpose of the test described in this section is to determine whether a coating sorbs borate.

One test coupon of each of the five coating systems was exposed for one month at 65°C in 750 ml of a solution containing 0.37M sodium tetraborate, 0.13M sodium thiosulfate, and 0.20M sodium hydroxide. Test coupons used were 2 x 1-1/2 x 3/16 inch rectangles. The solution was analyzed before and after the test for boron. Polyethylene containers were used to hold the solution, and polyethylene stirrers were used. Stirring was performed at such a rate as to form only a slight vortex.

As shown in Table V, this treatment produced about the same results as deluging the coatings for one month with the same solution. The treatment produced only minor damage to all coating systems. None of the coating systems sorbed a significant amount of boron. This test could be made more sensitive by decreasing the ratio of the volume of boron containing solution to the surface area of coating.

TABLE V

PERFORMANCE OF COATINGS IMMERSSED IN A STIRRED SOLUTION OF DILUTE BASE
AT 65°C FOR ONE MONTH

Coating	Deterioration Noted	Percent of Total Coating Weight Lost from Coupon During Treatment	Percent Loss or Gain of Boron from Dilute Base During Treatment	Performance Rating
Amercoat 66	[a] [b]	0.9	1.0 lost	Satisfactory
Phenoline 368	[a]	gained 2.2	1.0 gained	Satisfactory
Phenoline 302	[a]	3.3	1.0 gained	Satisfactory
Tanclene	none	0.5	0.3 lost	Satisfactory
Epoxy-Zinc Silicate System	[b]	1.2	2.1 lost	Satisfactory

[a] =Discoloration

[b] =Unbroken blisters, # 4 in size or smaller, medium or few in density[3]

VIII. CONTAINMENT INTEGRITY, SEALING

It is essential that volatile radioelements released from a reactor during an accident be prevented from escaping the containment structure to the atmosphere. Efforts are made to fabricate the structure as tightly as possible to prevent such leaks. A test is described in this section to determine the ability of a coating to fill random penetrations in a containment structure wall.

The five coating systems were tested in the equipment shown in Figure 4. The test coupons consisted of blind flange covers for the pressure vessel. The covers contained seven 1/32-inch diameter holes; the inside of the covers were painted with the protective coating under test. Observations were made to determine whether the cover containing the holes sealed the test vessel so as to maintain 10 psig of steam pressure within the vessel for one week. In testing a coating, 200 ml of water were added to the vessel, the lid was secured to the vessel using the neoprene gasket for a tight seal, the resulting equipment was weighed, the vessel was maintained at 10 psig for one week using heating tape, the vessel was cooled to 1 psig, the needle valve was opened to prevent a vacuum from being formed in the vessel, the vessel was weighed, the cover was removed, and the coating on the inside of the cover was inspected.

Figure 4 Equipment for the containment integrity test.

The results of the test are shown in Table VI. All five coating systems satisfactorily filled the seven 1/32-inch diameter holes so as to maintain 10 psig of steam pressure within the test vessel for one week. In all the tests, very little water escaped from the vessel; coatings were not excessively deteriorated. The holes in the test coupons could be made larger for a more meaningful test.

TABLE VI
CONTAINMENT INTEGRITY TEST FOR COATINGS

Coating	Deterioration Noted	Percent by wt. of Water Lost From Vessel During Test	Ability of Coat- ing to Hold Pressure
Amercoat 66	[a] [b] [c]	0.25	Satisfactory
Phenoline 368	[d]	0.08	Satisfactory
Phenoline 302	none	0.30	Satisfactory
Tanclene	[b]	0.06	Satisfactory
Epoxy-Zinc Silicate System	[b] [c] [e]	0.12	Satisfactory

[a]=Discoloration

[b]=Unbroken blisters, # 4 in size or smaller, medium or few in density[3]

[c]=Dimples over some of the 1/32 inch diameter holes

[d]=Contained large unbroken blister which disappeared upon standing at ambient conditions

[e]=Slight wrinkling in some areas

IX. RESISTANCE TO HIGH PRESSURE STEAM JETS

Relatively small areas of any coating system applied to the internal surfaces of a containment structure which may be located near the site of a primary system rupture in a pressurized or boiling water reactor may be subjected to a high pressure steam jet during loss of coolant from the reactor. The dimensions of the affected area would probably be less than one-tenth of a percent of the internal surface area of the structure, and steam pressures inside the reactor would decrease from an initial 2200 psia to 100 psia in about ten seconds during a pressurized water reactor accident and from 1000 psia to 100 psia in about 30 seconds during an accident in a boiling water reactor[5].

The test used to determine if a coating is resistant to a high pressure steam jet utilized the equipment shown in Figure 5. The pressure vessel was of stainless steel and had a maximum operating pressure of 1800 psig. The vessel had a capacity of one liter and was 10-1/4 inches in length and 3-1/2 inches in diameter. Water was put into the vessel through the tubing leading to the pressure gauge. Heat was supplied to the vessel by heating tapes (controlled with a variac) wrapped around its external surfaces. The needle valve was changed after every test.

In the test, steam was directed for 5 minutes through a 0.0135-inch diameter orifice onto a coated coupon (1-1/2 x 3/4 x 3/16 inch) located one inch from the orifice. The coupon was at right angles to the nozzle. The initial pressure of the steam jet was 1000 psig which decayed to 250 psig in five minutes..

All five coatings gave satisfactory performances in this test. Two coatings (Amercoat 33HB and Turco 5302) that give poor resistance to steam[1,2] were also tested. The Amercoat 33HB was unaffected by the steam jet. The steam jet made a crater about 3 mils in depth in Turco 5302; this coating becomes soft and fluid when subjected to pressurized steam for several hours. The steam jet was directed on Amercoat 66 coupons held at right angles to and at an

Figure 5 Equipment for high pressure steam jet test.

angle of 45° from the axis of the spray. The steam jet scorched the specimens slightly when located at right angles to the coupons but not at an angle of 45° ; thus, the former orientation results in the more extreme exposure. The results of this test indicate that steam-resistant coatings would not be harmed by a jet of moderate pressure (1000-250 psig) steam directed on them for a short period.

In order to confidently scale-up the results of this test to the effects of a full scale blowdown, further tests with larger equipment and panels would have been required. This was outside of the scope of the program. Until such scale-up tests are done, the resistance of a coating in a simple autoclave test should be taken as an index of resistance to a steam-water jet.

X. CONCLUSIONS

The use of commercially available protective coatings for finishing the inside of nuclear reactor containment structures with the expectation that they will effectively survive a loss-of-coolant accident and will in no way interfere with the proper operation of the reactor safety systems during and after the accident represents a very severe service for these materials. Tests to evaluate protective coatings for this service are in some cases different from those used to evaluate coatings for more conventional service. All but one of eight new tests studied were successful in determining whether or not the standard coating systems had the desired characteristics. The results obtained with the specific coatings tested clearly indicate that there are products available which can give the performance required in nuclear reactor containment structures under accident conditions. Except for the steam jet test, the test conditions and the levels of performance sought all appear to be significant in terms of the projected application and the capabilities of commercially available coatings. All coatings that have good steam resistance, and possibly a majority of the coatings that do not, would be resistant to the short duration steam jet test; this test probably would not predict the resistance of a coating to actual steam jets because of scale-up uncertainties. While test conditions in the other seven tests served to display distinct differences in coating performances, in three of the tests these conditions were unrealistically severe. In these three tests, it was felt that the test conditions could be made less severe and still indicate whether a coating was sufficiently satisfactory in the characteristic being tested to perform satisfactorily in a reactor accident.

XI. REFERENCES

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