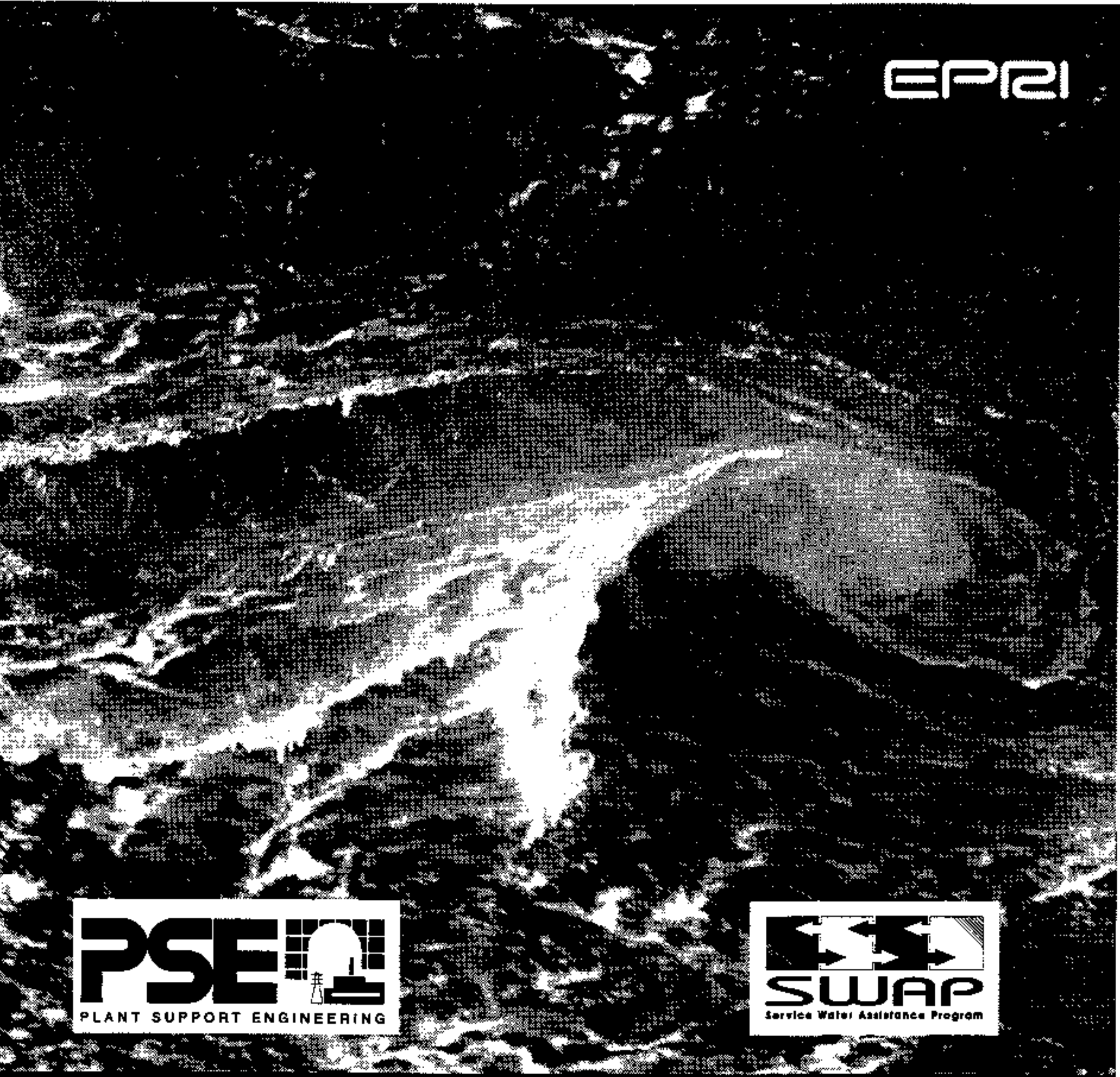

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COATING FAILURE AT MILLSTONE 3 - LESSONS LEARNED

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Abstract

Protective coatings installed in service water systems can potentially impact the reliability and operability of safety-related functions in the event of a coating failure. Licensees are addressing this issue as a result of documented coating failures and increased NRC attention (NRC IE Notice 97-13, pending NRC Generic Letter). EPRI has developed a guidance document (Guidelines on Nuclear Safety-Related Coatings) to assist licensees in addressing the issue. This paper discusses a service water system coating failure case history at the Millstone Nuclear Plant and addresses lessons learned that may apply to other plants.

Northeast Nuclear's Millstone Unit 3 uses protective epoxy coatings on the internal surfaces of Service Water piping for erosion and corrosion protection. After two failures of coatings in safety-related applications were discovered, the original safety evaluation assumptions and application methods were determined to be inadequate. Actions were taken to flush the system to ensure all delaminations were removed and a significant program was developed to inspect and test 100% of safety-related epoxy coating installations. New procedures were developed to address environmental controls and to ensure proper curing of newly installed coating systems. Testing was performed to determine failure mode and mechanism as well as ongoing material suitability. Extensive analysis indicated inadequate work practices during field application were the cause, and not a failure of the ARCOR epoxy product itself. Coating installation was determined to be an Unreviewed Safety Question and a License Amendment was submitted.

Service Water Lining Installation

The Service Water System at Millstone Unit 3 (MP3) is a combination of solid 90/10 copper nickel concrete encased (underground) piping and carbon steel clad with 90/10 CuNi for 18" through 30" headers. Small bore (12" and less) is solid 90/10 CuNi with recent repairs made using Monel 70/30 CuNi. Two 100% independent trains take suction from Long Island sound using raw water for cooling throughout the plant.

Unit 3 Service Water was placed in service July 1985 with plant commercial operation in May 1986. Cladding degradation was noted in a relatively short time after startup. In 1991, various epoxy coatings were evaluated for use in repairing degraded cladding conditions and providing erosion/corrosion protection to base metal repairs.

Products from Speeco, Belzona, ARCOR and Plastocor were used in limited demonstration amounts to coat the internal surfaces of pumps, piping and selected components. MP3 selected ARCOR as the primary protective coating for installation in the Service Water system based on its high ratings in blister resistance testing and the effective use of this category of products worldwide in mitigating circulating water system corrosion problems (1). ARCOR coatings are "Novolac" multifunctional epoxy resins with ring-type amine curing agents. This combination produces cured coatings that are generally superior to conventional "Bis-A" epoxies. Desirable characteristics of these non-solvent emitting (NSE) epoxy formulations (also referred to as "one hundred percent solids") include sustained flexibility over time, excellent surface wetting and enhanced resistance to moisture penetration (2).

MP3 Service Water lining materials used prior to 1997 were purchased non-QA, but installation was performed using a "QA" maintenance procedure. The System Engineer was the designated authority based on need, and determined what and when to coat. Major piping lining projects were performed as design changes to the plant with 10CFR50.59 Safety Evaluations determining that these changes were safe because failure was not credible. The Safety Evaluations assumed that any failure that did occur would be limited to small flakes which would have no impact on the ability of the system to perform its safety function. Limited scope coating applications were performed on components as enhancements to corrosion protection often by Nonconformance Report which required no 50.59 screening.

ARCOR provided MP3 with a general application specification, a technical specification sheet for each coating product and quality control guidelines for documenting conditions and coating system characteristics. Included was guidance on material handling and storage, surface preparation, mixing ratios and methods, application techniques, recoat/cure times and inspection criteria. This information was incorporated into the MP3 maintenance procedure for surface preparation, application and testing requirements of ARCOR coating products, issued September 1991 (3). This procedure was used by plant personnel and contractors to apply the lining. The procedure did not contain requirements for applicator familiarization with the lining products or formal application training. Informal training was provided to the applicators by an ARCOR representative who was requested to be on-site during some, but not all, of the installation efforts (1).

The ARCOR products were applied in the large bore piping at MP3 by a contractor in accordance with the maintenance procedure. The procedure called for a 30-60 mil thick dry film lining applied in two coats. The products were generally rolled or brushed on. An ARCOR rebuild product was also used that was troweled on. Documentation called for by the procedure included start/stop times, substrate temperatures and relative humidity. Actions not specified in the procedure included contamination testing, humidity control and wet or dry film thickness testing. The procedure specified that a low voltage spark test be performed after completion of coating installation to detect voids, skips and holidays (3).

Although ongoing heat exchanger and piping inspections found evidence of minor flaking and chipping of the lining, it was considered to be expected and not found to be a reason for concern. Eventually extensive areas of coating were installed throughout the Service Water system. A written spool pieces matrix log was used to keep track of where coatings had been used and to record a chronological history of installations.

Lining Failures

In May 1995, a delamination was found in a 30" header at the outlet of a RBCCW heat exchanger during a planned outage inspection. The location had no safety impact as all debris would be flushed overboard and the isolated case was attributed to improper application as no bonding between coats had occurred.

In July 1996, following a flush of the Containment Recirculation Heat Exchangers, sufficient quantities of ARCOR were found that the function of the heat exchangers could have been affected. The total area of material recovered exceeded the tube plugging limits assumed in the accident analysis calculations. A Licensee Event Report (LER) was written. As corrective action to the LER, the delaminated coating was located and repaired. The source was a 30" elbow located in the supply header to the train at the intake structure. Although the area of missing coating was only a few square feet, the entire remaining topcoat of the elbow showed questionable adhesion during base coat preparation, indicating additional failure was likely.

Root Cause Investigation

A root cause investigation was conducted, which resulted in the finding that 1.) no formal plant organization "ownership" of the coating program existed, 2.) procedures for field installation of coatings were inadequate and 3.) training of applicators was insufficient.

A review of the lining application procedure determined that it was inadequate and in some cases contained information that was incomplete or incorrect (4). The lack of consistency in the requirements for ARCOR application was attributed to not fully understanding application requirements, such as recoat windows being not well understood or strictly adhered to. Subsequently, a new site-wide application procedure was developed which provided consistent and adequate technical information for all three units. The procedure was configured to adequately address personnel qualifications, environmental condition control, surface preparation standards, proper application techniques and thorough documentation.

The procedure also dramatically increased the requirements for QA coating applications. Painter and Inspector qualifications were delineated for all site activities involving safety-related painting. A minimum level of applicator expertise and experience with coatings was stipulated. Painters were required to be certified through formal training and performance-based assessment. A proficiency demonstration was introduced as part of the evaluation process. Maintenance of training records was specified along with personnel recertification on a periodic basis.

In early 1997, all coating applications were reviewed for applicability under Reg Guide 1.54, QA Requirements for Protective Coatings in Water Cooler Nuclear Power Plants. A Material Equipment Plant Listing (MEQL) evaluation was performed which determined that coating installations, whose failure could affect QA components, are QA and require QA material and procedures.

Commercial Grade Dedication of the ARCOR safety-related lining products was implemented under the MP3 Appendix B program. Critical characteristics were defined in the areas of physical attributes, performance characteristics and product identification. Source inspections were initiated which involved MP3 inspectors witnessing formulation and batching of product destined for safety-related application.

Another corrective action required that all safety evaluations be revisited for previous lining applications to determine if assumptions were still valid. Because failure was still attributed to improper application, the safety evaluation review concluded the original evaluations were valid. The Design Engineering organization could not assume improper application in the evaluation. A similarity was drawn to a welding installation which could fail if improperly executed. The evaluation would assume proper welding to code requirements.

Another recommendation from the root cause investigation was that program ownership be mandated for the installation of linings due to the safety significance. Consistency throughout the three unit site was required and control established under a single, experienced group. A decision was made in May 1997, per the recommendation of the root cause evaluation, that the entire coating program be controlled by the Nuclear Materials group whose expertise included application of linings. For the first time, a sitewide program was developed and procedures were revised with input from industry experts retained as consultants. A position of Responsible Lining Engineer (RLE) was developed to have a point contact for all questions relating to coatings.

In April 1997, during an inspection of piping previously coated with ARCOR, a spool was found to have approximately 15 square feet of top coat missing or delaminated. As a result, a high velocity flush was performed on the affected train of Service Water to ensure no coating material remained which could impact heat exchanger function. Heat exchanger inspection showed no debris. All ARCOR coatings had now become suspect. A commitment was made to perform 100% inspection of all QA installations (upstream of QA heat exchangers).

A new 10CFR50.59 Safety Evaluation was prepared by the Nuclear Materials group incorporating all the new information and programmatic commitments. The determination was made that addition of linings to the Service Water System could increase the probability of previously evaluated malfunctions of equipment important to safety. This resulted in the determination that coating application was an Unreviewed Safety Question (USQ). However, it was also determined to be "SAFE" based on the high confidence that gross failures had been eliminated.

Credit was taken for weekly heat exchanger surveillances to detect failures in addition to scheduled GL 89-13 visual heat exchanger inspections to discover failed linings. MP3 accident scenarios are not more dynamically challenging to the system than normal operation and so no initiating coating failure events are caused by accident mitigation system alignments. Additional details of the SE can be obtained from the authors.

A License Amendment including the USQ determination has been submitted to the NRC for approval. An Operability Determination, per the requirements of GL 91-18 R1, Degraded and Nonconforming Conditions, was prepared to allow plant entry into Mode 4 and above while the USQ is outstanding.

Field Inspection and Testing

In order to demonstrate that the coating installed in the large bore SW piping would not come off in service, a field test program was devised. A project was undertaken from May through September 1997 to inspect and, as necessary, replace all QA linings. As part of this project, an attempt was made to determine a method to inspect installed coatings for delamination without destructively testing the surface. It was understood that visual inspection was not adequate to find delaminated layers which appeared intact. Hammer "thumping" was eliminated as too imprecise.

Testing initially consisted of adhesion pull testing to show that there were no large areas that did not have proper adhesion. A test methodology was developed to use "pull tabs" glued to the surface which could be pulled off using a calibrated instrument at a set value. An optimally applied coating consisting of several coats should fail cohesively (e.g. not between coats) when tested to ultimate strength. The proof load used was approximately 50% of the ultimate strength of the coating. Results of lab tests of properly and improperly applied coating were factored into the proof load. Test plates were specially prepared with (1) proper application, (2) intercoat contaminated by dust and moisture, and (3) deliberately overcured intercoat. Testing was done to determine the strength of "good" and delaminated coatings to determine a value to be used in the field for validation.

Problems occurred with the pull test method; in one case, tabs pulled to satisfactory strength in the field resulted in failed coatings when tabs were removed. As a result , a decision was made to destructively test all coatings using "X-cuts" to ensure unquestionable quality of installed linings. This method involved cutting a two inch long "X" incision and using a putty knife to probe the apex of each incision in an attempt to force disbondment (5). Each spool piece (nominally 10 feet linear length) had three X-cuts made including each end and center. These tests were determined to be subjective in nature and results were based on interpretation by the various inspectors and engineers involved.

Failure Mode and Mechanism Analysis

The possibility of both product failure and application failure were considered in efforts to identify the cause of delamination. Extensive analysis indicated inadequate work practices during field application were the cause, and not a failure of the ARCOR epoxy product itself (6).

Prior to the discovery of the large scale delamination at MP3, coating specialists associated with the power industry assumed that non-solvent epoxy linings fail in small chips (2). This was based on evidence from laboratory testing and field experience. August, 1991, May, 1993, and August, 1994 MP3 Safety Evaluations stated that "Past experience with ARCOR coating at MP3 has shown that, rather than failing in large sheets which might impact downstream components (RO's, heat exchangers), ARCOR tends to fail in small flakes" (1).

Contrary to this widely held belief, evidence indicates that ARCOR did fail in larger than small chips at MP3 via intercoat delamination (6). A two square foot piece of ARCOR material was removed from a service water spool piece. Another 15 ft^2 area was found to be missing its topcoat (it's uncertain whether this piece failed as a single piece or broke up into chips). The cause of the intercoat delamination was believed to be the result of improper bonding between the first and second coats during application (6).

Initially, it was thought that the cause of the intercoat delamination was "missed" recoat windows. Recoat window is the minimum and maximum amount of time specified by the manufacturer for application of an additional coat on top of an existing coat. Industry practices have long recognized that polymeric linkages between a prior coat of a system and fresh material will be impaired if the prior coat advances to a level of cure that inhibits cross linkage. The overcoat window for the ARCOR products used at MP3 is between six and eight hours when applied by brush or roller at 72° F. Lab adhesion tests at MP3 revealed that that overcoat window is not a critical control variable for the ARCOR products. Samples were prepared in which the prime coat was fully cured by heating at 200° F for 24 hours, well beyond ARCOR's stated required conditions for complete curing. The data from the forced cured intercoat showed an average tensile strength 25% greater than samples with proper intercoat curing (7).

In an attempt to identify an alternative cause of the intercoat delamination, a literature review was initiated. One study published in 1994 indicated that aliphatic amine-cured epoxies such as ARCOR are prone to intercoat adhesion failure at moderate relative humidity when ambient temperatures are at the lower end of the allowable range (8). At room temperature of 21° C (70° F), the rate of reaction between the amine curing agent and the epoxy prepolymer is rapid, resulting in the formation of coatings with good intercoat adhesion. At lower temperatures, the cross-linking reaction rate decreases, creating an opportunity for moisture to permeate the coating and form a solution with the amine. In this soluble state, the amine can react with carbon dioxide in the atmosphere to form carbamate salts. These salts are incapable of reacting with the prepolymer to form proper cross-linking between coats. Test results showed that intercoat adhesion failure can be observed at a relative humidity level of 40% when temperature is 13° C (55° F).

Lining Material Suitability Determination

The potential for ARCOR product failure was evaluated through qualification testing and a study of product performance in similar applications. A study of ARCOR applications in cooling water systems at other plants was conducted in order to determine if instances of intercoat delamination had occurred. The study concluded that the ARCOR products were suitable for installation as linings in raw water systems (9).

No industry-wide test standard currently exists for evaluating the suitability of linings in cooling water systems (2,10). This may be due in part to the challenge of creating a standard that addresses the range of site-specific conditions potentially impacting the suitability for any particular plant. Millstone generated its own ad hoc guidelines by taking into consideration a range of test results when evaluating the ARCOR product suitability.

Qualification testing results considered by Millstone in its justification to continue using ARCOR after the intercoat delamination included:

- Permeability Testing (per ASTM E96)
- Immersion Environment Testing - Atlas Cell (Modified NACE TM 0174)
- Reverse Impact Testing (ASTM D2794)
- Flex Testing
- Adhesion Testing (ASTM D4541)
- Cathodic Disbondment (ASTM G-42)
- Dielectric Strength (ASTM D115)
- Mandrel Bend (ASTM D522)
- Direct Impact (ASTM G14)
- Abrasion Resistance (ASTM D4060)

The testing which best replicates service water system immersion conditions is Atlas Cell. The primary objective of such testing is to assess the affect of "cold wall" conditions (temperature differential between the coating surface and substrate) on product performance. Moisture tends to migrate toward the coolest location in the coating/substrate. If the substrate is significantly cooler than the coating surface, the rate of moisture permeation through the coating toward the substrate will be accelerated. This leads to premature blistering and failure of the coating (10). A study of cold wall resistance was commissioned by Northeast Utilities (and several other utility companies) to obtain information on resistance to cold-wall blistering. ARCOR and several other polymer coatings were tested in thin and thick film versions under expected and accelerated conditions. The ARCOR product performed best overall, providing MP3 with a highly defensible basis for suitability (11).

There are numerous manufacturers of these types of polymer coatings, with formulation differences in terms of stoichiometry; additives; batching process and other variables. With the determination that coatings used outside containment in immersion service are QA Category 1, each licensee using these products may have to justify the suitability of existing or proposed lining materials. Development of an industry-wide standard would establish a consistent basis for determination of suitability, enhancing efficiency for both the licensee and coating manufacturer.

Any standard should take into consideration the change in physical properties that results due to long term immersion of the coating. Physical characteristics of saturated coatings can be quite different than in the dry state. The standard should also ensure that the material being tested and the material ultimately used are one in the same.

Tables 1 & 2 contain a matrix of suitability testing requirements proposed for use at Millstone. Tests are grouped as Phase 1 and Phase 2. For a given lining product category, as indicated in the Description column, material used in the performance of all Phase 1 testing must be from the same batch numbers. Materials used in Phase 2 testing may be from batch numbers different that those used in Phase 1.

In the proposed Phase 1 suitability testing, each component of material (base resin, curing agent) to be used is initially "finger printed" by Fourier Transform Infrared (FTIR) analysis. This provides a baseline for analysis of each batch of material received for future use, in order to verify composition of the material. Additional tests performed include permeability, 180 day atlas cell with intermittent AC impedance, sample weight measurement and total organic carbon measurement of the test solution. On completion of the 180 day atlas cell test, the samples are dried and subsequently tested for reverse impact, flex and adhesion. The Phase 2 testing consists of analyzing various mechanical properties including cathodic disbondment, dielectric strength, mandrel bend, radiation resistance, direct impact and abrasion resistance.

Lessons Learned

Lessons learned as a result of the Millstone service water lining experience include:

- Delamination failure mode is plausible. A two foot by four foot sheet of delaminated ARCOR coating was retrieved from the MP3 SW system. Previously, coating suppliers and industry experts assumed that epoxy coatings in immersion service would fail in pieces too small to block safety-related equipment. Although the cause of the intercoat delamanination could not be determined, or replicated in the lab, failure mode assumptions should be revisited.

- Ownership of the safety-related coatings program must be defined and assigned by plant management to ensure accountability;

TABLE 1: Proposed Material Qualification Testing

Phase 1 Tests

Description	FTIR	Permeability	Atlas Cell Air-to-Water	Atlas Cell Water-to-Water	AC Impedance Air-to-Water	AC Impedance Water-to-Water	Panel Weight	TOC	Reverse Impact	Flex	Adhesion
Liquid-Grade 24 mils		X									
Paste-Grade 24 mils		X									
Liquid-Grade											
Paste-Grade											
Liquid-Grade 10 mils											
Paste-Grade 10 mils											
Thin Film 24 mils	X		X	X	X	X	X		X	X	X
Thick Film 200 mils	X		X	X	X	X	X		X	X	X
Each Test Cell Solution								X			
Fluid-Grade Elastomeric	X	X									
Paste-Grade Elastomeric (1)	X	X									
Gun-Grade Elastomeric	X	X									

(1) If different from Gun-Grade material.

TABLE 2: Proposed Material Qualification Testing

Phase 2 Tests

Description	Cathodic Disbondment	Dielectric Strength	Mandrel Bend	Radiation Resistance	Direct Impact	Abrasion Resistance
Liquid-Grade 24 mils	X					
Paste-Grade 24 mils	X					
Liquid-Grade		X				X
Paste-Grade		X				X
Liquid-Grade 10 mils			X			
Paste-Grade 10 mils			X			
Thin Film 24 mils				X	X	
Thick Film 200 mils				X	X	
Each Test Cell Solution						
Fluid-Grade Elastomeric	X			X		
Paste-Grade Elastomeric (1)	X			X		
Gun-Grade Elastomeric	X			X		

(1) If different from Gun-Grade material.

- Procedural control of coating installation is critical to ensuring proper surface preparation, contamination detection/removal, application and quality control;

- Applicators should be certified for application of a specific coating system in a safety-related situation;

- Safety-related coating suppliers should provide active technical customer support during installation and on a continuing basis to evaluate the performance of coatings in safety-related application.

- A comprehensive immersion coating/lining test standard currently does not exist for evaluating suitability of commercial products. The suitability testing standards referenced in EPRI's Guidelines on Nuclear Safety-Related Coatings are a good start. Utilities should consider joint development of an industry-wide test standard that contains specific protocols and works toward establishing acceptance criteria. Development of such a standard would provide a consistent industry-wide licensing basis for lining material suitability.

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