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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

CORROSION BEHAVIOUR OF MILD STEEL COATED WITH EPOXY-ZINC OXIDE NANOPARTICLES FILM IN 3.5% WT NaCl SOLUTION

This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering

By

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APPROVAL

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ABSTRAK

Lapisan cat epoksi adalah salutan cat biasa yang digunakan dalam industry kerana sifatnya adalah pelekat yang baik serta penebat haba yang tinggi tetapi ianya mudah menyerap kakisan yang agresif menyebabkan kadar kakisan naik. Penambahan pengisi di dalam sistem salutan cat epoksi digunakan untuk meningkatkan perlindungan dari sistem salutan daripada kakisan. Oleh itu, kajian ini dijalankan untuk menyiasat kelakuan kakisan keluli ringan yang bersalut dengan nanopartikel epoksi-zink oksida. Penyediaan bahan akan melibatkan epoksi resin, pengikat dan zink oksida sebagai pengisi dalam system salutan. Salutan cat nanopartikel epoxy-zink oksida telah digunakan pada substrat keluli ringan dengan menggunakan kaedah berus tangan dan membiarkannya kering selama satu hari dalam suhu bilik. Pengisi nanopartikel zink oksida telah dimasukkan ke dalam sistem salutan cat epoxy dengan rumusan kandungan zink oksida berbeza bermula dengan 0% wt, 5% wt, 10% wt, 15% wt, 20% wt dan 25% wt berat nanopartikel zink oksida .Tindak balas kakisan salutan epoxy-zink okdisa ini ditentukan dengan menggunakan ujian Terbuka Potensi Terbuka (OCP) dan ujian Polarisasi. Penambahan sampel keluli ringan 0% wt dan 5% wt zarah zink oksida dapat memberikan perlindungan yang lebih baik sebagai penghalang terhadap aktiviti kakisan manakala sampel salutan dengan penambahan 10% wt, 15% wt, 20% wt dan 25% wt zarah oksida zarah tidak dapat memberikan penghalang kakisan yang tepat. Sampel dengan penambahan zat oksida 25% wt zink menunjukkan aktiviti kakisan terburuk dan telah dibuktikan oleh pemerhatian makroskopik dan mikroskopik selepas semua sampel telah direndam dalam 3.5% wt larutan NaCl selama 30 hari. Kedua-dua ujian OCP dan Polarisasi yang dijalankan juga menunjukkan bahawa penambahan 25% wt zink oksida partikel ke dalam sistem salutan epoksi tidak dapat memberikan penghalang kakisan yang lebih baik dan menyebabkan substrat keluli ringan untuk menghakis.

ABSTRACT

The epoxy paint coating is the common paint coating used in industries because of its properties are high thermal and good adhesion but its permeable to aggressive agent caused the corrosion rate increase.Addition of filler inside the epoxy paint coating system used toimprove the corrosion protection of the coating system. Thus, this study was conducted to investigate the corrosion behaviour of the mild steel coated with epoxy-zinc oxide nanoparticles. The preparation of the material will involve with epoxy resin, hardener and zinc oxide particles as the filler in coating system. The epoxy-zinc oxide nanoparticles paint coating was applied on the mild steel substrate by using hand brush method and let it cure for one day in room temperature. Zinc oxide nanoparticles filler was added into the epoxy paint coating system with different formulate of zinc oxide content started with 0% wt, 5% wt, 10% wt, 15% wt, 20% wt and 25% wt by weight of zinc oxide nanoparticles. The corrosion behaviour of these epoxy-zinc oxide coating was determined using Open Circuit Potential (OCP) test and Polarization test. The mild steel sample coated addition of 0% wt and 5% wt of zinc oxide particles able to provide better protection as a barrier against the corrosion activities while coating sample with addition of 10% wt, 15% wt, 20% wt and 25% wtof zinc oxide particles are not able to provide the proper corrosion barrier. Sample with addition of 25% wt zinc oxide particles shows the worst corrosion activities and had being proven by both macroscopic and microscopic observation after all samples were immersed in 3.5% wt of NaCl solution for 30 days. Both of OCP and Polarization test conducted also showed that the addition of 25% wt of zinc oxide particles into the epoxy coating system are not able to provide better corrosion barrier and causes the mild steel substrate to corrode.

DEDICATION

To my beloved parents

Sahatim Bin Chu Baharuddin and Laili Binti Harun

To my final year project supervisor

Dr. Mohd Shahadan Bin Mohd Suan

To all my friends and lecturers

for giving me moral support, cooperation, encouragement and also understandings

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LIST OF ABBREVIATIONS AND SYMBOLS

NACE	-	Nation Association of Corrosion Engineers
Fe	-	Iron
H_20	-	Water
UV	-	Ultraviolet
NaCl	-	Sodium Chloride
HCL	-	Hydrochloric acid
OCP	-	Open Circuit Potential
Pt	-	Platinum
E_{corr}	-	Corrosion potential
I_{corr}	-	corrosion current
R_{ct}	-	Coating resistant
Cdl	-	Capacitance double layer
ZnO	-	Zinc oxide
OH^-	-	Hydroxide
SiC		Silica Carbide
Ag/AgCl	-	Silver/silver chloride
μm	-	Micrometer
mm	-	Millimeter
cm	-	Centimeter
%	-	Percentage
Wt%	-	Weight percentage
^0C	-	Celsius temperature

CHAPTER 1

INTRODUCTION

1.1 Background of study

The metal work application had been introduced for a long time in industry sector since British colonization, where the metal work helped to develop the country and many infrastructures are made from metal. Until now the metal is still relevant to use in industry sector because of the structure of metal is hard and high tensile strength. Therefore, metal is appropriate to use in building bridge, producing the vehicles and other that related to metal work. There are many types of metal use in industry but the commonly use is mild steel. Mild steel is not a pure metal which the composition of mild steel is primarily carbon with a low amount of alloy and other elements. Thus, mild steel is widely use in steel frame buildings, machinery parts, cookware and pipelines because the composition of alloy element increase the mechanical properties of mild steel. The characteristics of mild steel that is high tensile strength, malleability, and good conductor of electricity make it favoured to be used in industry. Besides, the cost for mild steel is cheaper compared to other steel and that is the reason why mild steel is being used.

However, the mild steel is poor resistance towards corrosion because of it consists high amount of carbon where the carbon is the cause of corrosion. Corrosion is a serious problem for mild steel where it leads to material properties deteriorate and the physical condition of the mild steel become worst when brownish colour appear on the surface and it is called rusting. So, the mild steel material structure tendency to broken apart or destroyed if rusting is not prevent. In fact, American Galvanizers Association stated that the annual direct cost of metallic corrosion worldwide is about $ 2.2 Trillion USD. This shows that the corrosion not only affecting the physical structure of metallic but it also effect on economy where many industrial facing losses due to corrosion. Thus, the corrosion need to be overcome in order to improve the material properties, increase the safety use of mild steel and help to improve the economy of industry. So, many studies were conducted to research a method on preventive the corrosion and one of the methods is coating painting. From the report of National Association of Corrosion Engineers (NACE), the annual cost study of corrosion and prevention in US was estimated up to $ 276 billion.

Paint coating is a common method use to prevent the corrosion and the cost for paint coating is cheaper compared to other preventive method. This technique is simple and easy to use in industry. There are several paint coating available in market and one of them is epoxy resin coating. Epoxy resin is widely being use in industry such as aircraft, automotive and construction as an adhesive material use to attach the part together or as the coating for the parts surface. Epoxy resin characteristic provide an excellent mechanical properties, good electrical insulating and strong adherence to substrate which help to reduce the corrosion by creating a physical barrier in the material. Nonetheless, the application of paint coating in industry is limited due to poor impact resistance and stress cracking (Conradi et al., 2014). Due to the lack of preventing stress cracking, many studies were conducted to improve epoxy resin properties by adding additive material into epoxy resin.

In this study, the material used to add into epoxy resin is Zinc Oxide (ZnO) nanoparticles. ZnO nanoparticles is an inorganic compound and has being use as additive in numerous material. It possesses great physical and chemical properties such as high chemical stability, high electrochemical coupling coefficient and broad range of radiation absorption (Renjanadevi et al., 2016). The purpose of adding ZnO nanoparticles is to improve the exterior durability of epoxy coating as the current epoxy coating cannot withstand with high exposure of UV. Due to that, the metal surface is exposed to the corrosion environment and the crack is occurred on coating surface (Shalini et al., 2016). Thus, adding ZnO nanoparticles into epoxy helps to resist the corrosion and overcome the stress corrosion cracking.

1.2 Objectives

1. To fabricate epoxy-ZnO nanoparticles as coating material on the mild steel by varying the composition of ZnO weight percentages.
2. To characterize the microstructure and physical properties of epoxy-ZnO coating film.
3. To investigate the corrosion behavior of the mild steel coated with of epoxy/ZnO film.

1.3 Problem statement

Corrosion is a natural process which the process of something such as metal deteriorate because of oxidation which a chemical reaction that created oxides that flake away from the base of the materials (Alhumade et al., 2015). The effect of corrosion cannot be neglect as it can cause a big impact towards technology, safety and economics. Thus, because of the corrosion it leads to a serious problem in industries especially the industries involved with mild steel work application. The corrosion cause the metal surface become rusting and in any longer time the rust will cause the metal to be broken apart or destroyed especially at oil shore place. So, it can endanger the human life if the structural of the mild steel is rusting because the strength of the material is decrease by the corrosion.

There are many mechanism have been reported to prevent corrosion mild steel such as cathodic protection, sacrificial anode and supply of the electrical current where the cathodic protection is the easiest yet the most efficient method (Yuyu et al,.2017). In this mechanism, cathode or the mild steel surface will be protected from any contact against corrosion agents include oxygen, water and oxidation ions. The protective materials used to cover the mild steel must have good adhesion to the surfaces, and able to withstand particular working environmental conditions. Since, the mild steel employed in industrial is always in a large scale, the cathodic protective material should have these properties such as easy to be applied, consumed less energy, and available in low cost. Thus, epoxy film which had all of these properties attracted most attention to be selected as protective materials on the mild steel surface. Epoxy is known as the thermoset material which have excellent resistance against moist water and high temperature environment (Clayton, 1988). It is applicable on many surfaces and easily available at low cost.

However, it still not enough to overcome the crack propagation as it is poor resistance of crack propagation and permeable to aggressive agent which can increased the corrosion rate (Hsieh et al., 2011). There also been reported that, the corrosion of the mild steel can still occurred under the epoxy protective layer because of agglomeration of static ions between conductive and non-conductive surface. Hence, in this work, ZnO nanoparticles which renowned of their semi-conductivity properties was added with several composition into epoxy coating film in order to increase physical properties of the epoxy film and to discharge out the static ions and further improve corrosion protection of mild steel. Then, the exterior durability of coating can be increased due to capability of ZnO to absorb some of wavelength from UV.

1.4 Scope of study

1. Develop mild steel coated with epoxy-ZnO nanoparticles (ZnO wt%: 5, 10, 15, 20 and 25).
2. Characterize the microstructure of the developed film by using optical microscope
3. Investigate the corrosion behaviour of mild steel in solution 3.5wt% NaCl solution by using open circuit potential (OCP) and polarization test.
4. Observing the physical and microstructural of the mild steel surface after the corrosion test by using optical microscope.

CHAPTER 2

LITERATURE REVIEW

2.1 Corrosion

Corrosion can be defined as the gradual natural destruction or deterioration of material (usually metals) by chemical or electrochemical reaction with their environment. Usually metal is easily to corrode especially in applications which involved high temperature, corrosive atmosphere and sea water environment. Common examples of metal corrosion are rusting of irons, tarnishing of silver, dissolution of metals in acid solution and growth of patina in copper. Those examples of metal corrosion can become the serious threads and effect the economic growth of industries. So, the prevention control needs to applied in metal work industries in order to overcome the corrosion plus to save the cost of damage. Based on NACE study, it state the implementing corrosion prevention helps to save the cost damage between $375-$875 billion (USD) (Inspectioneering.com, 2018).

2.1 Basic corrosion mechanism

Other term used to call corrosion is electrochemical process (Perez, 2018).Usually, corrosion get through operation of half cell reaction which oxidation-reduction reaction happened within the substances where the electron is gain by reduction at cathode while oxidation causing the loss of electron to other substances at anode (McCafferty, 2010).This electrochemical process can be investigate through electrochemical cell which consist of two electrodes that are connected to the electrical circuit(Lewis, n.d.)The electrodes usually contact with electrolyte such as acidic solution to initiated the electrochemical process for the metal substances where the oxidation reaction is occurred at anode. Besides, electrolyte also containing positively and negatively charge ion that make the electrolyte as conductive solution where the electrolyte can carry out the ion release from the metal electrode to other. The reaction at anode metal is shown in Equation 2.1

$$Fe\ (s) \rightarrow Fe^{2+}\ (aq) + 2e^- \qquad \text{Equation 2.1}$$

At anodic reaction, the loss of electron leaves positively charged ions at the anode which the electron travel from anode to cathode by electrolyte and it carry the positive current. Thus, the corrosion is occurs at anode as electrode at anode has loss some weight due to loss of electron to other high concentration of ion. Then, the release electron from anode will be received at cathode. The following reaction at cathode is shown in Equation 2.2 and Equation 2.3

$$2H^+(aq) + 2e^- \rightarrow H_2\ (g)\ \text{(in acidic solution)} \qquad \text{Equation 2.2}$$

$$0_2(g) + 2H_20 + 4e^- \rightarrow 4OH^-(aq)\ \text{(in basic solution)} \qquad \text{Equation 2.3}$$

At cathode, the reduction reaction caused the number of oxidation at cathode is reduce. The number of oxidation is difference depend on the type of electrolyte contact with metal. During the electrochemical process, the positive ion in electrolyte will approach at cathode and attach. It will form film layer on cathode surface so it is call electrodeposited.

2.3 Form of corrosion

The corrosion on metallic resulted to the degradation of material property due to electrochemical reaction with surrounding environment. Usually corrosion occurred on metal is visible on the outer layer of metal. Generally, the form of corrosions that are identified on metallic corrosion such as general corrosion, galvanic corrosion, stress cracking corrosion, crevice corrosion, intergranular corrosion, selective leaching corrosion, pitting corrosion and erosion corrosion.

2.3.1 General Corrosion

General corrosion also known as uniform corrosion is refers to its corrosion form on the exposed metal surface where the corrosion is degraded unevenly over the entire surface. The change of general corrosion on metal can be easily foreseen and it doesn't cause a serious thread. The general corrosion is occurs when the surface of material undergo electrochemical process when metal surface is contact with corrosive environment. The anode and cathode for this electrochemical process is determined from the distinction in composition or position between small areas on material surface.

2.3.2 Galvanic Corrosion

Electrochemical reaction between two dissimilar metals which the metals in contact and revealed in the same electrolyte so this form of corrosion is called galvanic corrosion (Schneider *et al.*, 2014). There are many factors that can affect the performance of galvanic corrosion such as temperature, the anode-cathode area and spacing. The potential difference between metals or alloys in molten fluoride salts urged the galvanic corrosion to rapidly corrode. The metal at cathode is called noble as the cathode part is resistance to corrosion while anode metal is an active part where corrosion is take place in the presence of electrolyte (Wang, Liu and Zeng, 2014).

2.3.3 Crevice Corrosion

Crevice corrosion also known as concentration cell corrosion happened because of the environment difference surrounding the metal. This type of corrosion normally occurred within crevices or at shielded surfaces with the presence of stagnant solution. The crevice corrosion is consider harmful due to corrosion can occurred in alloy elements that usually rare to corrode and also happened on the area that slightly visible by naked eye (Rashidi, Alavi-soltani and Asmatulu, 2007).The mechanism of crevice corrosion is happened when the higher concentration of chloride inside crevice (anode) is easily to corrode the metal compared to the outer crevice (cathode) which form ferrous chloride at anode. There are many consideration that affecting crevice corrosion such as type of crevice, geometry for crevice, environmental condition and material composition.

2.3.4 Pitting Corrosion

Pitting corrosion caused the cavities or holes form on the material and it caused a serious damage to the material as shown in Figure 2.1. The damage that caused by pitting corrosion is not easily to detect on the outer surface of metal as the total serious damage occurrence underneath the metal layer. Thus, it conducts the engineering system to failure. Environmental factors such as temperature, humidity, rainfall and airbone influence the presence factors of pitting corrosion. So, pitting corrosion start to occur when the concentration of chloride ion increasing and reach the critical value each time electrolyte layer is drying (Tsutsumi, Nishikata and Tsuru, 2007).

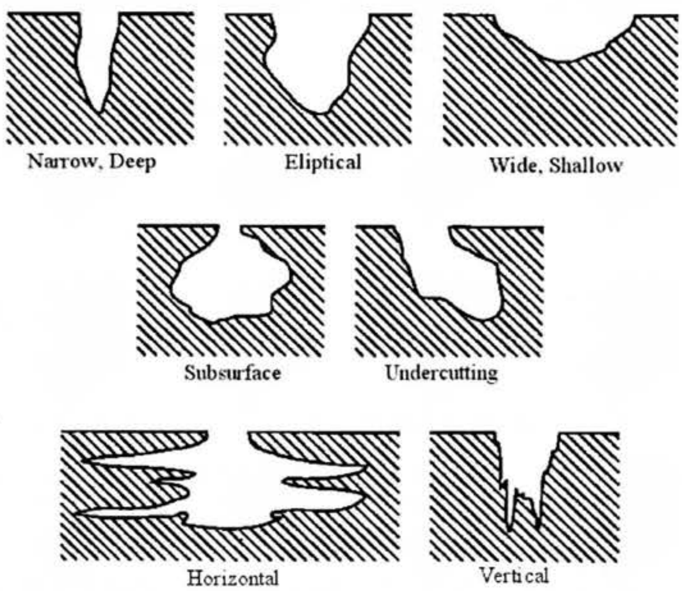

Figure 2.1: ASTM G-46 standard visual chart for rating of pitting corrosion (Corrosion Clinic, 2010).

2.3.5 Intergranular Corrosion

Intergranular corrosion is refers to the corrosion attack occurred along the grain boundaries and adjacent areas due to impurities enhance at grain boundaries while the bulk of material is remain passivation. The cause of intergranular corrosion vulnerability is from non-uniform decomposition at grain boundaries. Non-unifrom decomposition is form as the result of continuous precipitation of anodic phases or from depleting of zones adjacent (Sinyavskii, Ulanova and Kalinin, 2004). The process such as welding, annealing, heat treatment or overheating in service lead to the intergranular corrosion occurred deep into region where those process can produced of microscopic and compositional inhomogeneities.

2.3.6 Erosion Corrosion

Erosion corrosion refers to the acceleration or increasing the rate of degradation on metal because of the relative movement between the corrosive fluid and metal. The substrate of metal is removed from the surface by two forms either in the form of solid corrosion products or in the form of dissolved ion. The factors that affecting the mechanism of erosion corrosion, such as turbulent flow, droplets of liquid in gas flow, and impact of suspended solid particles. The erosion rate is rapidly increasing as the turbulence produce by pitting on the internal surface of tube is increasing and thus it caused a leaking for a pipe made from steel (Nace.org, 2018).

2.3.7 Stress Corrosion Cracking

Stress corrosion cracking (SCC) is a type of corrosion failure where the cracking is forming due to process involving simultaneous action of conjoint corrosion and sustained tensile stress (Practice and Control, n.d.).The cracking is occurred when there are presences of material susceptibility, applied tensile stress and aggressive environment. The basic mechanism of stress corrosion cracking can be identified into three that are active path dissolution, hydrogen embrittlement, and film-induced cleavage. For mechanism of active path