

Corrosion Performance of Epoxy-Cu-Zn-Al₂O₃ Nanocomposite as Coating for Welded Steel

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A proposal submitted in fulfilment of the requirements for the degree of Bachelor of Applied Science (Material Technology) with Honors

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DECLARATION

I declare that this thesis entitled "Corrosion Performance of epoxy-Cu-Zn-Al₂O₃ Nanocomposite as Coating on Welded Steel" is the results of my own research except as cited in the references.

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Corrosion Performance of Epoxy-Cu-Zn-Al₂O₃ Nanocomposite as Coating for Welded Steel

ABSTRACT

Applying an epoxy coating can provide corrosion protection to welded steel. The use of an epoxy-composite filler coating is a promising solution that may facilitate homogenous dispersion over the surface of a welded joint by giving long-term protection and preventing microcrack problems from occurring. The epoxy-Cu-Zn-Al₂O₃ coating and its corrosion performance on welded steel are developed and characterized in this work. The aim of this study is to evaluate the effectiveness of epoxy coating added Cu-Zn-Al₂O₃ powder at different compositions (0.5wt%, 0.7wt%, 1.0wt%, and 2.0wt%). Cu-Zn-Al₂O₃ reinforcement was milled at 300 rpm for 40 h, then combined with epoxy to create a film that was applied on the welded steel surface. The as-milled Cu-Zn-Al₂O₃ and epoxy-Cu-Zn-Al₂O₃ nanocomposite films were characterized for their phase identification, morphology, and functional group. An immersion test was carried out for 30 days for all the coated welded steel. The study indicated that the Cu-Zn-Al₂O₃ nanocomposite powder undergoes structural and morphological changes during the milling process. The particles are homogenously distributed in the epoxy and play an important role in changing its structure, shape, and functional group. The nanocomposite with 2.0 wt% Cu-Zn-Al₂O₃ in epoxy has the highest hardness that is 50.2H and is suitable for a welded steel protective coating due to its hardening effect. The higher amount of Cu-Zn-Al₂O₃ improves the hardenability of the reinforced phase and contributes to slightly reduced weight loss during immersion in NaOH.

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Prestasi Kakisan Nanokomposit Epoksi-Cu-Zn-Al₂O₃ sebagai Salutan untuk Keluli Terkimpal

ABSTRAK

Aplikasi salutan epoksi boleh memberikan perlindungan kakisan kepada keluli terkimpal. Penggunaan salutan pengisi komposit epoksi adalah penyelesaian yang menjanjikan kemudahan penyebaran homogen ke atas permukaan sambungan terkimpal dengan memberikan perlindungan jangka panjang dan menghalang masalah retakan mikro daripada berlaku. Salutan epoksi-Cu-Zn-Al₂O₃ dan prestasi kakisannya pada keluli dikimpal dibangunkan dan dicirikan dalam kerja ini. Matlamat kajian ini adalah untuk menilai keberkesanan salutan epoksi yang ditambah serbuk Cu-Zn-Al₂O₃ pada komposisi yang berbeza (0.5wt%, 0.7wt%, 1.0wt%, dan 2.0wt%). Pengisi Cu-Zn-Al₂O₃ dikisar pada 300 rpm selama 40 jam, kemudian digabungkan dengan epoksi untuk menghasilkan filem yang digunakan pada permukaan keluli terkimpal. Filem nanokomposit Cu-Zn-Al₂O₃ terkisar dan epoksi- Cu-Zn-Al₂O₃ telah dicirikan untuk pengenalpastian fasa, morfologi dan kumpulan berfungsi. Ujian rendaman telah dijalankan selama 30 hari untuk semua keluli yang dikimpal bersalut. Kajian menunjukkan serbuk nanokomposit Cu-Zn-Al₂O₃ mengalami perubahan struktur dan morfologi semasa proses pengisaran. Partikel ini disebarkan secara homogen dalam epoksi dan memainkan peranan penting dalam mengubah struktur, bentuk dan kumpulan berfungsinya. Nanokomposit dengan 2.0 wt% Cu-Zn-Al₂O₃ dalam epoksi mempunyai kekerasan tertinggi iaitu 50.2H dan sesuai untuk salutan pelindung keluli yang dikimpal kerana kesan pengerasannya. Jumlah Cu-Zn-Al₂O₃ yang lebih tinggi meningkatkan kebolehkerasan fasa tetulang dan menyumbang kepada pengurangan sedikit penurunan berat semasa rendaman dalam NaOH.

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LIST OF ABBREVIATIONS

Cu Copper

Zn Zinc

Cu-Zn Copper-Zinc

Al₂O₃ Alumina

Al Aluminum

MEKP Methyl Matrix Nanocomposite

WH Williamson Hall

XRD X-Ray Diffraction

SEM Scanning Electron Microscope

FTIR Fourier-Transform Infrared Spectroscopy

NaOH Sodium Hydroxide

PVC Polyvinyl Chloride

UV Ultraviolet

PTFE Polytetrafluoroethylene

LBE Liquid-Based Epoxy

O-H Oxygen-Hydrogen

C-H Carbon-Hydrogen

C-O Carbon-Oxygen

KELANIAN

LIST OF SYMBOLS

B_r Overall Broadening

α Alpha

β Beta

% Percentage

wt% Weight Percentage

• Degree

•C Temperature (Celsius)

 θ Theta

g Gram

kg Kilogram

kV Kilo Volts

s Seconds

mL Millilitre

nm Nanometre

μm Micrometre

mm Millimetre

min Min

rpm Rotate per minute

h Hour

± Plus minus

M Molarity

Cos Cosine

Sin Sine

Cps Counts per second

HD Unit hardness

H Hardness

GPa Gigapascals

CHAPTER 1

INTRODUCTION

1.1 Background of study

The polymer coating has been long established as an anticorrosion and protective layer for metals. The process of coating involves applying polymer materials onto a substrate. The use of polymer coatings helps to prevent direct contact of steel with the environment, lowering the rate of corrosion. Polymer coatings can give varying levels of protection based on the thickness of the coating, the type of polymer utilized, and the environmental conditions to which the structure is exposed. A thicker coating can give more corrosion protection, but it also raises the expense of the coating. The type of polymer coating is among the elastomers or other polymeric materials. Several examples of polymer coating are polyester, epoxy, polyethene, Polyvinyl Chloride (PVC), and polymethyl methacrylate. These polymers are applied based on the conditions and applications.

Epoxy coatings are frequently utilized for welded steel surfaces resulting from their excellent adhesion and corrosion resistance. When steel is welded, the heat and associated chemical reactions can form a coating of oxidation on the surface, rendering it prone to corrosion (Hammi et al. 2021). Preparing the surface for the application of an epoxy coating on welded steel includes removing any impurities such as rust or oil and roughening the surface to increase adhesion. After that, the epoxy coating is sprayed on the surface with a spray gun or roller. Over time, the coating cures and hardens, forming a strong, protective covering. However, epoxy coatings prone to microcracks after long time UV exposure. Epoxy coatings can be customized with a variety of additives to give extra features like slip

resistance, UV resistance, and chemical resistance to minimize corrosion welded steel problems, (Hammi et al. 2021). It is critical to choose an epoxy coating that is specifically intended for steel surfaces and fits the application criteria, such as temperature range, exposure to chemicals or water, and abrasion resistance, (Hammi et al., 2021). Surface preparation and application techniques are also critical for a successful and long-lasting epoxy coating on welded steel surfaces.

Copper-zinc (Cu-Zn) is in polymer coatings to improve durability and improve corrosion resistance. Moreover, Cu and Zn are known to have powerful antibacterial activity against a wide variety of bacteria, fungi, and viruses (Salah et al., 2021). By integrating Cu-Zn particles into a polymer coating, the coating can provide an additional layer of protection against microbial growth as well as expanding the lifespan of the coating. Also, incorporating the alumina (Al₂O₃) into Cu-Zn composite could enhance the mechanical properties of the epoxy coating. The inclusion of Al₂O₃ can improve the Cu-Zn composite strength, hardness, wear resistance, and corrosion resistance, making it appropriate for a wide range of structural applications.

Therefore, the development of an epoxy coating containing Cu-Zn-Al₂O₃ composite particles will be the primary focus of this work to enhance corrosion performance and make the coating more durable and robust for use on welded steel. Their corrosion performance and thermal stability will also be discussed in this study.

1.2 Problem Statement

The strong barrier performance and mechanical qualities of epoxy coatings make them a popular choice for corrosion protection coatings; nevertheless, polymeric coatings frequently struggle with long-term durability and may not protect steel sufficiently. Epoxy coatings can also deteriorate too soon because of the initial flaws that the curing process introduces. These flaws lower the barrier performance and hasten the production of

microcracks. As a result, traditional coatings including the epoxy or other protective coatings may fail to provide long-term protection, and the coating may be susceptible to damage during construction or usage, resulting in localised corrosion in the affected area.

Regarding the fact that polymer coatings have been utilised to protect steel in concrete constructions, their use is limited due to their protective and endurance properties (Chhetri et al., 2019). These limits underscore the need for continued research to create high-performance coatings that can protect steel throughout time. Adding fillers such as Cu-Zn-Al₂O₃ to epoxy coatings can improve their performance and extend their lifespan. The specific type and amount of filler used will depend on the intended use and the desired properties of the coating. Furthermore, Cu-Zn are both recognised for their corrosion resistance, which may provide additional protection to the underlying substrate when utilised as a filler in epoxy coatings. Epoxy coating containing Cu-Zn-Al₂O₃ could provide thermal stability, making it more resistant to heat transfer and lowering the risk of microcracking.

1.3 Expected Outputs

It is anticipated that this project's epoxy-Cu-Zn-Al₂O₃ nanocomposite will be used to create thin films and a coating for welded steel. A study on the characteristics of epoxy-Cu-Zn-Al₂O₃ nanocomposite for corrosion resistance of welded steel is expected to show that adding Cu-Zn-Al₂O₃ nanoparticles to the epoxy matrix can improve the coating's mechanical properties, such as its hardness, strength, and flexibility.

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1.4 Objectives

The objectives of the study are:

- 1. To prepare Cu-Zn-Al₂O₃ nanocomposite as fillers in epoxy using high-energy ball milling.
- 2. To evaluate the corrosion performance and properties of epoxy-Cu-Zn-Al₂O₃ nanocomposite on welded part at different compositions.

1.5 Significant of Study

The corrosion performance of epoxy Cu-Zn-Al₂O₃ nanocomposite coating on welded steel is noteworthy due to the extensive use of welded steel in numerous industries. Corrosion can reduce the structural integrity and service life of welded steel, resulting in considerable economic losses owing to repair or replacement expenses. Epoxy coatings have been used to protect welded steel from corrosion, although they have some limitations. The study is to increase the corrosion resistance and longevity of welded steel in various industrial applications by investigating the use of Cu-Zn-Al₂O₃ nanocomposite in epoxy coatings, providing a cost-effective and environmentally friendly alternative. Furthermore, the research can help to enhance the fields of materials science and corrosion engineering by offering vital insights into the creation of novel corrosion-resistant coatings for various industrial uses. The findings have broad ramifications for industry, the environment, and the scientific community.

1.6 Scope of Study

Examining the efficiency of the coating in preventing corrosion of welded steel under various environmental circumstances will be the main goal of the study on the corrosion performance of epoxy Cu-Zn-Al₂O₃ nanocomposite coating on welded steel. The Cu-Zn-Al₂O₃ nanocomposite will be prepared for the investigation and added to an epoxy coating. After that, various corrosion tests, including as salt spray exposure, humidity exposure, and

immersion in acidic solutions, will be applied to the coated welded steel samples. The study will also assess the coatings' adherence, mechanical characteristics, and durability. The study's findings will shed light on the efficacy of Cu-Zn-Al₂O₃ nanocomposite additives in enhancing epoxy coatings' corrosion resistance as well as on their suitability for shielding welded steel in a variety of industrial applications. Since only laboratory tests were performed as part of the study, the outcomes could not accurately represent how well the coating performs in actual applications. The study, however, will serve as a starting point for additional investigation and the creation of fresh, corrosion-resistant coatings for welded steel.

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CHAPTER 2

LITERATURE REVIEW

2.1 Polymer Coating for Metal Protection

A polymer coating is a protective thin layer that is applied to the surface of a substrate, usually a metal, to offer protection and improve its performance. Polymer coatings have significantly increased in popularity in the metal protection industry due to their remarkable qualities and broad range of uses. To increase the lifespan of metal surfaces, these coatings must offer a strong barrier against environmental elements such corrosion, oxidation, and abrasion. Anticorrosion protection can be provided by polymer coating systems applied to metal surfaces. However, in structural applications, polymer coating easily develops microcracks, lowering lifespan, hence early detection, diagnosis, and repairs of microcracks are critical. Polymeric coatings or paints are adhesion and corrosion-resistant coatings or paints. In the polymeric coating process, an elastomeric or other polymeric substance is applied to a supporting substrate (Zhang, 2014).

Polymer coatings are made of polymers, which are big molecules with repeating subunits and are intended to form a barrier between the substrate and the environment (Matt Sokol, VP Sales, and Marketing, 2022). Depending on the polymer employed, these coatings have different qualities like resistance to corrosion, chemicals, abrasion, and weathering. To establish a consistent and adherent layer on the substrate, polymer coatings are applied using techniques including spraying, dipping, or electrostatic deposition. The coating material can be customised to fulfil a variety of specifications, such as those related to substrate adherence, flexibility, hardness, and aesthetic appeal. The lifespan of the substrate can be greatly increased

by coating it with a polymer, which also shields it from damage brought on by elements including moisture, UV rays, chemicals, and mechanical wear (Liu et al., 2021).

2.1.1 Type of Polymer Coating

Various polymer coating has been employed for metal protection. These include polyester, epoxy, polyethene, PVC, and polymethyl methacrylate (Motlatle et al., 2022). They are mainly among the elastomers or polymer-based materials. However, the level of protection provided by these various polymer coating types varies, and they are chosen based on the needs of the application. In outdoor applications such as automobile coatings, architectural coatings, and protective coatings for infrastructure, polyurethane coatings are frequently utilised because they provide higher weathering resistance (Fang et al., 2022). The outstanding weather resistance, colour preservation, and glossy appearance of acrylic coatings make them very popular. They offer defence against moisture, UV rays, and outside factors. Architectural uses for acrylic coatings include metal roofs, facades, and signage (Zhang et al., 2021). The non-stick qualities, chemical resistance, and high-temperature stability of fluoropolymer coatings like polytetrafluoroethylene (PTFE) are well recognised. In applications involving moving parts or components subjected to harsh circumstances, such as those in the automotive, food processing, and electronics sectors, PTFE coatings produce a low-friction surface that makes them appropriate (Wang et al., 2018).

Polymers come in a wide variety of kinds and have a range of characteristics. For their additional durability and anti-corrosion properties, they are highly regarded. Pumps, pipelines, and valves that are part of industrial equipment are among the many types of parts that these coatings are quite useful for protecting. Ceramics, metals, and synthetic materials can all benefit from polymer coatings. At about 535°F (280°C), they can withstand high temperatures. To protect multi-head scales, containers, and pans, for example, several of these coatings have

received FDA approval for use in the food manufacturing business. The following are some further choices for polymeric corrosion-resistant coatings: For farm equipment and other industrial products that need effective corrosion protection at a fair price, alkyds and acrylics are frequently employed as coatings. Tugboats, conveyor equipment, road construction machinery, polyurethane coatings, and motorcycle parts are all examples of their utilisation. Railroad hopper cars may be covered in urethanes having abrasion resistance properties. A metal surface's ionic resistance is enhanced by the application of a polymeric coating that resists corrosion. To prevent corrosion, polymeric coatings must stick strongly to a substrate without easily flaking or degrading when exposed to moisture, heat, chemicals, or salt. These coatings must also be highly robust and resist degradation from these elements (secoaTECH, 2022).

2.2 Epoxy Coating

Epoxy coatings are a popular option for metal protection because of their many advantages. Epoxy coatings are known for their outstanding adherence to a variety of surfaces, including metals. In steel structure application, epoxy coatings create a robust and long-lasting protective layer that can endure challenging circumstances and shield metal surfaces from corrosion (Kong et al., 2022). They have exceptional UV protection, abrasion resistance, and flexibility. These coatings are made of epoxy resins, which are mixed with a curing agent to create a tough, protective layer on metal surfaces. Epoxy coating provide securely adhered layer to the metal surface due to these powerful connections and long-lasting (Liu et al., 2021). Rust and corrosion are successfully retarded by the epoxy coatings because they keep moisture and other corrosive materials from getting to the metal substrate (Kumari et al., 2021).

Epoxy coatings are made of thermoset epoxy resins, which have a wide range of uses as protective films for structural and speciality metallic components. Epoxy resins are highly

changeable matrices that can be made with or without solvents. The epoxy technology is used in a variety of industries, such as household goods, automotive, aerospace, and manufacturing, to name a few. It can be used in composites or films. Epoxy coatings made of solvents are inexpensive and offer a variety of benefits, including outstanding processability, great adherence to metallic substrates, and excellent chemical resistance. There are numerous processes and a variety of cure times to create epoxy resins. The curing substances could be cationic salts, quaternary phosphonium salts, amine-boron trihalide complexes, nitrogen, oxygen, sulphur-containing compounds, or cationic compounds (Ulaeto et al., 2023). Epoxy resins were first produced using this method in the 1930s by reacting bisphenol A with epichlorohydrin. Since then, castings and coatings have both been made with epoxy resins. Additionally, they are frequently employed as potting materials, binders, adhesives, and wraps. Aromatic glycidyl amines, cycloaliphatic, and phenolic glycidyl ethers are the groups of widely used and commercially available epoxies.

2.2.1 Development of Epoxy-Based Coating

Epoxies are the most popular polymeric coatings due to their superior corrosion resistance, exceptional adhesive qualities, low shrinkage, and superb mechanical and thermal properties. Despite having many advantageous qualities, epoxy resins are extremely brittle, have poor impact resistance, and are not very flexible. As a result, mechanical damage causes countless microscopic pores and microcracks to form in the resin.

Moreover, consistent exposure to corrosive ions causes the epoxy matrix to be hydrolysed, opening a pathway for corrosive ions to enter the thin protective layer (Chhetri et al., 2019). Thus, the addition of a filler or composite based material as a protective barrier or inhibitor is necessary to increase the durability of the epoxy-based coatings. Epoxy coatings are enhanced with fillers to improve a variety of properties, including mechanical strength,

thermal conductivity, flame retardancy, dimensional stability, and abrasion resistance. The filler able to resist corrosion by lengthening the corrosive ions' diffusion paths and preventing the development of micropores and cavities (Chhetri et al., 2019). The initial epoxy matrix resin used in liquid-based epoxy (LBE) coating systems is in liquid form. Numerous kinds of nanofillers have been widely used to enhance the load bearing, friction, and wear characteristics of pure epoxy resins. As can be seen, fillers can be roughly categorised as lubricant, mineral silicate, carbon-based, metallic, polymer-based, ceramic, and metal-based fillers. These fillers can be used singly, in pairs, or in combinations to strengthen the epoxy matrix. The term "hybrid system" is frequently used to describe a system that combines different kinds of nanofillers. When introduced in specific compositions chosen through a series of incremental tests, hybrid systems have been shown to give the best qualities of each of the fillers. Different nanofillers have improved the tribological properties significantly by lowering the coefficient of friction and reducing wear of the mating interfaces under sliding conditions, whether they were introduced as a stand-alone component or as a hybrid (Baig & Samad, 2021).

2.2.2 Fillers

Fillers are essential in epoxy formulations because they alter the characteristics and functionality of the epoxy coating. In most cases, they can be in solid particles that are homogenously disperse throughout the epoxy matrix. Various choices filler which could come from type of ceramic and metal. Ceramic filler that commonly used in epoxy composite includes aluminum oxide (Al₂O₃) (Saber et al., 2022) silica (Linec & Mušič, 2019) and silicon carbide (Sanya et al., 2019). Meanwhile, metal fillers range from pure metals and alloys. Al, Cu, and Zn are the most common metal fillers for epoxy composites. The development on using metal-metal fillers improve the anticorrosion properties of epoxy coating. For example, incorporation of both Al and Cu particles within the epoxy composites produced overall wear loss was almost 50% of the neat epoxy resin (Srivastava & Verma, 2015). In the study of

(Hussin et al., 2022) on the epoxy-brass composite the compressive strength and thermal conductivity has been improved.

To improve a material's performance or meet a particular need, additives are compounds that are added in small quantities. In most cases, these substances are chemical mixtures that have advantages for the material. A material's final attributes, such as mechanical strength, chemical resistance, or UV stability, can be improved with the application of additives, much as processing properties, such as flowability or curing time, can be improved. The following are a few types of additives: plasticizers, stabilisers, flame retardants, antioxidants, UV absorbers, and colourants. To provide the intended improvements, additives frequently collaborate with the base substance. On the other hand, fillers are solid components that are added in higher amounts to a substance to change its properties or attain features. Most fillers are inert, and they barely react at all with the base substance. They can also give dimensional stability, thermal conductivity, or barrier qualities, as well as enhance mechanical attributes including strength, stiffness, or impact resistance. Polymers, composites, coatings, and adhesives frequently employ fillers. Silica, glass fibres, carbon black, calcium carbonate, and alumina are a few examples of fillers (Ramesh et al., 2022).

2.3 Cu-Zn composite as Filler in Epoxy-Based Coating

Cu-Zn alloy or brass can possibility used as filler for epoxy composite which serve to increases the mechanical properties and thermal conductivity of epoxy coatings. Moreover, Cu-Zn composite could a promising material as filler for epoxy composites. They offer good corrosion resistance, thermal conductivity, and high strength with electrical properties. By promoting electron transmission across the coating, the synergistic action of Cu and Zn in the composite helped to improve electrical conductivity. The coating performed better overall because to the inclusion of the Cu-Zn composite fillers, making it appropriate for use in

applications that call for both mechanical and electrical conductivity (Hamed et al., 2021). However, Cu-Zn composite have been less investigated as fillers for epoxy composites. Epoxybased materials can be enhanced with metallic fillers, such as copper-zinc (Cu-Zn) alloys, to provide characteristics and develop composites with specialised uses. These fillers, which often take the form of powders or particles, are combined with epoxy resin to improve properties of the finished composite material. Cu-Zn alloys come with a variety of useful qualities, such as excellent electrical conductivity, thermal stability, and corrosion resistance. The resulting composite can have increased electrical conductivity because to the addition of Cu-Zn metallic fillers, making it appropriate for uses that call for electrical grounding or shielding. Additionally, the addition of Cu-Zn fillers can improve the epoxy-based material's thermal characteristics, assisting in dissipation of heat and thermal management. Additionally, the corrosion resistance of Cu-Zn alloys helps to the endurance and toughness of the epoxy composite in harsh environmental circumstances. Cu-Zn metallic fillers can be used to create multifunctional composites with improved electrical and thermal properties, which make them appropriate for usage in a variety of applications in the electronics, aerospace, and automotive industries (Author links open overlay panel Mukesh Kumar 1 et al., 2013).

2.3.1 Incorporation of Al₂O₃ in Cu-Zn composite

It is a well-known idea that a high-performance second phase can be incorporated into a traditional engineering material to create a composite with properties that are not possible from the individual components alone (Chhetri et al., 2019). In this case of Cu-Zn composite, Al₂O₃ is favoured for another reinforced material due to low cost and reputation for having high strength, good hardness, and great corrosion resistance. By creating a thin surface coating on the Cu-Zn composite that may potentially be used for metal coating, the addition of Al₂O₃ to the Cu-Zn composite increased. Ceramic particles often added into Cu-Zn matrix to increase strength and durability of the composite. Al₂O₃ is preferred because of inexpensive and known

to possess high strength, good hardness, and excellent corrosion resistance. Addition of Al₂O₃ into Cu-Zn composite increased by forming thin surface coating on the Cu-Zn composite which potentially be used in metal coating. Thus, because of these advantages, Al₂O₃ will be used as reinforced phase in Cu-Zn composite.

To improve steel's barrier qualities, nanocomposite coating is being researched (AV et al.). Epoxy coatings see reduced coating delamination and blistering when nanofillers are used. This is explained by the increased ability of tiny particles to fill the holes. Furthermore, the presence of nanoparticles smaller than 100 nm won't affect the polymeric coating's transparency (Sharifi Golru a et al., 2014). Additionally, alumina (Al₂O₃) fillers have been extensively employed to improve associated properties of epoxy matrices. These fillers are distinguished by their strong mechanical properties. The food sector uses numerous nanomaterials, including nanocomposites and nano polymers, at different degrees. In comparison to coatings made entirely of polymers, coatings containing Al₂O₃ nanofillers demonstrated improved mechanical characteristics (J et al., 2019).

2.3.2 Preparation techniques for Cu-Zn- Al₂O₃ nanocomposite

Techniques like mechanical alloying, powder metallurgy, or electrodeposition are frequently used to fabricate metal-based nanocomposites. These techniques make it possible for fillers to be evenly distributed throughout the metal matrix, ensuring that the reinforcements are distributed uniformly and producing a suitable composite structure.

Mechanical alloying that uses high energy ball milling could be to produce Cu-Zn-Al₂O₃ nanocomposite. This method creates surface diffusion as well as a uniform dispersion of reinforced material for creating metal-matrix nanocomposites. At the processing stage, very small reinforcement particles may form inside the matrix. As a result of the oxygen and other contaminants not being picked up by the approach, either oxidation or gas absorption cannot

be detected (Baghani, Aliofkhazraei & Askari, 2017), leading to a composite with clean interfaces between reinforcement particles and matrix (B. Stalin, M. Ravichandran, S. Jasper, & C. Ramesh Kannan, 2020).



CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Materials

Materials used in this study are epoxy resin, Cu, Zn, Al₂O₃ and methyl ethyl ketone peroxide (MEKP). Cu (> 99.9% purity, average particle size > 20 μm), Zn (> 99.9% purity, average particle size > 20 μm) and Al₂O₃ (>99.9% purity, average particle size 20 μm) will be purchased from Sigma Aldrich. Epoxy resin and MEKP aqueous solution will be obtained from Revertex Sdn. Bhd.

3.2 Methods

The experiment started with preparation Cu-Zn-Al₂O₃ reinforcement, followed by preparation of epoxy-Cu-Zn-Al₂O₃ nanocomposite coating and preparation of coated welded steel. Overall research flow is shown in Figure 3.1.

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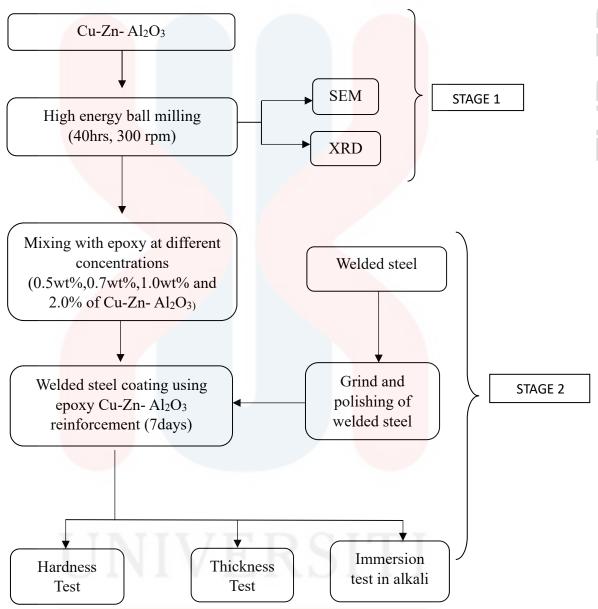


Figure 3.1: Overall research flow

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3.2.1 Preparation of Cu-Zn- Al₂O₃ Reinforcement

Cu-Zn-Al₂O₃ nanocomposite produced using a planetary ball milling (DECO-PBM-V-0.4L). The composition of 70 wt.% Cu-Zn and 30 wt.% of Al₂O₃ mixture used and calculated using rule of mixture. Then, the powder mixture milled using high energy ball milling for 40 h and milling speed 300 rpm. Zirconia with diameter size 10 mm and balls to powder of 10:1 used.

3.2.2 Preparation of Epoxy-Cu-Zn-Al₂O₃ Nanocomposite Coating

The composition used to produce the epoxy-Cu-Zn-Al₂O₃ nanocomposites was made of 0.5wt%, 0.7wt%, 1.0wt%, 2.0wt% of Cu-Zn-Al₂O₃ particles. Mix epoxy and hardener 4:1 with each composition and stir with bamboo spoon until the mixture homogenize.

3.2.3 Preparation of Epoxy-Cu-Zn-Al₂O₃ thin film

After mixing process, the epoxy-Cu-Zn-Al₂O₃ thin film was produced by applying the composite using bamboo spoon on the petri dish at room temperature. The thin film thickness maintained less than 1 mm. Then, the thin films cured for 24 h at 70°C in an oven.

3.2.4 Preparation of Coated Welded Steel

The welded steel substrates were ground and polished with silicone carbide paper (400-1000 grit) to ensure any oxide layers removed. Then, the epoxy coating was applied on the welded steel surface using a bamboo spoon and cured for 24 h at 70°C in an oven and have tested for hardness and corrosion test.

3.3 Characterizations

After the preparation of composite was done, they were characterized for phase analysis (XRD), scanning electron microscope (SEM) and Fourier-transform infrared spectroscopy (FTIR). The hardness, thickness, and corrosion behaviour of coating on welded steel substrate also have been determined.

3.3.1 Phase Analysis

Bruker D2 Phaser X-ray Diffraction (XRD) was used to characterise the Cu-Zn-Al₂O₃ nanocomposite for phase identification. The step size of 0.02° with 20° to 90° has been used. Software DIFFRAC.EVA was used to determine the phase identification of the composite. The information from XRD patterns was analysed based on Williamson- Hall (WH) method for crystallize size and internal strain of Cu.

3.3.2 Microstructure

An integrated JEOL JSM IT100 Scanning Electron Microscope (SEM) system was utilized to analyse the morphologies of Cu-Zn-Al₂O₃ nanocomposite and welded steel coated with epoxy-Cu-Zn-Al₂O₃ nanocomposite. The measurement was performed at 5kV and at magnifications of 500X and 1000X magnifications.

3.3.3 Functional group

Fourier-transform infrared spectroscopy (FTIR) used to identify and characterize thin film of epoxy-Cu-Zn-Al₂O₃ with wavelength range within 400 cm⁻¹ and 4000 cm⁻¹ based on

their infrared absorption spectra. It works by measuring the interaction of infrared radiation with matter, providing about the molecular composition and structure of the thin film.

3.3.4 Thickness

Thickness gauge has been used to determine the coated film on welded steel. By placing a coil near a conductive substance, such as the metal substrate underneath the epoxy coating, this approach produces an alternating magnetic field. The induced eddy currents in the metal are influenced by the thickness of the coating, and this in turn affects the coil's impedance. This change in impedance is measured by the gauge, which then interprets the data to determine thickness. On the middle section of the specimen, five impressions were produced, and the mean thickness was calculated.

3.3.5 Hardness

The hardness of coated welded sample and composite thin film was evaluated using a Shore D Durometer. On the middle section of the specimen, five impressions were produced, and the mean hardness was determined. Shore D can measure the hardness of medium to harder materials such as epoxies, durable rubber, or hard plastics, typically those with a hardness between 40 and 90 on the shore D scale (Tucker, 2023).

3.3.6 Immersion Test

ASTM G31-72 has been carried for corrosion immersion process. All the samples diluted in 40 g sodium hydroxide (NaOH) with 1000 ml distilled water making 0.1 M. The

immersion test performed for total 30 days. The weight loss method utilised to calculate the corrosion rate and taken at 5 days interval. The weight loss method is the most common way of determining corrosion rates. The samples were immersed in NaOH solution and allowed to corrode. The corrosion rate was determining based on the weight loss of the sample as follows:

Corrosion rate =
$$\frac{\text{weight}}{\text{days}} \times 100$$

Equation 1.

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CHAPTER 4

RESULTS AND DISCUSSION

4.1 Phase Identification and Structural Properties

4.1.1 Cu-Zn-Al₂O₃ Nanocomposite Powder

X-ray diffraction (XRD) was used to identify the phase in the Cu-Zn-Al₂O₃ nanocomposite powder. Figure 4.1 shows the XRD pattern of the as-milled Cu-Zn-Al₂O₃, which has been milled for 40 hours, and the peaks of Cu, Zn, and Al₂O₃ were observed in the peak patterns. Cu exhibited strong diffraction peaks in the XRD pattern at $2\theta = 43.324^{\circ}$, 50.465° , and 74.196° (COD 5000216). Zn (COD 9008522) and Al₂O₃ (COD 9008081) were also characterized having crystalline structure.

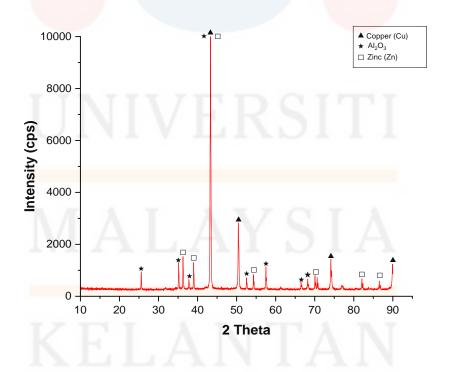


Figure 4.1: XRD pattern of as milled Cu-Zn-Al₂O₃ nanocomposite powder

4.1.2 Copper crystallite size and internal strain

The Cu crystallite size and internal strain was determined by using Williamson Hall (WH) method. The peaks broadening in this study was associated with both the size and strain. Linear regression was used to determine crystallite size and internal strain. The strain was extracted using the slope of the fit, and the crystallite size was determined using the y-intercept. There were two highest peaks used to determine crystallite size, which was located at $2\theta = 43.318^{\circ}$ (111) and 50.448° (200).

Table 4.1 Crystallite size and internal strain value

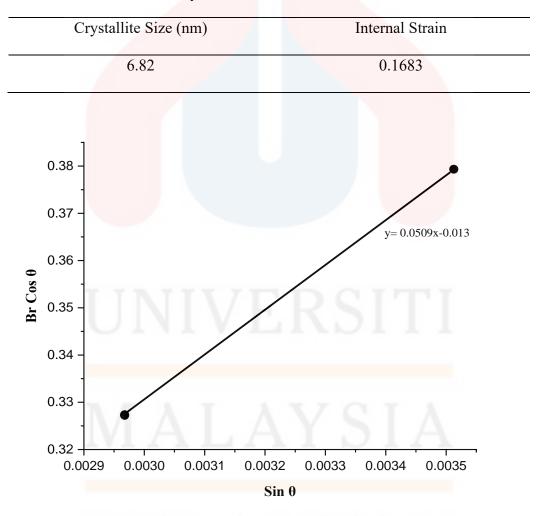


Figure 4.2 :Linear regression in determining the Cu crystallite size and internal strain using
Williamson-Hall method

4.1.3 Epoxy-Cu-Zn-Al₂O₃ Nanocomposite Film

The XRD patterns of the epoxy nanocomposite film at different amount of Cu-Zn-Al₂O₃ are displayed in Figure 4.3. The films were observed associated with crystalline Cu and Al₂O₃ as well as amorphous epoxy. Amorphous epoxy's broad peak was discovered at 21.1°, while the largest peak of Cu was observed at 43.5° and 50.7°. The peak intensity of Cu and Al₂O₃ increased as the composition of the Cu-Zn-Al₂O₃ increased. The strongest intensity of Cu and Al₂O₃ peaks were obtained with addition of 2.0 wt% Cu-Zn-Al₂O₃. In addition, Zn peaks were only detected with 2.0 wt% Cu-Zn-Al₂O₃ addition. This is because Zn exhibited small particles size than the Cu and Al₂O₃ that could be isolated by epoxy resin (Xiao et al., 2017).

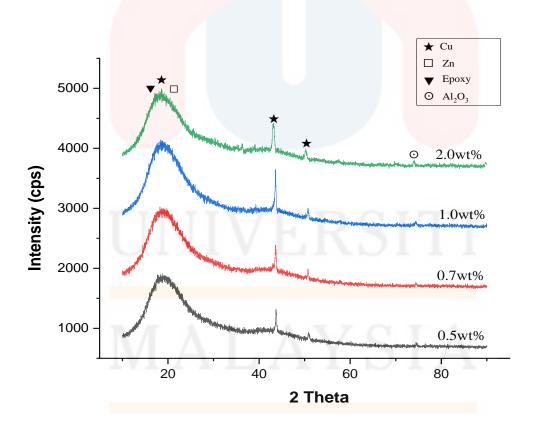


Figure 4.3: XRD patterns of epoxy-Cu-Zn-Al₂O₃ composite film at different Cu-Zn-Al₂O₃ compositions

4.2 Morphology

Figure 4.4 shows Cu-Zn-Al₂O₃ milled nanocomposite powder using 500x and 1000x magnification. It shows clear particle shape of each material which spherical shape indicates Cu and hexagonal shape indicates Zn meanwhile Al₂O₃ particle changes from angular to irregular shape.

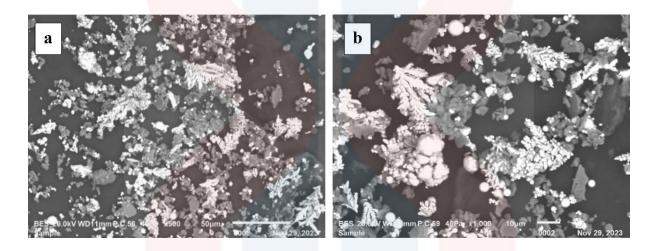


Figure 4.4: SEM images of Cu-Zn-Al₂O₃ nanocomposite at a) 500X and b) 1000X magnifications

SEM images of the as-milled Cu-Zn-Al₂O₃ and epoxy- Cu-Zn-Al₂O₃ are shown in Figure 4.5. At a higher magnification, the uneven form, size, and aggregation size of the particle become marginally visible. The red arrow in Figure 4.5 indicates porosity and the green arrow indicates nanocomposite agglomeration.

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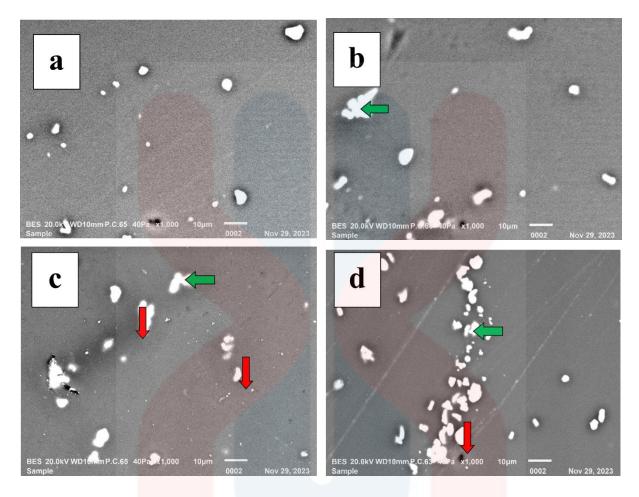


Figure 4.5: SEM images of epoxy-Cu-Zn-Al₂O₃ nanocomposite film at different Cu-Zn-Al₂O₃
a) 0.5wt% b) 0.7wt% c) 1.0wt% and d) 2.0wt% at 1000X magnifications

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4.3 Functional Group

FTIR spectroscopic analysis was conducted to identify the presence of any functional group in epoxy-Cu-Zn-Al₂O₃ nanocomposite film. Figure 4.4 shows the FTIR spectra of the epoxy nanocomposite film at different amount of Cu-Zn-Al₂O₃. Broad peak at 3380 cm⁻¹ is assigned for O-H stretch in secondary aliphatic alcohols, stronger bonds vibrate at higher frequencies (wavenumbers) because they require more energy to stretch or bend. It is common for the O-H stretching frequency to be broad, encompassing an extensive range of wavenumbers. The kind of alcohol (primary, secondary, or tertiary) and the existence of nearby functional groups can have an impact on the precise placement (Hossain, 2013).

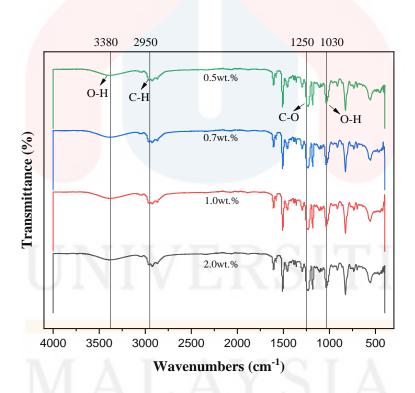


Figure 4.6: FTIR spectra of epoxy-Cu-Zn-Al₂O₃ nanocomposite film at different Cu-Zn-Al₂O₃ compositions

Sharp peak at 2950 cm⁻¹ represents C-H stretch in aliphatic hydrocarbon. The C-H stretching region for alkane C-H bonds is typically observed in the range of 2800-3000 cm⁻¹. Besides, another sharp peak at 1250 cm⁻¹ is for C-O, within the range of 1000–1300 cm⁻¹ is the typical C-O stretching vibration for single bonds in alcohols and ethers. The peak in alcohols is typically located between 1000 and 1050 cm⁻¹, but the peak in ethers can be observed between 1100 and 1150 cm⁻¹. Depending on the molecular environment and particular functional group, the precise location may change. Another peak located at 1030 cm⁻¹ is for O-H. In contrast to the broad O-H stretching peak, the O-H bending peak is frequently sharper and more defined.

4.4 Physical Properties

To generate design data, anticipate service performance, control quality, and occasionally investigate problems, physical qualities of materials must be monitored. It is impossible to successfully create new items without test results, which provide no evidence of quality. In a variety of disciplines, including materials science, engineering, manufacturing, and quality control, measures of physical attributes like hardness and thickness are crucial. These attributes give important details regarding the qualities and functionality of materials. The thickness of coatings on nonmetal substrates can be measured with ultrasonic gauges using the ultrasonic pulse-echo technique without causing any damage to the coating. An ultrasonic transducer built into the instrument's probe pulses through the coating. The transducer receives the pulse's reflection back from the substrate and transforms it into a high-frequency electrical signal. The thickness of coating is ascertained by digitising and analysing the echo waveform. In certain situations, it is possible to measure individual layers inside a multi-layer system (Zhang et al., 2021). A straightforward method for obtaining the distinctive microhardness

values of surface coatings that are too thin to be tested directly is described. Understanding the film thickness and performing standard microhardness tests on coated and uncoated substrates are prerequisites for applying the approach (Nishibori et al., 2002).

4.4.1 Thickness of Epoxy-Cu-Zn-Al₂O₃ Coating Film

Film thickness greatly influences steel protection and must be accurately measured (Ulaeto et al., 2023). The films thickness was measured using digital thickness gauge. By placing a coil near a conductive substance, such as the metal substrate underneath the epoxy coating, this approach produces an alternating magnetic field. The induced eddy currents in the metal are influenced by the thickness of the coating, and this in turn affects the coil's impedance. This change in impedance is measured by the gauge, which then interprets the data to determine thickness. Figure 4.5 shows the thickness of the hardness of the epoxy nanocomposite film at different amount of Cu-Zn-Al₂O₃. The film with 0.5wt% Cu-Zn-Al₂O₃ recorded the lowest than thickness all the nanocomposite films while the highest thickness was the film with 2.0wt% Cu-Zn-Al₂O₃. The thickness of all the composite was not uniform but having less than 1.0 mm. The non-uniform thickness might be caused by uneven steel surface and coated film. Thus, it may have an impact on the results of the hardness and corrosion performance.

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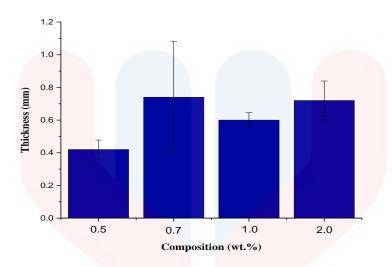


Figure 4.7: Thickness of the epoxy-Cu-Zn-Al₂O₃ nanocomposite film at different Cu-Zn-Al₂O₃ compositions

4.4.2 Hardness of Epoxy-Cu-Zn-Al₂O₃ Coating Film

Hardness of epoxy-Cu-Zn-Al₂O₃ nanocomposite film was observed using shore D durometer. Shore D can measure the hardness of medium to harder materials such as epoxies, durable rubber, or hard plastics, typically those with a hardness between 40 and 90 on the shore D scale (Tucker, 2023). The test was conducted by pressing a spring-loaded indenter into the epoxy-Cu-Zn-Al₂O₃ coated on welded steel surface and measuring the depth of indention. The harder the material, the less the indenter will penetrate, and the higher the shore D hardness value. Figure 4.4 shows the hardness of the epoxy nanocomposite film at different amount of Cu-Zn-Al₂O₃. The highest hardness was observed the film containing 2.0wt% Cu-Zn-Al₂O₃. The coated film containing the highest amount of Cu-Zn-Al₂O₃ is the hardest among other compositions. Cu-Zn-Al₂O₃ act as reinforcement hardens the composite film through interactions with the matrix. Cu-Zn-Al₂O₃ strengthen the mechanical interlocking between

reinforcement and matrix, increasing the cross-linking densities and improving the hardness of the coated film (Yilmaz & Buytoz, 2001).

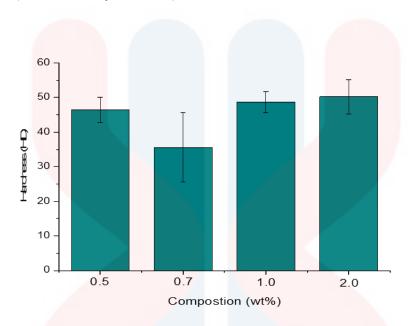
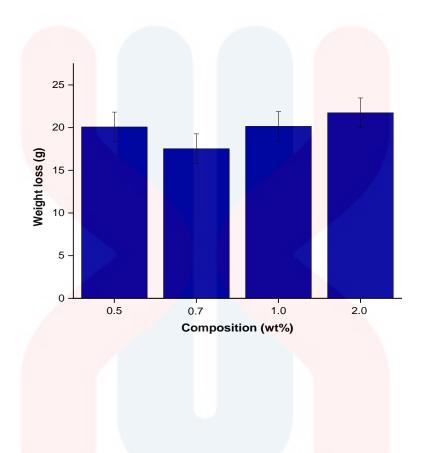


Figure 4.8: Hardness of the epoxy-Cu-Zn-Al₂O₃ nanocomposite film at different Cu-Zn-Al₂O₃ compositions

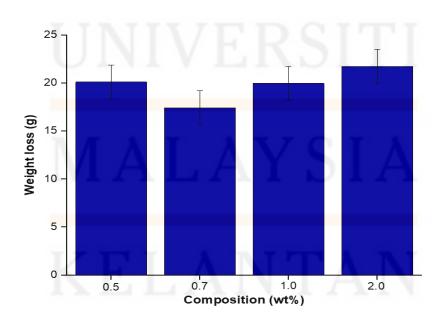
4.5 Immersion Test

The corrosion performance of epoxy-Cu-Zn-Al₂O₃ coated on welded steel was performed using immersion test. The tests were done within a month (30 days) with 5 days interval. All the samples were placed in the beaker filled with NaOH solution. The initial weight for 0.5wt% recorded 20.08 g, for 0.7wt% is 17.53 g, for 1.0 wt% is 20.15g, and for 2.0 wt% is 21.74 g. The weight loss of epoxy-Cu-Zn-Al₂O₃ film after immersion in NaOH solution is recorded in Figure 4.9. After 5 days of immersion, the film containing 0.5wt% Cu-Zn-Al₂O₃ was recorded increased to 20.10 g resulted from less amount of filler whereas the other films decreased. After 10 until 15 days, slight decreased on the film were determined with 1.0wt%, 0.7wt% and 2.0wt% of Cu-Zn-Al₂O₃. All the films were found showing weight loss from 20 to 30 days.

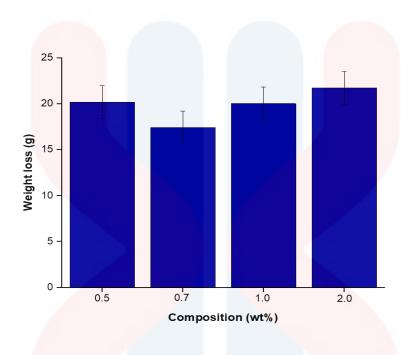
a)



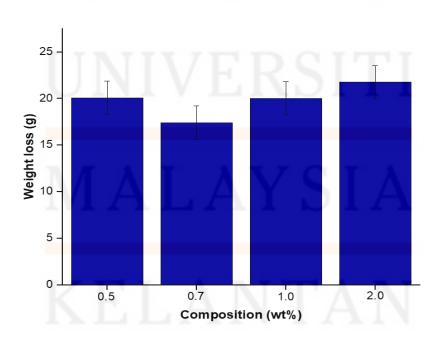
b)



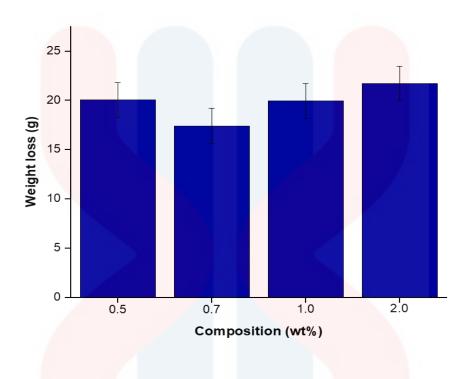
c)



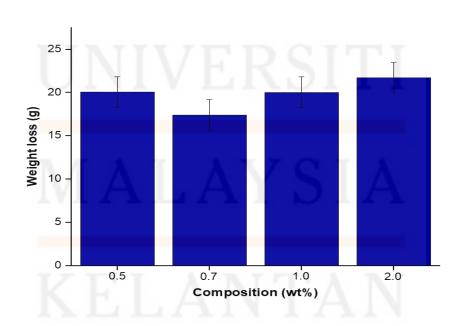
d)



e)



f)



g)

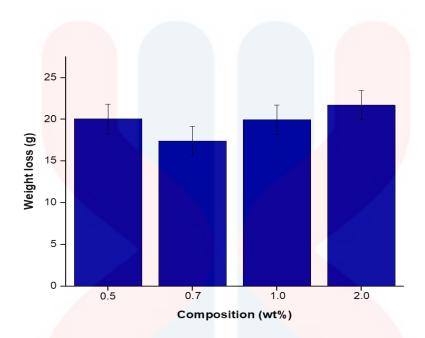


Figure 4.9: Weight loss of epoxy-Cu-Zn-Al₂O₃ nanocomposite coated film at different composition on welded steel immersed for a) 0 days, b) 5 days, c) 10 days, d) 15 days, e) 20 days, f) 25 days, g) 30 days.

Figure 4.10 summarize the weight loss of epoxy-Cu-Zn-Al₂O₃ film coated on welded steel at different Cu-Zn-Al₂O₃ compositions and immersion time. The film with 0.5wt% Cu-Zn-Al₂O₃ slightly occur at early few days due to many possibly reasons. Coated steel sheets' ability to withstand corrosion can be influenced by a wide range of factors, such as the type of substrate used, the paint type, the quantity and thickness of coating layers, the service environment in which the coated steel plate is used, and the methods used for machining, transporting, and storing the substrate (Limited, 2021). The coated film with lowest fillers amount obtained to be relatively slow to show the effects. Meanwhile, for the film with 0.7 wt% Cu-Zn-Al₂O₃ shows rapid rate of weight loss, opposite with film containing 1.0 wt% Cu-Zn-Al₂O₃ which exhibit non-uniform trend. The highest composition (2.0 wt%) of Cu-Zn-Al₂O₃ with the containing trends the poxylor of the coated steel plate is used.

Al₂O₃ in epoxy shows the slight reduction of weight loss on each interval time. The higher filler content produced decrement of corrosion protection of the welded steel. It can be concluded, in this study, the amount of Cu-Zn-Al₂O₃ was 2.0 wt% is suitable for steel protective coating.

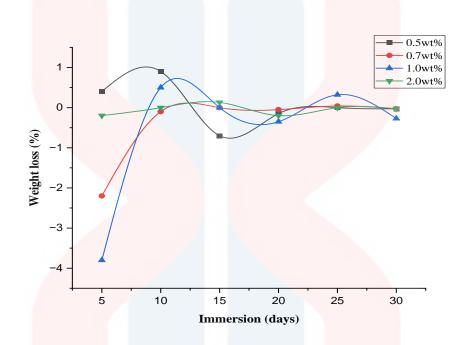


Figure 4.10: Corrosion rate of epoxy-Cu-Zn-Al₂O₃ film coated on welded steel at different composition for 30 days (5 days interval)

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CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In this study, epoxy-Cu-Zn-Al₂O₃ nanocomposites coating were successfully produced. Epoxy-Cu-Zn-Al₂O₃ nanocomposite were coated on welded steel to evaluate the corrosion performance in the alkaline environment. The conclusion drawn from this work are:

- a. After 40 h of milling, Cu-Zn-Al₂O₃ nanocomposite powder shows crystalline features with structural changes (crystallite size 68.2 nm and an internal strain 0.1683) and homogenous particle distribution. Their incorporation in the epoxy plays important role as they were found to alter the morphological and functional group of epoxies.
- b. The epoxy-Cu-Zn-Al₂O₃ nanocomposite having 2.0 wt% Cu-Zn-Al₂O₃ have the highest hardness due to hardening effect. The presence of higher amount of Cu-Zn-Al₂O₃ contributes slight decrease of weight loss in NaOH which related to the hardenability of the reinforced phase. This study shows the composition with 2.0 wt% in epoxy is suitable for welded steel protective coating.

5.2 Recommendations

In this work, milling is not enough to provide the powders with the energy required for internal refinement. When the milling period was 40 hours, the peak intensities of Cu, Zn, and Al₂O₃ rose. It is suggested that the milling time needs to be extended by more than 40 hours to supply enough energy for the internal refinement of composite powders. Furthermore, to

observe the sintered composite more clearly, the nanocomposite must be sintered after compaction. And last, mechanical testing on nanocomposites may be done in the future. This would involve examining how milling affects the strength of the materials.



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APPENDIX A

Name (mineral) Copper							
		2.08710	43.317	1000	1	1	1
		1.80750	50.449	450	2	0	0
Name (common)		1.27810	74.126	220	2	0	2
Status Status Unkr	nown						
Ambient Yes		1.09000	89.934	232	3	1	1
		1.04360	95.143	67	2	2	2
	_	0.90380	116.923	38	4	0	0
Lattice: Cubic	Mol. weight =						
s.g. : F m -3 m (225)	Volume [CD] = 47.24 Dx =	0.82930	136.514	175	3	1	3
	Dx =	0.80830	144.723	203	4	0	2
	I/Icor = 11.970						
= 1.00000 c/b = 1.00000 Primary Reference Swanson He, Tatge E, "Standard patterns", National Bureau of Stan (1953) 1-1.	X-ray diffraction powder dards (U.S.), Circular 359	RS					
Wavelengt h: 1.54060 SS/FOM:	Filter: Not specified d-spacing:	YS					

Figure A.1: COD XRD Pattern of Cu Nanocomposite Powder

Name (mineral) Zinc Name (common) Status Status Unknown Ambient Yes 2.30780 38.997 229 1 0 0	Formula	Zn			d	2θ	l fix	h	k	ı
Status Status Unknown Status Status Unknown Yes Status Status		Zino			2.47340	36.291	379	0	0	2
2.09140	• •	ZIIIC			2.30780	38.997	229	1	0	0
Amblent Yes 1.68730 54.327 155 0 1 2 1.34160 70.082 185 0 1 3 1.33240 70.638 120 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 1 0 1.33240 70.638 120 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		Status Unl	<mark>know</mark> n		2.09140	43.224	1000	1	0	1
1.34160 70.082 185 0 1 3 1.33240 70.638 120 1 1 1 0 1 1 1 0 1 1	Ambient	Yes						0		2
1.33240 70.638 120 1 1 0										3
Lattice: Hexagonal Volume (CD) = 30.42 Dx = Dm = 1.17300 82.096 142 1 1 2 2 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0										
S.G.: P63/mmc (194) Volume [CD] = 30.42 Dx = Dm = 1.17300 82.096 142 1 1 1 2 1 1 2 1 1 1 2 1 1 1 2 1 1 1 2 1 1 1 2 1 1 1 2 1 1 1 2 1 1 1 1 2 1 1 1 1 2 1 1 1 1 1 2 1					1.33240	70.638	120	1	1	0
Volume [CD] = 30.42 1.23670 77.052 29 0 0 0 0 0 0 0 0 0	Lattice: Hex	agonal	Mol. weight =							
Dra	S.G.: P 63	3/m m c (194)	_	30.42	4.00070	77.050	00	0	0	4
Vicor = 10.960 1.15390 83.758 17 2 0 0 0 0 0 0 0 0 0										4
a = 2.66480 c = 4.94670 a/b = 1.00000 c/b = 1.85631 Primary Reference Wyckoff R. W. G., "Second edition. Interscience Publishers, New York, New YorkHexagonal closest packed, hcp, structure", Crystal Structures 1 (1963) 7-83. Wavelengt h: 1.54060 Filter: Not specified d-spacing:				10.060	1.17300	82.096	142	1	1	2
1.09000 89,934 27 0 1 4			i/icor –	10.900	1.15390	83.758	17	2	0	0
c = 4.94670 a/b = 1.00000 c/b = 1.85631 Primary Reference Wyckoff R. W. G., "Second edition. Interscience Publishers, New YorkHexagonal closest packed, hcp, structure", Crystal Structures 1 (1963) 7-83. Wavelengt h: 1.54060 Filter: Not specified d-spacing:	a = 2.66480				1.12370	86.550	90	2	0	1
a/b = 1.00000 c/b = 1.85631 Primary Reference Wyckoff R. W. G., "Second edition. Interscience Publishers, New York, New YorkHexagonal closest packed, hcp. structure", Crystal Structures 1 (1963) 7-83. Wavelengt h: 1.54060 Filter: Not specified d-spacing:	o - 4.04670				1.09000	89.934	27	0	1	4
Primary Reference Wyckoff R. W. G., "Second edition. Interscience Publishers, New York, New YorkHexagonal closest packed, hcp, structure", Crystal Structures 1 (1963) 7-83. Wavelengt h: 1.54060 Filter: Not specified d- spacing:	a/b				1.04570	94.891	23	2	0	2
Primary Reference Wyckoff R. W. G., "Second edition. Interscience Publishers, New York, New YorkHexagonal closest packed, hcp, structure", Crystal Structures 1 (1963) 7-83. Wavelengt h: 1.54060 Filter: Not specified d- spacing:	= 1.00000									
	Wyckoff R. W. G. York, New YorkH Crystal Structures	, "Second edition exagonal close is 1 (1963) 7-83.	st packed, hcp, struc	ture",	RS YS					
		060	spacing:		_ ~					
	33/FUIVI.									

Figure A.2: COD XRD Pattern of Zn Nanocomposite Powder

Formula	Al2O3	d	2θ	l fix	h	k	I
Name Name (mineral)	Corundum	3.48000	25.577	664	1	0	2
Name (common)	Corundam	2.55090	35.152	984	1	0	4
Status	Status Unknown	2.37950	37.777	453	1	1	0
Ambient	Yes	2.16520	41.681	5	0	0	6
		2.08540	43.354	1000	1	1	3
		1.96420	46.179	14	2	0	2
Lattice: Hexago	onal Mol. weight =		101170		_		_
S.G.: R -3 c (ee.g	. =					
	Dx =	1.74000	52.553	480	2	0	4
	Dm = 1.000	1.60140	57.504	946	1	1	6
	1.000	1.54660	59.743	25	2	1	1
a = 4.75890		1.51480	61.130	31	2	1	2
c = 12.99100		1.51080	61.309	68	1	0	8
a/b		1.40450	66.522	354	2	1	4
= 1.00000		1.37380	68.209	546	3	0	0
c/b = 2.72983		1.33600	70.420	12	2	1	5
		1.27540	74.309	12	2	0	8
		1.2390	76.882	151	1	0	10
		1.23410	77.244	82	1	1	9
		1.19310	80.426	7	2	1	7
		1.18970	80.703	57	2	2	0
		1.16000	83.219	6	3	0	6
		1.14720	84.360	47	2	2	3
		1.13870	85.137	3	3	1	1
		1.12580	86.349	34	3	1	2
		1.12410	86.512	23	2	1	8
		1.09890	89.010	66	2	0	10
		1.08260	90.719	17	0	0	12
		1.07820	91.193	77	3	1	4
Primary Reference	Haar V M IIDaffaaaaa (1)	1.04630	94.820	2	3	1	5
Ti2O3, V2O3 and C	Haan Y. M., "Refinement of the alpha Al2O r2O3 structuresLocality: synthetic", Zeitsch		95.251	156	2	2	6
fur Kristallographie	117 (1962) 235-237.	1.01760	98.397	21	4	0	2
Wavelengt	Filter: Not specified d-						
h: 1.5406 SS/FOM:		NIT					

Figure A.3: COD XRD Pattern of Al₂O₃ Nanocomposite Powder

APPENDIX B

Calculation of weight loss

Formula
$$\Rightarrow$$
 Corrosion rate = $\frac{\text{weight final-weight initial}}{\text{days}} \times 100$

Table B.1: Weight sample for 30 days

Table B.1. Weight sample for 30 days									
Days/	0.5wt%	0.7wt%	1.0wt%	2.0wt%					
Composition									
D-0	20.08g	17.53g	20.15g	21.74g					
D-5	20.10g	17.42g	19.96g	21.73g					
D-10	20.19g	17.41g	20.01g	21.73g					
D-15	20.08g	17.41g	20.01g	21.75g					
D-20	20.05g	17.40g	19.94g	21.71g					
D-25	20.05g	17.41g	20.02g	21.71g					
D-30	20.04g	17.40g	19.94g	21.70g					

Example: Corrosion rate =
$$\frac{20.10g-20.08g}{5 \text{ days}} \times 100$$
$$= 0.4\%$$

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APPENDIX C

Calculation of crystallite size

Copper peak 1

X = 0.0031, Y = 0.3278

Y=mx+C

0.3278 = 0.0020 (0.0031) + C

C = 0.00244

Copper peak 2

X=0.3787, Y= 0.0033

0.0033 = 0.0020 (0.3787) + C

C = 0.002543





Figure C.1: Image of samples after 30 days of immersion