FAILURE ANALYSIS OF CONDUCTIVE PAINT COATING USING ELECTROCHEMISTRY METHOD

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

by

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APPROVAL

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ABSTRACT

This report covers the failure analysis of conductive paint coating by using electrochemistry method. The paint coating used in this analysis is the combination of epoxy resin and carbon black and also polyamide as a hardener. The formulation of pure epoxy paint coating is modified by adding carbon black filler of 5%, 10%, 15% and 20% of its weight. This conductive paint coating is then molded by using plastic pipe to form a pellet. Corrosion test by using Tafel Extrapolation method is conducted in order to determine the corrosion rate of the sample. The analysis methods used in this project research are Scanning Electron Microscopy (SEM) for microstructures observation, Energy Dispersive x-ray Spectroscopy (EDS) for elemental composition and Fourier Transform Infrared spectroscopy (FTIR) which is to characterize the chemical bonding are conducted before and after the corrosion test. Moreover, pH test also conducted to investigate the corrosion behavior in different corrosive environment. As a result, the modified conductive paint coating with 20%CB is noted as a severe corrosion failure compared to the other samples while the pure epoxy paint coating is the one that behave as a good corrosion resistance. It can be proved that the corrosion rate for the modified conductive paint coating with 20%CB is found to be the highest due to the particle size of the carbon black used which is not in powder form. This condition also occurred to another corrosive environment which is in acidic and alkali environment. Furthermore, the surface morphology of degraded samples showed that corrosion failure in form of weak spots and blisters for the 20%CB sample. The epoxy paint coating is suggested to combine with the polyaniline emeraldine salt (PAni-ES) replacing the carbon black filler in order to obtain excellent corrosion resistance of this coating.

ABSTRAK

Laporan ini merangkumi analisis kegagalan salutan cat yang berkonduktif dengan menggunakan kaedah elektrokimia. Salutan cat digunakan dalam analisis ini adalah hasil gabungan resin epoksi dan karbon hitam dan juga poliamida yang merupakan sejenis pengeras kepada salutan ini. Perumusan salutan cat epoksi tulen diubah dengan menambahkan pengisi karbon hitam sebanyak 5%, 10%, 15% dan 20% dari beratnya. Kemudian, salutan cat berkonduktif ini dibentuk menjadi sample palet dengan menggunakan paip plastic. Ujian kakisan dengan menggunakan kaedah *"Tafel Extrapolation"* dijalankan untuk menentukan kadar kakisan bagi sampel tersebut. Kaedah-kaedah menganalisis yang digunakan dalam penyelidikan projek ini adalah seperti *"Scanning Electron Microscopy" (SEM)* iaitu untuk pemerhatian pada mikrostruktur, *"Energy Dispersive x-ray Spectroscopy" (EDS)* untuk mengkaji komposisi unsur yang terbentuk dan *"Fourier Transform Infrared spectroscopy" (FTIR)* bagi mencirikan pengikatan kimia terbentuk di mana ia telah dijalankan sebelum dan selepas ujian kakisan dilakukan. Tambahan lagi, ujian pH juga dijalankan untuk mengkaji sifat kakisan dalam persekitaran menghakis yang berbeza. Sebagai hasil, salutan cat berkonduksi yang diubahsuai dengan penambahan 20% karbon hitam jelas menunjukkan kakisan yang teruk berbanding dengan sampel yang lain sementara cat epoksi tulen bertindak sebagai penghalang pada kakisan. Ia dapat dibuktikan dengan kadar kakisan pada sampel 20% adalah tertinggi berhubung dengan saiz partikel karbon hitam adalah besar iaitu bukan dalam bentuk serbuk. Tambahan pula, morfologi permukaan selepas ia terhakis menunjukkan kegagalannya dalam bentuk tompokan bagi sampel 20%. Salutan cat yang disarankan bersama dengan epoksi adalah *"polyaniline emeraldine salt" (PAni-ES)* bagi menggantikan pengisi karbon hitam untuk mendapatkan penghalang kakisan yang terbaik.

DEDICATION

For my beloved mother and family.

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LIST OF ABBREVATIONS, SYMBOLS & NOMENCLATURE

%	–	percentage
°C	–	degree Celcius
µm	–	micrometer
AES	–	Auger Electron Spectroscopy
Ag-AgCl	–	Silver-Silver Chloride
ASTM	–	American Society for Testing & Materials
ATR	–	Attenuated Total Reflectance
C	–	Carbon
CB	–	Carbon Black
CE	–	Counter Electrode
Cl^-	–	Chloride ion
DGEBA	–	Diglycidyl Ethers of Bisphenol A
DGEBF	–	Diglycidyl Ethers of Bisphenol F
e^-	–	Electron
E_{corr}	–	Corrosion potential
EDS	–	Energy Dispersive X-ray Spectroscopy
EIS	–	Electrochemical Impedance Spectroscopy
FCC	–	Face Centre Cubic
FTIR	–	Fourier Transform Infrared
H^+	–	Hydrogen ions
H_2	–	Hydrogen gas
H_2O	–	Water
HCl	–	Hydrogen Chloride
i_{corr}	–	Corrosion current densities
i_{ox}	–	Oxidation current density
i_{red}	–	Reduction current density
KCl	–	Potassium Chloride
mA	–	mili ampere
N	–	Nitrogen

NaCl	–	Sodium Chloride
NaOH	–	Sodium Hydroxide
O	–	Oxygen
O_2	–	Oxygen gas
OH	–	Hydroxyl
PAni	–	Polyaniline
PAni-EB	–	Polyaniline Emeraldine Base
PAni-ES	–	Polyaniline Emeraldine Salt
PPy	–	Polypyrrole
RE	–	Reference Electrode
SCE	–	Saturated Calomel Electrode
SEM	–	Scanning Electron Microscope
UV	–	Ultra Violet
VOC	–	Volatile Organic Compounds
WE	–	Working Electrode
XPS	–	X-ray Photoelectron Spectroscopy
XRD	–	X-Ray Diffraction
Zn	–	Zinc
Ω	–	Ohm

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The conductive paint coating used is like the combination of epoxy resins and the additional agent which is carbon black. The materials are coated on its surface with paint or some other non-conducting coating. This prevents the electrolyte from reaching the metal surface if the coating is complete. Scratches exposing the metal will corrode with the region under the paint, adjacent to the scratch, to be anodic. Other prevention is called passivation where a metal is coated with another metal such as a tin can. Tin is a metal that rapidly corrodes to form a mono-molecular oxide coating that prevents further corrosion of the tin. The tin prevents the electrolyte from reaching the base metal, usually steel (iron). However, if the tin coating is scratched the iron becomes anodic and the can corrodes rapidly (Anonymous 2008).

Electrochemistry is a branch of chemistry that studies chemical reactions which take place in a solution at the interface of an electron conductor (a metal or a semiconductor) and an ionic conductor (the electrolyte), and which involve electron transfer between the electrode and the electrolyte or species in solution. If a chemical reaction is driven by an external applied voltage, as in electrolysis, or if a voltage is created by a chemical reaction as in a battery, it is an electrochemical reaction. In general, electrochemistry deals with situations where oxidation and reduction reactions are separated in space or time, connected by an external electric circuit to understand each process (Anonymous 2008).

In chemistry, epoxy or polyepoxide is a thermosetting epoxide polymer that cures (polymerizes and cross links) when mixed with a catalyzing agent or "hardener". Most common epoxy resins are produced from a reaction between epichlorohydrin and bisphenol-A. "Two part waterborne epoxy coatings" are used as ambient cure epoxy coatings. These two-part epoxy coatings are developed for heavy duty service on metal substrates and use less energy than heat cured powder coatings. These systems use a more attractive 4:1 by volume mixing ratio. The coating dries quickly providing a tough, UV resistant, protective coating with excellent ultimate hardness and abrasion resistance. They are designed for rapid dry protective coating applications. Ambient cure 2 part waterborne epoxy coatings provide excellent physical properties in exterior applications. These products have excellent adhesion to various metal substrates. Their low Volatile Organic Compounds (VOC) and water clean up makes them a natural choice for factory cast iron, cast steel, cast aluminum applications and reduces exposure and flammability issues associated with solvent borne coatings (Anonymous 2008).

Carbon black is a material produced by the incomplete combustion of heavy petroleum products such as FCC tar, coal tar, ethylene cracking tar, and a small amount from vegetable oil. Carbon black is a form of amorphous carbon that has a high surface area to volume ratio, and as such it is one of the first nano materials to find common use, although its surface area to volume ratio is low compared to activated carbon. It is similar to soot but with a much higher surface area to volume ratio. Carbon black is used as a pigment and reinforcement in rubber and plastic products. Short-term exposure to high concentrations of carbon black dust may produce discomfort to the upper respiratory tract, through mechanical irritation (Anonymous 2008).

1.2 Problem Statement

There are many types of paint coating failures for which the coatings or corrosion engineer has little or no control over. These types of failures are related to the formulation of the coating itself. If the coating system that is selected is formulated inadequately, the coating will most likely fail regardless of all efforts made in an optimal application. These formulation related failures occur as a result of the ingredients used and their formulation in the paint coating. The majority of paint coating failures can be attributed to many primary causes. All the causes of paint coating failure will be investigated depending on the material selected and their formulation in the paint coating.

Ordinary polymer paint coating that usually used is not conductive. Thus, the paint coating used cannot be transmitted current and will own a lower conductivity value. This conductive paint coating may also have big problem like it has corrosion inhibitor material which have inability to inhibit corrosion to the steel materials. Some paints also can produce galvanic action like zinc rich paints (Orlikowski *et al.*, 2002).

The paint produced also has its own special system and will present a certain property. This property will indicate the changes of conductivity value, electrochemistry value and other values. The filler that included in the polymers also indicates different nature paint polymer. The carbon black used depends on their particle size, types of filler material, and shapes of filler size either in the particles form, pieces or others (Orlikowski *et al.*, 2002).

Failure paint coating frequently occurs in paint production such as micro fraction, lump up and many more that always make troubles in paints. However, just a few information and research carried out to study the failure analysis of conductive paint coating using electrochemistry method such as potential time and impedance spectroscopy, yet this method have successfully proved the failure paint coating at the right time. The information found from this method will be analyzed and studied again based on the failure happens.

1.3 Objective

i. To analyze the failure of the carbon black/epoxy resin conductive paint coating such as its microstructure, size, morphology, element composition and chemical bonding.
ii. To investigate the failure behavior/characteristics of conductive paint coating of carbon black/epoxy resin by using the electrochemistry method.

1.4 Scope of Study

To investigate a failure, and analyze the conditions that promoted the failure, important information must be collected on the failed paint coating. The conductive paint coating involved is the combination of epoxy resins and the additional agent which is carbon black. The sample is undergoing the corrosion testing and pH testing by using electrochemistry method which is Tafel Extrapolating in order to determine the corrosion rate of the paint coating. The morphology of the testing sample is then observed by using Scanning Electron Microscopy (SEM) while the chemical bonding observation is done by Fourier Transform Infrared spectroscopy (FTIR) before and after corrosion test in order to differentiate between them. This research also responsible to make recommendations for reducing the failure and suggests possible improvements of the coating analysis.

CHAPTER 2

LITERATURE REVIEW

2.1 Failure Analysis

Failure analysis is the process of collecting and analyzing data to determine the cause of a failure and how to prevent it from recurring. It is an important discipline in many branches of manufacturing industry, where it is a vital tool used in the development of new products and for the improvement of existing products (Bayer *et al*., 2004).

Coating failures can occur for dozens of reasons, although they are typically a result of poor application, a defective coating, or an inadequate specification. A determination of the fundamental causes behind coating failures is critical. Not only does this help in assigning financial responsibility, but knowing how a coating has failed is often the first step in planning how to fix it. To investigate a failure, and analyze the conditions that promoted the failure, important information must be collected on the failed paint coating. The conditions that promoted the failure are essential in identifying the underlying factors that may have initiated the failure. Other elements that may not be readily acknowledged in failure analysis, yet are no less important, are common sense, a critical and unbiased mode of thinking, experience, knowledge, and experimental observation (Bayer *et al*., 2004).

There is a wide variety of testing methods currently available for failure analysis of paints and coatings. Sophisticated and highly calibrated laboratory equipment can detect the slightest imperfections on a specimen, and accurately identify the inherent characteristics. A chemical analysis of the paint or coating, as well as the substrate and corrosion products is usually the next step. Chemical analysis techniques typically used in the laboratory for paint and coating failure analysis are Fourier

Transform Infrared spectroscopy (FTIR) for organic functional group analysis, Scanning Electron Microscopy (SEM) with associated Energy Dispersive X-ray Spectroscopy (EDS) for elemental analysis, Auger Electron Spectroscopy (AES) and X-Ray Diffraction (XRD) (Bayer *et al*., 2004).

2.2 Paint Coating

Coating is a covering that is applied to an object. The aim of applying coatings is to improve surface properties of a bulk material usually referred to as a substrate. One can improve amongst others appearance, adhesion, wetability, corrosion resistance, wear resistance, scratch resistance, etc. They may be applied as liquids, gases or solids (Anonymous 2008).

Coating protection is one of the most popular methods of anticorrosion protection of reinforced concrete structures. In this case the aim of the coating is to ensure a tight barrier from access of moisture and diffusion of aggressive ions such as chlorides, sulphates, nitrates to the porous structure of concrete. Protective coatings also ensure protection from access of aggressive gases such as carbon dioxide, sulphur dioxide etc. Due to direct contact with the surrounding environment protective coatings applied on concrete surfaces should be characterized by special physical and chemical properties such as resistance to water and moisture, gases present in the atmosphere, water vapor, ultraviolet light and erosion. Most of the presented requirements are fulfilled by coatings based on epoxide resin binder. Investigations performed by Flounders et al. indicate, however, that the epoxide coating applied as anticorrosion protection of concrete should be made up of many layers with application of differentiated chemical and physical properties of each layer. Application of protective coatings directly on reinforcing bars is another form of anticorrosion protection of reinforced concrete (Orlikowski *et al.,* 2002).

Investigations of Kilareski and Walter confirm that this type of solution is the most effective form of protection of newly constructed bridge structures. Due to difficulties in painting of openwork reinforcement, reinforcing bars are painted with powder paints. Application of coating protection does not always ensure full