

# An Argument for Judicious Chemical Cleaning: Effect of Sulphide Surface Contaminant Removal on Coating Performance

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Worldwide, many thin - and thick - film polycyclamine-cured epoxy linings have performed admirably in the oil industry for high-temperature protection of tank and vessel internals. Notwithstanding the ever-increasing temperatures and pressure and chemical resistance requirements in oil and gas environments, the demands placed upon linings are becoming more stringent.

This article investigates whether the performance of these linings used in maintenance works could be enhanced by first abrasive blasting the steel substrate and then providing a subsequent application (and removal) of a unique chemical reagent to remove deleterious sulphide contaminants, thus improving lining performance in aggressive immersion service conditions, and potentially extending the life-cycles of the applied linings.

Accelerated laboratory investigations were carried out on a set of reagent treated, and untreated, carbon steel test panels. Sets of panels were lined with a three coat thin film solvent-borne epoxy novolac coating or a single coat solvent-free, thick film polycyclamine cured epoxy.

Proper surface preparation prior to the application of lining systems is fundamentally crucial to long-term coating success. Carbon steel that has been abrasive blasted to an SSPC-SP 5/ NACE No. 1 (Sa1) "White Metal Blast Cleaning" standard is the benchmark for lining performance when it is ensured that soluble chlorides and sulphates are kept below threshold levels where they would initiate lining failure. Predictors of successful, or failure-prone, lining applications in the real world include the influences of surface profile, peak height and peak count density, and cleanliness of the steel substrate. Importantly, from a surface profile and morphology viewpoint, a deeper profile with a greater surface area is required for these linings<sup>1-4</sup>. Furthermore, in earlier investigations the authors contended that with single-coat and solvent-free thick-film lining applications, the peak height of the profile should preferably be 3-to-4 mils (75-100 microns) and have a jagged pattern (peak height, peak density and coating rheology are all important factors)<sup>1</sup>. From a surface cleanliness viewpoint, much has been discussed in the literature about the deleterious effects on lining performance by non-visible contaminants, notably soluble salts<sup>5-8</sup>. With an industry emphasis on an understanding of the effect of anions such as chlorides, sulphates and more recently nitrates (for example flash rusting, osmosis and blistering of linings), the effect of cations and insoluble sulphides (e.g., iron and manganese), has received comparatively scant attention.

Iron sulphide species are an ever-present reality in the oil and gas, mining and wastewater industries. Given that iron sulphide is cathodic to steel, and often the scourge of many industrial processes, this investigation was carried out to evaluate lining performance on carbon steel that had been intentionally contaminated by iron sulphide. Iron sulphide may form a thin adherent protective layer on steel surfaces, or a thick, porous detached layer depending upon the pH, H<sub>2</sub>S concentration, temperature, flow rate and pressure<sup>9</sup>. When iron sulphide-fouled surfaces are intended to be coated, it is imperative that any residual iron sulphide or other contaminants are removed. Any deposits remaining would act as both a barrier between the coating and substrate that may affect coating adhesion and become a possible corrosion initiation site due to a cathode-anode reaction when sufficient permeation of an aggressive solution through the coating occurs.

Previous autoclave and EIS studies carried out by the authors on coated panels subjected to 149 C in acidic gases (10% H<sub>2</sub>S, 10% CO<sub>2</sub>, 80% CH<sub>4</sub>), 5% aqueous sodium chloride solution, and sweet or sour crude oil, had indicated that a proprietary post-abrasive blast, water-based cleaner and surface decontamination

process appeared to show considerable merit<sup>10</sup>. However, studies reported elsewhere on soluble salt removal from steel and coating performance concluded that the same cleaner and decontamination process (using tap water and not deionized water) had neither a positive nor negative effect on the performance of ten atmospheric coating systems or on four internal lining systems<sup>11, 12</sup>.

The primary interest of the present investigations centred on (a) the efficacy of the post-blast chemical cleaning treatment to remove insoluble iron sulphide contaminants and the difference in lining performance with or without the chemical treatment, and (b) comparing and contrasting the metal surface after abrasive blasting and post-blast chemical treatment.

The cleaner and decontamination procedure was claimed to remove both soluble salts, and *insoluble sulphides* (authors' emphasis), carbonates and oxides, and produce a residue-free and so-called passivated iron layer on the steel substrate. Furthermore, it appeared to have improved the performance of a thin-film epoxy novolac tank lining system applied to an SSPC-SP 5<sup>10</sup>. The epoxy novolac lining blistered under the test conditions on a white metal surface that had not received the chemical treatment. In contrast, the same epoxy novolac lining did not blister after having been applied to the white metal surface post treated with the surface cleaner decontamination procedure. However some questions remained,

1. Were the results repeatable and therefore, other things being equal, able to genuinely demonstrate the efficacy of the post-abrasive-blast applied, water-based cleaner to enhance the adhesion and performance of the lining system?
2. Did the cleaner and decontamination process serve as an effective, complementary and arguably necessary treatment to abrasive blasting? Did they decontaminate the surface and remove non-visible and insoluble iron sulphide contaminants?
3. Did the cleaner "passivate steel" as reported in the literature?<sup>13</sup>
4. Was the steel surface modified by the cleaner in some unknown way?

The answers are given below.

## Test procedure

### Surface decontamination

The proprietary two-step decontamination process was stated to remove deleterious soluble salts, flash rust and insoluble metal sulphides from carbon steel substrates. This process was said to be accomplished first by application of a proprietary acidic cleaner, then followed by the application of an alkaline wash to neutralise the substrate prior to a coating application.

## Coatings

Based on earlier studies, a thin-film multi-coat solvent-borne epoxy novolac and a thick-film single-coat solvent-free polycyclamine-cured hybrid epoxy coating were chosen for the present investigations. Each lining is known to be well-suited to the oil and gas industry for new construction as well as maintenance and repair projects for tanks, vessels and pipe spools.

The solvent-borne, thin-film epoxy novolac (Coating 1) was applied in three-coats to achieve approximately 12 - 15 mils (305 - 380 microns) total dry-film thickness (DFT). This coating has an extensive worldwide track record in the lining of tanks and vessels in the oil and gas industry. It possesses excellent hydrolytic, thermal (up to 121C) and chemical resistance, and cures at temperatures as low as 10C.

Coating 2 was a new-generation, rapid-cure, solvent-free epoxy lining with a longer pot life than most rapid-curing, single-coat solvent-free epoxy linings, and it was applied in one coat at 16-25 mils (406 – 635 microns) by single-leg spray. As with Coating 1, it possesses excellent hydrolytic, thermal (up to 149C) and chemical resistance, and cures at temperatures as low as 5C.

## Lining Applications

Both coatings were applied in accordance with the lining manufacturer's instructions to the three sets of panels.

## Test methods for coating evaluation

### Autoclave

The primary screening test most commonly employed by facility owners in the oil industry for tank and vessel linings is NACE TM0185, "Evaluation of Internal Plastic Coatings for Corrosion Control of Tubular Goods by Autoclave Testing."<sup>14</sup> The test environment consists of three phases: a gas phase mixture of 10% H<sub>2</sub>S, 10% CO<sub>2</sub>, 80% CH<sub>4</sub>, a hydrocarbon phase of sour crude, and an aqueous phase of a 5% NaCl solution. The tests were conducted at 149 C and at a total pressure of 250 psig.

### Electrochemical Impedance Spectroscopy (EIS)

EIS was used as a diagnostic tool to compare barrier properties before and after autoclave exposure. Essentially, the low frequency impedance is related to the permeability of the coating to water, organic molecules, and small gaseous molecules such as H<sub>2</sub>S and CO<sub>2</sub>. The log Z value, where Z is impedance at 0.01 Hz is typically used as the basis for comparison of a coating's barrier properties<sup>15, 16</sup>. The higher the impedance of a coating, the higher barrier properties it has, thus the more protective the coating is. A basic rule of thumb is that the barrier performance of a coating is excellent, good or marginal when log Z values are on the order of 10, 8 and 6, respectively.

### Adhesion and Visual Rating

After the coated panels were removed from the autoclave they were evaluated visually for any defects such as blistering per ASTM D714 or cracking. The coatings' adhesion was also assessed per ASTM D6677 and the DFT was measured.

## Measurements on Bare Steel Before lining Application

### Surface Profile Measurements

Surface profile measurements were performed using high-temperature, extra-course replica tape. Three measurements were taken on each sample. No surface profile measurements were carried out on panels that had heavy black iron sulphide deposits.

### Conductivity Measurements

Total dissolved salts (TDS) conductivity was carried out in accordance with SSPC Guide<sup>15,16</sup>. A 500ml liquid sample was taken from each steel panel using the laboratory boiling extraction method.

### SEM-EDX/ X-Ray Diffraction

SEM can be used for the examination of surfaces and EDX detects elemental composition. One limitation of this analysis is that it will not determine the compounds present, only the elements present. Hence, X-ray diffraction was used to further characterize the crystallographic phase of compounds present on the surface. Analyses were performed on the surface of test panels in Sets B and C.

## Results and discussion

The results of the autoclave testing of Coating 1 and Coating 2 at 149 C in sour crude are summarised in Tables 1 and 2, in which 1A refers to the front side of a given panel from the A panel set, and 2A refers to the back side of the same panel. The same nomenclature applies to panels B and C.

Post-autoclave test panels are shown in Figures 2 and 3. A coating was deemed to have failed in the autoclave test if it blistered, cracked or flaked in any of the three phases. Adhesion ratings of the coating films were considered of lower importance than the presence, or absence, of film blistering. Coatings may provide sufficient adhesion and sub-film corrosion resistance even when water has been permeated and reached the coating/metal interface.

## Experimentation

### Preparation and Treatment of Steel Panel

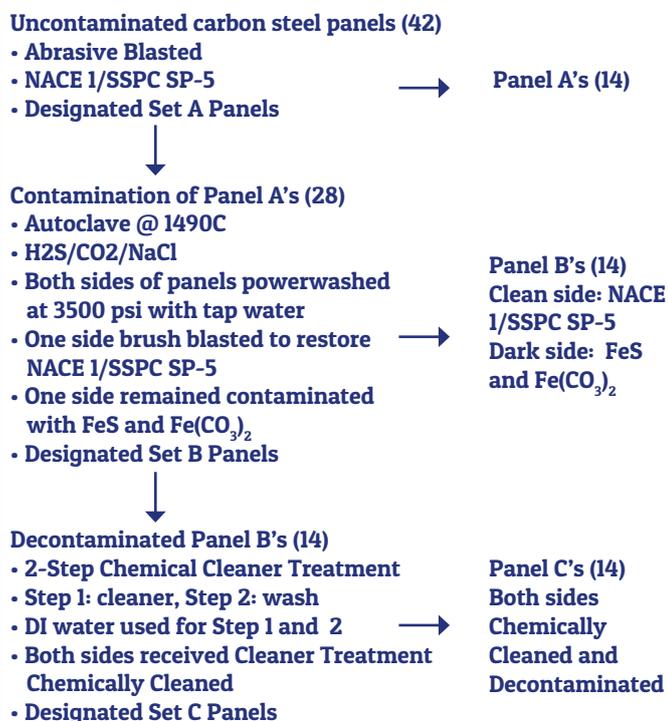


Figure 1. A schematic outline of the preparation of the 42 carbon steel (4.5 x 1.5 x 11/8 inch) test panels.

### Uncontaminated carbon steel panels

Using staurolite 20/40 abrasive the panels were abrasive blasted to SSPC-SP 5/NACE No. 1 white metal blast to achieve a sharp angular profile in the range of 2.5-to-4 mils (63.5 - 100 microns). Fourteen of these panels were set aside and designated as Set A.

### Deliberately contaminated carbon steel panels

The remaining 28 bare panels were deliberately contaminated by exposing them to a gaseous phase of 10% H<sub>2</sub>S, 10% CO<sub>2</sub>, 80% CH<sub>4</sub> and an aqueous phase of 5% NaCl, in an autoclave for four days at 149C and 250 psig. This contamination procedure was undertaken to ensure that a significant amount of black iron sulphide was formed on the steel panels and that the steel was also contaminated with iron carbonate and chlorides.

After taking conductivity measurements, the 28 deliberately contaminated panels were then pressure washed on both sides at 3,500 psi with tap water. One side of the panels was then abrasive swept to restore the clean and original SSPC-SP 5/NACE No. 1 condition and the other side left alone and which turned dark over time. Fourteen of these panels were set aside and designated as Set B.

### Decontaminated carbon steel panels

Both sides of the remaining 14 panels were designated as Set C, and treated with the cleaner and surface decontamination gel almost two weeks later.

The test panels were treated by exposing the entire panel to the acidic gel cleaner in a ziplock bag for 30 minutes. These panels turned the gel dark grey. The panels were then power washed on both sides with a 1% solution of alkaline rinsing aid in deionised water using a low-pressure, 4,000 psi pressure washer. The panels were left exposed under indoor ambient conditions.

**Table 1 – Coating 1 Autoclave Analysis**

149°C, 250 psig, 10% H <sub>2</sub> S, 10% CO <sub>2</sub> , 80% CH <sub>4</sub> , Sour Crude, 5% NaCl in Distilled Water, 96hrs												
Coating 1	Avg. DFT	Adhesion Rating (ASTM D6677)				Blistering (ASTM D714)			Impedance (log Z @ 0.1 Hz)			
		Pre-Test	Water	HC	Gas	Water	HC	Gas	Pre-Test	Water	HC	Gas
1A	13.0	8	10	10	8	N	N	N	10.29	7.85	9.99	10.08
2A	14.3		10	10	8	N	N	N		7.74	10.05	10.33
1B	14.0	8	10	8	8	N	N	N	10.10	7.79	10.33	10.09
2B	13.8		8	8	8	F#2	N	N		7.63	10.60	10.75
1C	16.4	10	10	10	8	N	N	N	10.39	7.91	10.78	11.01
2C	15.0		10	10	8	N	N	N		6.61	10.10	10.71

Notes for Table 1 and 2:

1. DFTs were measured for each coating and each phase. Average DFT reported;
2. HC = hydrocarbon;
3. Panels A – Applied to NACE 1/SSPC SP-5 abrasive blasted steel;
4. Panels B – Applied to panels above which were then deliberately contaminated with gaseous H<sub>2</sub>S (10%) and NaCl solution (5%), washed with tap water, and brush blasted on one side;
5. Panels C – Applied to Panels B treated with the chemical cleaner.

**Table 2 – Coating 2 Autoclave Analysis**

149°C, 250 psig, 10% H <sub>2</sub> S, 10% CO <sub>2</sub> , 80% CH <sub>4</sub> , Sour Crude, 5% NaCl in Distilled Water, 96hrs												
Coating 2	Avg. DFT	Adhesion Rating (ASTM D6677)				Blistering (ASTM D714)			Impedance (log Z @ 0.1 Hz)			
		(ASTM D6677)	Blistering	HC	Gas	Water	HC	Gas	Pre-Test	Water	HC	Gas
1A	21.1	8	10	10	6	N	N	N	9.96	7.07	10.68	10.49
2A	21.0		10	10	6	N	N	N		6.98	7.19	6.98
1B	24.5	8	8	4	6	N	N	N	10.04	7.44	10.57	10.28
2B	24.5		6	4	4	N	N	N		7.50	10.29	10.58
1C	16.7	8	10	10	8	N	N	N	10.28	6.54	10.05	10.01
2C	17.4		10	10	10	N	N	N		7.45	10.11	10.60



Figure 2, Coating 1 panels, post-autoclave.



Figure 3, Coating 2 panels, post-autoclave.

The solvent-borne Coating 1 showed no blistering in all phases regardless of panel type, with the exception for panel 2B which showed F2-sized blisters in the water phase. Coating 1 maintained or in many cases improved its adhesion compared to its pre-test value. The coating showed excellent pre-test impedance and maintained high log Z of greater than 10 values post-test in the gas and hydrocarbon phases. However, these values decreased in the water phase to log Z of 7-to-8 indicative of the more aggressive water phase.

Inspection of the EIS values showed that the performance of Coating 1 slightly improved in the gas phase on chemically treated Set C panel surfaces compared to Set A and Set B panel surfaces.

In earlier studies, the performance of Coating 1 at 149C was excellent when applied to a NACE 1/SSPC SP-5 white metal blasted surface that had been post treated with the chemical cleaner and subject to a surface decontamination process; the performance of Coating 1 at 149 C was very poor when applied to a surface prepared to a NACE 1/SSPC SP-5 standard. In the present study, however, the performance of Coating 1 under similar conditions was very good on both types of surfaces. This result indicates that a certain inconsistency of coating performance can arise on steel that has been prepared to a white metal standard alone. This is not surprising since the appearance of a white metal surface does not signify the absence of residual soluble or insoluble moieties, nor detrimental materials.

The solvent-free Coating 2 had as high pre-test impedance values as did Coating 1 and showed similar post-test impedance behaviour. With Coating 2 on white metal Set A panel surfaces there was an inconsistency of EIS values not seen on the chemically cleaned Set C panels. Adhesion of Coating 2 on Set B panels decreased significantly in all phases when compared to panels from sets A and C which maintained, or slightly changed, vis-à-vis adhesion rating. Overall, the adhesion ratings of Coating 2 were lower than Coating 1 indicating superior wetting out of the solvent-borne Coating 1. Interestingly, the adhesion ratings for Coating 2 improved significantly in the gas phase on the chemically prepared Set C panel surfaces.

The adhesion of Coating 2 was markedly improved on Set C panel surfaces versus Set B panel surfaces. This showed that the chemical cleaner had performed well in achieving a contaminant-free surface, removing both soluble and insoluble moieties. In the case of Set C panels with the chemical cleaner treatment, as evidenced by the log Z values, the excellent

barrier performance was consistent on both panel sides (Table 2). However, in the case of Set A panels with only the SSPC-SP 5/NACE No. 1 abrasive blast, there was good barrier performance on one side of the panel but poor performance on the other side. Hence, as seen in our earlier studies, the efficacy of the chemical treatment appears to lead to an increasing consistency of superior performance when using the cleaner as a complementary (and to re-iterate, arguably necessary) treatment for the abrasive blast-cleaning prior to coating application.

The chemical treatment improved the adhesion of the solvent-free Coating 2 on decontaminated steel, compared to the Coating 2 adhesion on either SSPC-SP 5/NACE No. 1 white metal or contaminated, power washed and white metal surfaces that were subsequently brush-blasted (Table 2).

The results of the conductivity and surface profile measurements are shown in Table 3. The surface profile of two panels that were abrasive blasted to an SSPC-SP 5/NACE No. 1 white metal standard averaged 2.63 mils (67 micron) for the first panel, and 2.50 mils (63.5 micron) for the second panel. In marked contrast, the surface profile of two panels that were abrasive blasted to SSPC-SP 5/NACE No. 1 white metal, contaminated in the autoclave prior to a low-pressure water wash at 3,500 psi, and then given the chemical cleaner treatment, averaged 3.63 mils (92 micron) for the first panel and 3.53 mils (90 micron) for the second panel. This increase in profile depth occurred as a result of the corrosion of the steel in the autoclave since, in separate work, the cleaner itself was shown not to increase the profile depth on bare steel panels abrasive blasted to SSPC-SP 5/NACE No. 1. Application of the cleaner and its subsequent neutralisation and removal with low-pressure washing removed all abrasive media embedded on a panel from Set C (whereas the abrasive-blasted white metal panel from Set A was full of embedment). The removal of embedded detrital material was not achieved by pressure washing alone.

An uncontaminated panel from Set A and a chemically treated panel from Set C both had conductivity measurements of zero. The unwashed and deliberately contaminated panel had a conductivity of 14  $\mu\text{S}/\text{cm}$ . The deliberately contaminated panel that was low-pressure washed at 3,500 psi with tap water had a conductivity of 32  $\mu\text{S}/\text{cm}$ . (This tap water was later determined to have a conductivity of 348  $\mu\text{S}/\text{cm}$ ). It is important to note that the manufacturer stipulates that to achieve proper chemical decontamination only deionized water should be used. The efficacy of this two-step cleaning process is not based on the application of corrosion inhibitors where inhibitor moieties are intentionally left on the steel surface and can affect the performance of the coating. Rather, this system is designed to clean the steel surface without leaving any chemicals which can interfere with the coating performance or participate in corrosion.

## SEM-EDX

Bulk surface analysis performed on un-coated panels (from Set A) with EDX indicated the presence of iron, oxygen and

carbon with aluminium, silicon and titanium, suspected oxides from the embedded blasting media (Figure 4).

Residual oxides and sulphides were present on a panel from Set B, especially the back surface which also exhibited a dark surface. The amount of surface oxide and sulphide constituents was greatest on this sample (Figure 5).

The post abrasive-blast, water-washed and chemically treated surface of a panel from Set C was extremely clean and possessed greater angularity and profile depth compared to the blasted and water-washed surface of panels from Set B (Figure 6). This profile difference is a direct result of the corrosion of the abrasive-blasted steel in autoclave conditions and not the cleaner treatment itself.

The results from XRD confirm the findings of the EDX analysis. The chemically cleaned pewter-coloured panel shows significant cleaning with negligible surface film, no sulphide deposits, no carbonate deposits or any apparent oxide formation. Both EDX and XRD penetrate the surface somewhat, and there is a possibility that a very thin surface film may be present that is not detectible using these techniques. It was speculated that the surface is not passivated per se, in terms of a protective oxide layer, but to be in a passive state given that it appears to essentially consist of pure iron.

Also of significance, is that the back surface of panel B (a side not reblasted) which exhibited a large amount of surface contamination, was essentially a bare steel substrate after the chemical cleaning process. The surface was approximately 97% iron after cleaning, with small concentrations of other crystallographic constituents present.

## Conclusions

The lining performance on steel panels subject to the chemical cleaner decontamination procedure after first abrasive blasting to an SSPC-SP 5/NACE No. 1 white metal standard is equal to (or marginally superior in the case of the solvent-free polyclamine-cured hybrid epoxy coating) the performance observed on steel prepared to a white metal standard.

The chemical treatment afforded discernible performance increments in terms of adhesion for the increasingly popular solvent-free coating, Coating 2.

Based on conductivity measurements, abrasive blasting to a white metal standard showed no difference between coated panels tested with or without the chemical decontamination procedure. The efficacy of the proprietary cleaner used as a means to decontaminate abrasive blasted steel was clearly demonstrated in the SEM-EDX and XRD studies. The cleaner was not deleterious to the carbon steel substrate even during extended surface contact of the cleaner with steel as tested. It readily removed ample amounts of insoluble iron sulphide species, iron carbonate and iron oxide from the substrate.

Based on the authors' current and previous studies the performance of the linings on post chemically treated abrasive blasted steel surfaces has been shown to be more consistent and

**Table 3 – Conductivity and Surface Profile Measurements of Test Panels Pre-Coating Application**

Test Panel from Set:	General Characteristics and Comments	Total Dissolved Salts (TDS)	Average Surface Profile (mils)
<b>A: no contamination abrasive blast</b>	<b>NACE 1/SSPC SP-5 white metal blast standard</b>	<b>0 <math>\mu\text{S}/\text{cm}</math></b>	<b>2.63 (1st panel) 2.50 (2nd panel)</b>
<b>B: deliberate contamination, washed*, not re-swept</b>	<b>Black surface: uniform rusting throughout (FeS/iron oxide)</b>	<b>32 <math>\mu\text{S}/\text{cm}</math></b>	<b>N/A</b>
<b>B: deliberate contamination unwashed, not re-swept</b>	<b>Black surface: uniform rusting throughout (FeS/iron oxide)</b>	<b>14 <math>\mu\text{S}/\text{cm}</math></b>	<b>N/A</b>
<b>C: chemical treatment to a Panel B that was washed</b>	<b>NACE 1/SSPC SP-5 white metal blast standard</b>	<b>0 <math>\mu\text{S}/\text{cm}</math></b>	<b>3.63 (1st panel) 3.53 (2nd panel)</b>

Table 4 – Summary of XRD Results (wt%)

Sample ID	Iron	Silicon Oxide	Aluminum Oxide	Iron Carbonate	Iron Sulphide	Zinc Sulphide	Calcium Carbonate	Iron Phosphide	Iron Oxide	Magnesium Hydroxide
Sample B	94.0	1.0	4.3	--	--	--	--	---	--	0.7
Sample B2	2.3	1.4	--	53.5	36.4*	4.1	--	---	1.5	---
Sample C	99.4	0.1	--	--	--	--	0.1	0.3	--	0.1
Sample C2	96.6	0.3	--	0.8	1.3	--	0.4	0.4	*0.2	--

FeS consists of Troilite (33.2%), Rudashevskyite (3.2%), and Mackinawite (0.8%)

Figure 4

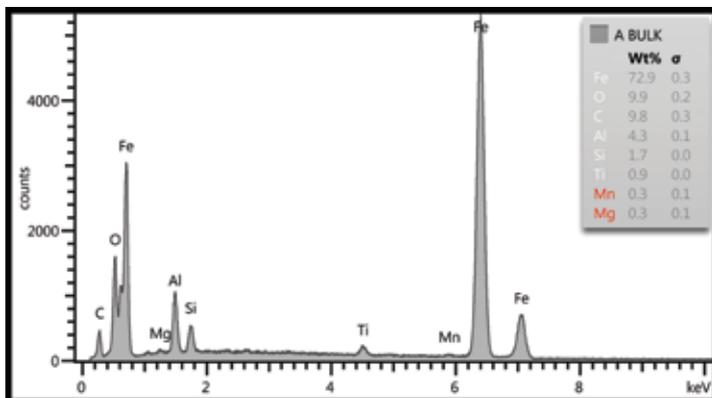


Figure 5

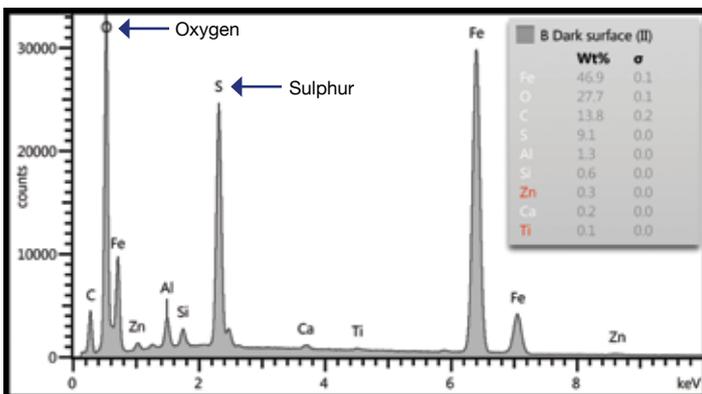
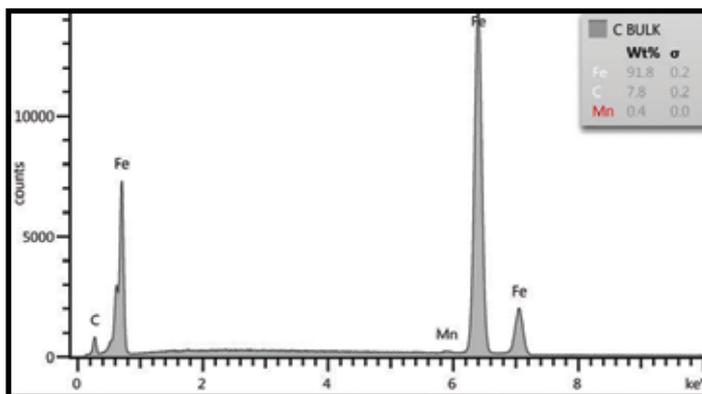


Figure 6



Bare steel: Figure 4 Panel A – Gold Standard for 70 yrs SSPC-SP5/NACE No.1, Figure 5 Panel B – Panel A contaminated, pressure washed, reblasted, Figure 6 Panel C – Panel B chemically cleaned, not reblasted.

predictable than that on the industry benchmark of a white metal blast. In addition, under the conditions tried, the chemical treatment proved as-good-as or better for lining performance even when challenged with contaminated surfaces.

To the answers to questions posed at the outset of this study. First, some of the autoclave, EIS and adhesion trends seen in previous studies were not repeated in this work. The cleaner did demonstrate an advantage using the post-abrasive blast applied treatment to enhance the adhesion of the solvent-free system, but not the solvent-borne system. Second, the cleaner decontaminated the carbon steel surface and removed both non-visible (and ample amounts of visible) iron sulphide, iron carbonate, iron oxides and soluble salt contaminants. Third, the cleaner afforded a passive state surface (perhaps as opposed to passivation vis-à-vis an oxide layer) of iron alone on the carbon steel. Fourth, the cleaner did not show any evidence of etching the steel surface.

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