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**Autoclave Test
of
CeraKote™ E-100 Coating
(Final Report, Revision 1)**

Project Reference Number: 2023529-1

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Prepared for:

NIC Industries, Inc.
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EXECUTIVE SUMMARY

A Plus Coating Solutions Inc. was retained by NIC Industries, Inc. to evaluate the performance of CeraKote™ E-100 coating using Autoclave test. E-100 is an oven cure thin film ceramic polymer based coating. The Autoclave test is designed to simulate the aggressive field service environments in controlled laboratory conditions. High temperature and pressure environments can put a great deal of stresses on a coating, affecting its physical performance and also leading to structural changes, such as softening, electrical impedance reduction, adhesion loss, and alteration of permeability, etc. Therefore, it is important to determine how well a coating will perform under these adverse conditions, so as to determine the suitability of the coating for a particular application. In this work, CeraKote™ E-100 all-side coated (3"×6") panels were submitted by the client for testing. The panels were exposed to a gas/hydrocarbon/water multiphase environment, with 6% H₂S, 8% CO₂, and 86% CH₄ as gas phase, Toluene: Kerosene (1:1) as hydrocarbon phase, and 5% NaCl solution as water phase, at temperature of 200°F/93°C and pressure of 3300 psi/22.8 MPa for 48 hours. Before and after the Autoclave test exposure, the change in appearance and adhesion were employed to assess performance characteristics of the coating.

The test results are summarized below and detailed in Table 1.

- ✓ The pre-test adhesion as per ASTM D3359-17 Method B was rated "5B".
- ✓ After the 48-hour autoclave exposure, all three test panels retained good appearance with no blistering, cracking or delamination. The adhesion evaluation confirmed no change in adhesion in all three phases.
- ✓ The results suggest the autoclave test conditions did not cause any noticeable change in coating film integrity.
- ✓ The only noted change was very slight change in color in the water phase as a result of testing.

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1.0 Objective

The objective of this study was to evaluate the performance of CeraKote™ E-100 coating in Autoclave test under a multiphase environment, using 6% H₂S, 8% CO₂, and 86% CH₄ as gas phase, Toluene: Kerosene (1:1) as hydrocarbon phase, and 5% NaCl solution as water phase, at temperature of 200°F/93°C and pressure of 3300 psi/22.8 MPa for 48 hours.

2.0 Scope of Work

The scope of this work included:

- NIC Industries, Inc. provided the test and parameters.
- NIC Industries, Inc. submitted CeraKote™ E-100 coated panels for testing.
- A Plus Coating Solutions Inc. evaluated the performance of the coating according to the test requirements.

3.0 Samples as Received

Total of six (6) 6"×3" CeraKote™ E-100 coated panels were received from NIC Industries, Inc. (see Figure 1). The samples were properly packed and the package was in good condition when arrived.



Figure 1. The CeraKote™ E-100 Coated Panels Received on July 28th, 2023

4.0 Test Methodologies

4.1 Autoclave

The test was performed according to NACE TM0185: 2006 “Evaluation of Internal Plastic Coatings for Corrosion Control of Tubular Goods by Autoclave Testing” using the apparatus as shown in Figure 2. After the film thickness measurements and visual examination, three test panels were positioned in a 1-L beaker, the pre-prepared Toluene: Kerosene (1:1) and 5% NaCl solution were added, and the beaker was placed into the autoclave vessel. The vessel was then assembled and secured, and the gases were loaded as per the pre-determined partial pressures. The heater was turned on and the system was monitored until the target temperature and pressure were reached. During the duration of the testing, the temperature and pressure were checked and recorded. The Autoclave test conditions are shown below:

- Temperature: 200°F/93°C
- Pressure: 3300 psi / 22.8 MPa
- Gas Phase: 6% H₂S, 8% CO₂, 86% CH₄ (1/3 of the panel length)
- Hydrocarbon Phase: Toluene: Kerosene (1:1) (1/3 of the panel length)
- Water Phase: 5% NaCl solution (1/3 of the panel length)
- Duration: 48 hours



Figure 2. Example of the Autoclave Test Setup

After the completion of the test, the autoclave heater was turned off to allow the test vessel to cool to 32.8°C. Then, the pressure was released to atmospheric pressure in 3 minutes 55 seconds at the temperature. The panels were removed examined for film thickness change, blistering, cracking, delamination, and color change. After that, the adhesion (see section 4.2) was determined in all three test phases of the coating.

4.2 Tape Adhesion

The test was conducted in accordance with ASTM D3359-17 “Standard Test Methods for Rating Adhesion by Tape Test”, Method B. Since the applied coating film was less than 2 mils, a lattice pattern with eleven cuts in each direction was made through the film to substrate on each selected test side. After that, a soft brush was used to lightly brush the surface to remove any detached flakes or ribbons of coatings.

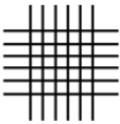

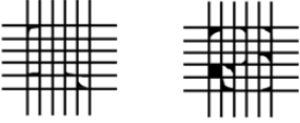
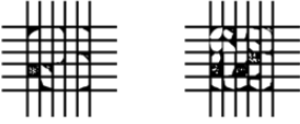
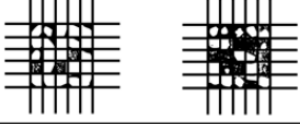
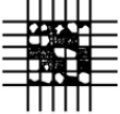
CLASSIFICATION OF ADHESION TEST RESULTS		
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 – 15%	
2B	15 – 35%	
1B	35 – 65%	
0B	Greater than 65%	

Figure 3. Classification of Adhesion Test Results for Method B of ASTM D3359-17

The required length of the tape was removed from the roll of the pressure-sensitive tape. After placed the center of the tape over the grid and in the area of the grid, the tape was carefully smoothed into place by finger in the area of the incisions to ensure no entrapped air under the tape. The surface of the tape was then rubbed firmly over the surface of the tape with the pressure until the color was uniform in appearance, indicating good and uniform contact between the tape's adhesive and the coating surface. Within 90 seconds of application, the tape was removed by seizing the free end and rapidly back upon itself at an angle close to 180°. After that, the grid area was inspected for removal of coating from the steel substrate, and the classification of the adhesion was rated in accordance with the scale shown in Figure 3.

5.0 Results and Discussion

Figures 4 - 5 illustrate the test results for CeraKote™ E-100 before and after the 48-hour Autoclave test exposure. As shown in Figure 4, the coating on the three panels A1 to A3 was smooth and appeared uniform black color finish prior to testing. The pre-test adhesion evaluation was carried out on one of the received extra panels. Despite the coating film was thin, it was noted during the preparation of the cross-cut (eleven cuts in each direction) that the coating was hard with good scratch resistance, as it was difficult to cut through to steel substrate and the blade of the cross-hatch cutter was worn-out much quicker than normal testing of other types of coatings with similar film thicknesses. The pre-test adhesion of the coating was rated "5B".

After the Autoclave test for 48 hours, all three panels retained good appearance (see Figure 5). Except very slight change in color in water phase, the coating showed no blistering, cracking, or delamination in any of the test phases. The film thickness measurements revealed no apparent swelling. The tape adhesion evaluation was completed on panel A3, and the adhesion was rated "5B" in all three phases as indicated by 0% of the coating was detached. The results suggest the autoclave test conditions did not cause any noticeable change in coating film integrity.

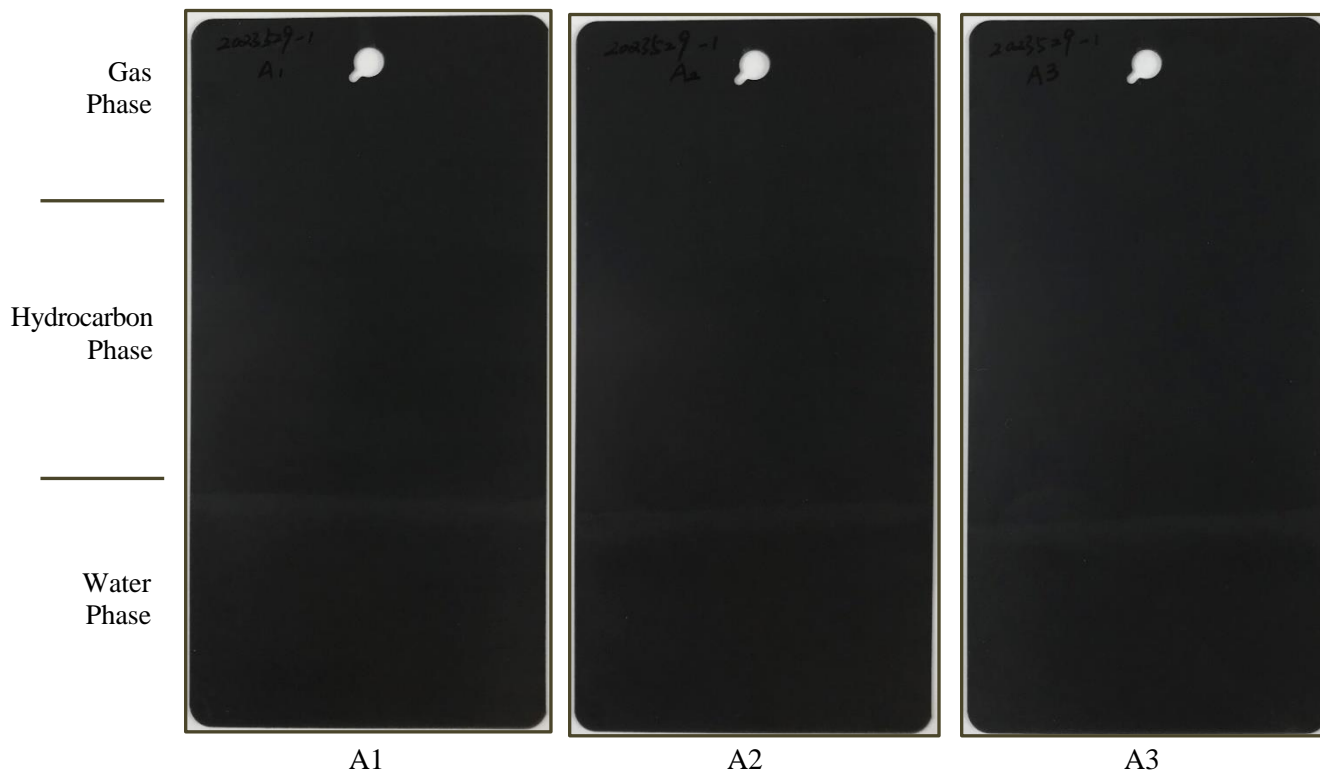


Overview Appearance before the Test Exposure

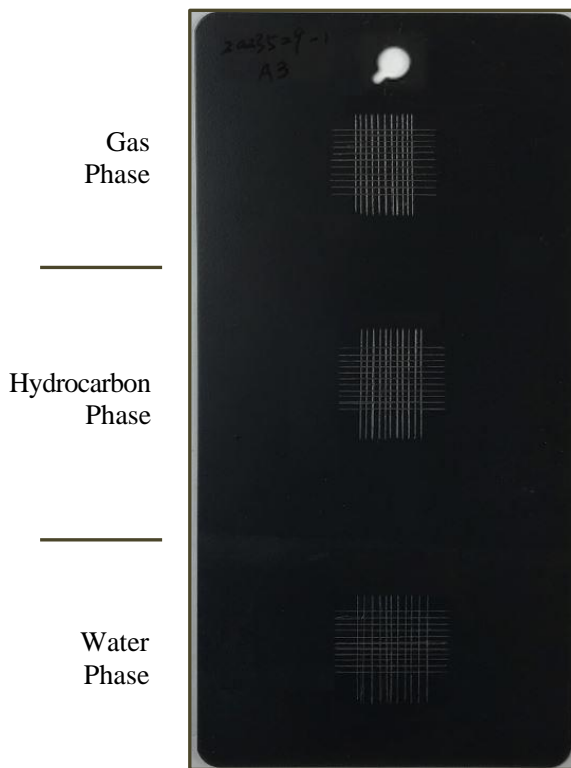


Pre-test Adhesion on the Received Extra Panel

Figure 4. Evaluation of CeraKote™ E-100 Coated Panels before the Autoclave Exposure



Overview Appearance after the Test Exposure



Tape Adhesion on Panel A3

Figure 5. Post-test Evaluation of CeraKote™ E-100 Coated Panels after the Autoclave Exposure NACE TM 0185- 2006: 1/3 (6% H₂S, 8% CO₂, and 86 % CH₄), 1/3 (Toluene: Kerosene (1:1)), and 1/3 (5% NaCl Solution), at 200°F/93°C and 3300 psi/22.8 MPa for 48 Hours

AUTOCLAVE TEST DATA SHEET



Project Number:	2023529-1	Test Method:	NACE TM 0185-2006
Temperature:	200°F/93°C	Gas Phase:	6% H ₂ S, 8% CO ₂ , and 86% CH ₄
Pressure:	3300 psi/22.8 MPa	Hydrocarbon Phase:	Toluene: Kerosene (1:1)
Date:	August 8 - 10, 2023	Water Phase:	5% NaCl Solution
Duration:	48 Hours	Release Temperature:	32.8°C
Release Pressure:	From 2450 psi to atm.	Tested By:	Gary Q., Sherry R.
Release Time:	3 minutes 55 seconds	Rated By:	Sherry R.
Coating	CeraKote™ E-100		

Panel ID	Test Phase	Pre-test		Post-test				
		Film Thickness (Range/Average, mils)	Film Thickness (Range/Average, mils)	Color Change	Blisters (ASTM D714)	Cracking	Delamination	Adhesion (ASTM D3359)
A1	Gas	1.1 - 1.5 / 1.3	1.1 - 1.4 / 1.3	No	None	No	No	/
	Hydrocarbon	1.0 - 1.2 / 1.2	1.1 - 1.3 / 1.2	No	None	No	No	/
	Water	1.1 - 1.2 / 1.1	1.1 - 1.3 / 1.1	Very Slight	None	No	No	/
A2	Gas	1.4 - 1.8 / 1.6	1.5 - 1.8 / 1.7	No	None	No	No	/
	Hydrocarbon	1.2 - 1.6 / 1.4	1.2 - 1.6 / 1.5	No	None	No	No	/
	Water	1.3 - 1.6 / 1.4	1.4 - 1.6 / 1.5	Very Slight	None	No	No	/
A3	Gas	1.2 - 1.6 / 1.4	1.3 - 1.7 / 1.5	No	None	No	No	5B
	Hydrocarbon	1.1 - 1.6 / 1.4	1.2 - 1.6 / 1.4	No	None	No	No	5B
	Water	1.3 - 1.6 / 1.4	1.4 - 1.6 / 1.5	Very Slight	None	No	No	5B

*Pre-test adhesion was completed on one of the received extra panels and was rated as “5B”.

Rating Keys:

Degree of Blistering as per ASTM D714	
Size	Frequency
No. 2	Dense, D
No. 4	Medium dense, MD
No. 6	Medium, M
No. 8	Few, F

Rating as per ASTM D3359 for Method B:

- 5B The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
- 4B Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
- 3B Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
- 2B The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
- 1B The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
- 0B Flaking and detachment worse than Classification 1B.

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