IGS

Crude Distillation Unit Column Overhead Corrosion Protection

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Contents

Abstract

Repair and outage costs of refinery Crude Distillation Units (CDUs) can be significant, involving the complexity of shell replacement or weld overlay and associated PWHT. High-Velocity Thermal Spray (HVTS) cladding technology has a well-documented history of shell protection in sour conditions where low, or locally low, pH results in material wastage. These modified superalloy systems are resistant to both caustic and acidic conditions at high temperatures. They are effective in providing an impermeable metallurgical barrier, protecting the underlying substrate,and can be efficiently applied without impact to the existing shell or the need for post thermalprocessing. However, unlike many service vessels found in upstream and refining, CDUs presentunique challenges due to graduated temperature and corrosivity profiles, as well as the uniquechemical characteristics of the feed, which can vary throughout the column. Extensive testing was undertaken in corrosion autoclaves using raw crude oil obtained from functioning wells. Thesematerials were characterized and enhanced in terms of corrosivity to mirror more aggressive feeds. Distinct corrosion and test environments were created to represent the differential CDU conditions. This paper discusses the performance of modified HVTS systems for corrosion protection in the varied environment of a typical CDU column.

Key words:
High Velocity Thermal Spray, Crude Distillation Unit, Overhead Corrosion, Naphthenic Acid, Ammonium Chloride, Amine Corrosion

Introduction

Crude Distillation Units (CDUs) are mission critical units and often the primary bottleneck in refinery output and outage timelines. At their most basic, CDUs separate raw crude into useful fractions based on boiling point; these distillates are then further processed into refined products such as jet fuel, gasoline, kerosene, etc.[1] Following arrival from well sites, raw crude oil is typically pre-heated and desalted to remove water and inorganic salts; this reduces the corrosivity of crudes, however, many corrosives such as organic chlorides, naphthenic acids, and sulfides can remain.[2,3,4] Desalted crude is then heated to approximately 350°C to 400°C and fed into to the primary CDU column, where specific fractions condense based on boiling point and flow out on condensation trays. Naphtha and light vapors travel to the top of the column before being drawn off via overhead lines.[1,4]

Crude Distillation Units are indispensable to refinery operation; however, they are subject to corrosion attack from a variety of sources due to varied regional feeds and conditions. Raw crude oil, especially "opportunity crudes" sourced from lower quality wells, is extremely corrosive due to elevated acid, salt, and sulfur content. Rapidly changing crude sources can result in an everchanging and varied regional corrosion environment.[6] Additionally, due to the vastly different temperature gradients and local fractionalized media, the sources and intensity of corrosion is variable throughout the column. Generally, however, there are two major sources of significant corrosion in CDUs: high temperature sulfidation and naphthenic acid corrosion on the column trays and walls, and overhead corrosion due to HCl condensation and amine/ammonia salts.[4,7]

Overhead Corrosion

CDU overhead corrosion occurs at the top of the column, the overhead lines, and surrounding infrastructure such as the accumulator and heat exchangers. This environment is characterized by lower temperatures ranging from approximately 130°C down to below 100°C, allowing for the condensation of corrosive hydrochloric acid and the corresponding formation and deposition of salts such as ammonium chloride (NH_4Cl) and amine hydrochlorides.[1,2] HCl is typically formed from the hydrolysis of chloride salts at temperatures above 125°C and crude chloride concentrations above 20 PPM.[8]

1 $NaCl + H_2O = NaOH + 2HCl$
2 $MgCl_2 + 2H_2O = Mg(OH)_2 + 2\ HCl$
3 $CaCl_2 + 2H_2O = Ca(OH)_2 + 2\ HCl$
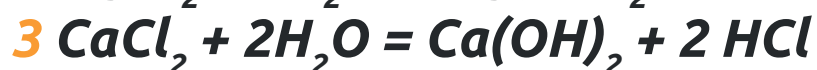
Once formed, HCl can undergo additional reactions with ammonia and amines to form ammonium chloride (NH_4Cl) and amine hydrochloride salts.

4 $Fe^2+ + 2Cl = FeCl_2$
5 $NH_3 + HCl = NH_4Cl$
6 $RNH_2 + HCl = RNH_3Cl$
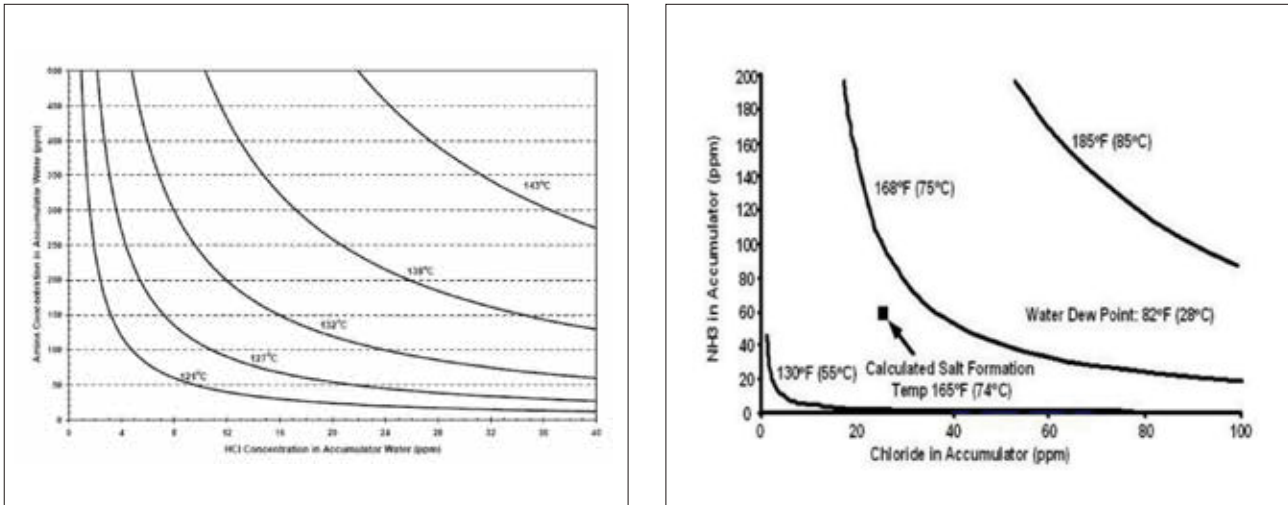**7** $RNH_3Cl + H_2O = RNH_3^+ + Cl$

Figure 1: Salt formation temperatures for amine salts (left) and NH$_4$Cl (right) [1]

Both ammonia and amines are common contaminants in crude oils, as well as hydrocarbon streams that are blended into crude before processing. NH$_3$ is also injected as a pH neutralizer in the CDU overhead, while "tramp amines" are the amines that are typically injected at crude production stage, as corrosion inhibitors, filmers, and H$_2$S scavengers. Both NH$_4$Cl and amine hydrochloride salts are extremely corrosive and hygroscopic, leading to severe localized corrosion in addition to deposition and plugging issues.

In a 2004 case study at the Irving Oil Refinery, a 300,000+ BPD refinery in Canada, routine UT monitoring revealed corrosion rates in the range of 0.5-1.0 mm / year (20 – 40 mpy) were occurring at the top of the #3 CDU; additional monitoring suggested that NH$_4$Cl salt deposits had formed and under deposit corrosion was occurring. An investigation revealed that, during winter months, the temperature of the header area was operating close to the formation temperatures for NH$_4$Cl and, combined with higher than recommended levels of HCl in the overhead drum water, led to deposition formation. Ultimately, the damage necessitated a full replacement of overhead infrastructure and major process changes at a significant monetary cost.[10]

Naphthenic Acid and Sulfur Species Corrosion

In contrast to overhead corrosion, naphthenic acid and sulfur species corrosion are high temperature phenomena, and typically occur in the lower to mid-column CDU area, resulting in wall thinning and corrosion on the condensation trays. Naphthenic acids (RCOOH) are complex groups of carboxylic acids that are naturally present in crude oils, ground water, and oil sands. These acids become corrosive on carbon steel around 180-220°C, then become increasingly corrosive as temperature increases, with a maximum corrosion occurring between 280 and 385°C. Above 400°C Naphthenic acids break down and no corrosion occurs. Generally, the naphthenic acid content is quantified in the crude oil TAN, with a TAN of greater than 0.5 indicating potential corrosivity.[6, 11, 12]
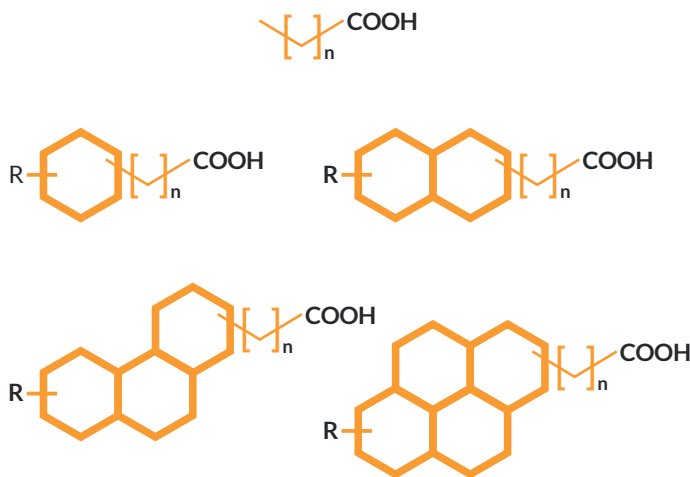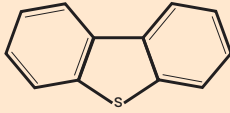
n-Cetyl mercaptan	$CH_3(CH_2)_{15}SH$
n-Decyl sulfide	$CH_3(CH_2)_9S(CH_2)_9CH_3$
Di-*n*-octyl disulfide	$CH_3(CH_2)7S-S(CH_2)7CH_3$
Dibenzothiophene	

Figure 2: Common naphthenic acid (left) and organosulfur (right) forms found in crude oil [13, 14]

3. Naphthenic and Sulfide Autoclave Testing

A corrosion autoclave was used to test coupons in a high temperature naphthenic acid and sulfidation environment. Enhanced crude oil was stored in a reservoir under atmospheric temperature and pressure and continually fed into a 700 ml stainless steel autoclave through a positive displacement peristaltic pump. A low volume nitrogen feed was used to prevent the formation of a vacuum in the oil reservoir, with an air lock used to release excess nitrogen and prevent a pressure build. A mixer was run continuously at approximately 45 RPM to prevent separation and the accumulation of heavy crude fractions such as tar in the vessel. Specialty autoclave test coupons were fixed to the shaft, ensuring they were always exposed to fresh media and flowing crude oil. Spent crude was collected in a waste reservoir, while vapors

$$8 \quad Fe + 2RCOOH \leftrightarrow Fe + (RCOO)_2 + H_2$$
$$9 \quad FE(RCOO)_2 + H_2S = FeS + 2$$
$$10 \quad Fe + H_2S = FeS$$
$$11 \quad FeS + 2RCOOH \rightarrow Fe(RCOO)_2 + H_2S$$

In one case study, a refinery was using alternating blends of crude oil with different TAN and sulfur levels. After a 5-year service period, severe corrosion of trays and an almost complete loss of column wall thickness was detected, with damage being especially pronounced on trays 24-25 (carbon steel) and tray 35 (410 SS). A failure investigation determined that light naphthenic acids with boiling points in the low 200°C range were being concentrated in the kerosene fractionalization trays (150 to 270°C), giving the distillate a high acidity (0.76 mg KOH/g), and corroding both carbon and stainless steels. Normally an iron and chromium sulfide scale could offer some protection, however alternating high sulfur crudes with high acid/low sulfur crudes led to destruction of the iron sulfide layer and exposed fresh metal to attack from both naphthenic acid and H$_2$S. [7]

High Velocity Thermal Spray Cladding Technology

To protect against CDU corrosion, novel High Velocity Thermal Spray (HVTS) systems were explored as a solution. HVTS is a corrosion mitigation technique where a corrosion resistant metal alloy is atomized and the droplets conveyed at high velocity to a substrate such as carbon steel. On impact they form flattened 'splats' and sequentially build up cladding thickness. This metal coating, commonly based on NiCrMo/Nb/W super-alloys with alloy modifiers specifically required for thermal spray processes, forms a corrosion resistant and mechanically superior barrier over the carbon steel substrate. Prior limitations of the technology have been addressed with alloy modification to eliminate inter-particle connected porosity and shut down any corrosion path to the base metal. Further alloy modification is required to enhance bond strength and reduce native as deposited stress state. Although material design is a key parameter in successful application; conveyance equipment, process controls, procedure qualification and technician certification are equally critical to providing large surface coverage and protection.
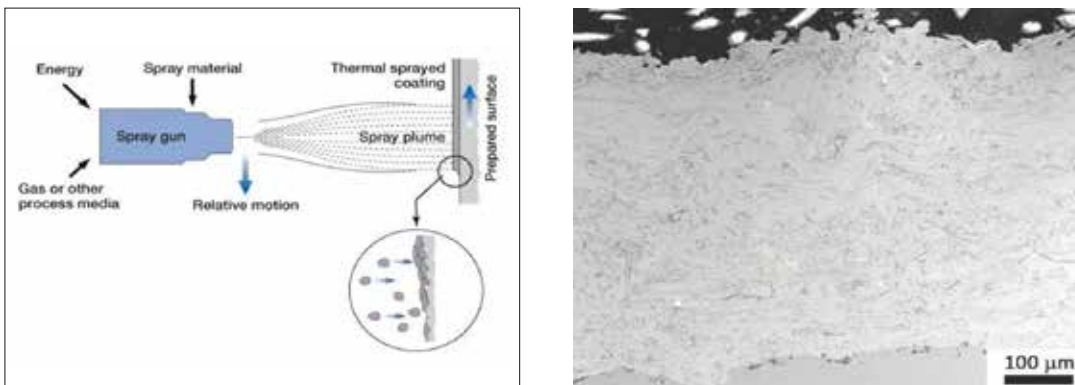

Figure 3: Example HVTS process diagram (left) and typical HVTS microstructure (right)

Experimental Procedure and Material

Experimentation focused on running a materially diverse set of HVTS protective claddings through aggressive testing designed to simulate both overhead CDU corrosion, with a focus on HCl, NH_4Cl, and amine salt corrosion, as well as high temperature sulfide and naphthenic acid corrosion due to corrosive regional feeds.

1. Cladding and Materials Tested

HVTS Systems were selected based on current NiCrMo-XX thermal spray cladding systems (designated 5450 and 5470) and a newly developed cladding technology called Metal Reinforced Polymer systems (MRP®500)+. This hybrid system employs a preformed porous titanium alloy cermet scaffolding, microfused to the substrate, and imbued with a tightly structured, high temperature polymeric system to provide improved corrosion resistance in a diverse range of environments. Control systems for the test included uncoated UNS G10180 carbon steel as well as an unmodified UNS N04400 thermal spray alloy used as a traditional weld overlay and thermal spray system.

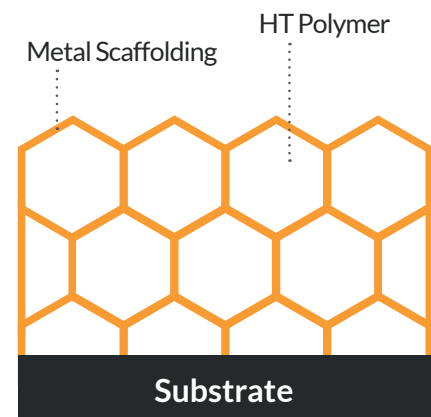| No'	System	Chemistry	Notes
1	Carbon Steel	Carbon Steel	Control system – no protective cladding
2	IGS 5315	UNS N04400 (Monel®400 (+) NiCu)	Traditionally used and applied alloy (unmodified)
3	IGS 5450	NiCrMoXX	Alloy modified HVTS system
4	IGS 5470	NiCrMoXX	Alloy modified HVTS system
5	MRP 500	TiXX	Titanium alloy cermet MRP system

Figure 4: Table 1 of test alloys (left) and a typical MRP idealized structure (right)

2. Media and Crude Oil Analysis

To simulate naphthenic acid and sulfur corrosion in a high temperature CDU environment, crude oil was used as a test media in a high temperature autoclave. A standard drum of blended Permian Basin crude was obtained from several wells in West Texas. To quantify the potential corrosivity of the media, a limited crude oil assay was performed.

Table 2 - Crude Oil Evaluation Testing

Test Performed	Standard	Notes
TAN Evaluation	ASTM D664	Acid Number of Petroleum Products by Potentiometric Titration
Water and Sediment in Crude (BS&W)	ASTM D4007	Water and Sediment in Crude Oil by the Centrifuge Method
Metals in Oil	ASTM D708	Determination of Nickel, Vanadium, and Iron in Crude Oils and Residual Fuels by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry
Total Sulfur in Petroleum Liquid	ASTM D4294	Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
Total Chlorine in Oil	ASTM D808	Chlorine in New and Used Petroleum Products

Following analysis, to increase the corrosivity of the crude oil test media to be representative of "worst case" opportunity crudes, pure naphthenic acid (CAS 1338-24-5) and a mixture of cetyl mercaptan and elemental sulfur were introduced to artificially raise the crude sulfur content and TAN level. Samples of doped crude were similarly assayed to verify the effectiveness of this technique, and a final formulation of >3.0% sulfur and > 2.0 mg KOH/g, was selected, in line with high acid sour crudes.

3. Naphthenic and Sulfide Autoclave Testing

A corrosion autoclave was used to test coupons in a high temperature naphthenic acid and sulfidation environment. Enhanced crude oil was stored in a reservoir under atmospheric temperature and pressure and continually fed into a 700 ml stainless steel autoclave through a positive displacement peristaltic pump. A low volume nitrogen feed was used to prevent the formation of a vacuum in the oil reservoir, with an air lock used to release excess nitrogen and prevent a pressure build. A mixer was run continuously at approximately 45 RPM to prevent separation and the accumulation of heavy crude fractions such as tar in the vessel. Specialty autoclave test coupons were fixed to the shaft, ensuring they were always exposed to fresh media and flowing crude oil. Spent crude was collected in a waste reservoir, while vapors were drawn off into a gas disposal system. Both feed and waste reservoirs were respectively filled and emptied multiple times throughout experimentation to maintain continuous flow. The system operated at a temperature of 300°C for two weeks for exposure time.
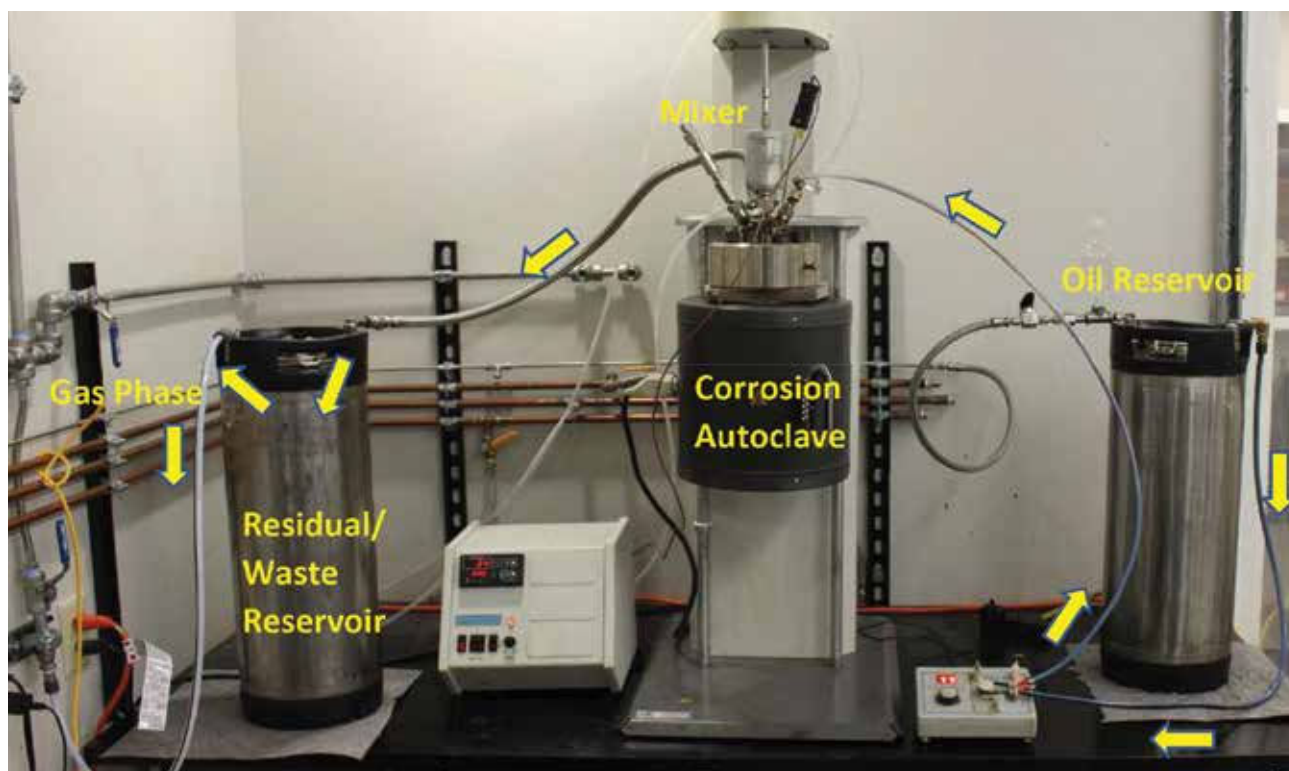

Figure 5: High temperature naphthenic acid and sulfidation autoclave setup

Test coupons consisted of a carbon steel insert welded into a NiCrMo, UNS N06625 sheath. This coupon design, intended for immersion and autoclave corrosion studies, allows for a carbon steel exposure test area while eliminating edge-effect issues that can lead to non-experimental coating failure on small coupons. Before HVTS coating, cleaned and profiled with a high specification aluminum oxide abrasive blast media. A profilometer was used to confirm that the carbon steel plates were profiled correctly before applying IGS thermal spray and other cladding systems. Coating thickness was confirmed using an electromagnetic standoff gauge. Control coupons were blasted with a low-profile garnet blast media to remove mill scale but not significantly increase surface area. Following exposure, coupons were cross-sectionally analyzed.

Table 3 - Autoclave Test Conditions

Pressure	Atmospheric
Temperature	300°C (572°F)
Exposure Time	High Acid Sour Crude
Flow Rate	300 ml/hr
Vessel Size	700 ml

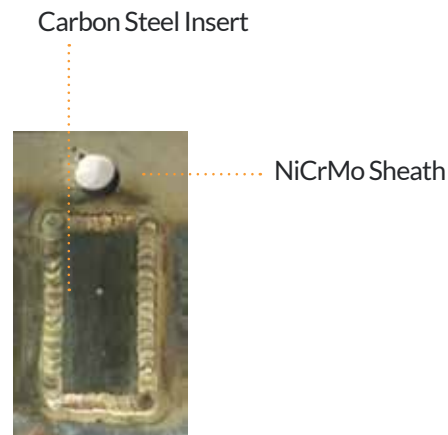Carbon Steel Insert

NiCrMo Sheath

Figure 6: Table 3 of autoclave test conditions (left) and standard IGS Immersion Coupon (right)

To prove efficacy of the setup and obtain a flowrate calibration for the peristaltic pump, the system was gradually brought up to the test temperature of 300°C. Waste samples were collected and measured for API and density for different feed rates. A linear trend of API gravity vs flow rates was seen, with all tests having lower APIs (higher densities) compared to the initial crude feed; this indicates that crude oil was successfully fractionalized and accurately simulated the processes occurring in a CDU. After the vessel was opened, no build-up of tar or condensed material was seen, indicating that fresh media was successfully displacing spent crude, and keeping levels of corrosive media high

Table 4 - Autoclave Flow Rates vs. Crude Output API

Condition	Flowrate (ml/hr)	API Gravity
As Received	-	30.2
1	A300	<10
2	700	4.2
3	1000	16.3
4	1300	28.9

Test Conditions

4. Overhead Corrosion – Exposure Cell Testing

Evaluation of the overhead CDU corrosion environment, which takes place at lower temperatures and is primarily HCl and salt based, was performed using exposure cells. The test cell solution was a deionized water mixture of 1% HCl (resulting in a room temperature solution pH of 0.8), 2.5% NH_4Cl, and 2.5% Methylamine Hydrochloride (CH_3NH_2HCl), a corrosive amine salt used to represent the range of amine salts that can form. The cells were kept in a furnace at 95°C. Test coupons consisted of 2" x 4" carbon steel panels coated with the MRP and HVTS protective claddings. As with the autoclave coupons, all panels were cleaned and profiled with a high specification aluminum oxide abrasive blast media. Coating thickness was confirmed using an electromagnetic standoff gauge. Control coupons were blasted with a garnet blast media.

Table 5 - Exposure cell test conditions

Pressure	Atmospheric
Temperature	95°C (200°F)
Exposure Time	2 Weeks
Test Solution	94% DI Water 1% HCl 2.5% NH4Cl 2.5% CH3NH2HCl

Figure 7: Table 5 of exposure cell test conditions (left) and standard exposure cell setup (right)

5. Coupon Analysis Procedure

On completion of the exposure testing, both the exposure cell coupons and autoclave test coupons were photographed, visually examined, and cut into cross-sections for metallographic evaluation. High precision cutting instruments and diamond blade saws, with cuts rotating into the coating, were used to minimize preparation damage to the cermet systems. Samples were then mounted in a clear, thermoset epoxy, ground, and polished using 180 to 1200 grit silicon carbide paper along with 3 um and 1 um diamond suspensions to create an accurate image of the coating microstructure without the presence of pullout or preparation defects. After preparation, test panels were cross-sectioned and analyzed using a Hitachi 3600N SEM operating at a 30 kV excitation voltage and equipped with Energy-dispersive X-ray spectroscopy (EDS) for elemental analysis.

Results

1. Crude Oil Evaluation

A partial assay was performed on the crude oil test media to ascertain the base corrosivity and properties of the oil. This included ASTM compliant testing for Acid Number (TAN), Metals content, Basic Sediment and Water (BS&W), Total Sulfur + Chlorine, and API gravity. The Permian basin crude blend had an API of 30.2 and sulfur content of 0.54 % wt., classifying it as a medium and slightly sour crude grade. A TAN of 0.75 mg KOH/g was reported as well as a chlorine content of 1372 mg/kg (ppm), classifying this as a moderately acidic crude oil. Metal content of the crude is within expected values, though higher levels of calcium (48mg/kg) and vanadium (2.8 mg/kg) do present a slight risk for eutectic salt or scale formation. Ultimately, this blend would likely be considered moderately corrosive. To increase corrosivity to represent typical worst case (opportunity) crude blends, naphthenic acid, cetyl mercaptan and elemental sulfur were added to artificially raise the TAN and sulfur content of the crude to 3.0% sulfur and > 2.0 mg KOH/g.

Table 6 - Received Raw Crude Metal Content

Metal*	Results	Units	Nominal Detection Limit
Calcium	48	mg/kg	0.1
Iron	89	mg/kg	0.1
Nickel	5.4	mg/kg	0.05
Sodium	<10	mg/kg	0.5
Vanadium	2.8	mg/kg	0.05
Zinc	<3	mg/kg	0.1
Zirconium	<10	mg/kg	0.5

*All other metals tested (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mg, Mn, Mo, P, K, Se, Ag, Sr, Sn, Ti were below <3 mg/kg

Table 7 - Received Raw crude corrosivity evaluation

TAN Acid Number	0.75	mg KOH/g
Sulfur	0.54	Mass %
	5.4	g/kg
Total Chloride	5.4	mg/kg
BS&W - Sediment	0.2	Volume %
BS&W - Water	<0.025	Volume %
API Gravity	3.02	

2. Autoclave Results

Following two weeks of exposure, coupons were removed from the corrosion autoclave and examined visually. The coupons were, as expected, covered in a layer of crude oil, which was removed with alcohol. The cleaned coupons remained stained black with dried crude, and limited observations could be drawn visually. Coupons were then cross-sectioned and analyzed via SEM EDS; pass/fail evaluations were determined based on the presence of any corrosive media at the cladding/carbon steel bond line or in the cladding, as well as any active corrosion.

Table 8 - Autoclave Test Results

No'	System	Status
1	Carbon Steel	Fail – Corrosion scale
2	IGS 5315	Pass
3	IGS 5450	Pass
4	IGS 5470	Pass
5	MRP 500	Pass

Figure 8: Table 8 of autoclave test results (left) and post-test coupons (right)

The cross-sectioned control coupon, a test surface of uncoated carbon steel within a NiCrMo sheath, showed a continuous corrosion scale approximately 50 – 75 μm (2-3 mils) in thickness. The scale was not universally well adhered, and in locations, the carbon steel surface showed additional evidence of pitting and granular corrosion. The scale itself was predominantly iron sulfide, with low levels of iron oxide, suggesting that, in this specific setup, iron sulfidation was the primary driver of corrosion over naphthenic acid.

As previously noted, iron sulfide scale can be easily destroyed by oxidation or naphthenic acid corrosion, and will not necessarily slow down continuing corrosion or provide protection.
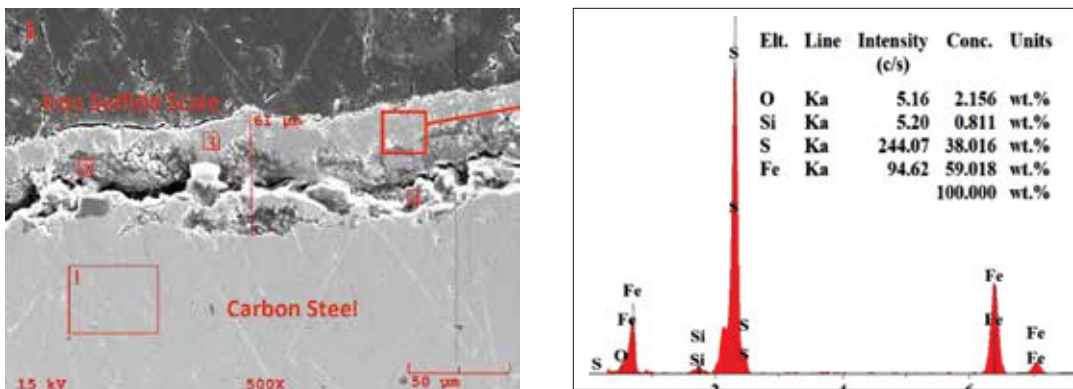

Figure 9: Control coupon corrosion scale (left) and EDS analysis (right)

As with the control coupon, the four experimental coupons were cross sectioned for analysis. Imaging and elemental analysis of the bulk coating systems was performed to ascertain the microstructural condition of the claddings, verify that the composition and oxide levels are within specification, and detect any corrosive media that has penetrated. Additionally, higher magnification images were taken at the bond line between the carbon steel and claddings, where corrosion and cladding disbondment is frequently seen. Results indicated that microstructural composition was within specification and all coatings were well-adhered. Some imbedded aluminum oxide blast media, used for surface profiling, can be seen at the carbon steel; this is considered normal and does not impact coating quality or adhesion. Porosity seen in the MRP system is also normal and a feature of the polymer filled metal scaffolding. No corrosion or corrosive media was seen at any of the bond lines, with no indication of HVTS or MRP degradation. Ultimately, all coatings passed the autoclave corrosion testing.
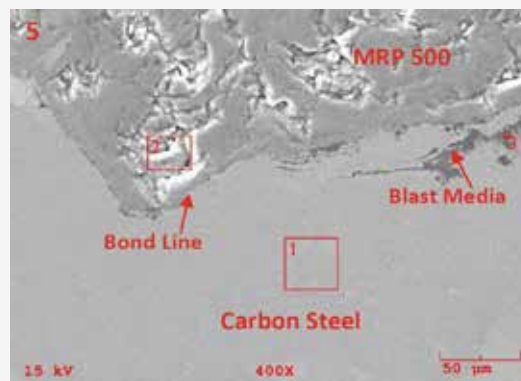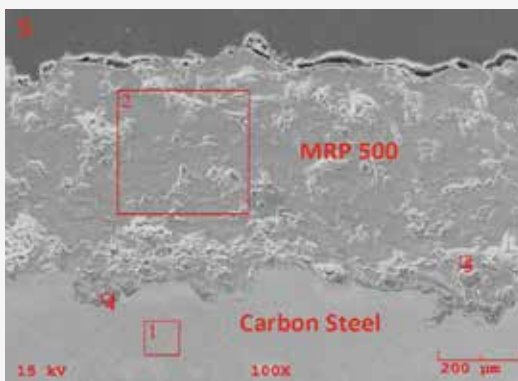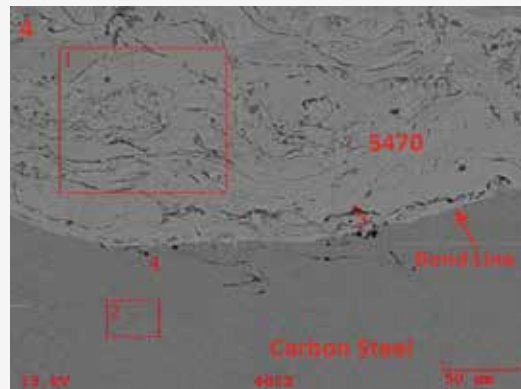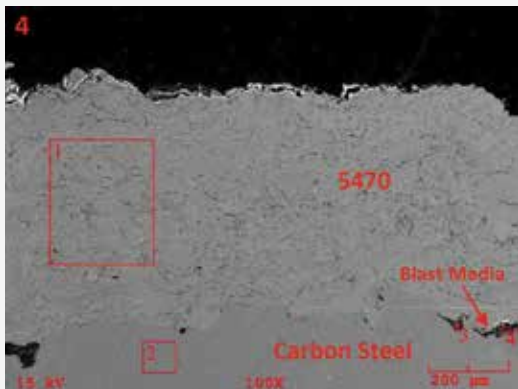
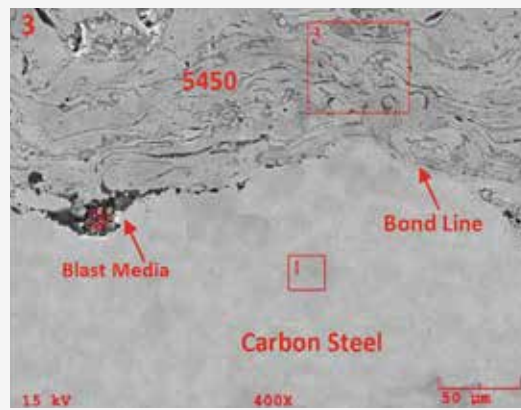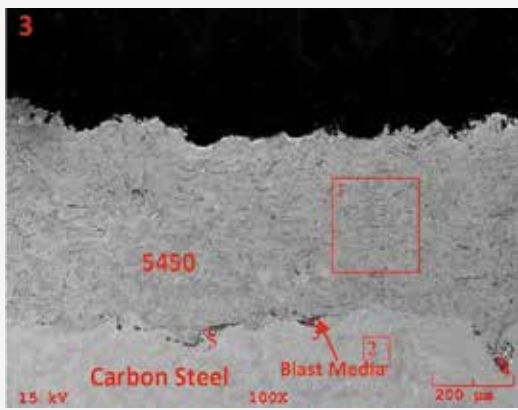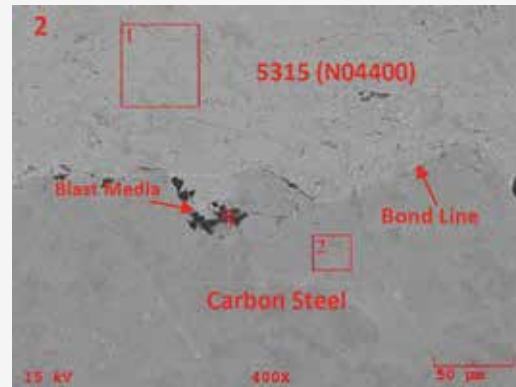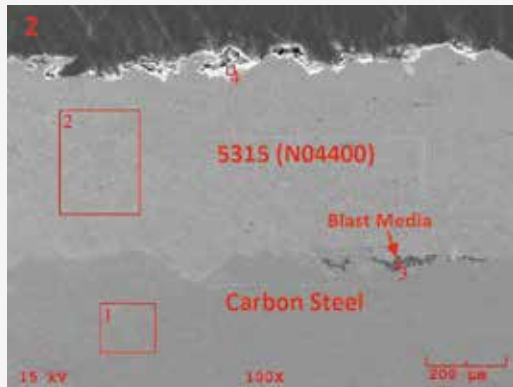100X Bulk Imaging   400X Bond Line Imaging

Figure 8: Table 8 of autoclave test results (left) and post-test coupons (right)

3. Exposure Cell Results

To simulate the CDU overhead corrosion environment, which involves lower temperatures and alternative corrosion species, coated 2"x4" carbon steel test panels were exposed to a solution of 1% HCl, 2.5% NH_4Cl, and 2.5% CH_3NH_2HCl at a temperature of 95°C for two weeks.Following exposure, panels were removed, inspected visually, then cross-sectioned and analyzed via SEM EDS. Pass/fail evaluations were determined based on the presence of any corrosive media at the cladding/carbon steel bond line or in the cladding, as well as any active corrosion or degradation.

Table 9 - Exposure cell test results

No'	System	Status
1	Carbon Steel	Fail – Corrosion scale
2	IGS 5315	Fail – Corrosion at Bond Line
3	IGS 5450	Pass
4	IGS 5470	Pass
5	MRP 500	Pass

Panels were inspected daily. Within 24 hours of exposure, the uncoated carbon steel test coupon showed the formation of a brown corrosion scale, which continually grew in thickness and density. After removal, the control corrosion scale was approximately 2 – 2.5 cm (0.5 – 1.0 in) thick. This scale was preserved using a clear, high wetting epoxy to protect it through the sample cutting and preparation process. SEM EDS confirmed that the scale was a mixture of iron chloride and iron oxide, and substantial material wastage had occurred. The rapid scale growth confirms the corrosivity of the test environment. The level of iron chloride noted confirms that a low oxygen level was maintained in the system and prevented breakaway oxidation.
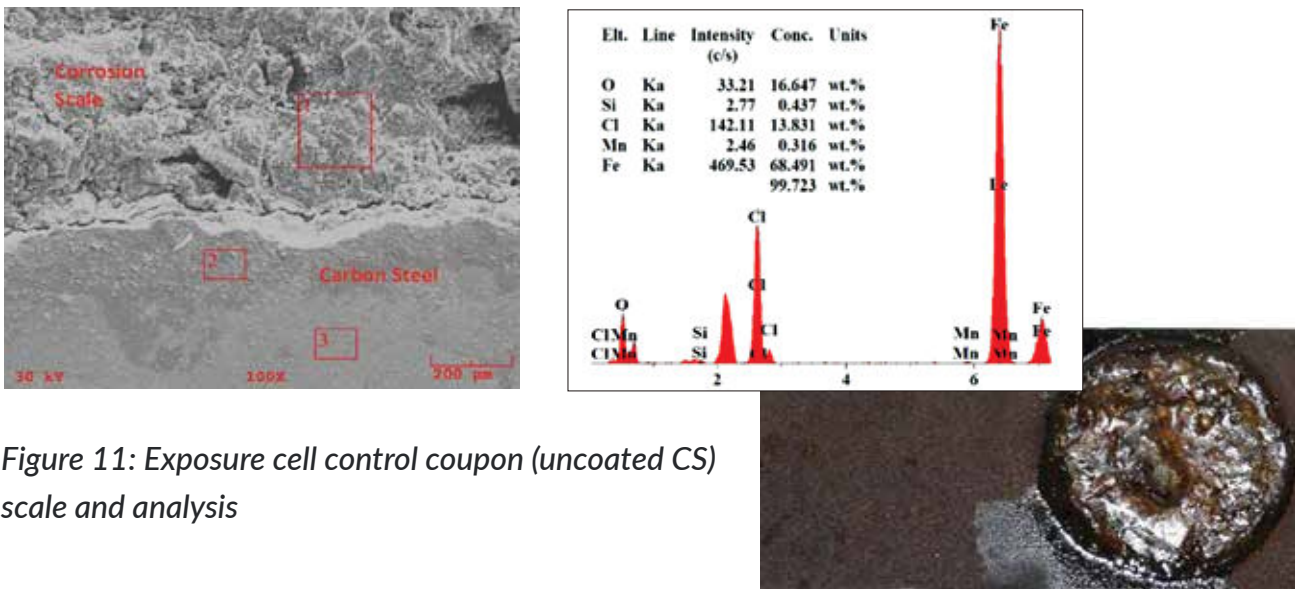
Figure 11: Exposure cell control coupon (uncoated CS) scale and analysis

As with the autoclave coupon analysis, cross-sectional imagining and EDS focused on HVTS microstructural form and composition, adhesion, and any corrosion scale at or near the bond line. SEM imaging revealed a corrosion failure on the 5315 (UNS N04400) system, with the formation of iron chloride-based corrosion scale at the bond line and partial disbondment of the cladding. The 5315 system is not alloy modified for thermal spray application. Intersplat adhesion is consequently poor, which when coupled with solidification stress, decreases interfacial adhesion of the system and results in fine permeation pathways. It is possible that excess porosity and nanometer sized intersplat cold lapping allowed for corrosive media, specifically chlorine, to penetrate the system and attack the interface.

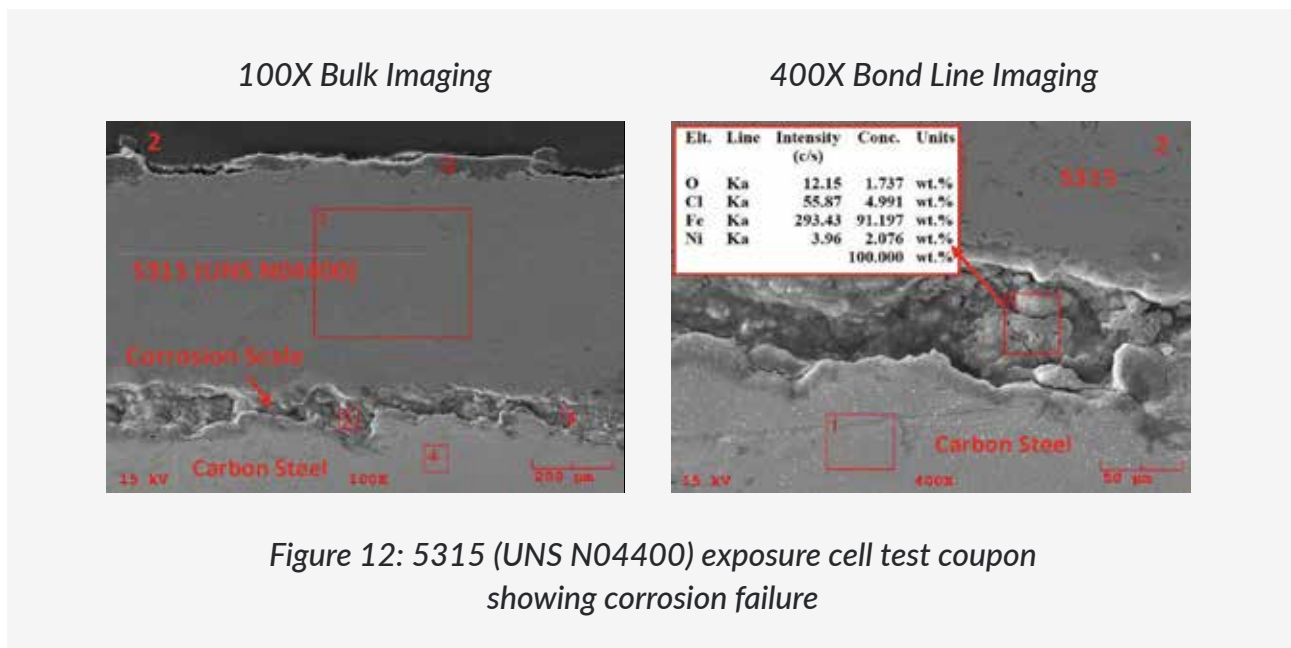### 100X Bulk Imaging   400X Bond Line Imaging

Figure 12: 5315 (UNS N04400) exposure cell test coupon
showing corrosion failure

The other three experimental systems (5450, 5470, MRP) passed corrosion testing as cross-sectional analysis revealed no material chemistry anomalies and no evidence of chlorine or other media penetration into the microstructures. Claddings remained well-adhered, and all bond lines were free of corrosion scale or chlorine. There was no indication of HVTS degradation at the surface, within the matrix or at the substrate interface
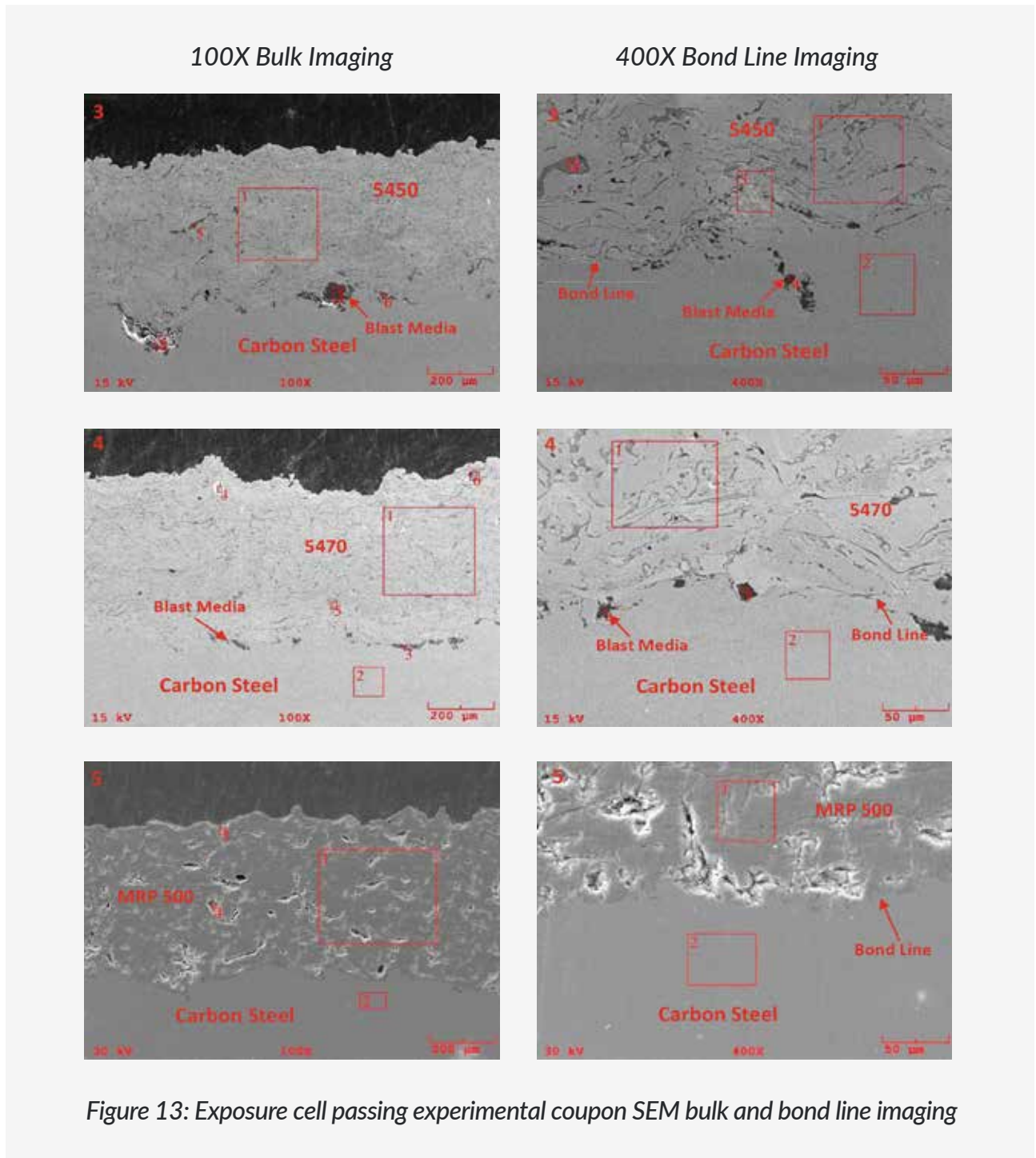

100X Bulk Imaging   *400X Bond Line Imaging*

Figure 13: Exposure cell passing experimental coupon SEM bulk and bond line imaging

Conclusions

IGS Technology Solutions successfully evaluated a materially diverse set of protective HVTS and MRP cladding systems in two unique simulated CDU environments. Traditional alloys that have previously been used in thermal spray and overlay, UNS N04400, were found to exhibit interlaminar porosity and failures in aggressive HCl and amine salt environments. This allowed for the penetration of chlorine species and the beginnings of cladding disbondment and corrosion scale formation from the metal substrate. Alloy modification resulted in improved High Velocity Thermal Spray claddings that show superior resistance to both high temperature naphthenic acid and sulfidation conditions as well as CDU overhead environments. The titanium cermet MRP system was similarly unaffected. All IGS modified NiCrMo-XX alloys and newly developed MRP systems showed complete corrosion resistance in both test phases rendering them suitable selections for substrate protection in a broad range of CDU environments.

IGS supports refineries and chemical plants with a wide range of efficiency and reliability objectives.

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