

A Trade-off Based Assessment Study on Possible Coating Alternatives for Armored Combat Vehicles

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ABSTRACT

Armored combat vehicles often encounter harsh service conditions, especially in environments like tropical weather, which in turn necessitates high corrosion resistance regarding durability and/or reliability requirements. Consequently, several solutions are employed to prevent corrosion, beginning from the design phase and extending to maintenance procedures. In many cases, appropriate coating processes are utilized during the design activities, and they can be complemented by specialized painting processes like Chemical Agent Resistant Coating. The selection of suitable coating systems depends on various parameters, including the material of the workpiece, design requirements, and cost-effectiveness. In this regard, the armored combat vehicle industry possesses a distinctive character due to its widespread use of high-strength materials. This reality highlights the phenomenon of hydrogen embrittlement, which can occur due to electroplating or environmental corrosion. If the dissolved hydrogen in the workpiece material reaches a certain threshold, it significantly impairs overall mechanical performance and leads to sudden catastrophic failures. This study aims to compare different coating alternatives in terms of their impact on hydrogen embrittlement in high-strength materials used in armored combat vehicles. To achieve this goal, samples of high-strength steel substrates coated with various alternatives were examined for their susceptibility to hydrogen embrittlement following ASTM F519 standards. To understand the effect of environmental corrosion on hydrogen embrittlement, advanced tests were designed based on preliminary results obtained from ASTM F519 tests, including salt spray tests according to ASTM B117.

Keywords: Corrosion prevention, hydrogen embrittlement, coating, sustained load test

1.0 INTRODUCTION

In terms of environmental and economic aspects or degradation of mechanical properties, corrosion has to be overcome or at least prevented for complete engineering component (see Figure 1). In addition to the potential problems in service life, corrosion can also cause extra maintenance or repair costs of nearly 3% of the total worldwide GDP (Urbahs et al., 2018). In defense industry, where a reasonable number of parts may have mission-critical functional applications, any premature failure due to corrosion and/or hydrogen embrittlement could lead to catastrophic failure/dramatic results (see Figure 2). In most cases, corrosion itself can be a source of free hydrogen, and the coatings used to prevent corrosion can trigger hydrogen embrittlement (HE) (Kamoutsi et al., 2006, Ibrahim, 2017, Sriraman et al., 2013). Particularly in some mechanical components such as transmission gear systems or compression connections, applying paint is nearly impossible due to the desired mechanical tolerances and clearances. In such cases, coating and/or electroplating processes come into play.



Figure 1: Corroded component from an underbody system of an armored combat vehicle.



Figure 2: A fractured spring of a component due to hydrogen embrittlement.

The procedures for sacrificial metal coatings, such as zinc (Zn) or cadmium (Cd), used to protect steel components from corrosion, are electroplating processes. Cadmium, primarily employed in the defense and aerospace sectors, has faced limitations on its use in recent years due to regulations stemming from its cyanide-based carcinogenic properties (Güleken, 2019). These disadvantages have created a necessary space for the development of more environmentally friendly Zn-based coatings. Similar to Cd coating, Zn alloy coatings, which are more commonly preferred in the automotive sector, are applied to steel through an electrodeposition process, and provide anodic corrosion protection through sacrificial corrosion protection (Sriraman et al., 2013). While these coatings extend the corrosion life of the applied metal surface, they can still lead to delayed hydrogen embrittlement failure when not properly supported by appropriate pre/post-processing steps. Evaluating these coatings should include considerations such as corrosion resistance, sufficient wear resistance to withstand torque during compression processes, and the potential for the coating processes to render the substrate material susceptible to hydrogen embrittlement. Additionally, the coating should exhibit good friction resistance during compression processes.

The phenomenon of HE, which results from hydrogen ingress-related issues, can be categorized into two categories: environmental and internal hydrogen embrittlement. Environmental hydrogen embrittlement (EHE) is, as the name suggests, the type that occurs due to the exposure of metal materials to hydrogen because of the surrounding environmental conditions. Examples of cases falling into this category include HE resulting from corrosion or hydrogen-rich environments. Sacrificial cathodic protection coatings generate hydrogen through corrosion. When the coating is damaged, cracked, or partially lost due to corrosion, any openings on the surface allow this generated hydrogen to be absorbed. Internal hydrogen embrittlement (IHE), on the other hand, results from residual hydrogen generated during production processes, including surface cleaning and preparation steps such as pickling, especially in coating processes. The reason why coating-related processes are primary contributors to IHE is that these stages are typically the final

production steps and act as a barrier preventing hydrogen, which tends to diffuse from within the steel to the outside. It's important to note that the hydrogen input derived from corrosive environments, which is considered the cause of EHE, can be categorized in the same category as IHE under the name "electrochemical hydrogen embrittlement" which includes processes such as electroplating and electrochemical hydrogen embrittlement (Brahimi, 2014).

When we examine the corrosion mechanism, it becomes clear that the oxidation of the metal occurs in a corrosive environment. This corrosive solution can be considered as an aggressive environment and may exist locally in environmental corrosion conditions or processes such as acid pickling or electroplating. Both coated and uncoated metals share the same corrosion mechanism, but the key difference lies in whether the oxidized material is the metal itself or not (see Eqn (1) and Eqn (2)).



or



When reactions are expressed as half-cell reactions, it is understood that the metal surface where corrosion occurs serves as the anode for the anodic reaction. Metal atoms lose electrons and undergo oxidation, forming metal ions. Eqn (3) provides a general overview of the anodic reaction in metal corrosion.



Typically, reduction reactions occur at the cathode, which usually represents a different region of the metal surface. The primary cathodic reaction is the reduction reaction of oxygen, as expressed in Eqn (4).



When dissolved oxygen is depleted, hydrogen ions are reduced, and hydrogen atoms capable of entering the metal are produced, which will subsequently exist molecularly.



As well-documented in the literature, it is known that high-strength steels are more susceptible to both IHE and EHE. However, in the armored combat vehicle industry, particularly in critical applications like fasteners, where the aim is to produce products that can withstand harsh environmental conditions, the use of high-strength steel becomes necessary. To protect these materials from failures related to HE processes such as pre-coating stress relief and post-plating heat treatment, referred to as baking, can be applied. When applied correctly, the baking process can make the hydrogen diffusing into the metal less harmful by either removing it from the lattice or forcing it to migrate into trap regions within the metal. Key factors affecting the effectiveness of the baking process, such as temperature, duration, and the coating's permeability, should be considered when determining appropriate baking conditions. To achieve the desired results, it is important to minimize the time between coating and baking, thereby maximizing the removal of hydrogen (Brahimi, 2007).

The issue of HE arising from coating processes is often overlooked due to its complex and challenging nature to fully comprehend. This situation has been reflected in industry standards, indicating that the prevention of HE may not have been adequately addressed. It can also be argued that existing preventive approaches are not consistently reliable. Some preventive measures include banning high-risk coating processes or restricting steps like pickling and promoting the use of inhibitory acids. In cases not covered by these approaches, the baking process becomes essential, and it's necessary to determine whether it is

sufficient to prevent HE. However, the specified baking times may not always deliver the desired results without thorough testing and verification. Therefore, comprehensive HE tests are crucial to confirm the effectiveness of the baking process in situations where it is applied.

From this perspective, this study will review the most commonly used coating alternatives in armored combat vehicles and evaluate their susceptibility to HE when subjected to post-heat treatment (baking). To detect and quantitatively assess this susceptibility, the Sustained Load Test (SLT) method defined in the ASTM F519 standard will be employed. In the initial set of tests, notch bar specimens with coatings commonly used in armored vehicles, including zinc phosphate (Zn-phosphate), zinc nickel (Zn-Ni), and cadmium (Cd) coatings, were conducted to directly detect HE attributed to the coatings. Following the preliminary results, to gain insights into HE that may occur due to corrosion, the prominent coatings identified in the SLT were applied to new specimens. After undergoing the Salt Spray Test (SST) as described in ASTM B117 standard, the HE susceptibility of these coatings was re-evaluated using ASTM F519 on the new specimens.

Section 2 of this paper describes the methodology followed, including the coating materials, coating processes, and test procedures. Section 3 presents and discusses the results by comparing the data from the tests. Section 4 summarizes the findings, and lessons learned, and outlines the areas of future study.

2.0 EXPERIMENTAL PROCEDURE

2.1 Methodology

ASTM F519, officially titled "Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments," is a defined test method used primarily to determine the susceptibility of a coating or material to HE. This test method is generally conducted under "Sustained Load" conditions. The standard for this test specifically defines the type of specimen to be used, emphasizing the importance of obtaining specimens that meet the defined criteria for a successful test. Throughout the test duration, the specimens are subjected to a sustained tensile load. This load, which will be applied for 200 hours, corresponds to 75% of the ultimate tensile strength (UTS) of uncoated notched specimens (see Eqn (6)). The evaluation of this test operates on a pass/fail basis and does not provide quantitative results. If no cracking occurs during the test, and the full 200-hour duration is completed, the test is considered a pass. However, if any cracking does occur, the test is deemed a failure, suggesting that the material or coating may be susceptible to hydrogen embrittlement and may not be suitable for high-strength applications (ASTM F 519 Standard).

$$\text{Sustained Tensile Load} = 0.75 \times \text{UTS of Uncoated Specimens} \quad (6)$$

ASTM F519 specifies a minimum of 4 specimens for the SLT. When evaluating specimens consecutively bonded according to ASTM F519:

- If none of the specimens break, it is assumed that there is no brittleness in the coating process or material.
- If one of the specimens breaks, the broken specimen is removed from the test, and the remaining specimens continue the test to complete 200 hours of loading. After completing 200 hours, if there is no further breakage, the load is increased in 5% increments every two hours until it reaches 90%. If there is no breakage within 2 hours after reaching 90% of the increased load, it is assumed that there is no brittleness.
- If two or more specimens break within the 200-hour test period, it is assumed that there is brittleness in the coating process or material.

Experiments have been designed to understand the ability of the baking process to reduce HE sensitivity in specimens. Priority coatings to be applied to the specimens have been determined. Sets of specimens were subjected to baking and non-baking processes, and SLT was performed. This allows for a concrete demonstration of the presence of HE and the effectiveness of the baking process as a preventive method.

Another stage of the test procedure in this study is aimed at substantiating the theory that hydrogen ingress can occur not only from the coating or processes applied but also from environmental sources due to corrosion. The Salt Spray Test described by the ASTM B117 standard was applied to baked coatings. Test duration is determined by specific coating standards that define test methods to characterize the properties of each coating. Subsequently, SLT will be conducted to observe whether fractures occur due to hydrogen ingress.

2.2 Substrate Materials

The substrate specimens described by ASTM F519 were used in this study: Coated and uncoated tensile notch bar AISI 4340 steel specimens with the composition given in Table 1 were used to examine the susceptibility to HE. The samples with the dimension given in Figure 3 were heat treated to 51-53 Rockwell C (HRC).

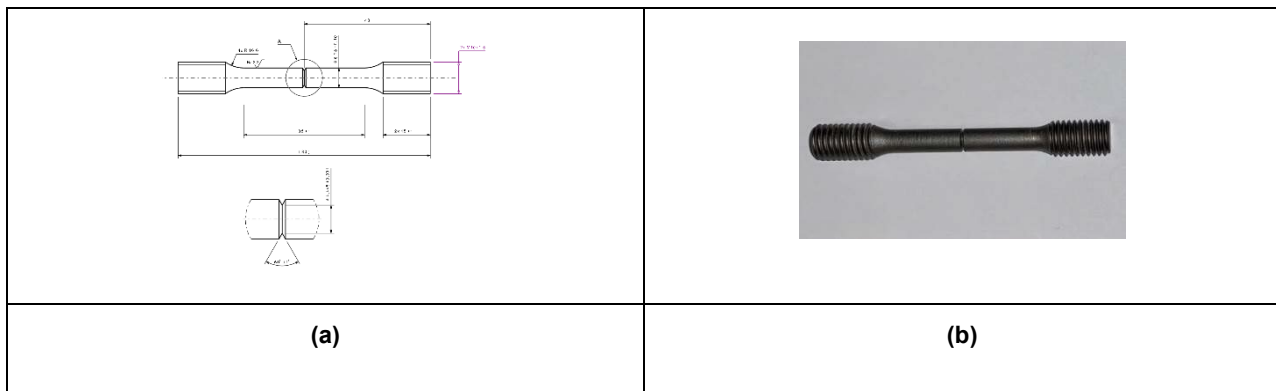


Figure 3: The specimen geometry in ASTM F519: a) Designed in the metric system, b) Test specimen.

Table 1: Spectral analysis of the specimen used in this study.

Fe	C	Si	Mn	P	S
95.690	0.427	0.231	0.648	0.012	0.017
Cr	Mo	Ni	Al	Co	Cu
0.671	0.201	1.900	0.020	0.034	0.052
Nb	Ti	V	W	Pb	Sn
0.021	0.014	0.014	0.026	0.001	0.010
Zn	B	Zr	Ta		
0.002	0.001	0.008	0.002		

2.3 Coating Processes

The sample substrates were coated by the electrodeposition of cadmium (Cd), zinc-nickel (Zn-Ni) and zinc phosphate (Zn-Phosphate) with respect to SAE-AMS-QQ-P-416, ASTM B841, MIL-DTL-16232 standards. As seen in Table 2, the coating thickness of the notch bars to be used in the ASTM F519 tests was maintained between 9 – 15 μm . Samples were baked at 160°C for 5 hours before coating and 23 hours at 160°C after coating.

Table 2: Substrate properties for each type of coating.

Material	Coating Thickness, μm
Zn-Phosphate, ASTM F519 1a.1	9.53 ± 0.48
Zn-Ni, ASTM F519 1a.1	14.87 ± 2.33
Cd, ASTM F519 1a.1	$11,19 \pm 0,25$

2.4 Mechanical Tests

The tests designed for the experimental studies started after the properties of the samples belonging to the tests were verified. Before starting the mechanical tests, the spacers of the tensile test (TT) set-up specially designed in compliance with ASTM F519 were produced to be compatible with the Instron 600LX (England) machine. Thanks to this design, both tensile tests and sustained loading tests (SLT) were performed with the same device and design tools. As the first step of ASTM F519 tests, a simple tensile test was performed with uncoated notch bars to calculate the loading described in the standard. In addition, the tensile test was also performed with coated samples to observe the direct effect of the coating on the mechanical response, although ASTM F519 does not provide a directive in this regard. As stated in the standard, 75% of the tensile test results obtained from the uncoated samples were preloaded for 200 hours on the apparatus shown in Figure 4.

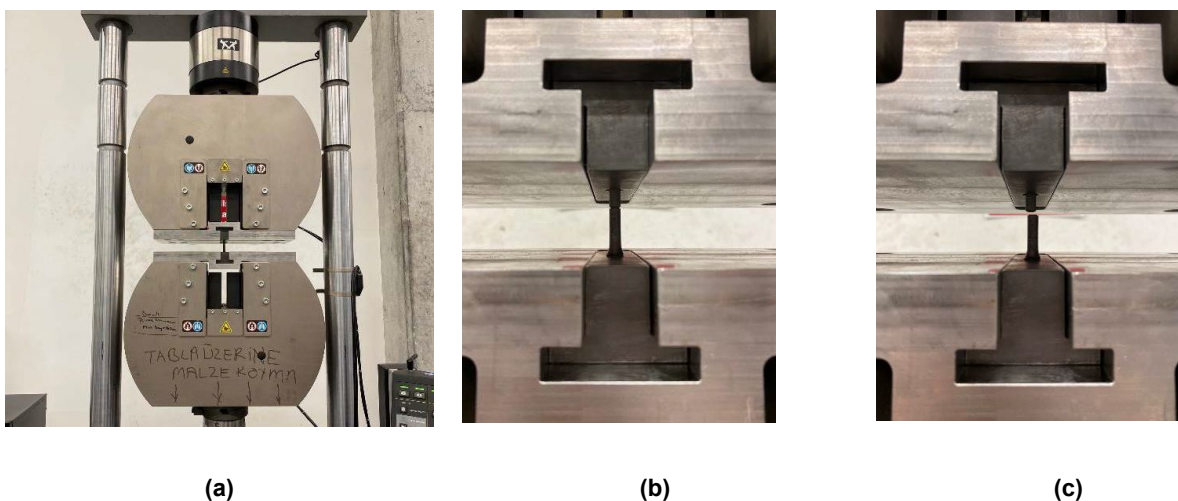


Figure 4: Tensile test: (a) Machine set-up, (b) Before fracture, (c) Fractured specimen of uni-axial tensile test.

2.5 Salt Spray Tests

Then the samples were tested on the Q-Fog SSP-1100 (USA) machine conforming to the ASTM B117 standard for salt spray test (SST). As stated in the standard, the environment during SST consists of 5% (± 0.50) NaCl and 95% water, and the ambient temperature is 35 °C. Figure 5 shows how the test specimens are positioned inside the SST apparatus during the test, and Table 3 indicates the specified test durations in the coating standards for ASTM B117 Salt Spray Test.

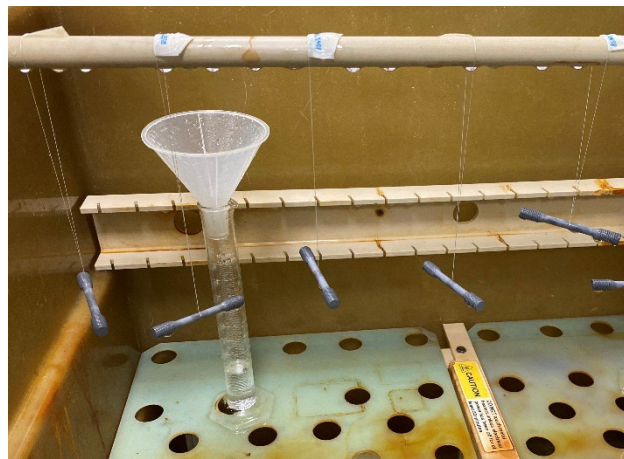


Figure 5: Specimens at the salt spray test set-up.

Table 3: Salt Spray Test requirements for selected coatings according to ASTM B117.

Material	Coating Standard	Requirement
Zn-Phosphate coated specimen	MIL-DTL-16232	24 hours
Cd coated specimen	SAE-AMS-QQ-P-416	96 hours
Zn-Ni coated specimen	ASTM B841	300 hours

3.0 RESULTS

3.1 Sustained Load Test (ASTM F519)

As the first step of the study, a simple tensile test was applied to the uncoated test specimens for the loading value defined in ASTM F519. When the values noted separately for each sample were evaluated, one of the three samples tested was out of the value range, so it was not included in the calculation in order to obtain a more consistent result. The load value to be used for ASTM F519 tests is 23.87 kN when 75% calculation is made over the final value obtained by averaging the test results (see Table 4).

Table 4: Data of tensile tests performed for ASTM F519 test and final load value.

Material	Load (kN)
	24.27*
Uncoated, ASTM F519 1a.1	32.65
	31.01
MEAN	31.83 ± 0.82
Calculated value for ASTM F519 (%75 of uncoated TT results)	23.87

*This value is out of range.

The direct effect of coatings on the mechanical response could be readily observed in tensile tests performed to obtain a prediction about the effect of coatings on brittleness. This finding was in line with the current literature data since it is a well-known fact that different coatings (platings) yield different amounts of hydrogen diffusion into the material, which leads to the unpredictable behavior of the coatings (Aiello et al., 2023, Shen et al., 2019).

Likewise, Table 5 presents the outcomes of tensile tests conducted to assess the influence of pre-firing and post-firing coating applications. It is evident that, in the case of each specific coating type, the tensile test results indicate an appreciable increase in resistance for samples subjected to pre-baking at 160°C for 5 hours and post-baking at 160°C for 23 hours subsequent to the coating application. In this way, a meaningful prediction is obtained about the fractures caused by the hydrogen input.

Table 5: Tensile test data for hydrogen baking and non-hydrogen baking coated ASTM F519 test specimens.

Material	Processes	Load (kN)
Zn-Phosphate, ASTM F519 1a.1	-	32.59
	160°C 5h/160°C 23h	38.46
Zn-Ni, ASTM F519 1a.1	-	21.45
	160°C 5h/160°C 23h	29.74
Cd, ASTM F519 1a.1	-	26.32
	160°C 5h/160°C 23h	29.72

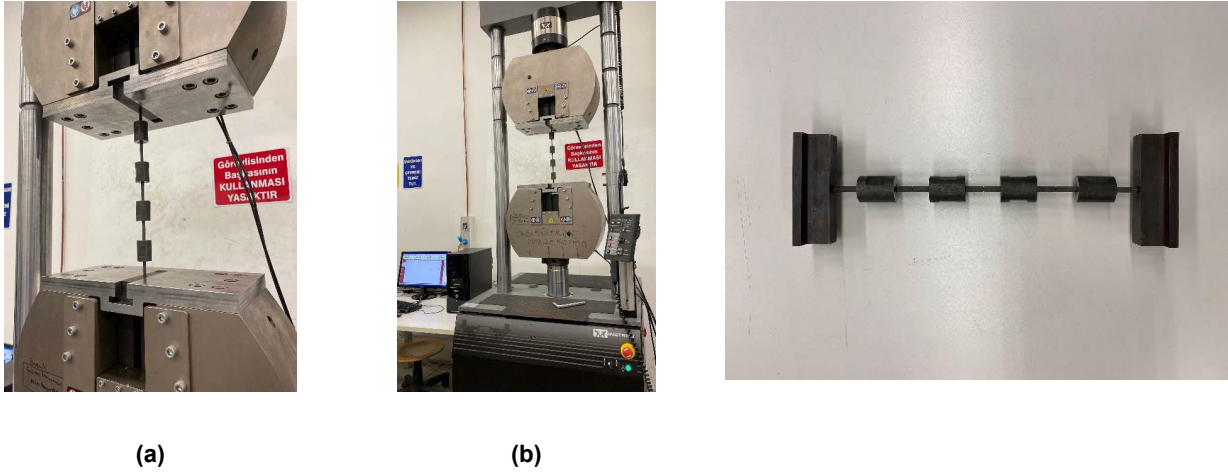


Figure 6: Sustained Load Test: (a) Machine set-up, (b) Specimens in apparatus design.

Following the foresights gained in this direction, ASTM F519 sustained load tests designed over 200 hours were carried out. The minimum four specimens rule recommended by the standard was applied with five specimens according to the FNSS internal requirements (see Figure 6). The reason for this is to increase the probability and ensure more accurate test results due to the excess number of samples during the test period. Since ASTM F519 tests are based on an evaluation method as passed/failed, there is no obstacle or non-standard point in this direction. The results of the tests performed with 23.87 kN loading in compliance with the standard are shown in Table 6.

Table 6: ASTM F519 1a.1 test results of AISI 4340 steel with different coatings.

Material	Condition	Result
Zn-Phosphate	160°C 5h, Coating	Fracture
	160°C 5h, Coating, 160°C 23h	No fracture
Zn-Ni	160°C 5h, Coating	Fracture
	160°C 5h, Coating, 160°C 23h	No fracture
Cd	160°C 5h, Coating	Fracture
	160°C 5h, Coating, 160°C 23h	Fracture

As shown in Table 6, all the coated samples without baking failed according to ASTM F519 tests. On the other hand contrary to Cd coated samples, baking of the Zn-phosphate and Zn-Ni coated samples at 160°C for 23 hours after coating reduced the susceptibility to HE. Hence, the prediction that hydrogen input occurs during the coating processes is confirmed. Zinc-phosphate coatings are inherently non-electrolytic, and exhibit a relatively reduced susceptibility to HE when compared to alternative coating methods

(Brahimi, 2014). This phenomenon is substantiated when evaluating the fracture resistance of specimens in the context of ASTM F519 sustained load tests. Hence the study showed that the baking process at 160°C for 23 hours after Zn-Ni and Zn-Phosphate coating reduced the HE effect. Furthermore, several metallographic inspections are carried out on the fractured samples to observe the microstructure and possible fracture mechanism. As a first attempt, after completing the metallographic sample preparation process, optical microscope (OM) images are taken at distinct magnifications. In Figure 7, a sample image is given for x100 magnification where the tempered martensite and retained austenite-dominated microstructure could be observed. In addition, scanning electron microscope (SEM) analysis on the fractured surface possessed a mixed-type fractographic character which consists of both ductile, brittle, and intergranular type fracture modes to some extent. In Figure 8, some voids (evidence of some ductility in the material, even it is insubstantial and intergranular character are detected.

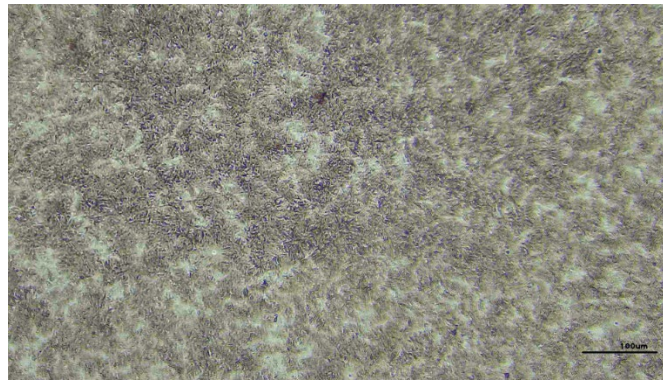


Figure 7: OM examination of the Zn-phosphate coated specimen without baking, where fracture was observed.

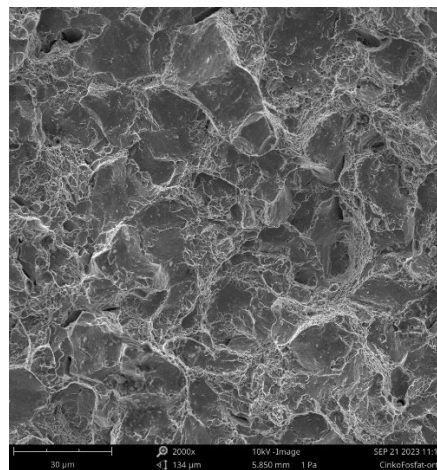


Figure 8: SEM examination of the Zn-phosphate coated specimen without baking, where fracture was observed.

3.2 Salt Spray Test Specimens

No fracture was observed in the Zn-phosphate and Zn-Ni coated AISI 4340 specimens which were baked at 160°C for 23 hours after coating. While it can be stated that the baking process is effective in preventing hydrogen input after the initiation of corrosion in the initial stage of the tests, it is also a well-established fact

in the literature that environmentally sourced hydrogen input occurs more slowly compared to internal hydrogen input. Hence, in order to understand environmental effect, salt spray tests according to ASTM B117 standard were conducted to Zn-phosphate coated and baked specimens (160°C, 23 hours). After the salt spray test, 5 samples were tested according to ASTM F519 sustained load tests. Similar to the Zn-phosphate coated and baked samples, no fracture has been observed in the salt sprayed test samples given in Figure 9.



Figure 9: Zinc-phosphate coated AISI 4340 specimen after 24 hour salt spray test.

4.0 CONCLUSIONS

As a result of all the conducted tests, it has been observed that despite cadmium electroplating process, a reliable zinc-nickel and zinc-phosphate coating processes have been developed for high-strength AISI 4340 steels, without deleterious hydrogen embrittlement. Baking treatment at 160°C for 23 hours after the coating process reduced the susceptibility to hydrogen embrittlement in Zn-phosphate and Zn-Ni coated samples. Despite the successful application of the baking process with temperature-time parameters for Zn-phosphate and Zn-Ni coating, further research is needed to determine the effect of baking process parameters for cadmium coatings.

While the success of the baking process may be inferred for IHE (Internal Hydrogen Embrittlement), it is important to note that the Salt Spray Test designed for EHE (Environmental Hydrogen Embrittlement) may not be sufficient. As an initial result, the success of the baking process is concluded, but as part of future work, ASTM F519 tests should be conducted on specimens exposed to more environmental hydrogen ingress after corrosion.

The phenomenon of hydrogen embrittlement is closely related to fasteners; therefore, the correct torque application is important to prevent overloading and underloading of bolts. Another one of the future works is to examine the relationship between different coatings and preload by applying proper torque procedures and calculating the coefficient of friction. Furthermore the detailed examination of microstructure analysis of "face-fractions" will continue in conjunction with ongoing tests.

5.0 ACKNOWLEDGEMENTS

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