UNIVERSITI MALAYSIA KELANTAN

PROPERTIES OF EPOXY Al_2O_3-TiO_2 NANOCOMPOSITE FOR WELDED STEEL CORROSION RESISTANCE

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Table of Contents

LIST OF TABLES .... **vii**

LIST OF FIGURES .... **viii**

LIST OF ABBREVIATIONS .... **ix**

LIST OF SYMBOLS .... **x**

ABSTRACT .... **xi**

ABSTRAK .... **xii**

CHAPTER 1 .... **1**

INTRODUCTION **1**

1.1 Background of Study 1

1.2 Objectives 3

1.3 Scope of Study 3

1.4 Significances of Study 4

CHAPTER 2 .... **5**

LITERATURE REVIEW **5**

2.1 Epoxy resin 5

2.1.1 Epoxy resin structure 6

2.1.2 Application of epoxy 7

2.2 Epoxy composite 9

2.2.1 Properties of epoxy composite 9

2.2.2 Nano materials reinforced for epoxy composite. 9

2.3 Epoxy composite coating for counter welded steel failure. 11

2.3.1 Failure causes 11

2.3.2 Prevention and solutions 12

2.4 Combination of Al2O3 and TiO2 fillers in epoxy composites 14

2.4.1 Role of Al_2O_3 Filler in Epoxy Composites 14

2.4.2 Role of TiO2 Filler in Epoxy Composites 16

CHAPTER 3 19

MATERIALS AND METHODS 19

3.1 Introduction 19

3.2 Materials 21

3.3 Epoxy-Al_2O_3-TiO_2 nanocomposite composition 21

3.4 Preparation of epoxy-Al_2O_3-TiO_2 thin film 22

3.5 Preparation of coated welded steel 23

3.6 Characterizations 23

3.6.1 X-ray Diffraction 23

3.6.2 Scanning electron microscopy 24

3.6.3 Hardness test 24

3.6.4 Thickness measurement 25

3.6.5 Corrosion test 26

CHAPTER 4 27

RESULT AND DISCUSSION 27

4.1 Phase Identification 27

4.1.2 Crystallite Size and Internal strain 28

4.1.3 Microstructure of As-milled Al_2O_3-TiO_2 Composites......................30

4.2 Epoxy-Al_2O_3-TiO_2 Nanocomposite Film..31

4.2.1 Phase Identification...31

4.2.2 Microstructure of Thin Film Al_2O_3-TiO_2 Nanocomposite................32

4.2.3 Functional Group..34

4.3 Epoxy Al_2O_3-TiO_2 Nanocomposite Coated on Welded Steel.........................37

4.3.1 Thickness..37

4.3.2 Hardness...38

4.3.3 Immersion test...39

CHAPTER 5...**42**

CONCLUSION AND RECOMMENDATION...**42**

5.1 Conclusion...42

5.2 Recommendation..43

REFERENCES..**44**

APPENDIX A..**48**

A.1 XRD analysis of 40 h milled Al_2O_3-TiO_2 nanocomposite powder.................48

A.2 XRD analysis of (0.5) thin film epoxy Al_2O_3-TiO_2 nanocomposite...............49

A.3 XRD analysis of (0.7) thin film epoxy Al_2O_3-TiO_2 nanocomposite...............49

A.4 XRD analysis of (1) thin film epoxy Al_2O_3-TiO_2 nanocomposite 50

A.4 XRD analysis of (2) thin film epoxy Al_2O_3-TiO_2 nanocomposite 50

A.4 FTIR analysis of (0.5,0.7,1 and 2) thin film epoxy Al_2O_3-TiO_2 nanocomposite51

APPENDIX B..**52**

B.A Hardness, thickness, and mass data throughout the corrosion test 52

3.7 B.B Weight change for each sample over 28 days. 53

LIST OF TABLES

PAGE

3.1 Sample designation and composition of Al_2O_3-TiO_2 in epoxy milled at different milling times. 22

LIST OF FIGURES

		PAGE
Figure 2.1	Crystal Structures of Bisphenol A	6
Figure 2.2	Chemical structures of epoxy resin	7
Figure 2.3	Epoxy coating for welded steel	8
Figure 2.4	Schematic illustration of three kinds of ceramic/ polymer composites: (a) dispersed, (b) physically contacted, and (c) chemically bonded.	10
Figure 2.5	Epoxy coating failure	12
Figure 2.6	Crystal structure of alumina oxide	15
Figure 2.7	Crystal structures of TiO2: (a) anatase, (b) rutile, and (c) brookite	16
Figure 3.1	The overall experimental process of this study	20
Figure 3.2	Shore D for measure thickness	25
Figure 3.3	Thickness gauge for measure thickness	26

LIST OF ABBREVIATIONS

Al_2O_3	Aluminium oxide
TiO_2	Titanium dioxide
BPA	Bisphenol A
BPS	Bisphenol S
BPF	Bisphenol F
R'	Organic groups
O	Oxygen atom
(CH2)n	Chain of n methylene units
Al	Aluminium
Ti	Titanium
Nm	Nanometre
UV	Ultraviolet
mm	millimetres
XRD	X-ray diffraction
SEM	Scanning electron microscopy
µm	Micrometers/ Micron
NaOH	Sodium hydroxide

LIST OF SYMBOLS

%	Percentage

PROPERTIES OF EPOXY Al_2O_3-TiO_2 NANOCOMPOSITE FOR WELDED STEEL CORROSION RESISTANCE

ABSTRACT

Aluminum oxide, commonly known as alumina (Al_2O_3), finds widespread application in high-performance contexts owing to its commendable attributes such as high corrosion and wear resistance. Nevertheless, its low bending strength poses a challenge. To overcome this limitation, researchers propose reinforcing Al_2O_3 with materials like TiO_2 to bolster its properties. This study aims to synthesize epoxy Al_2O_3-TiO_2 composite powder via the powder metallurgy route and assess its properties with a milling time of 40 hours for the coating sample. The composite powder was produced through high-energy ball milling at a constant speed of 300 rpm for the specified duration. Subsequently, it was combined with epoxy to create coatings for welded steel and thin films. Phase identification and morphology analysis were conducted using X-ray diffraction (XRD) and scanning electron microscopy (SEM) respectively, while Fourier-transform infrared spectroscopy (FTIR) was employed for characterization. Crystal size and internal strain of Al_2O_3-TiO_2 were determined using the Williamson-Hall method. Additionally, coated welded steel underwent immersion in NaOH for 28 days to assess changes in weight loss, hardness, and thickness, measured using tools such as the Shor D gauge, thickness gauge, and scales. Following the 28-day immersion period, significant variations were observed among the samples. Notably, the sample with a composition of Al_2O_3-TiO_2 at 1wt% exhibited the lowest weight loss. Furthermore, the epoxy Al_2O_3-TiO_2 composite at 1wt% experienced the least decrease in thickness during the immersion period, with the hardness test indicating higher hardness compared to others. These findings underscore the potential of epoxy Al_2O_3-TiO_2 composites in enhancing material properties, offering valuable insights for various industrial applications.

Keywords: Epoxy, Al_2O_3-TiO_2 composite, internal strain ,Williamson-Hall method, Phase identification, morphology, NaOH,

SIFAT-SIFAT NANOKOMPOSIT EPOXY Al_2O_3-TiO_2 UNTUK KETAHANAN HAKISAN KELULI KIMPALAN

ABSTRAK

Aluminium oksida, biasanya dikenali sebagai alumina (Al_2O_3), mendapat aplikasi yang meluas dalam konteks berprestasi tinggi kerana sifatnya yang terpuji seperti kakisan yang tinggi dan rintangan haus. Namun begitu, kekuatan lenturannya yang rendah menimbulkan cabaran. Untuk mengatasi batasan ini, penyelidik mencadangkan pengukuhan Al_2O_3 dengan bahan seperti TiO_2 untuk memperkukuh sifatnya. Kajian ini bertujuan untuk mensintesis serbuk komposit epoksi Al_2O_3-TiO_2 melalui laluan metalurgi serbuk dan menilai sifatnya dengan masa pengilangan selama 40 jam untuk sampel salutan. Serbuk komposit dihasilkan melalui pengilangan bebola tenaga tinggi pada kelajuan malar 300 rpm untuk tempoh yang ditetapkan. Selepas itu, ia digabungkan dengan epoksi untuk mencipta salutan untuk keluli dikimpal dan filem nipis. Pengenalpastian fasa dan analisis morfologi telah dijalankan masing-masing menggunakan pembelauan sinar-X (XRD) dan mikroskop elektron pengimbasan (SEM), manakala spektroskopi inframerah-transformasi Fourier (FTIR) digunakan untuk pencirian. Saiz kristal dan ketegangan dalaman Al_2O_3-TiO_2 ditentukan menggunakan kaedah Williamson-Hall. Selain itu, keluli dikimpal bersalut menjalani rendaman dalam NaOH selama 28 hari untuk menilai perubahan dalam penurunan berat, kekerasan dan ketebalan, diukur menggunakan alat seperti tolok Shor D, tolok ketebalan dan penimbang. Selepas tempoh rendaman 28 hari, variasi ketara telah diperhatikan di kalangan sampel. Terutama, sampel dengan komposisi Al_2O_3-TiO_2 pada 1wt% menunjukkan penurunan berat badan yang paling rendah. Tambahan pula, komposit Al_2O_3-TiO_2 epoksi pada 1wt% mengalami penurunan ketebalan paling sedikit semasa tempoh rendaman, dengan ujian kekerasan menunjukkan kekerasan yang lebih tinggi berbanding dengan yang lain. Penemuan ini menggariskan potensi komposit epoksi Al_2O_3-TiO_2 dalam meningkatkan sifat bahan, menawarkan pandangan berharga untuk pelbagai aplikasi perindustrian.

Kata kunci: Epoksi, komposit Al_2O_3-TiO_2, terikan dalaman, kaedah Williamson-Hall, Pengenalpastian fasa, morfologi, NaOH

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Epoxy resins have excellent chemical resistance to a range of solvents and other chemicals, which makes them useful in harsh environments. They are applied as polymer coating in a sealed chemical metal container. Epoxy composites have gained tremendous attention due to their high mechanical toughness and moisture absorption properties(Lapčík et al., 2023). However, the limitation of this material is its brittle nature (mainly due to the low stiffness and low fracture toughness of resin) that leads to reduced properties that are matrix dominated, including impact strength, compressive strength, in-plane shear, fracture toughness, and interlaminar strength.

Epoxy composite coating materials are made of a mixture of epoxy resin, filler, and additives. The addition of filler significantly affects the mechanical, thermal, and chemical properties of the composite. Besides, they also provide good wear resistance and stability to the composite. Various fillers that can be added to improve the properties of the matrix (Choi et al., 2021). Many types of reinforcement are used in epoxy composite including fiberglass, carbon fiber, wood fibers, microspheres, silica, talc, calcium carbonate, metal powders, and more. These fillers serve different purposes depending on the desired properties of the composite materials. Inorganic particles in organic polymers are of considerable interest because it makes it possible to create hybrid nanocomposite materials with improved properties such as heat resistance, fire resistance, reduced gas permeability, and resistance to chemicals (Bogdanova et al., 2020).

Aluminium oxide (Al_2O_3) is an excellent permeability barrier, and titanium dioxide (TiO_2) is chemically more durable and has high corrosion resistance (Staszuk et al., 2022). The Al_2O_3-TiO_2 nanocomposite can improve the epoxy composite's mechanical strength, hardness, wear resistance, and corrosion resistance (Choi et al., 2021). The high melting point and heat resilience of Al_2O_3-TiO_2 nanocomposites have excellent thermal stability of the epoxy composite. These characteristics qualify them for high-temperature applications where coatings must endure high temperatures without degrading. Epoxy Al_2O_3-TiO_2 nanocomposites could provide unique qualities that make them appropriate for a wide range of applications, including metal coatings. Their combination has synergistic effects that improve the coating's capacity to protect the substrate from environmental factors.

It is interesting to combine epoxy with Al_2O_3-TiO_2 nanocomposites filler as polymer coating materials. Apart from the less studied in the literature, they could provide good results on corrosion performance on welded steel protection. Thus, this study aims to prepare epoxy composite with the addition of different content of Al_2O_3-TiO_2 nanocomposites. The performance of the epoxy-Al_2O_3-TiO_2 nanocomposites as coating materials for welded steel will be discussed and evaluated.

1.2 Objectives

The objectives of this study are:

1. To prepare the epoxy nanocomposite with addition of Al_2O_3-TiO_2 nanocomposite powder milled at 40 hours.

2. To evaluate the physical properties and corrosion performance of the epoxy- Al_2O_3-TiO_2 nanocomposites at different filler loading.

1.3 Scope of Study

In this study, the influence of varying concentrations of Al_2O_3 and TiO_2 on the properties of the nanocomposite coating will be investigated. X-ray diffraction (XRD) analysis will be employed to determine the composition of the epoxy-Al_2O_3-TiO_2 nanocomposites. Understanding the precise composition of the coating will provide valuable insights into the potential of using epoxy-Al_2O_3-TiO_2 nanocomposites as a cost-effective and environmentally friendly solution for corrosion prevention in welded steel structures. By analysing the composition and evaluating the properties of the nanocomposite coating, this research aims to contribute to the development of effective protective coatings with enhanced corrosion resistance for welded steel structures.

1.4 Significances of Study

Epoxy-Al_2O_3-TiO_2 nanocomposites on welded steel should be studied which potentially to improve the anticorrosive characteristics of welded steel structures. A combination between epoxy and Al_2O_3-TiO_2 nanocomposite could offer an alternative material for metal protective coating. As a result, investigating the characteristics of epoxy-Al_2O_3-TiO_2 on welded steel can aid in determining the best circumstances for their application and assist to the creation of stronger and more corrosion-resistant welded steel structures. This might increase safety and dependability in industries that depend on welded steel structures, such as building and manufacturing.

CHAPTER 2

LITERATURE REVIEW

2.1 Epoxy resin

Epoxy resin is a type of synthetic resin that is derived from a class of polymers called epoxides. It is a versatile and popular material known for its strong adhesive properties, durability, and chemical resistance. Epoxy resin material is a three-dimensional network polymer formed by the interaction of epoxy group and curing agent (Wei et al., 2023).

The epoxy resin itself is a liquid polymer that is typically clear or transparent. It is derived from a reaction between bisphenol A (BPA) or bisphenol F (BPF) and epichlorohydrin. Bisphenol A (BPA) and its substitutes bisphenol S (BPS) and bisphenol F (BPF) are endocrine disrupting chemicals widely used in the production of polycarbonate plastics, epoxy resins and thermal papers (Bousoumah et al., 2021). Epoxy resins come in different viscosities, allowing for various applications ranging from thin coatings to thick laminates.

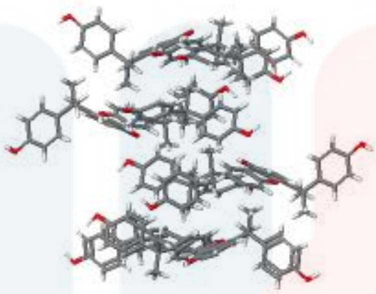


Figure 2.1: Crystal Structures of Bisphenol A

Source: PubChem (2005)

2.1.1 Epoxy resin structure

The structure of epoxy resin can vary depending on the specific type of epoxy used and the presence of additional functional groups. Epoxy resins, also known as polyepoxides, belong to the group of thermoset materials containing a reactive oxirane group (Matykiewicz et al., 2022). Epoxy resins can be modified with various additives, fillers, or reactive diluents to tailor their properties for specific applications. These modifications can alter the chemical structure of the resin and influence it perform ance characteristics. The general chemical formula of epoxy resin can be represented as:

R-O-(CH2)n-O-R'

Epoxy resin consists of a molecular structure represented by R-O-(CH2)n-O-R', where R and R' are organic groups, O denotes an oxygen atom, and (CH2)n represents a chain of n methylene units. This structure forms the building blocks of epoxy resin molecules. The organic groups R and R' can vary, giving different epoxy resins unique properties. The oxygen atoms form a three-membered ring known as an epoxy group or glycidyl group, which is highly reactive. The chain of methylene units provides flexibility and adjusts the length of the epoxy chain, influencing the overall characteristics of the resin. When multiple R-O-(CH2)n-O-R' units are linked together, they form a polymer chain, resulting in the epoxy resin. This molecular structure of epoxy resin is crucial in understanding its behaviour and the effects it imparts when added to materials like metal oxide.

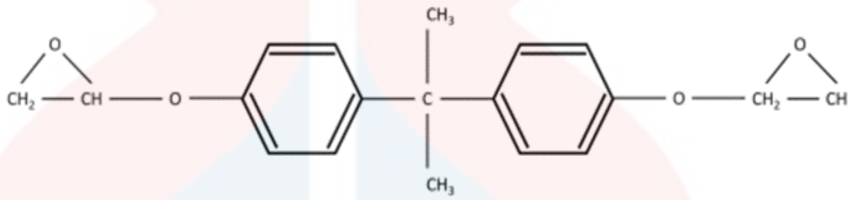


Figure 2.2: Chemical structures of epoxy resin

Source: Choi et al (2021)

2.1.2 Application of epoxy

Epoxy resin finds extensive use across industries due to its versatile properties. One common application is in adhesives and bonding, where it provides strong adhesion to a variety of substrates in construction, automotive, aerospace, and electronics (Zhao et al., 2022). It is also utilized in coatings and paints, offering durability, chemical resistance, and corrosion protection for surfaces in flooring systems, marine environments, and automotive applications (Yakdoumi et al., 2020).

Epoxy resin has diverse applications, including its use in composites and reinforcements. When combined with materials like fiberglass, carbon fiber, or aramid fibers, it forms lightweight and high-strength composite structures, widely employed in aerospace, automotive, and sporting goods industries. Additionally, epoxy resin is effective in mold making and casting, enabling the production of intricate replicas and laminated materials for composite mold manufacturing. In the realm of art and crafts, epoxy resin finds use in creating jewelry, art pieces, and decorative items, offering flexibility in design by incorporating pigments, dyes, and additives to achieve desired colors and effects. The addition of nano-fillers to modified epoxy resin further enhances its interfacial properties, leading to improved tribological and mechanical performance. Overall, epoxy resin exhibits versatility across multiple industries and creative endeavors (Cao J et al., 2022).

Figure 2.3: Epoxy coating application for welded steel

Source: Denso (2023)

2.2 Epoxy composite

Epoxy composites refer to materials that are composed of epoxy resin as the matrix and reinforced with fibers or particles to enhance their mechanical properties. The reinforcement materials can vary, but commonly used options include fiberglass, carbon fibers, aramid fibers, and various types of particles such as metal oxide.

2.2.1 Properties of epoxy composite

The combination of epoxy resin with reinforcing materials results in a composite material that exhibits improved strength, stiffness, and durability compared to the pure epoxy resin. Good tribological properties can be obtained for polymers filled with nano-scale fillers compared to that filled with micro-scale particles (Abass, 2019).

2.2.2 Nano materials reinforced for epoxy composite.

Epoxy has been widely used as polymer matrix for composite applications (Zhao et al., 2022). The choice of reinforcement and filler materials for epoxy composites depends on the specific requirements of the application. One common option for reinforcement is alumina (Al_2O_3), a ceramic material that offers excellent mechanical properties and high temperature resistance. Alumina particles provide strength, stiffness, and improved thermal stability to the composite. Another option for reinforcement is titanium dioxide (TiO_2), a versatile material known for its high strength and durability. When nano TiO_2 material is reinforced with metal matrix composites, it has the potential to produce materials suitable for high temperature applications due to its high thermal conductivity, outstanding mechanical properties, and attractive damping properties (Raghu et al., 2022).

As for filler materials, alumina (Al_2O_3) particles are often used in epoxy composites to improve mechanical properties, reduce cost, and enhance dimensional stability. These particles can increase the hardness, wear resistance, and thermal conductivity of the composite. Titanium dioxide (TiO_2) particles, on the other hand, are chosen for their excellent UV resistance and ability to improve the mechanical and thermal properties of epoxy composites. They can enhance the strength, stiffness, and heat resistance of the composite material.

Furthermore, the combination of Al_2O_3 and TiO_2 fillers in epoxy composites can lead to synergistic effects. The Al_2O_3 particles act as reinforcement, providing strength and stiffness to the composite, while TiO_2 particles contribute to improved adhesion between the filler and the epoxy matrix. This synergistic effect can lead to enhanced overall performance and durability of the composite material. By adding fillers to epoxy resin to strengthen the mechanical properties of epoxy resin composite materials, the tribological performances of epoxy resin composites are improved (Cao J et al.,, 2022).

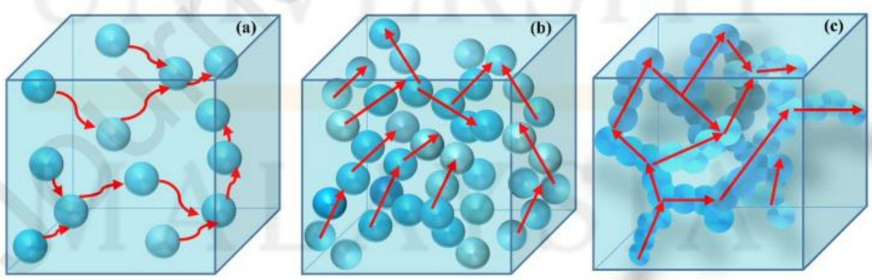


Figure 2.4: Schematic illustration of three kinds of ceramic/polymer composites: (a) dispersed, (b) physically contacted, and (c) chemically bonded.

Source: Hao et al (2019)

2.3 Epoxy composite coating for counter welded steel failure.

Epoxy coatings for counter welded steel are protective layers applied to steel surfaces that have undergone welding, particularly in applications where two welded steel components meet or counter each other.

2.3.1 Failure causes

Epoxy composite coating failures on counter welded steel can occur due to various factors. One common cause is improper surface preparation. Inadequate surface cleaning, insufficient removal of rust, mill scale, or contaminants, and poor surface roughening can hinder the adhesion of the epoxy coating, leading to premature failures. Another factor is the welding-induced heat. The high temperatures generated during welding can affect the underlying epoxy coating. According to Junjie Wen (2022), increasing temperature, the behavior of the composite begins to change from interface debonding to resin matrix failure. Excessive heat can cause thermal degradation of the epoxy, resulting in reduced adhesion, blistering, or cracking of the coating.

Poor weld quality is also a significant contributor to epoxy coating failures. If the welding process is not performed correctly, it can introduce defects such as weld spatter, undercutting, or porosity. These defects can compromise the integrity of the epoxy coating, allowing moisture or chemicals to penetrate, leading to coating delamination and corrosion of the steel substrate.

Mechanical stress is another critical factor. Counter welded steel structures can experience mechanical stresses due to load-bearing or dynamic forces. Excessive flexing, vibration, or impact can cause the epoxy coating to crack or peel, exposing the steel to potential corrosion. According to Wu (2022), the three-dimensional network structure of epoxy resin leads to poor fracture toughness, which easily causes cracking and consolidation under complex loads conditions, resulting in fatigue damage and ultimate failures.

In summary, the process of destruction gives rise to flaws, making it easier for corrosive elements like oxygen, water molecules, and ions to penetrate the substrate's surface via cracks and openings. This ultimately leads to corrosion, undermining the effectiveness of long-term corrosion protection measures.

Figure 2.5: Epoxy coating failure

Source: Dai et al (2018)

2.3.2 Prevention and solutions

To prevent epoxy coating failures on counter welded steel, a comprehensive approach involving various measures can be implemented. Firstly, proper surface preparation is essential. This involves meticulously cleaning the steel surface to remove rust, mill scale, and contaminants. Additionally, ensuring proper surface roughening promotes strong adhesion of the epoxy coating.

Controlling welding parameters is another critical step. By implementing appropriate welding techniques and maintaining proper heat input, the adverse effects on the epoxy coating can be minimized. It is important to minimize the heat-affected zones during welding, as excessive heat can lead to thermal degradation of the epoxy coating, resulting in reduced adhesion, blistering, or cracking.

Quality welds are crucial for preventing epoxy coating failures. This requires following proper welding procedures, providing adequate training to welders, and conducting thorough inspections to ensure minimal defects such as weld spatter, undercutting, or porosity. High-quality welds contribute to the integrity of the epoxy coating and prevent moisture or chemicals from penetrating the coating.

Optimal application of the epoxy coating is vital. It is essential to apply the coating with the recommended thickness and ensure full coverage on all welded surfaces, particularly critical areas where two components meet. Inadequate coating thickness or coverage can lead to localized areas where the steel is not adequately protected, making those areas prone to corrosion and coating failures.

In high-stress or corrosive environments, considering additional protective measures becomes important. This may involve using corrosion inhibitors, which can provide added protection against corrosion. Applying multiple layers of epoxy coating can offer enhanced durability. Alternatively, exploring alternative coating systems that are better suited to specific environmental conditions can be beneficial.

Regular inspection and maintenance play a crucial role in preventing epoxy coating failures. Implementing a scheduled inspection program helps identify any coating damage or corrosion at an early stage. Prompt repair of damaged coatings and addressing underlying issues in a timely manner prevents further coating failures and corrosion of the steel substrate.

By implementing these preventive measures, including proper surface preparation, controlled welding parameters, quality welds, optimal coating application, considering additional protective measures, and regular inspection and maintenance, the risk of epoxy coating failures on counter welded steel can be minimized. This ensures improved durability and protection of the steel structures over the long term.

2.4 Combination of Al2O3 and TiO2 fillers in epoxy composites

The combination of Al_2O_3 (aluminium oxide) and TiO_2 (titanium dioxide) fillers in epoxy composites offers several advantages and can enhance the properties of the composite material. Hybrid nanofillers evidence that, the physical and mechanical characteristics of nanocomposites enhance better compared to using one type of fillers (Nabhan et al., 2023). When these fillers are incorporated into the epoxy resin matrix, they interact synergistically to create a composite with improved mechanical, thermal, and electrical properties.

2.4.1 Role of Al_2O_3 Filler in Epoxy Composites

Aluminium oxide exhibits a ceramic composition characterized by a hexagonal crystal lattice. Within this lattice, the oxygen anions form a hexagonal close-packed structure, while the aluminium cations occupy approximately two-thirds of the octahedral sites present in the hcp lattice. Due to the substantial charge (3+) carried by the aluminium cations, they strive for maximum separation within the structure. A diagram illustrates the structural unit cell. Notably, the basal planes of the cell contain vacant Al-sites, resulting in a repetitive pattern every six layers, giving rise to a c0 value of 1.299 nm.

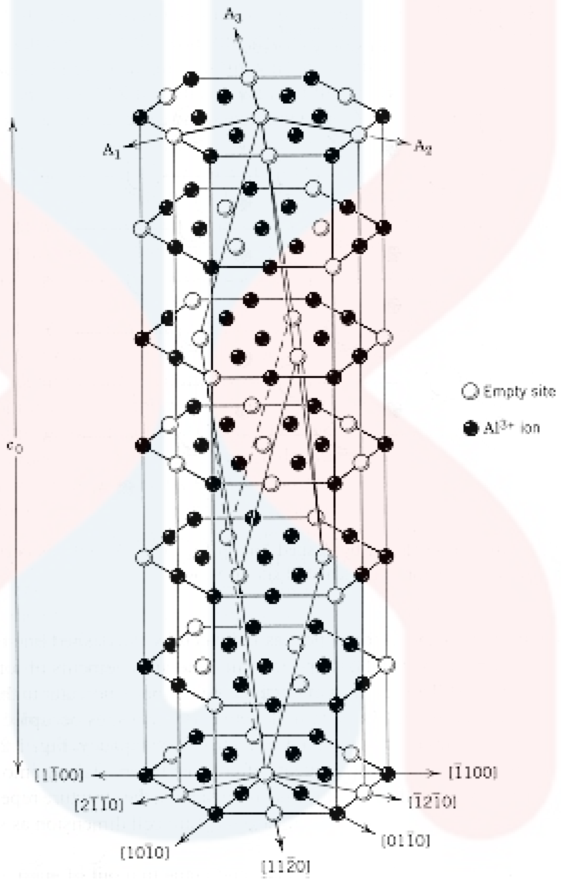


Figure 2.6: Crystal structure of alumina oxide

Source: MIT (1997)

Aluminium oxide filler is commonly used in epoxy composites due to its excellent mechanical and thermal properties. When incorporated into the epoxy resin matrix, Al_2O_3 fillers serve multiple roles in enhancing the overall performance of the composite material. Al_2O_3 nanoparticles is one of metal nano-oxides has good enough properties to play a main role in increasing and improving the various properties of polymer composite (Nabhan et al., 2023). Firstly, Al_2O_3 fillers act as reinforcing agents, improving the stiffness and strength of the epoxy composite. The high hardness and rigidity of Al_2O_3 particles provide resistance against deformation and crack propagation, resulting in improved mechanical properties such as increased tensile strength and flexural modulus. Moreover, Al_2O_3 fillers enhance the abrasion resistance of the composite, making it suitable for applications that require wear resistance, such as coatings, linings, and tooling.

Additionally, the presence of Al_2O_3 fillers in epoxy composites contributes to improved thermal conductivity. Aluminium oxide is known for its high thermal conductivity, allowing for efficient dissipation of heat within the material. This property is particularly advantageous in applications where thermal management is critical, such as electronic devices or heat sinks. By enhancing thermal conductivity, Al_2O_3- fillers help to dissipate heat and prevent localized temperature buildup, thus improving the thermal stability and resistance to thermal degradation of the epoxy composite.

2.4.2 Role of TiO2 Filler in Epoxy Composites

Titanium oxide (TiO2) exhibits several crystal structures, with the most common ones being rutile, anatase, and brookite. Each of these structures has unique arrangements of titanium and oxygen atoms.

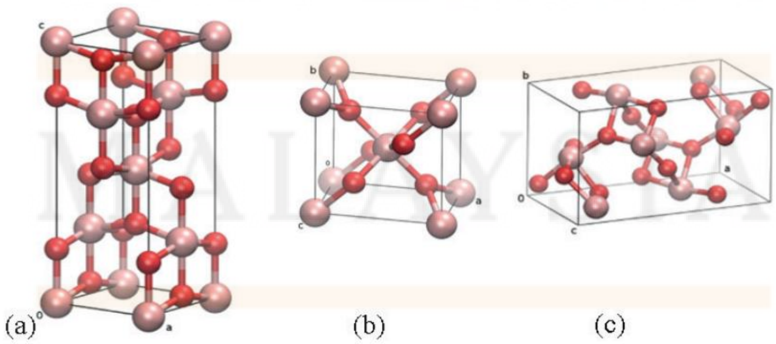


Figure 2.7: Crystal structures of TiO2: (a) anatase, (b) rutile, and (c) brookite

Source: Scarpelli et al (2018)

The rutile crystal structure consists of Ti atoms octahedrally surrounded by oxygen atoms, and each oxygen has three Ti atoms as neighbours (Mashimo et al., 2017). Rutile is the most stable and commonly occurring form of titanium dioxide. It has a tetragonal crystal structure, characterized by octahedrally coordinated titanium atoms surrounded by oxygen atoms. The titanium atoms are arranged in a three-dimensional network, forming a distorted lattice structure.

Anatase is another polymorph of TiO2 and has a tetragonal crystal structure. It consists of octahedrally coordinated titanium atoms surrounded by oxygen atoms. Anatase has a different arrangement of atoms compared to rutile, resulting in distinct physical and chemical properties.

Brookite is the least common polymorph of TiO2. It has an orthorhombic crystal structure, with titanium atoms coordinated by six oxygen atoms. The arrangement of atoms in brookite is different from rutile and anatase, leading to unique crystal properties. These different crystal structures of titanium oxide contribute to variations in its physical and chemical properties, including optical properties, photocatalytic activity, and stability (Scarpelli et al., 2018).

Titanium dioxide filler is widely utilized in epoxy composites due to its unique properties and versatility. When incorporated into the epoxy resin matrix, TiO_2 fillers offer several benefits that enhance the performance of the composite material. TiO_2 nanoparticles are worth mentioning due to their versatile properties including high thermo-mechanical stability, wear resistance, corrosion resistance and wide bandgap (Sagar et al., 2022). One key role of TiO_2 fillers is their contribution to improved electrical insulation. Titanium dioxide is a semiconductor material with excellent dielectric properties. It exhibits high resistivity and low dielectric constant, making it suitable for applications requiring electrical insulation, such as electronic components, circuit boards, and insulating coatings. According to Ilhamdi (2021) TiO_2 is widely used because of its mechanical, optical, dielectric, anti-corrosion, and biocompatibility. By incorporating TiO_2 fillers, the epoxy composite can achieve enhanced dielectric strength and insulation capabilities, reducing the risk of electrical breakdown or leakage currents.

Furthermore, TiO_2 fillers can provide UV resistance to epoxy composites. Titanium dioxide has inherent UV-blocking properties, effectively absorbing and scattering ultraviolet radiation. Nano TiO_2 in the epoxy resin greatly enhanced, and the mechanical and heat resistance properties of the composite materials (Xia et al., 2023). This characteristic is beneficial for applications exposed to sunlight or UV radiation, as it helps to prevent degradation and discoloration of the epoxy composite caused by UV exposure. The UV resistance offered by TiO_2 fillers contributes to the long-term durability and colour stability of the composite material.

CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

The preparation of the epoxy-Al_2O_3-TiO_2 nanocomposite involved two stages. First, the Al_2O_3-TiO_2 nanocomposite was prepared using high-energy ball milling. Subsequently, the Al_2O_3-TiO_2 nanocomposite was mixed with epoxy. Thin films and coatings were produced on welded steel to evaluate their properties and corrosion performance, respectively. The overall experiment in this study is depicted in Figure 3.1.

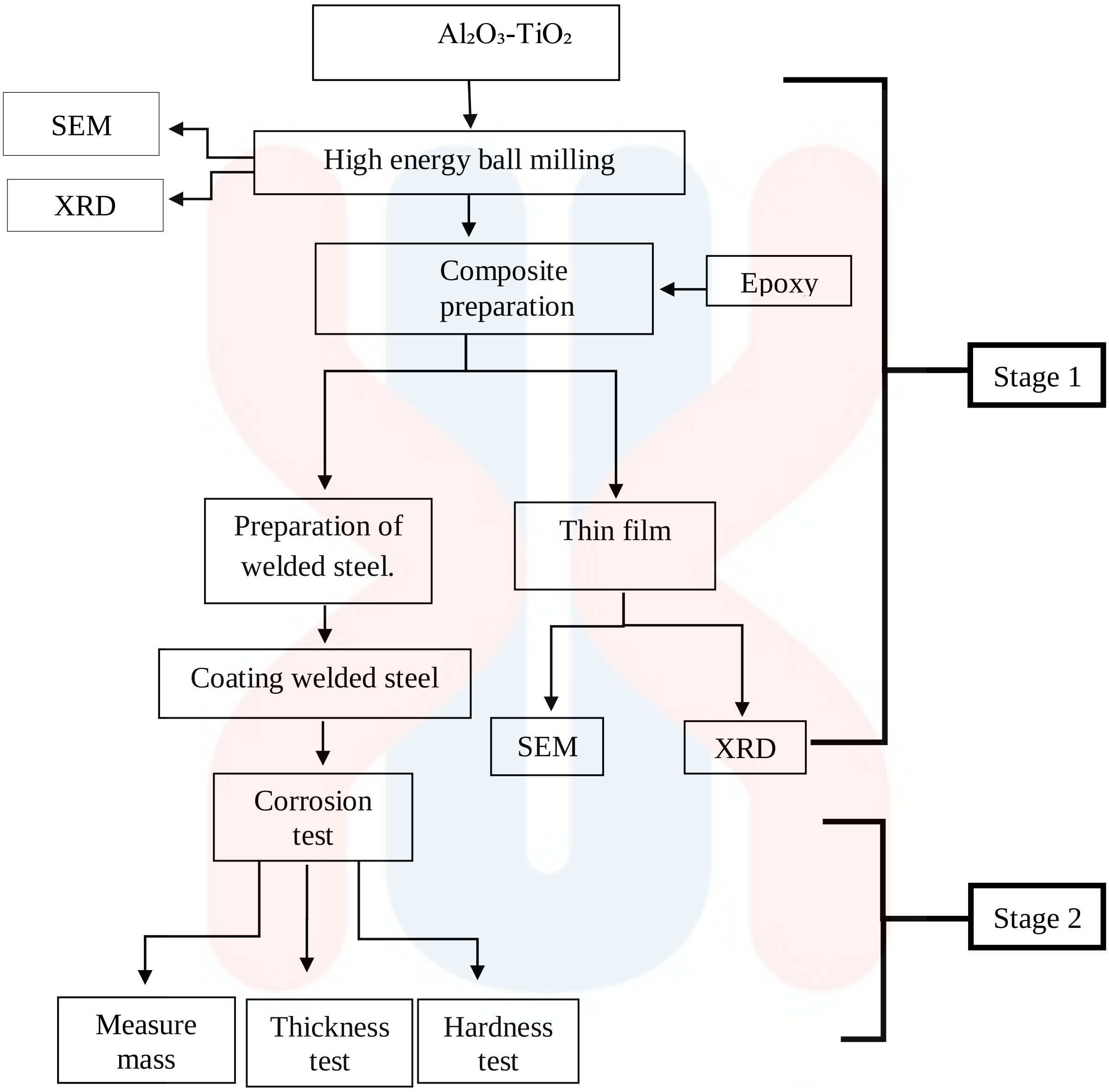


Figure 3.1: The overall experimental process of this study

3.2 Materials

The main materials of this study were alumina powder (Al_2O_3) and titania (TiO_2), both obtained from the material technology lab. This material was used as an additive for coating and thin film, with epoxy serving as a filler for the samples. The Al_2O_3-TiO_2 composite served as a versatile material with potential applications in various fields. In this study, it was combined with epoxy, which acted as a filler for the samples. This combination aimed to harness the unique characteristics of Al_2O_3 and TiO_2 while taking advantage of epoxy's bonding and structural properties.

3.3 Epoxy-Al_2O_3-TiO_2 nanocomposite composition

The preparation of the epoxy-Al_2O_3-TiO_2 nanocomposite involved the dispersion of Al_2O_3-TiO_2 nanocomposite particles in the epoxy matrix. The epoxy was mixed in speed 300 rpm with Al_2O_3-TiO_2 at different compositions, as shown in Table 3.1. Subsequently, the epoxy-Al_2O_3-TiO_2 nanocomposite was prepared for thin film and coated on welded steel.

Table 3.1: Sample designation and composition of Al_2O_3-TiO_2 in epoxy milled at different milling times.

Sample designation	Epoxy (wt%)	Al_2O_3 and TiO_2 (wt%)	Milling time (h)
E-0.5 AT	99.5	0.5	40
E-0.7 AT	99.3	0.7	40
E-1 AT	99	1	40
E-2 AT	98	2	40

3.4 Preparation of epoxy-Al_2O_3-TiO_2 thin film

The purpose of preparing the epoxy-Al_2O_3-TiO_2 nanocomposite thin film was to determine the dispersion of Al_2O_3-TiO_2 in the epoxy. A thin layer of epoxy-Al_2O_3-TiO_2 nanocomposite was applied on the petri dish to a thickness of at least 1.0 mm. The thin film was subjected to X-ray diffraction (XRD) and scanning electron microscopy (SEM). Additionally, a hardness test and thickness test was also performed.

3.5 Preparation of coated welded steel

Prior to applying the epoxy-Al_2O_3-TiO_2 coating on welded steel, proper surface preparation processes were taken to ensure perfect adhesion and coating performance. Several critical processes were involved in the surface preparation process.

The first step was to carefully clean the welded steel surface to eliminate contaminants that may interfere with efficient adhesion, such as dirt, oil, grease, corrosion, scale, or previous coatings. This was done using a proper cleaning solution, solvent, or degreaser to ensure that all contaminants were completely removed. After cleaning, the surface was rinsed with clean water to remove any residue.

A grinding and polishing method was employed to obtain a surface with a mirror-like finish. The grinding was performed using silicon carbide paper with 600 to 1200 grit. Then, after washing and roughening the surface, it was important to fully dry the surface. This step ensured that any residual moisture or detergents were removed. Moisture on the surface could affect adhesion and risk the performance of the epoxy-Al_2O_3-TiO_2 coating.

3.6 Characterizations

3.6.1 X-ray Diffraction

XRD (X-ray diffraction), a technique commonly employed to analyze the crystal structure and phase composition of materials, particularly thin films, was utilized in the case of epoxy-Al_2O_3-TiO_2 composite thin films to offer valuable insights into their structural properties.

By examining the diffraction patterns obtained from the thin films, various pieces of information were deduced. The primary focus was usually on the crystal structure and phase composition of the material. XRD enabled the identification of different crystalline phases present in the thin films, such as the Al_2O_3 and TiO_2 crystal structures, as well as any changes caused by the epoxy matrix.

Furthermore, XRD provided significant details about the orientation and preferred orientation of crystal planes within the thin films. This information could offer insights into the growth mechanism and texture development of nanocomposite thin films during the fabrication process.

3.6.2 Scanning electron microscopy

Scanning Electron Microscopy (SEM) was another powerful technique that complemented X-ray diffraction (XRD) in the characterization of materials, including epoxy-Al_2O_3-TiO_2 composite thin films. While XRD focused on the crystal structure and phase composition, SEM provided detailed surface morphology and topographical information at a micro- to nanoscale level.

3.6.3 Hardness test

The Shore D hardness test was a frequently employed method for assessing the hardness of thin film epoxy-Al_2O_3-TiO_2 nanocomposites. It entailed the use of a durometer equipped with a sharp indenter to penetrate the material's surface and determine its hardness.

Figure 3.2: Shore D for measure thickness

3.6.4 Thickness measurement

To measure the thickness of epoxy-Al_2O_3-TiO_2 coatings, a thickness gauge was utilized. The most used unit of measurement for coating thickness was the micrometer (µm) or micron (µm), which is equivalent to one millionth of a meter. Micrometers were widely employed in the industry for precise and accurate coating thickness measurements.

Figure 3.3: Thickness gauge for measure thickness

3.6.5 Corrosion test

To carry out corrosion tests for epoxy-Al_2O_3-TiO_2 coatings and evaluate their performance, immersion in a 1 molar NaOH (sodium hydroxide) solution was used. During immersion in the NaOH solution, coated samples were monitored and evaluated periodically for any visible signs of coating deterioration, such as blistering, delamination, discoloration, or loss of adhesion. A minimal mass change indicated that the coating effectively protected the substrate from alkali corrosion, while a significant increase in mass suggested coating degradation and possible penetration of the corrosive media.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Phase Identification

The as-milled Al_2O_3-TiO_2 nanocomposite powders' structural characteristics were determined through X-ray diffraction (XRD). Diffract.Eva software was employed to establish matched patterns for the nanocomposite powders. The data extracted from the peak patterns were utilized to compute both the crystallite size and internal strain.

4.1.1 Phase Identification of Milled Powder composite

The XRD pattern for the as-milled nanocomposite powder milled at 40 h is shown in Figure 4.1. The most intense peaks in the XRD pattern at around 2θ = 35°, 43°, and 52° which can be attributed to aluminum oxide (COD9009671). This indicates that Al_2O_3 is the major phase in the composite. The smaller peaks at around 2θ = 36°, 38°, and 45° can be assigned to titanium dioxide (COD 5000223). The broadening of the peaks, particularly those of TiO_2, is due to the small size of the crystallites in the composite. Collisions such as ball-to-ball and ball-to-cotainer walls during ball milling processes can break down larger crystals into smaller ones, resulting in broader peaks in the XRD pattern.

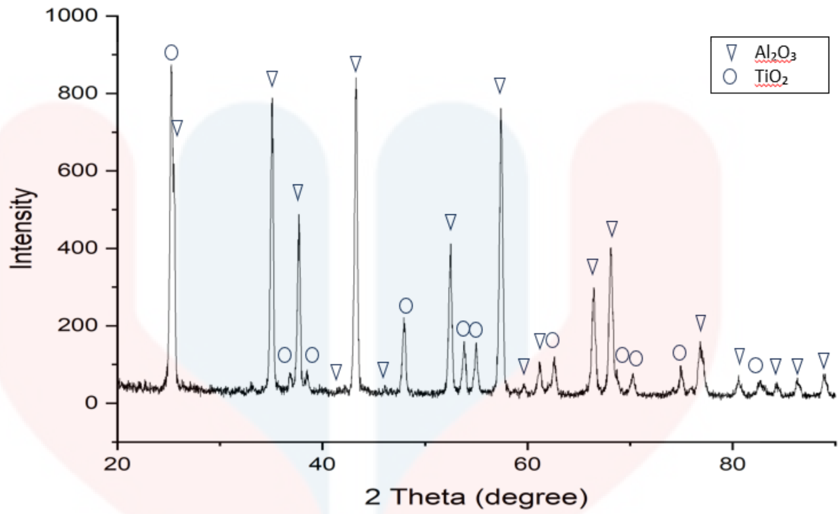


Figure 4.1: XRD pattern of Al_2O_3-TiO_2 composite powder at 40 h

4.1.2 Crystallite Size and Internal strain

The results obtained from the XRD analysis (Figure 4.1) were used to predict the crystallite size and internal strain of the Al_2O_3-TiO_2 composite powder by calculating broadening area of the peak using a Williamson-Hall (WH) method. In this study, the Al_2O_3, crystallite size was determined from full width at half maximum (FWHM) and angle was extracted from obs.max in Diffrac.EVA software of hkl (1, 0, 4) and (1, 1, 3). The plotted graph of $B_r cos\ \theta$ against $sin\ \theta$ can be used to observe the relationship between the Al_2O_3-crystallite size and the internal strain. The crystallite size was presented by y-intercept and strain was extracted from the slope of the lines.

The data derived from WH method was plotted as $B_r cos\ \theta$ against $sin\ \theta$ is shown in Figure 4.2. The plot was drawn linearly indicating that the structural properties of Al_2O_3-TiO_2 nanocomposite powder are influenced by both crystallite size and internal strain. The Al_2O_3 crystallite size and internal strain of as-milled Al_2O_3-TiO_2 powder milled at 40 h is presented in Table 4.1. During the milling process, the powder undergoes continuous deformation, resulting in an accumulation of dislocations and continuous refinement of crystallite size. As a result, there is an observed inverse variation between Al_2O_3 internal strain and crystallite size for all milling times.

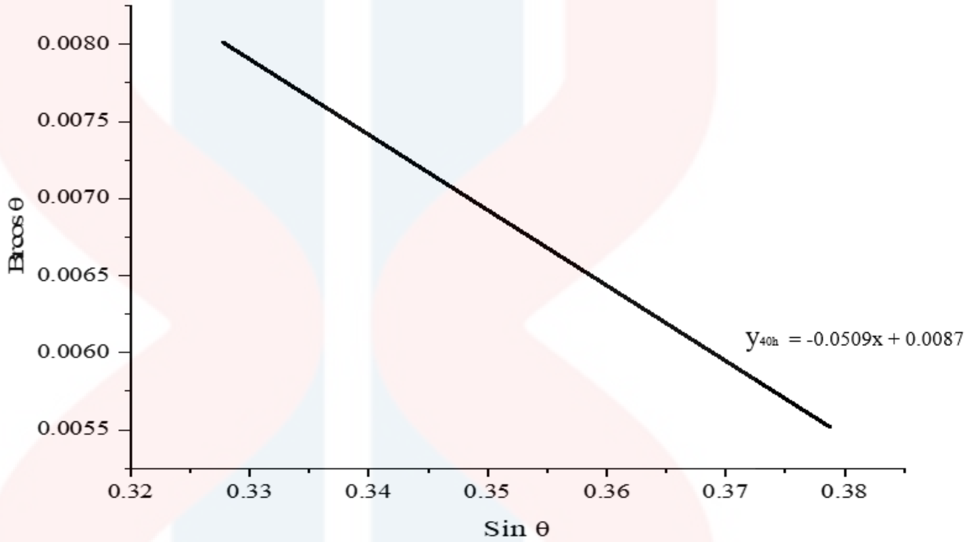


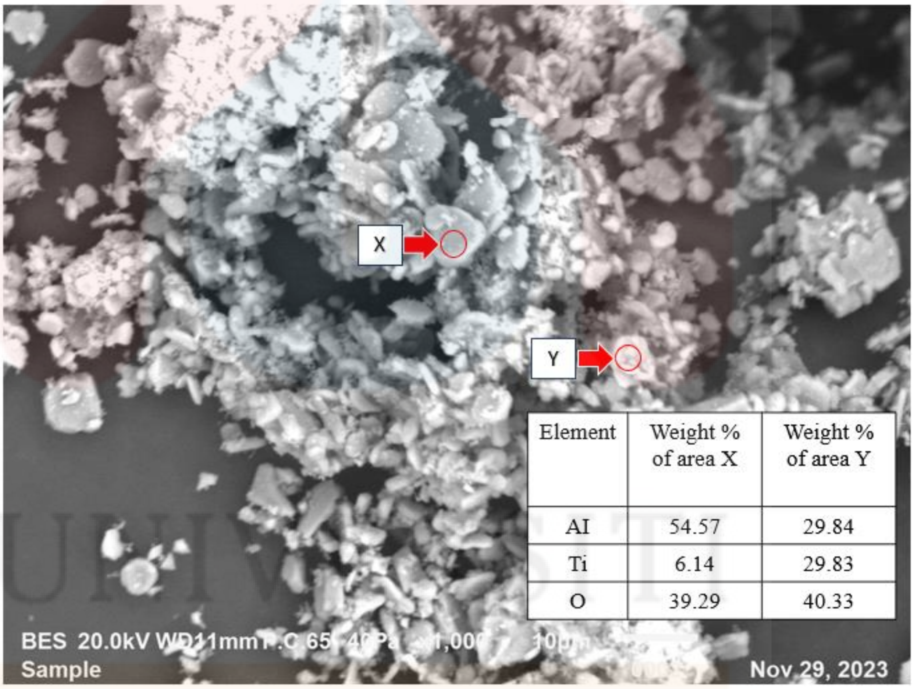
Figure 4.2: Plot of $B_r cos\ \theta$ against *sin θ* for calculation of crystallite size and internal strain of milled powder

Crystallite Size (nm)	Internal Strain (%)
24.1	0.0490

Table 4.1: Al_2O_3 crystallite size and internal strain of as-milled Al_2O_3-TiO_2 powder milled at 40 h

4.1.3 Microstructure of As-milled Al_2O_3-TiO_2 Composites

SEM and EDX werre used to observe microstructure and elemental composition in the as-milled Al_2O_3-TiO_2 nanocomposites. The SEM and corresponding element analysis is shown in Figure 4.3. In this figure, the observation was focused on the shape and size of the Al_2O_3-TiO_2 nanocomposite particles. The microstructure consists of flake particles that can be described as spherical agglomerates. The milling process for 40 h had broken down the larger particles into smaller pieces due to high impact energy. The grey-coloured particles (X region) refer to rich-side Al_2O_3 and the white particles refer to rich-side TiO_2 (region Y) as confirmed by EDX analysis.



Element	Weight % of area X	Weight % of area Y
Al	54.57	29.84
Ti	6.14	29.83
O	39.29	40.33

Figure 4.3: SEM image and EDX analysis of as-milled Al_2O_3-TiO_2 nanocomposite powder

4.2 Epoxy-Al_2O_3-TiO_2 Nanocomposite Film

Epoxy-Al_2O_3-TiO_2 nanocomposite was produced to investigate the phase identification, functional group, and morphology of coating film. The films were characterized for X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) with EDX, and Fourier Transform Infrared Spectroscopy (FTIR).

4.2.1 Phase Identification

Figure 4.4 shows the XRD patterns of the epoxy-Al_2O_3-TiO_2 nanocomposite films. The presence of a broad peak around $2\theta = 20°$ (COD 9009671) in all nanocomposites could be attributed to the epoxy matrix or an amorphous phase formed during processing. The small peaks corresponding to Al_2O_3 (COD 9009671) and rutile TiO_2 (COD 5000223) phases indicates a well-dispersed arrangement of Al_2O_3 and TiO_2 particles within the epoxy matrix, with no significant reaction between them. The intensity of Al_2O_3 peaks around $2\theta = 40°$ increases proportionally with the rising Al_2O_3-TiO_2 concentration, in line with expectations due to the higher Al_2O_3 content. Similarly, the intensity of TiO_2 peaks around $2\theta = 25°$ and 35° shows an increase with increasing Al_2O_3-TiO_2 nanocomposite concentration. This suggests a potential difference in size or concentration between the TiO_2 and Al_2O_3 particles. No noticeable shift in peak positions across samples indicates that the lattice parameters of the Al_2O_3 and TiO_2 phases remain largely unaffected by their integration into the epoxy matrix.

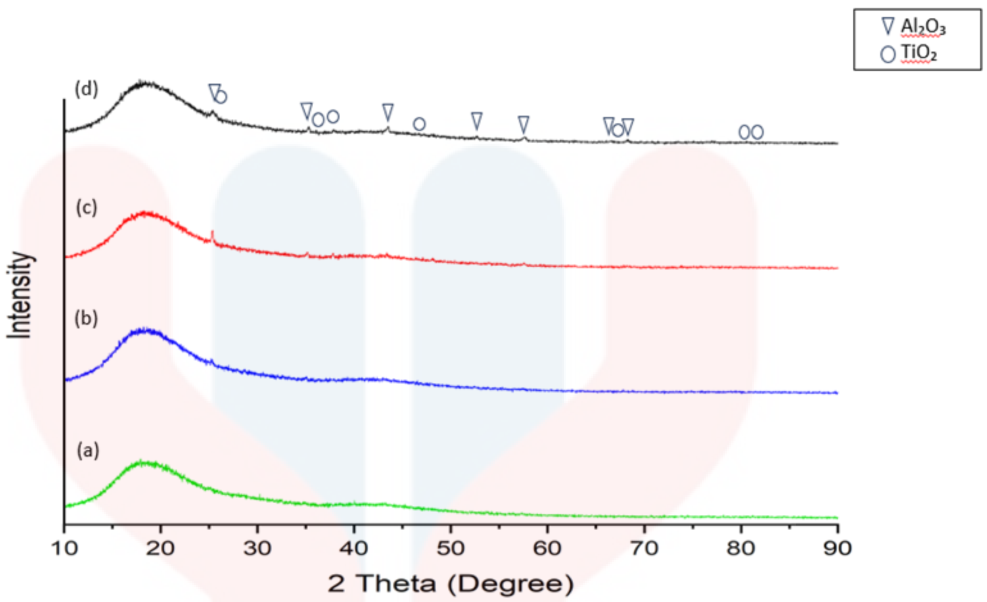


Figure 4.4: XRD patterns of epoxy-Thin Film Al_2O_3-TiO_2 nanocomposite film at different Al_2O_3-TiO_2 amount a) E-0.5 AT, b), E-0.7 AT, c) E-1 AT and d) E-2AT

4.2.2 Microstructure of Thin Film Al_2O_3-TiO_2 Nanocomposite

The used of SEM also confirmed that the morphology of thin film epoxy Al_2O_3-TiO_2 nanocomposite varies according to composition. Figure 4.5 shows the SEM images of epoxy-Al_2O_3-TiO_2 nanocomposite film. The film containing 0.5wt% Al_2O_3-TiO_2 exhibits a smooth surface texture compared to all compositions, suggesting minimal disruption to the epoxy matrix. This may offer lower initial reactivity but potentially reduce physical barriers to aggressive agents. The distribution Al_2O_3-TiO_2 particles in the film containing 0.7wt%, 1wt% and 2wt% Al_2O_3-TiO_2 are obviously observed with rougher surface texture. This surface roughness can provide good interfacial interaction with steel substrate by acting as a physical barrier, preventing the penetration of corrosive media into the steel substrate. The resulting surface roughness not only contributes to the debonding phenomenon but also provides an advantageous interfacial interaction with the steel substrate (Hao et al., 2019). Acting as a physical barrier, this roughness effectively prevents the penetration of corrosive media into the steel substrate.

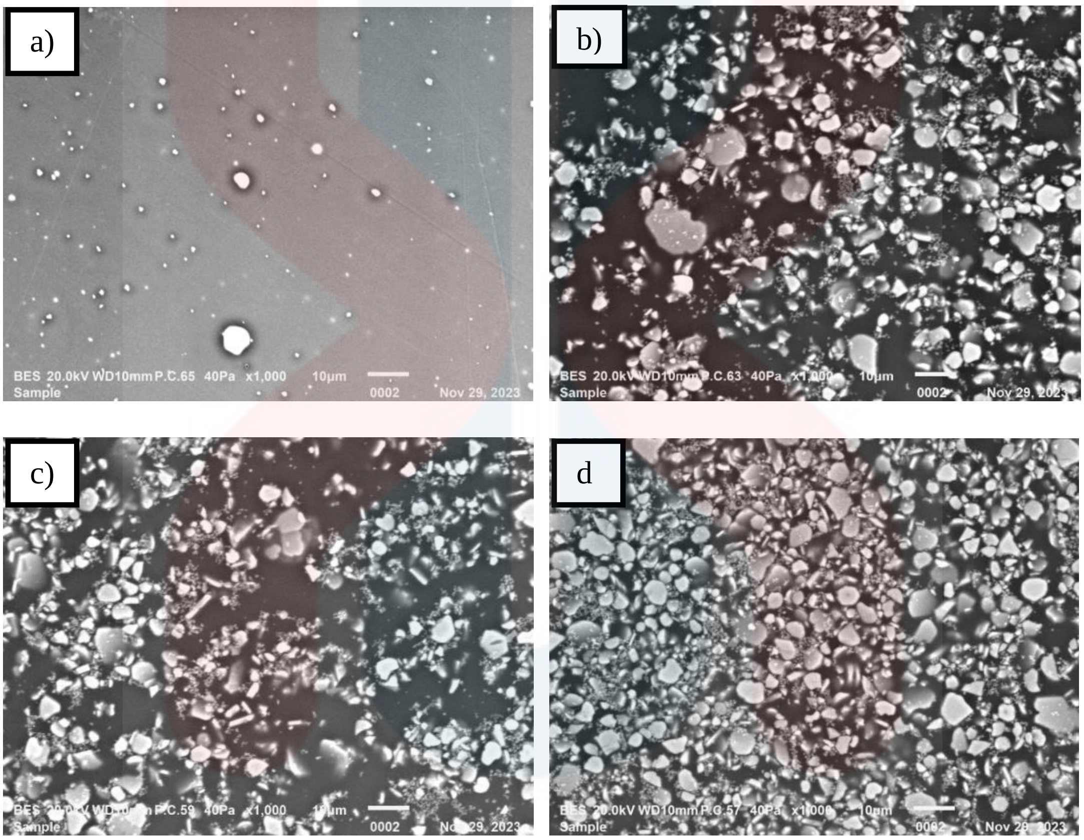
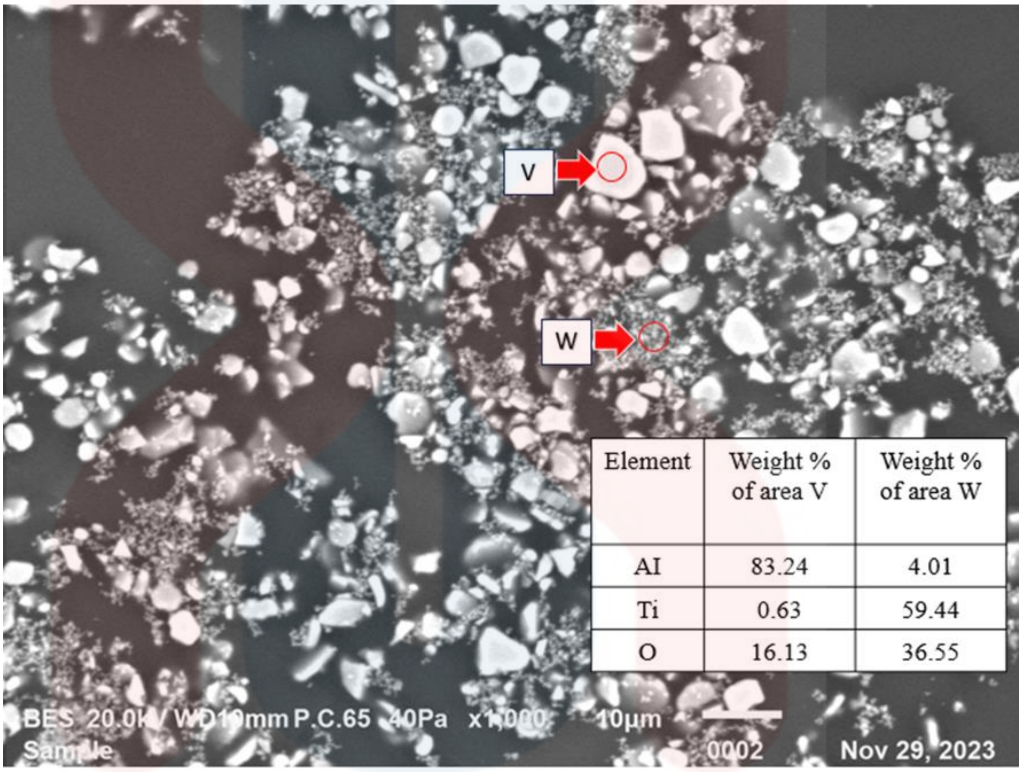


Figure 4.5: SEM images of epoxy-Al_2O_3-TiO_2 nanocomposite film at different composition of Al_2O_3-TiO_2 a) E-0.5 AT, b) E-0.7 AT, c) E-1 AT and E-2 AT under x1000 magnificent

The corresponding EDX analysis from SEM images in Figure 4.5 is shown in Figure 4.6. A well-dispersed distribution of rich Al_2O_3 (white) and rich TiO_2 (grey) particle within the epoxy matrix. This indicates effective mixing and incorporation of particles, maximizing their surface area contact with epoxy and potentially enhancing their synergistic effect on corrosion.



Element	Weight % of area V	Weight % of area W
Al	83.24	4.01
Ti	0.63	59.44
O	16.13	36.55

Figure 4.6: EDX analysis of epoxyAl_2O_3-TiO_2 nanocomposite film corresponding to Figure 4.5

4.2.3 Functional Group

Figure 4.7 shows the FTIR spectra of epoxy-Al_2O_3-TiO_2 nanocomposite film. Consistently, all the films exhibit similar peaks in their FTIR spectra. This uniformity arises from the samples sharing the same type, differing solely in composition. According to Khan (2023), the Fourier transform infrared spectra, curing agent accelerator and cured epoxy resin is coresponding to stretching vibration in the cured epoxy resin and the absence of corresponding absorption peaks. The summary of FTIR spectra in epoxy-Al_2O_3-TiO_2 nanocomposite film is shown in Table 4.2. The presence of hydroxyl groups is indicated by the O-H stretching vibrations at 3384 cm^{-1}. The broadening of these peaks also related to Al_2O_3-TiO_2 filler in the epoxy matrix. The Al_2O_3-TiO_2 has hydroxyl groups attached to its surface, causing the peak to broaden due to the moisture trap. The peak at 1507 and 1606 cm^{-1} is attributed to C-C and C=C, respectively due to stretching vibrations in aromatic rings of benzene rings. The $–CH_2$ stretching vibration modes of aromatic is assigned located at 2924 cm^{-1}. The peaks found at 1232, 1180, and 1033 cm^{-1} are identified to the stretching vibrations of C-O-C and C-O, respectively. According to the findings of Khan (2023) research, the FTIR spectrum reveals the presence of an absorption band like the one illustrated in Figure 4.7, depicting the FTIR spectra of the epoxy-Al_2O_3-TiO_2 nanocomposite film. These vibrations are linked to the ether linkage. The recorded peak at a wavenumber of 432.90 cm^{-1} provides evidence for the formation of Al-O bonds. This formation can be attributed to the reaction between hydroxyl groups present on the surface of Al_2O_3 particles and the epoxide rings of the resin (Ramírez-Herrera et al., 2021).

The film with a higher content of Al_2O_3-TiO_2 exhibits broader peaks, suggesting that the presence of these fillers has a significant impact on the chemical properties of the nanocomposite. According to Khudhair (2023), reinforcing with TiO2 and Al2O3 enhances both the bending strength and elasticity compared to pure epoxy. However, hybrid nanocomposites exhibit lower bending strength and elasticity compared to pure epoxy. The existence of dimers or high molecular weight species indicates the potential for unique chemical properties and a complex molecular structure.

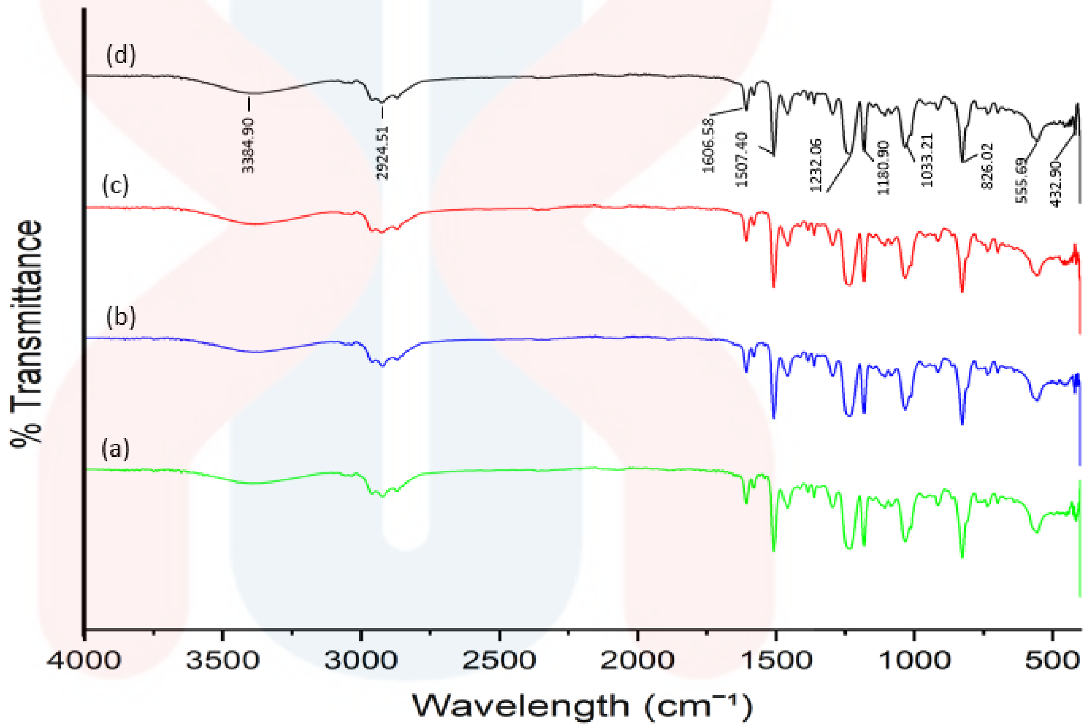


Figure 4.7: FTIR spectra of epoxy-Al_2O_3-TiO_2 nanocomposite film at different Al_2O_3-TiO_2 composition a) E-0.5 AT, b) E-0.7 AT, c) E-1 AT and d) E-2 AT.

Table 4.2: Summary of FTIR spectra in epoxy-Al_2O_3-TiO_2 nanocomposite film

Frequency (cm^{-1})	Assign Peak	Functional Group
3384.90	O-H stretch	Hydroxyl
2924.51	C-H stretch	Alkane
1606.58	C=C stretch	Aromatic
1507.40	C-C stretch	Aromatics
1232.06	C-O-C stretch	Aromatic
1180.90	C-O stretch	Aliphatic
1033.21	C-O-C stretch	Ethers
826.02	-CH deformation	Aromatic
555.69	C-H	Aromatic
432.90	Al-O	Al_2O_3

4.3 Epoxy Al_2O_3-TiO_2 Nanocomposite Coated on Welded Steel

The performance of steel coated epoxy-Al_2O_3-TiO_2 was performed for thickness, hardness, and immersion test. The characterization was carried out associated with the use of various compositions of Al_2O_3-TiO_2 in epoxy matrix.

4.3.1 Thickness

Figure 4.8 shows the average thickness of epoxy-Al_2O_3-TiO_2 nanocomposite coated on welded steel. The thickness of the coating film based on epoxy-Al_2O_3-TiO_2 was showed having less than 1.0 mm and non-uniform. The non-uniform thickness may impact the hardness and corrosion performance due to uneven steel surface and coated film.

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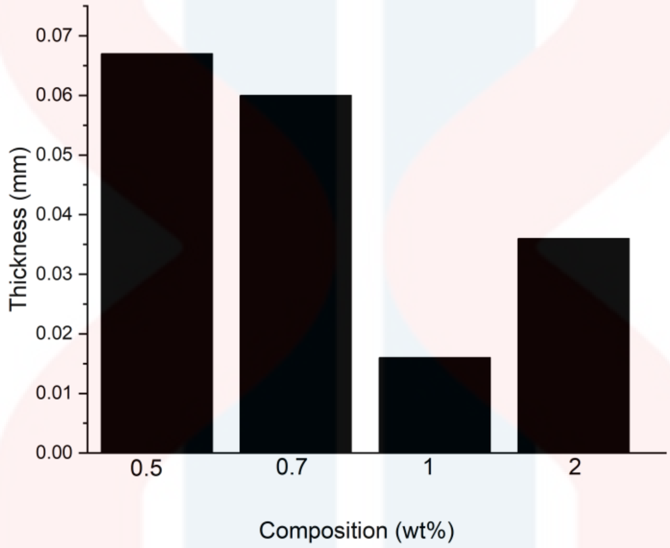


Figure 4.8: Thickness of coated welded steel with epoxy-Al_2O_3-TiO_2 at different Al_2O_3-TiO_2 concentrations

4.3.2 Hardness

Figure 4.9 shows the hardness of coated welded steel with epoxy-Al_2O_3-TiO_2 at different Al_2O_3-TiO_2 concentrations. The hardness was measured using Shore D durometer. It was observed that the coating with higher thickness (1wt% and 2wt% Al_2O_3-TiO_2) have a higher hardness than the coating with the lower thickness (0.5wt% and 0.7wt% Al_2O_3-TiO_2).

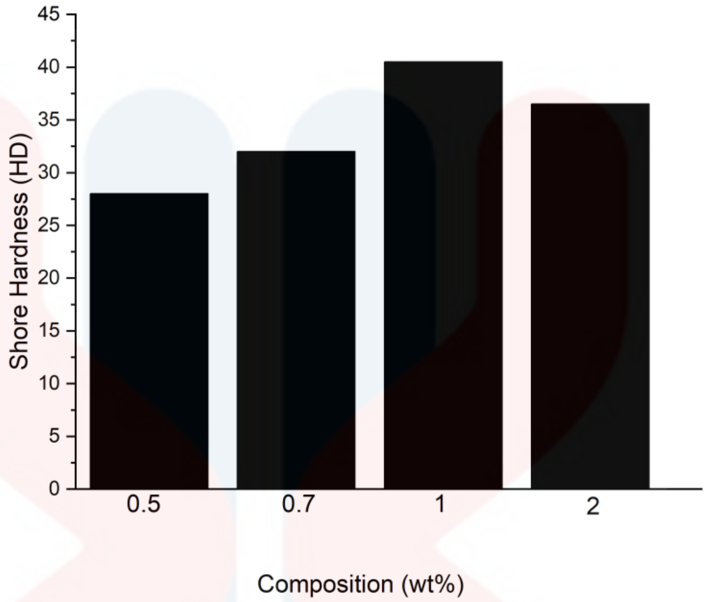


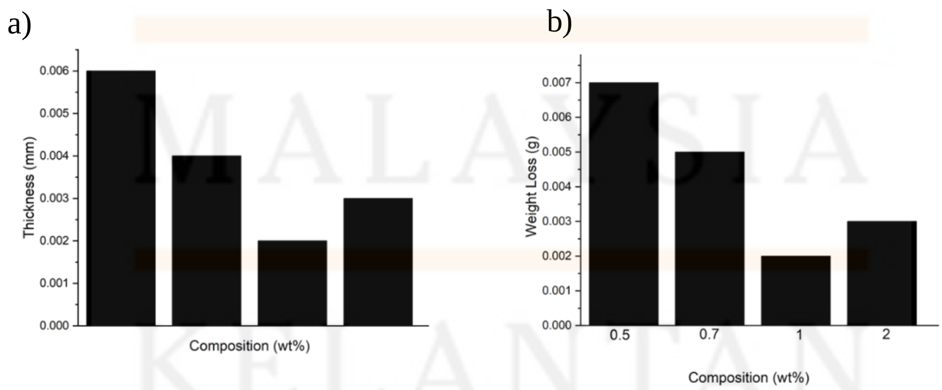
Figure 4.9: Hardness of coated welded steel with epoxy-Al_2O_3-TiO_2 at different Al_2O_3-TiO_2 concentrations

4.3.3 Immersion test

Figure 4.10 shows the weight loss of with epoxy-Al_2O_3-TiO_2 at different Al_2O_3-TiO_2 concentartions after 28 days of immersion. Over a 28-day period as the NaOH solution gradually degrades the epoxy-Al_2O_3-TiO_2 coating layer. The observed weight loss exhibits a consistent trend from day 5 to day 28, with the 0.5wt% showing the highest trend compared to the other coating. In contrast, the 1wt% sample displays the lowest degradation, remaining below the others on days 0, 5, 10, 16, 20, 24, and 28 days.

There are a few possible explanations for why the coating weight decreased after being soaked in the NaOH solution. The NaOH solution may have attacked the epoxy resin in the coating, causing it to degrade. The Al_2O_3-TiO_2 may also have reacted with the NaOH solution, leading to the formation of soluble products that washed away from the coating. According to Rehman (2020),discovered that as the concentration of NaOH increases, not only does the thickness of the coating on aluminum decrease, but also its roughness decreases. Additionally, the alkaline nature of the NaOH solution may have caused the steel substrate to corrode, which could have led to the coating detaching from the steel. It can be concluded that epoxy-Al_2O_3-TiO_2 nanocomposite coatings able protect welded steel from corrosion in 1 M NaOH solution. The extent of the decrease in thickness depends on the percentage of Al_2O_3-TiO_2 used in the coating.

The rate of weight loss is influenced by both the type and composition of the matrix. The concentration of Al_2O_3-TiO_2 particles in the epoxy coating plays a crucial role in determining its barrier properties and resistance to NaOH. Specific compositions with higher concentrations tend to result in a denser and more protective coating, potentially leading to reduced weight loss. The use of 1wt% Al_2O_3-TiO_2 in the epoxy coating is more effective than the other compositions. Additionally, the concentration of the NaOH solution also contributes to its corrosivity, with higher concentrations generally accelerating the degradation of the coating(Rehman et al., 2020).



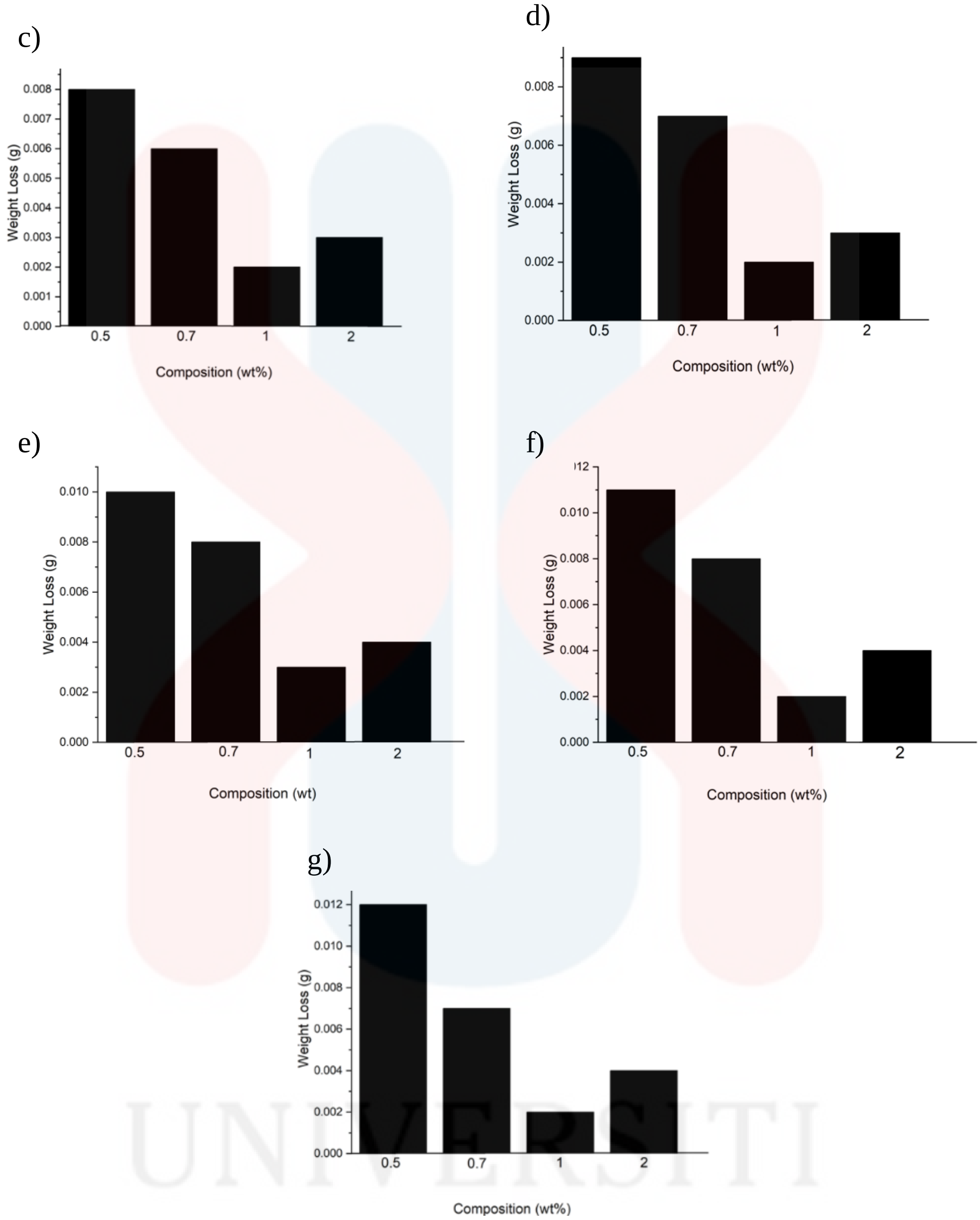


Figure 4.11: Weight loss of coated welded steel with epoxy-Al_2O_3-TiO_2 film at different Al_2O_3-TiO_2 concentrations after immersion in day a) 0, b)5, c) 10, d) 16, e) 20, f) 24, and g) 28

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This study has successfully synthesized Al_2O_3-TiO_2 epoxy with varying compositions (E-0.5 AT, E-0.7 AT, E-1 AT and E-2 AT). The prepared Al_2O_3-TiO_2 epoxy was applied as a coating on welded steel, subjected to immersion in a 1 molar NaOH solution. The research has yielded several noteworthy conclusions:

1. Despite a reduction in thickness, the nanocomposite epoxy coatings exhibit commendable efficacy in safeguarding welded steel from corrosion, even in the challenging environment of a 1 M NaOH solution.

2. Further investigations have highlighted that specific compositions within the Al_2O_3-TiO_2 range contribute to enhanced barrier properties, imparting greater resistance against NaOH-induced degradation. Notably, the utilization of 1% Al_2O_3-TiO_2 in the epoxy coating emerges as particularly effective in minimizing weight loss.

In summary, this research underscores the versatility and protective capabilities of Al_2O_3-TiO_2 epoxy coatings, demonstrating their potential for corrosion inhibition and structural resilience, especially when tailored with specific compositions.

5.2 Recommendation

Based on the findings of this study, several recommendations are proposed to enhance the quality and comprehensiveness of future research efforts. Firstly, extend the duration of corrosion tests to capture a more comprehensive understanding of the long-term effects. Prolonged exposure periods can bring out subtle changes in the samples, providing a clearer depiction of the corrosion behavior over time.

Secondly, ensure a thorough and uniform mixing of the Al_2O_3-TiO_2 nanocomposite with epoxy. An even distribution is crucial to avoid inconsistencies in the SEM images, as poorly blended mixtures may result in sections of the thin film lacking Al_2O_3-TiO_2 particles. This precaution is particularly relevant since SEM imaging often focuses on specific areas of the sample.

Lastly, Integrate mechanical testing methodologies such as Rockwell hardness, Vickers hardness, or flexural strength into the research protocol. These tests can provide valuable insights into the strength and hardness characteristics of the epoxy Al_2O_3-TiO_2 nanocomposite samples, allowing for a more comprehensive assessment of their mechanical properties across different compositions.

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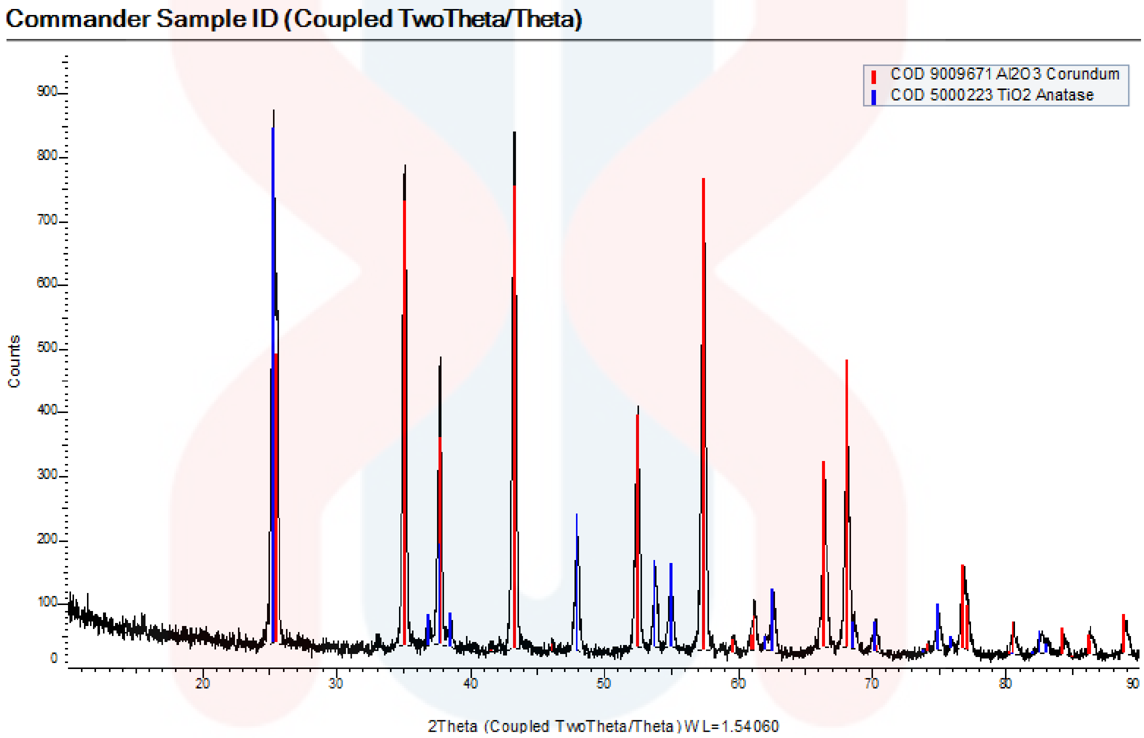
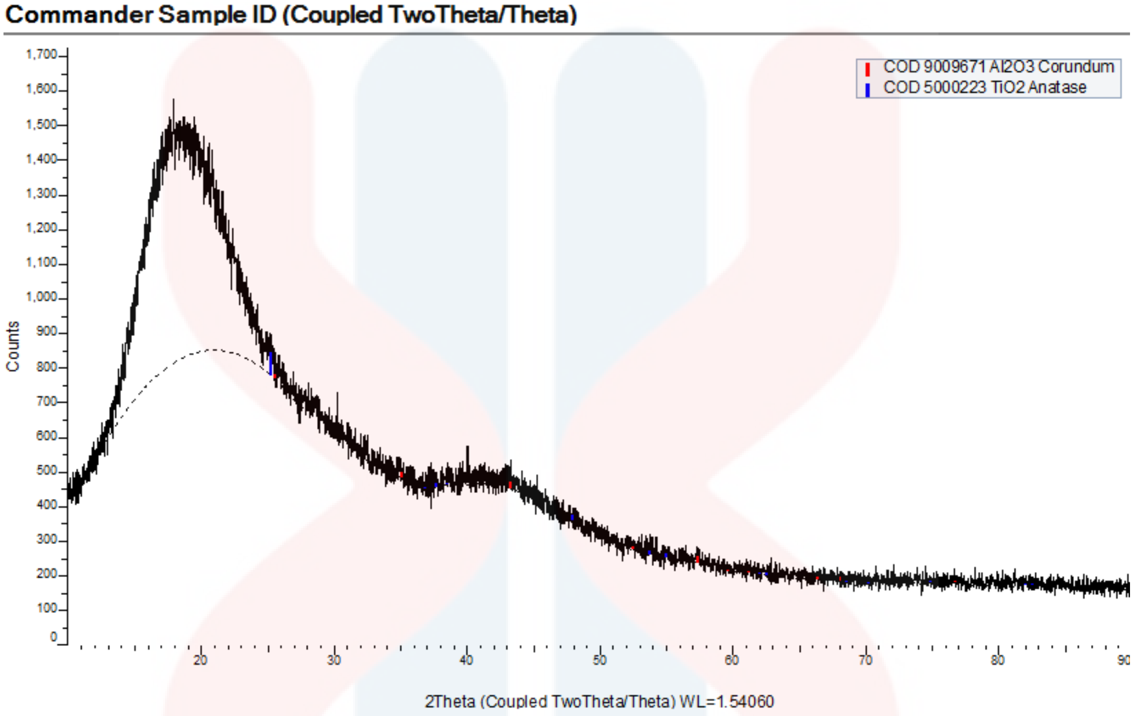
APPENDIX A

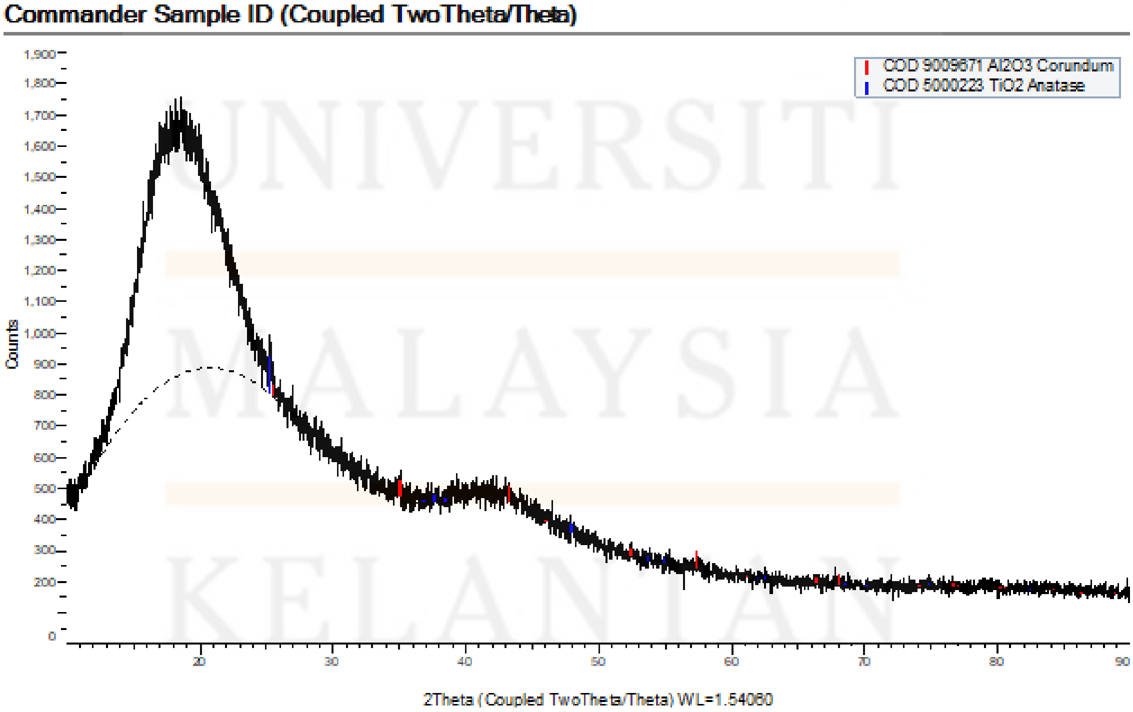
A.1 XRD analysis of 40 h milled Al_2O_3-TiO_2 nanocomposite powder



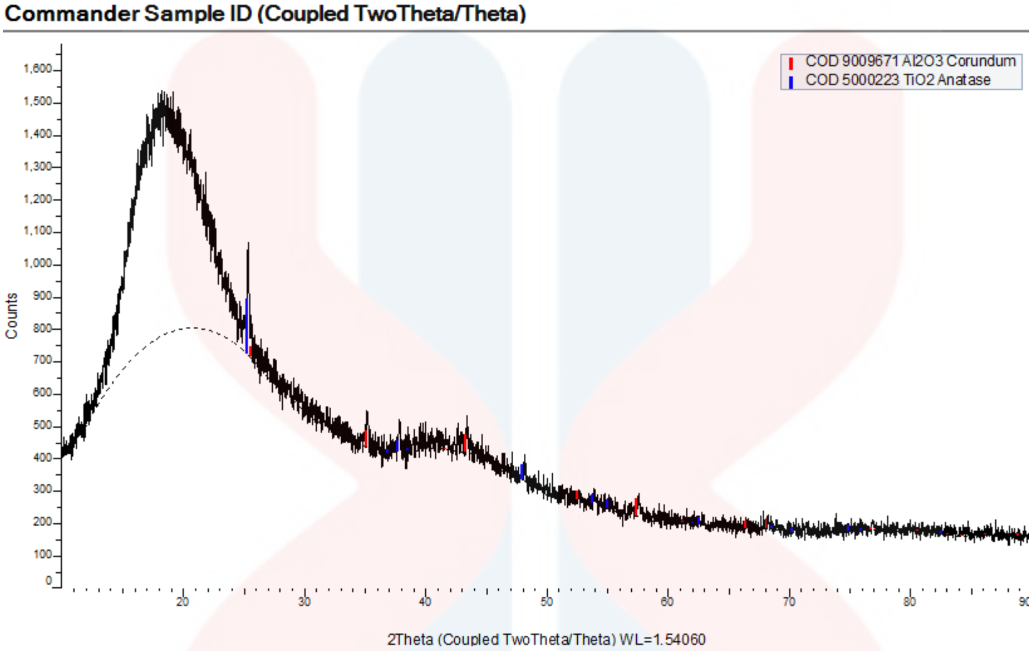
A.2 XRD analysis of (0.5) thin film epoxy Al_2O_3-TiO_2 nanocomposite



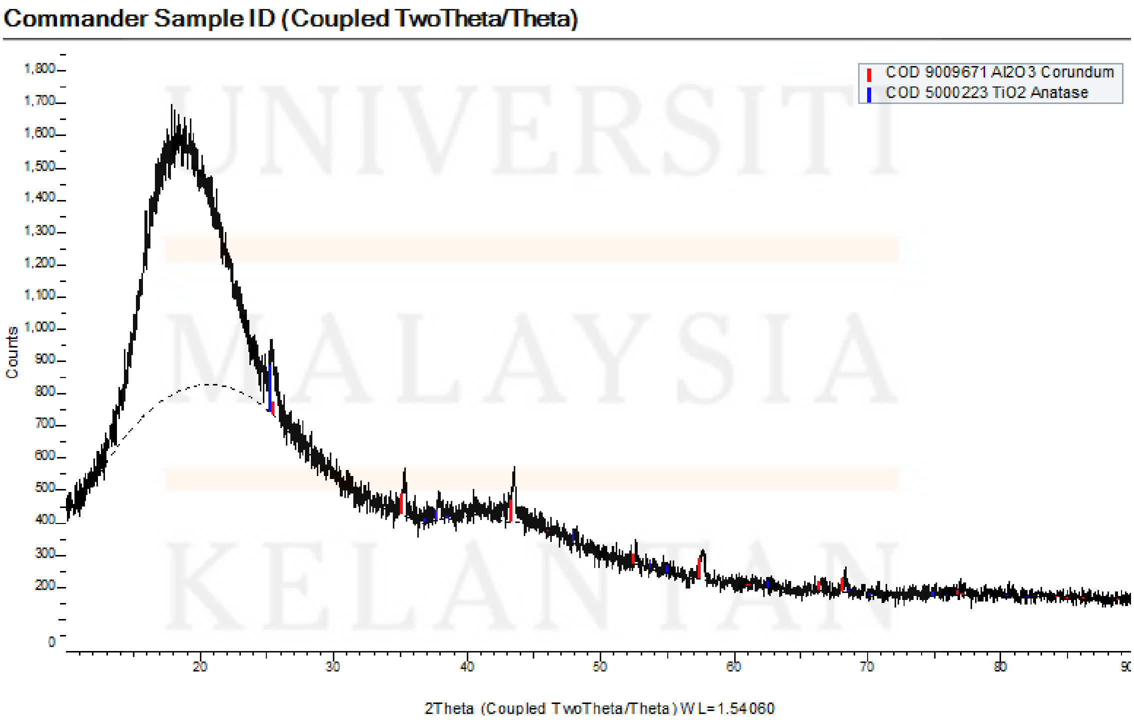
A.3 XRD analysis of (0.7) thin film epoxy Al_2O_3-TiO_2 nanocomposite



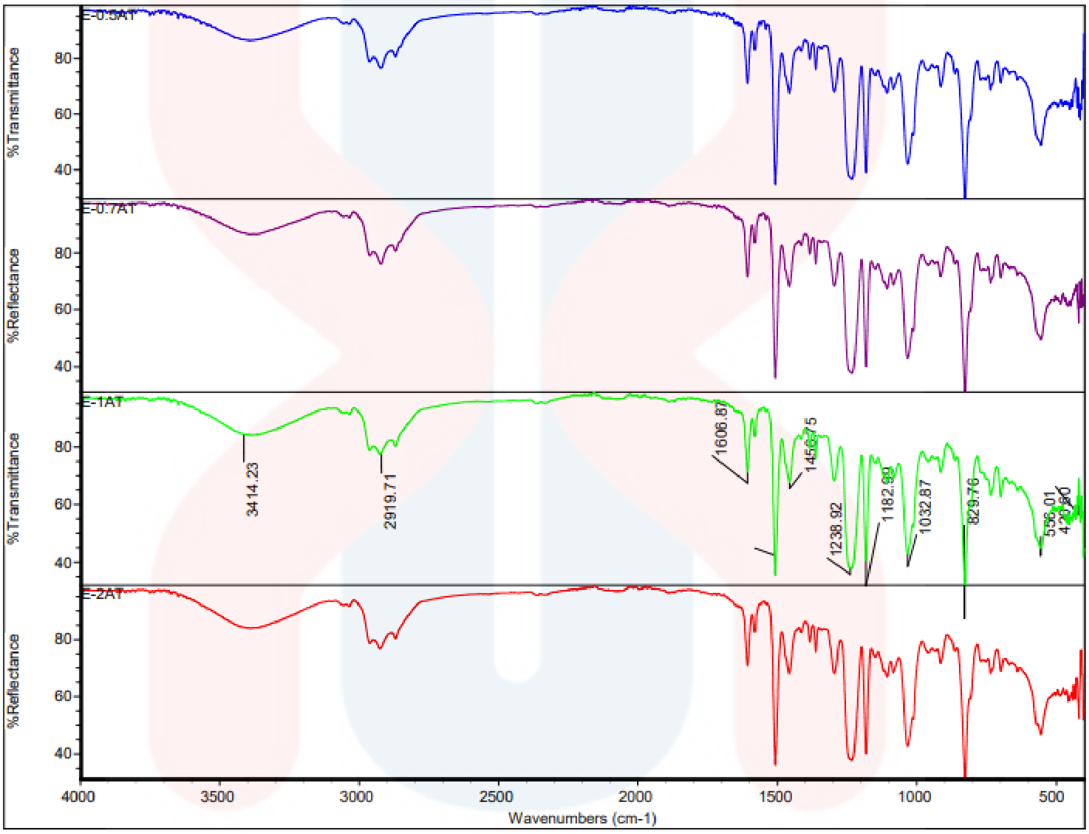
A.4 XRD analysis of (1) thin film epoxy Al_2O_3-TiO_2 nanocomposite



A.4 XRD analysis of (2) thin film epoxy Al_2O_3-TiO_2 nanocomposite



A.4 FTIR analysis of (0.5,0.7,1 and 2) thin film epoxy Al_2O_3-TiO_2 nanocomposite



APPENDIX B

B.A Hardness, thickness, and mass data throughout the corrosion test

SAMPLE	TEST	DATE						
		22.10.2023	26.10.2023	30.10.2023	5.11.2023	9.11.2023	13.11.2023	16.11.2023
0.50%	Shor D (HD)	30.5	30	30	29.5	29	28.5	28
	Thickness (mm)	0.562	0.537	0.528	0.512	0.51	0.5	0.495
	Mass (g)	22.419	22.412	22.404	22.395	22.385	22.374	22.362
0.70%	Shor D (HD)	33	33	33	32.5	32.5	32.5	32
	Thickness(mm)	0.546	0.532	0.512	0.504	0.5	0.493	0.486
	Mass (g)	17.479	17.474	17.468	17.461	17.453	17.445	17.438
1%	Shor D (HD)	40	40	40	40	40	40.5	40.5
	Thickness (mm)	0.596	0.594	0.592	0.59	0.587	0.583	0.58
	Mass(g)	18.238	18.236	18.234	18.232	18.229	18.227	18.225
2%	Shor D (HD)	37	37	37	37	36.5	36.5	36.5
	Thickness (mm)	0.582	0.575	0.543	0.543	0.544	0.55	0.546
	Mass(g)	20.229	20.226	20.223	20.22	20.216	20.212	20.208

B.B Weight change for each sample over 28 days.

Sample/ Date	0 Days	5 Days	10 Days	16 Days	20 Days	24 Days	28 Days
0.50%	0.006	0.007	0.008	0.009	0.01	0.011	0.012
0.70%	0.004	0.005	0.006	0.007	0.008	0.008	0.007
1%	0.002	0.002	0.002	0.002	0.003	0.002	0.002
2%	0.003	0.003	0.003	0.003	0.004	0.004	0.004