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Improving the Durability of Packaging Materials using Vapor Phase Corrosion Inhibitors

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ABSTRACT

On a global scale, the packaging industry is an enormous economic generator. The global market value for the packaging industry will reach $1 trillion by 2020; an increase from $839 billion in 2015. This immense growth rate is driven by consumer and industry trends. Reduce, reuse and recycle is part of the green commitment and ecological fight to being responsible in the world. The packaging industry recognizes its responsibility in attaining a closed loop cycle for these materials through the use of green corrosion inhibitors. Application of green corrosion inhibitor in packaging materials (wrapping paper, films, and corrugated boxes) is one of the best options. In this study several green inhibitors impregnated papers and film were investigated. Qualification tests of these products were conducted using MIL-STD-3010C including the vapor-inhibiting ability (VIA) (similar to the NACE TM 208-2018) before and after exhaustion test of VCI (volatile corrosion inhibitor) impregnated materials. VIA tests showed very satisfactory results for all the impregnated wrapping paper and film prior to the exhaustion test procedure (grade 3-4 according to NACE TM 208 grading criteria). The VIA grading decreases to 2 or 1 after the exhaustion tests. When the steel samples were wrapped with the VCI impregnated wrap paper and then subjected to exhaustion test conditions, post VIA tests showed good results (grades of 3). It is more realistic approach to conduct the exhaustion tests on the wrapped steel samples with VCI packaging materials and subject those samples to the VIA tests. These tests demonstrate that the VCI adsorbed compound can maintain its attachment to the surface during the exhaustion tests and protect steel samples against corrosion later.

Key words: Packaging papers, corrosion inhibitors, vapor-inhibiting ability, exhaustion test

INTRODUCTION

Packaging is an industry term for the technology protecting any product intended for storage, and shipping. Statistics indicate that the world market value for packaging industry will reach US $1 trillion by 2020 [1]. During manufacturing, storage and transport, humidity, salts and other corrosive substances in the environment as well as temperature fluctuations can make metal parts more susceptible to corrosion. The likelihood of corrosion can be minimized either by controlling the environment or by insulating the metal from the destructive surroundings using suitable packaging materials containing corrosion inhibitors. Anti-corrosion packaging is an important trend in the packaging market because it provides optimum protection and extends the shelf life of the products. Due to their effectiveness, flexibility, low cost and ease of application, the packaging technology finds its way into a wide range of products. VCI papers have been used to protect sensitive electronic components, metals in various forms, automotive parts, machinery, engines and tools. The demand for VCI packaging has significantly increased; the VCI packaging market is expected to exceed US $680 million by 2023 compared to US $480 million in 2015 and US $540 million in 2018.

Corrosion is an undesirable electrochemical reaction for metals and alloys in response to an aggressive environment. A corrosion inhibitor is a chemical substance which when added in small concentrations to an environment, can minimize or decelerate the corrosion process. Generally, an efficient inhibitor is compatible with the environment, economical to use and produces the desired effect when present in small concentrations. Most VCIs are amine derivatives that release free amine or its hydroxyl form upon hydrolysis. The laboratory investigations confirmed that volatile corrosion inhibitors are an effective replacement for toxic chemicals. Corrosion prevention is one of the major reasons for applying coatings to the surface of metals. However, sometimes, it is not possible to directly apply a coating on the surface [3]. In the past, a protective oil coating was used to prevent corrosion during transport and storage. The use of oil for corrosion protection involves its disadvantages. The oil coating needs to be removed before welding or painting. The removal of a protective oil coating also requires solvents. The disposal of used solvents has its own environmental restrictions. VCI packaging techniques facilitate corrosion protection of metal parts in light of the aforementioned complications.

VCI papers are highly effective and common packaging materials for protecting metals. They are cost-effective and easy to use for both ferrous and non-ferrous metals. These papers display good tensile strength, and are mostly scratch/abrasion resistant. VCI papers are environmentally friendly, allowing for biodegradability and sustainability by recycling into other types of paper products such as boxes and cardboard. Furthermore, they are void of hazardous ingredients such as nitrites, phosphates, silicones, chromates, or other heavy metals. VCI-coated papers were intended for wrapping metals during shipment, storage and process operations. They are an effective means to protect dissimilar metals against galvanic corrosion.

Volatile corrosion inhibitors are bio-based organic chemical compounds with significant vapor pressure, allowing vaporization of inhibiting molecules and further adsorption onto metallic surfaces. VCI molecules move from the paper directly to the surface of metal, condense and adsorb onto the metal surface, forming an extremely thin molecular protective layer of crystals over the metal surface. This thin layer effectively inhibits corrosion on the metal by preventing air, moisture, salt, oxygen and other corrosive elements from depositing on the metal surfaces. In the presence of even trace amounts of corrosive substances, VCI molecules dissolve and immediately develop strong bonding with the metal substrate. This layer separates the metal from the environment. VCI compounds are impregnated onto the packaging materials and released when they come into contact with the metal products within an enclosed space. Along with the corrosion protection, VCI packaging materials also act as moisture and dust barriers due to the hydrophobic nature of non-polar groups in organic VCI inhibitors. VCIs protect both accessible and hard-to-reach surfaces of the metal from attack by corrosive species. Providing a clean protection with no residue or aroma. The vapor released by the coating is odorless and harmless to humans. VCI packaging does not change the properties of metal in any way and thereby, it has no effect on the electrical or mechanical properties of the protected material. After the metal part is taken out of the package, the VCI molecules float away, and the metal is ready for immediate use, without any cleaning required.

Paper-based packaging materials possess low barrier properties against oxygen and water vapor penetration compared with plastic based packaging materials [5]. In this work the principles of superhydrophobicity and the microstructure of Kraft paper were used to control the VCI diffusion rate in the packaging papers. A state of superhydrophobicity (or the tendency of a solid surface to strongly repel water penetration by forming distinct droplets) by static contact angles higher than 150° and sliding angles less than 10° is commonly achieved by low surface energy modification and introduction of a hierarchical micro-and nano-scale structure. Superhydrophobicity can be created by embedding nanoparticles or by creating porosity. Thus, the inherent porosity of the papers were used to create semi-moisture barriers. Although the rough and porous surface of paper is necessary for creating high water contact angles, it also assists in the spreading and sorption of liquids through the fibrous geometry. The water contact angles of cellulose roughly varies between 17° and 47° [2], while the range of the typical roughness of uncoated paper is within the range of 3 - 5 μm [9]. The papers used in the experimental procedure are Kraft papers, which consist of almost pure cellulose fibers. Cellulosic surfaces are comprised of hydrophilic fibers that make up a capillary system. Cellulose chemistry facilitates the penetration of aqueous fluids due to the abundance of surface hydroxyl groups and their ease of forming hydrogen bonds with water molecules in the polysaccharide-rich material or its derivatives. Aspects such as the capillarity and the hydrophilic nature of the cellulose fibers increase the absorbency of the Kraft paper. Inherently, Kraft paper displays a rough and inhomogeneous surface, which draws itself from the intertwining and flocculation of the fibers in the slurry state from the paper formation. This non-uniformity is seen on the order of the fiber dimensions. The surface is actually an ill-defined boundary which depends on the degree of fiber compaction and collapse [6]. Thus, the internal cavity of the cellulose fibers, surface roughness and the pore networks are accountable for liquid or gaseous permeability in the paper. Moisture can migrate in the paper by a number of transport mechanisms: vapor-phase diffusion in the inter fiber pore space, Knudsen diffusion in pores of diameters less than 100 Å, surface diffusion over fiber surfaces, bulk-solid diffusion within fibers, and capillary transport [7]. The movement of materials into and out of the paper takes place in either solution or gaseous forms. It is not clear which physical processes are involved; however the movement is through imbibition and diffusion. Water flow in fibrous layer will move along the fiber first and then fill the pore between them [8]. The pores of the substrate were impregnated through spray-coating of the inhibiting solution. During the first spray VPCI compounds saturated the pores of the cellulose fiber, both inter and intra. The purpose of the second spray (superhydrophobic coating) allowed for the lowering of surface energy by altering the surface chemistry through silane derivatives (Si-based), while further micro and sub-micro roughness was introduced. The increase of roughness adds to the tortuosity of both gaseous and liquid materials. In conjunction with air always being present in the matrix of the paper, migration is believed to occur by diffusion of vapor through the void spaces as well as fiber cell walls- thus the travel is an increased path length [16]. Hence the moisture migration occurring by diffusion of water vapor through the void spaces and in condensed form through the fiber cell walls must now travel an increased path length [10]. It is evident from the results that the micro-roughness arising from the position of the fibers in the network followed with a hydrophobization treatment hindered the vapor phase transport through the void spaces by increasing the tortuous paths.

In the packaging industry, papers with a high level of water resistance are desirable. Controlling the surface wettability of cellulosic substrates is important in paper wrapping applications because paper may lose physical and mechanical strength through absorbing water during shipment and storage [14]. Due to the possibility of minimizing the contact area between liquids and a surface, superhydrophobic coatings can offer great water repellency [15]. Surface energy and surface roughness are the key factors contributing to the formation of superhydrophobic surfaces. Superhydrophobic coatings not only provide controlled diffusion of VCI inhibitor and protect paper substrate against moisture infiltration, but also help to resist oil and grease absorption. To achieve superhydrophobicity as well as controlled release of underlying VCI inhibitor, highly porous microstructures with large surface areas are required.

EXPERIMENTAL PROCEDURE

The objective of this project was to study the corrosion behavior of carbon steel in the presence of VCI coated packaging papers. In particular, the inhibiting ability of VCI coatings was evaluated by electrochemical polarization tests and vapor inhibiting ability (VIA) tests that were conducted before and after exposure to elevated temperature and controlled humidity (exhaustion tests). Visual examination of the metal surface was done using digital light microscopy and scanning electron microscopy (SEM) to assess the corrosion grade for the steel specimen.

Control VIA tests

Figure 1: VIA test setup for VCI impregnated wrapper corrosion effectiveness evaluation.

VCI A and VCI B are two impregnated wrap papers commercially available. Additional VCI coated papers were prepared by applying VCI C, VCI D and VCI E on natural Kraft paper (Standard 50-lb Kraft paper has a weight ~60-64 gram/m^2) and drying the coated papers at 40°C. One set of prepared VCI coated papers were sprayed with hydrophobic moisture repelling barrier coating after drying. Table 2 lists the different coated papers that were investigated. The vapor inhibiting ability of these VCI impregnated wrap papers were investigated by using 2 x 8 inch paper samples for the exhaustion tests. The exhaustion test were conducted at 60ºC (140ºF) for 12 days as per MIL-STD-3010C Test Method 4031 for Exhaustion Test of Packaging Materials. During this test, air was maintained at ~50% relative humidity (%RH) and pumped at a rate of ~100 cm^3 per minute into the test tube. To achieve the proper relative humidity, air passed through approximately 26% by volume mixture of glycerin/distilled water. Four exhaustion tests were conducted on each sample and after completion of the exhaustion test, paper was cut to 1x6 inch specimens for the VIA tests.

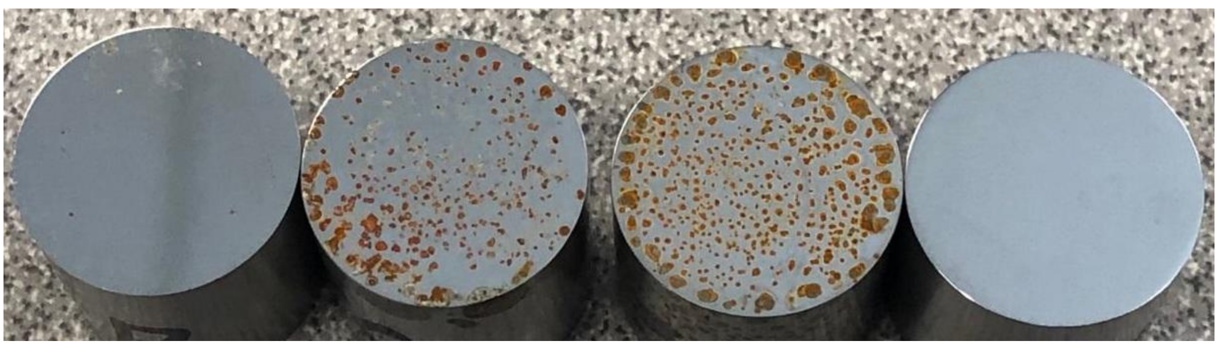
The corrosion behavior of carbon steel (UNS G10100) samples per Mil-STD 3010C VIA test method were subjected to VIA tests (one control sample, three with impregnated paper and six after exhaustion tests; total 10 samples for each VCI product). The VIA corrosion test method provides for standard conditions in a test jar of water-saturated, warm air without the presence of any corrosive species. Prior to testing, all steel samples were prepared by polishing with 240, 320, 400, and 600 grit silicon carbide papers and a final polish with 1 µm powder. The VIA tests require 1) sample conditioning for 20 hours at 22°C, 2) cooling cycle at 2°C, 3) pre-warming at 50°C, and 4) followed by three more hours at 22 °C. Figure 1 shows the test setup for the VIA test. After this process, the steel samples were inspected for visible water condensation and the surface of each was visually examined by microscope to determine its corrosion rating. The corrosion criteria for rating steel specimens consist of grade 0 (worst case, heavy corrosion) through grade 4 (no noticeable corrosion). For a valid VIA test, the control sample must have grade 0 (heavy corrosion). The control samples consistently rated a grade 0 for all VIA tests; therefore, test was valid. Relative humidity and the temperature of each test jar were monitored by sensors and data logging software. Samples subjected to the exhaustion test paper were inspected to ensure none had any surface pits that exceeded 300 micrometers in diameter.

RESULTS

The electrochemical polarization behavior of the steel specimens was investigated per ASTM G61 Cyclic Polarization standard test method. Electrochemical-based measurements determine information regarding the rate of corrosion and mechanism of corrosion protection. Through electrochemical measurements, the corrosion potential (E_c), break down potential (E_b) and corrosion current density (i_c) are measured and converted into a corrosion rate. The majority of tested samples in this study showed active-to-passive transition, through which the anodic polarization increased the corrosion rate initially and then caused a reduction in the corrosion rate when transforming into the passive range. In this test, steel samples were tested in solutions containing 0.05%, 0.075%, and 0.1% by volume inhibitor. Furthermore, a series of cyclic polarization tests were performed in temperatures ranging from room temperature to 50°C. Table 1 shows electrochemical behavior for UNS G10100 steel in different concentrations of inhibitor at different temperatures. These results showed a VCI concentration of ~0.075% was required to provide acceptable protection. A typical post-VIA test observation is shown in Figure 2. The test showed a heavily corroded surface for the control sample (Grade 0), and satisfactory corrosion protection (Grade 3-4) for all VCI impregnated papers. This shows that impregnated papers were able to protect steel samples from corrosion attack prior to exhaustion tests.

Table 1: Electrochemical behavior for UNS G10100 steel in different concentrations of inhibitor at different temperatures. These results showed a concentration of ~0.075% VCI was required to have good protection.

Temp ^oC	VCI %	Ec, mV	Ic, uA/cm2	Eb, mV	CR, mpy
23	0.050	-435	0.960	-112	0.40
23	0.075	-352	0.520	153	0.22
23	0.100	-280	0.210	810	0.09
30	0.050	-414	2.040	-254	0.86
30	0.075	-342	0.592	133	0.25
30	0.100	-294	0.367	636	0.15
40	0.050	-411	3.210	-273	1.35
40	0.075	-359	1.060	168	0.45
40	0.100	-291	0.553	239	0.23
50	0.050	-392	4.280	-221	1.80
50	0.075	-383	1.800	-157	0.76
50	0.100	-314	0.743	166	0.31



VCI-A after VIA test   Exhausted + VIA   Control-VIA   Sample before VIA

Figure 2: Comparison of VIA test results for non-tested sample; As received paper and after exhaustion tests on the VCI A. Grade is zero for samples after exhaustion test, while the test results for the as received paper is satisfactory (better than grade 3).

VCI A, VCI B, VCI C and VCI D samples that were subjected to exhaustion cycles suffered severe corrosion and showed surface pits, Figures 3-8. The VIA corrosion grading per TM-208 of these samples were Grade 1-2, (based on NACE method or grade 4 based on the Mil-STD 3010C criteria). VCI E was the only sample that passed both VIA tests before and after exhaustion cycles. Its grade after VIA tests was a satisfactory 3-4. These tests results create concerns over the feasibility of the exhaustion test criteria.

To improve the corrosion performance of these products, several coatings were investigated. These coatings were polyvinyl alcohol, PVA, (1.0% and 5.0%), PVA (1.0%) + bentonite (1.0%), and a superhydrophobic coating (as shown in Table 1). VCI B paper

Table 2: Different VCI papers evaluated in this project

VCI-Paper	Paper Weight gram/m^2	VIA test Pre-Exhaustion	VIA test Post-Exhaustion	Wrapped in Paper Exhaustion, VIA
VCI A	64.2	Passed	Failed	Passed
VCI B	64.3	Passed	Failed	Passed
VCI B+ coated with Super hydrophobic	66.3	Passed	Passed	
VCI B+ PVA (5.0%)	68.7	Passed	Failed	
VCI B+ PVA (1.0%) + Bentonite (1.0%)	96.3	Passed	Failed	
VCI C	64.4	Passed	Failed	Passed
VCI C + coated with Super hydrophobic	68.1	Passed	Passed	
VCI D	63.5	Passed	Failed	Passed
VCI D+ coated with Super hydrophobic	68.4	Passed	Passed	
VCI E	63.8	Passed	Passed	Passed
VCI E + coated with Super hydrophobic	67.6	Passed	Passed	
Kraft Paper , base*	60.1			

*Standard 50 lb paper (3000 sq ft) has~60-64 gram/m2

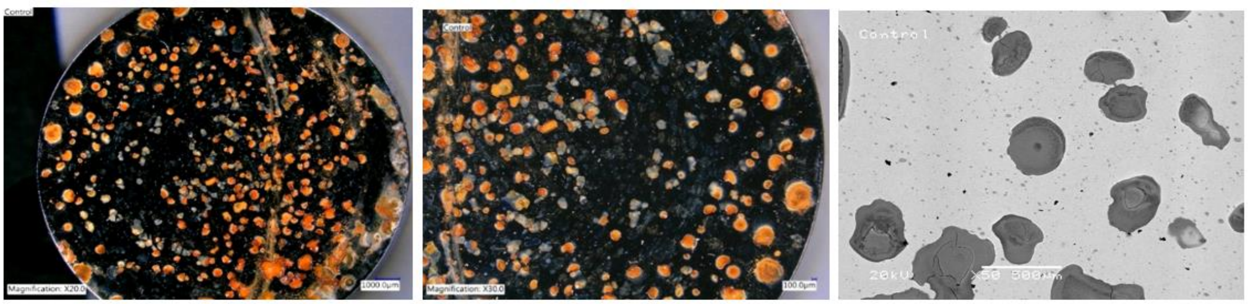
coated with 1.0% or 5.0% PVA, and PVA 1.0% + bentonite 1.0% samples that were tested after exhaustion cycle, suffered some corrosion showing surface pits. The corrosion grading per TM-208 indicated that the control sample had Grade 1-2. It is possible that the nonuniformity and some internal defects (porosity and microcracks) of the applied coating interfered with proper performance. Another disadvantage for these coatings was their heavy weight (~92-94 grams/m^2) and resulting discoloration of the Kraft paper that disqualified them. Papers coated with the super hydrophobic coating (the superhydrophobic coating included methyl isobutyl ketone, butyl acetate, mineral spirits and nanoparticles of silicon oxide) showed very promising results and coated samples passed the post exhaustion VIA with grading of 3-4. None of the superhydrophobic coated papers showed any surface pitting exceeding 300 um after exhaustion VIA tests (Mil-STD 3010C requirement).

Two commercially available papers (VCI A and VCI B) failed the post exhaustion VIA tests, creating concerns about the exhaustion test criteria. One possible explanation for the failure is the exhaustion test conditions; 60 ^{o}C for 12 days while circulating air at 50% RH and a flow rate of 100 cm^3 per minute can result in the loss of the VCI component on the wrap paper. Therefore, following exhaustion tests, the VCI quantity is not sufficient to protect the steel sample during the VIA tests. The other issue is the mechanism of VCI surface adsorption. When the VCI adsorbed onto the steel surface its hydrophobic nature does not allow wetting of the surface that results in better corrosion protection. The exhaustion test on the wrapping materials removes (depletes) the protective VCI compounds and results in unacceptable VIA grading. It is a more realistic approach to conduct the exhaustion tests on the wrapped steel samples with VCI packaging materials and subject those samples to the VIA tests. These tests demonstrate whether the VCI adsorbed compound can maintain its attachment to the metal surface during the exhaustion tests, protecting steel samples against corrosion. By modifying the exhaustion tests to include the steel samples in the test chamber (wrapping steel samples with the VCI impregnated papers during exhaustion cycle), VIA tests showed a very satisfactory performance for VCI A, VCI B, VCI C, VCI D, VCI E with grading of 3-4 (Figures 4, 6 and 8). This modification in exhaustion cycle, allowed the VCI molecule to be absorbed on the steel surfaces; improved corrosion performance was seen for these VIA tests. Adsorption mechanism based on the Langmuir adsorption isotherm approach showed that these VCIs mainly form a physical adsorption (physisorption) mechanism with roughly adsorption energy of ~18,200 j/mol. Therefore, any evaporated inhibitor molecule during exhaustion cycle can be adsorbed on the steel samples, allowing them to perform well during VIA tests.

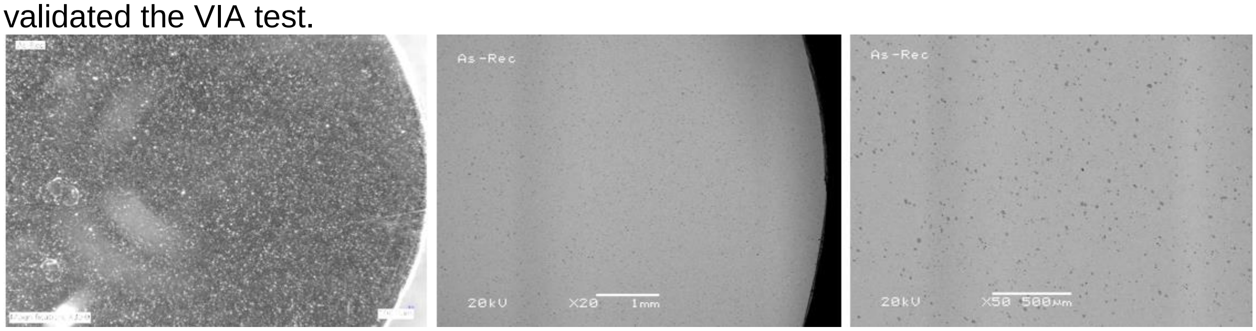
CONCLUSIONS

In summary, VCI E impregnated papers showed satisfactory corrosion protection in the VIA test method after exhaustion tests and steel samples achieved grade 4. Applying superhydrophobic coating, showed a satisfactory performance for all impregnated papers (VCI B, C, D and E). Polyvinyl alcohol, PVA and PVA+ bentonite coatings on VCI B after exhaustion cycle, suffered some corrosion and surface pits (Grade 1). It is possible that the non-uniformity and some internal defects (porosity and microcracks) of applied coating interfered with performance of these coating. Unfortunately, these coatings had a heavy weight and caused discoloration of the Kraft paper that made these coatings undesirable. A modified combination of PVA+ bentonite should be investigated to improve uniformity and a lighter weight coating.

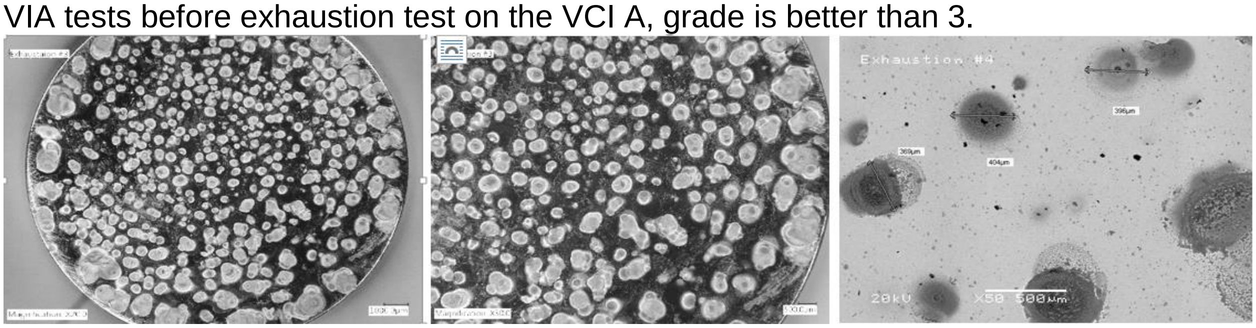
Two commercially available papers (VCI A and VCI B) failed the post exhaustion VIA tests. This created concern over the feasibility of the exhaustion test criteria. Conducting the exhaustion tests on the wrapped steel samples with VCI packaging materials and subjecting those samples to the VIA tests demonstrated satisfactory results of grade 3-4. Therefore, Mil-STD 3010C VIA and exhaustion test method might need to be re-evaluated and the exhaustion cycle procedure modified.



Control sample in the VIA tests. Grade is 0, most pits are larger than 300 um. Results validated the VIA test.

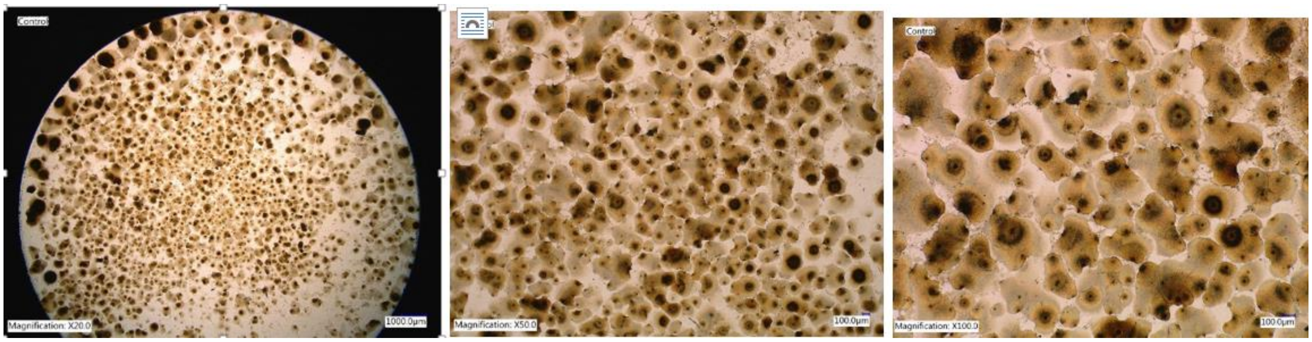


VIA tests before exhaustion test on the VCI A, grade is better than 3.

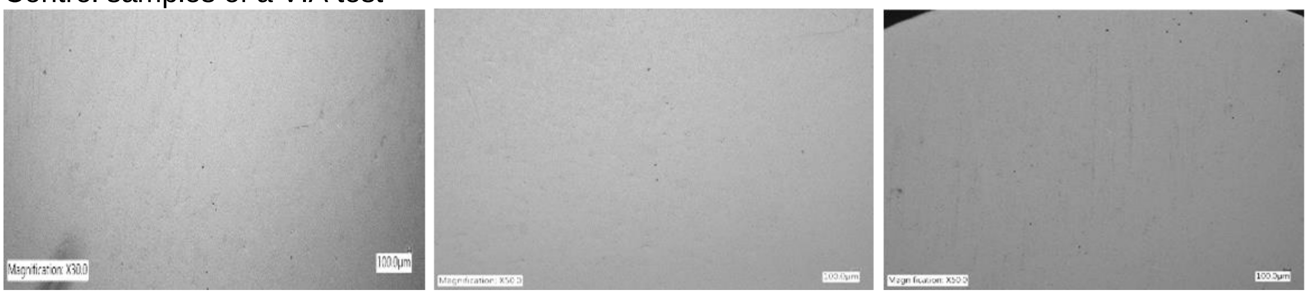


VIA tests after exhaustion test on the VCI A, grade is 0. Most pits are larger than 300 um.

Figure 3: Optical micrographs of samples after VIA test on VCI A post-exhaustion test, Grade is 0, most of surface pits are larger than 300 um.

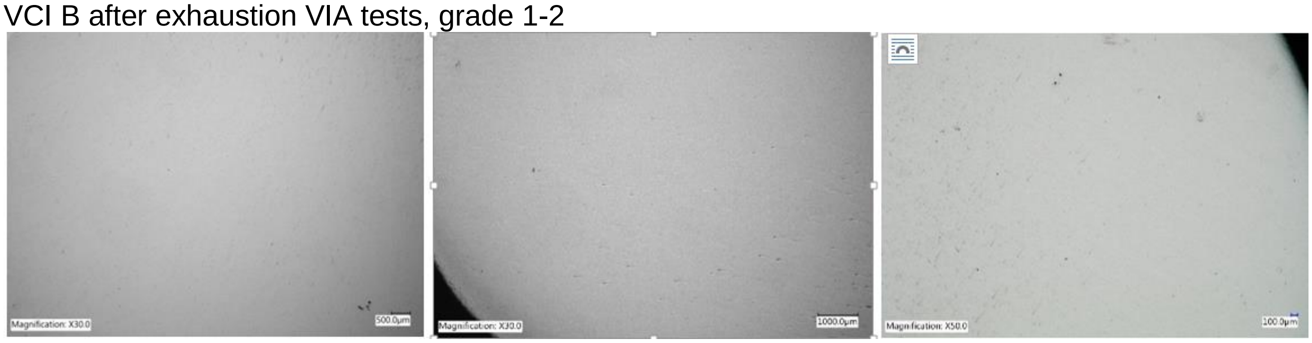


Control samples of a VIA test

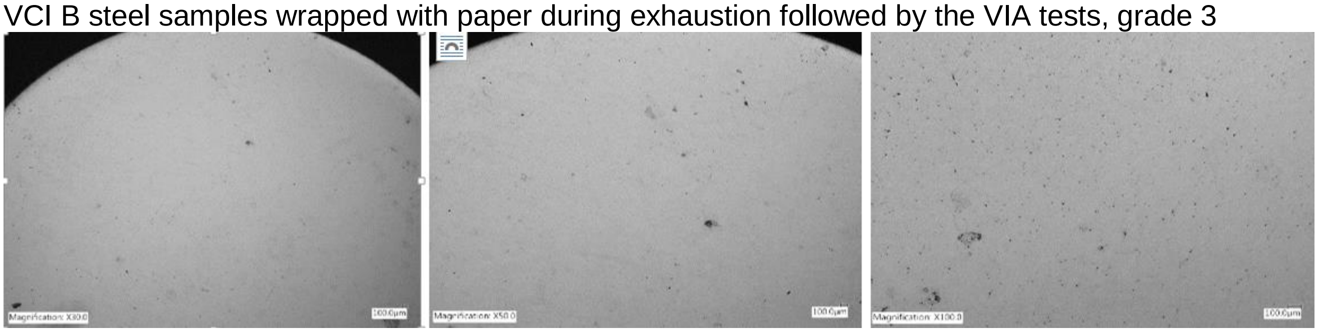


VCI B after VIA test, grade 3.

VCI B after exhaustion VIA tests, grade 1-2

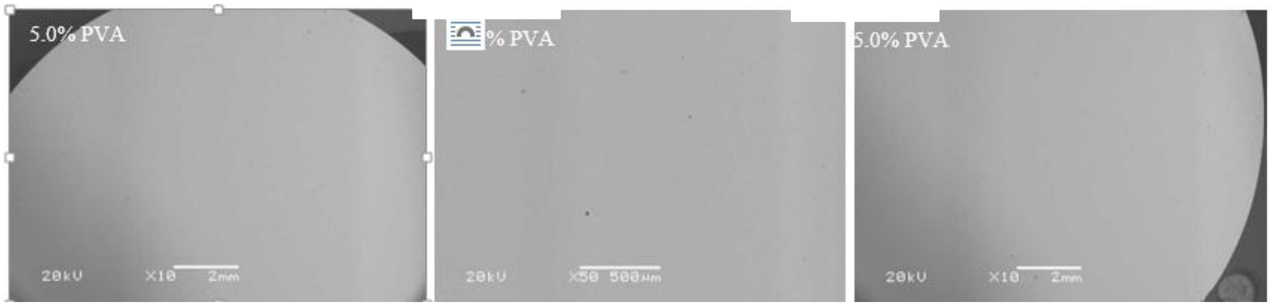


VCI B steel samples wrapped with paper during exhaustion followed by the VIA tests, grade 3

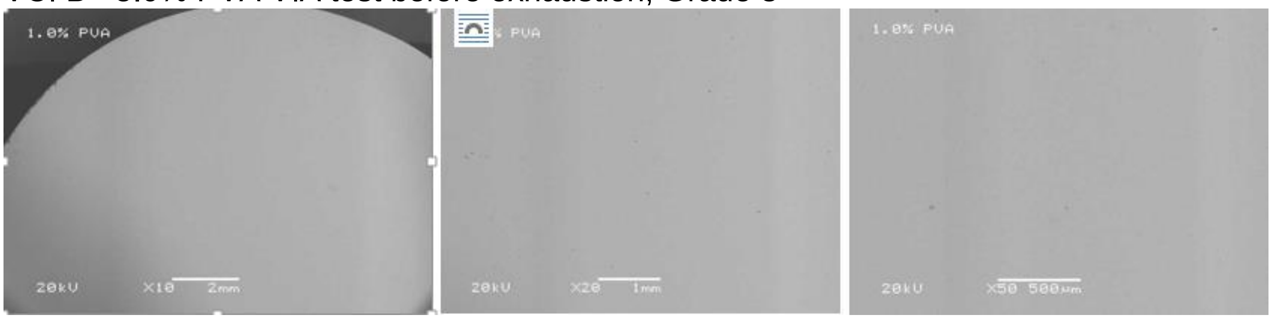


VCI B+super hydrophobic coating after exhaustion VIA tests, grade 3.

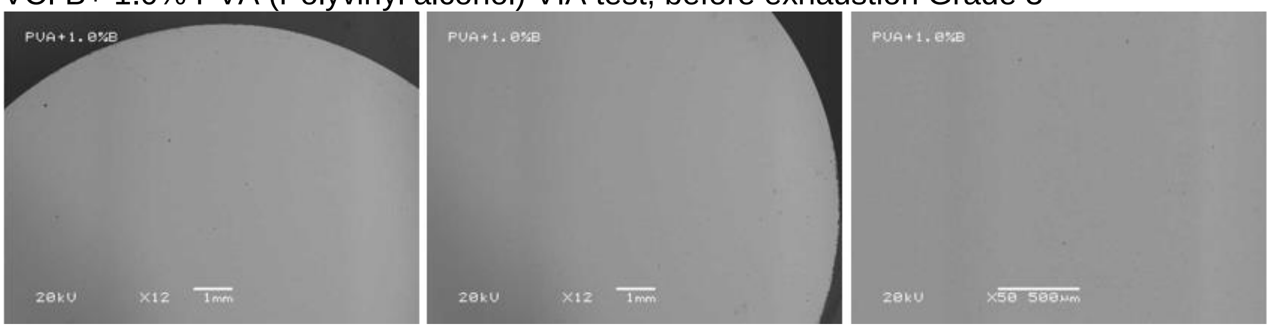
Figure 4: Comparison of VCI B and control corrosion behaviors after VIA test, grade 3.



VCI B+ 5.0% PVA VIA test before exhaustion, Grade 3



VCI B+ 1.0% PVA (Polyvinyl alcohol) VIA test, before exhaustion Grade 3



VCI B+ 1.0% PVA + 1.0%B VIA test, before exhaustion, Grade 3

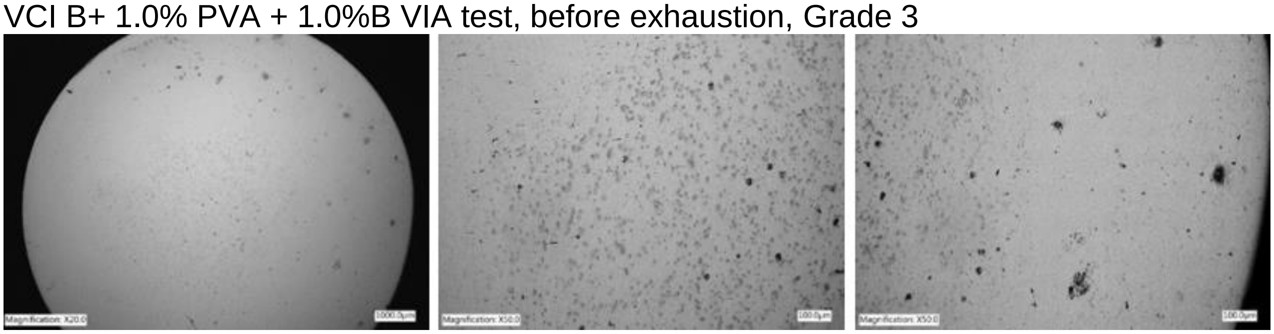
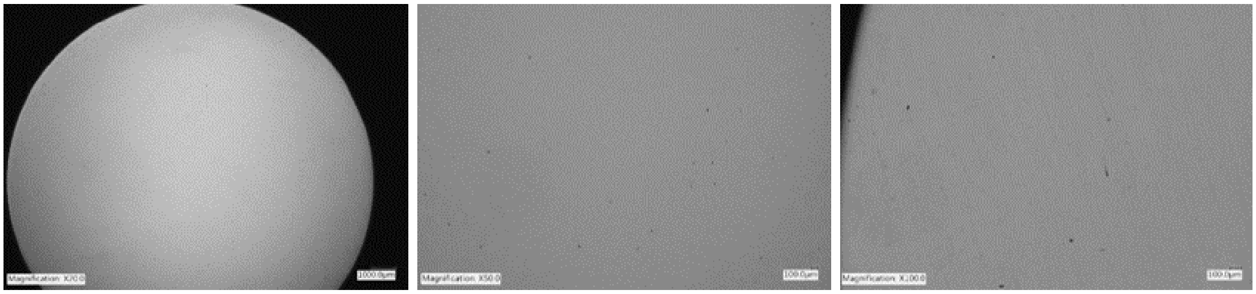
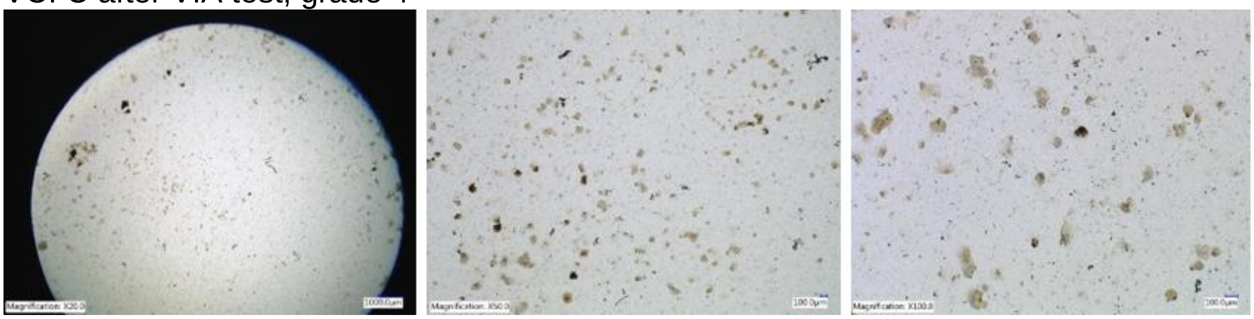

VCI B+ 1.0% PVA + 1.0%B VIA test, after exhaustion, Grade 1-2

Figure 5: Comparison of VCI B corrosion behaviors using different coating after VIA test.

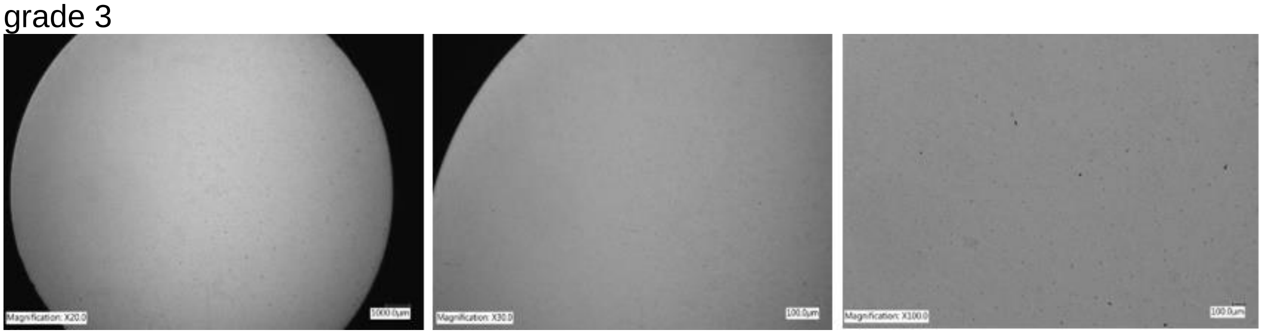


VCI C after VIA test, grade 4



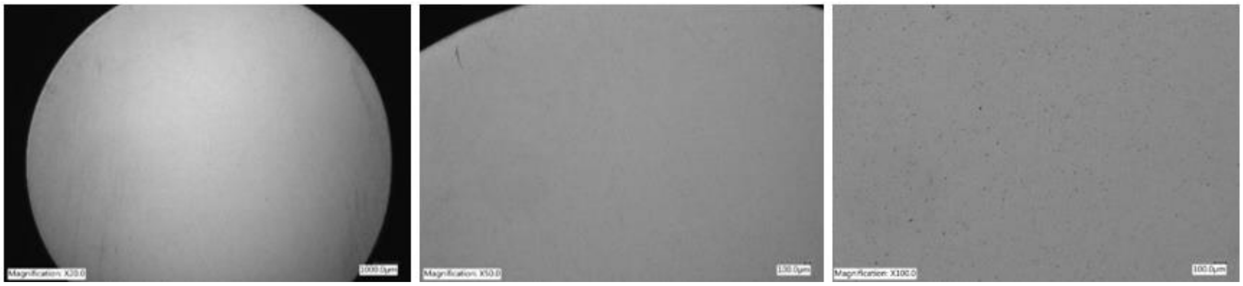
VCI C after exhaustion test and VIA test, grade 2

VCI C steel samples wrapped with paper during exhaustion followed by the VIA tests, grade 3

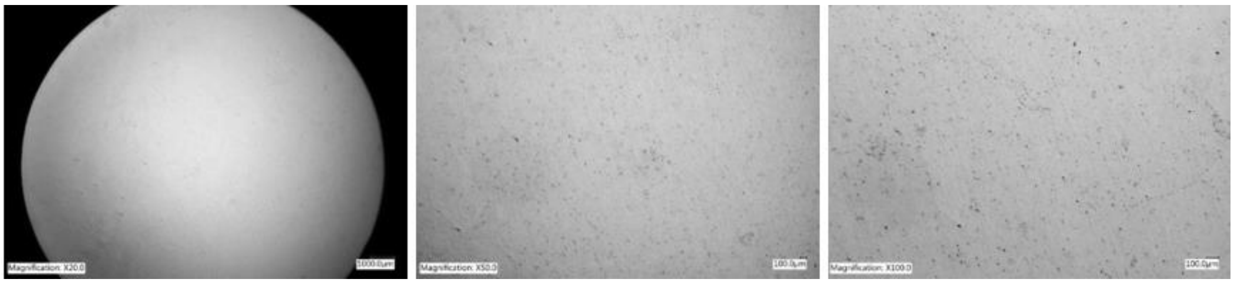


VCI C + super-hydrophobic VIA tests after Exhaustion. Grade 4.

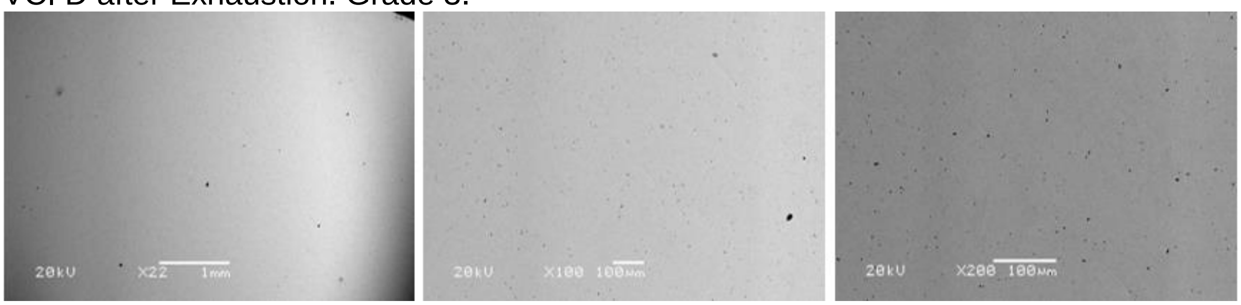
Figure 6: Comparison of VCI C corrosion behaviors using different coating after VIA test.



VCI D after VIA test. Grade 4.

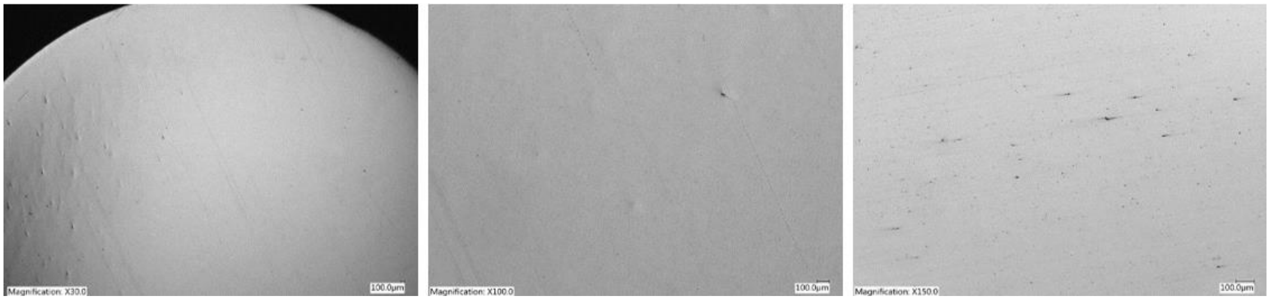


VCI D after Exhaustion. Grade 3.



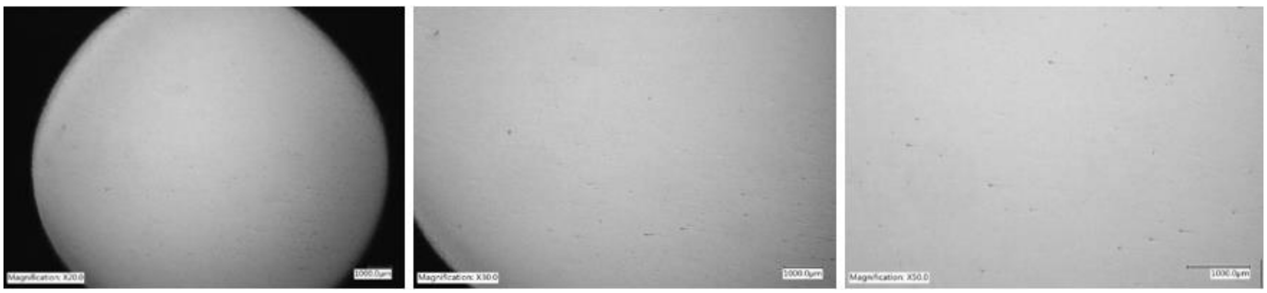
VCI D + superhydrophobic after Exhaustion VIA test, Grade 4.

Figure 7: Comparison of VCI D corrosion behaviors using different coating after VIA test.

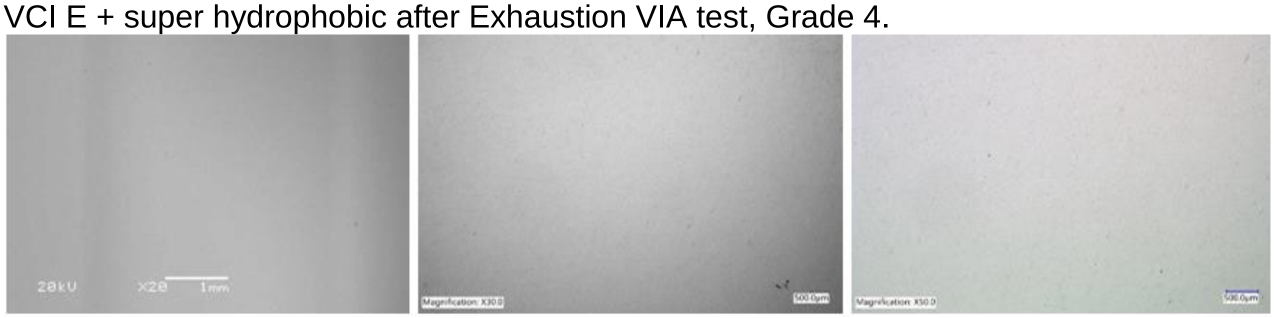


VCI E after VIA tests, Grade 4.

VCI E after Exhaustion. Grade 3.



VCI E + super hydrophobic after Exhaustion VIA test, Grade 4.



VCI E steel samples wrapped with paper during exhaustion followed by the VIA tests, grade 4

Figure 8: Comparison of VCI E corrosion behaviors using different coating after VIA test

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