



INCREASING THE CORROSION RESISTANCE OF LOW-CARBON STEEL USING COATINGS

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STEEL USING COATINGS**

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I certify that in my opinion the thesis submitted by Maha Yousif MOHSIN titled “INCREASING THE CORROSION RESISTANCE OF LOW CARBON STEEL USING COATINGS” is fully adequate in scope and in quality as a thesis for the degree of Master of Science

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“I declare that all the information within this thesis has been gathered and presented in accordance with academic regulations and ethical principles and I have according to the requirements of these regulations and principles cited all those which do not originate in this work as well.”

Maha Yousif MOHSIN

ABSTRACT

Master Thesis

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The corrosion challenge in industrial equipment and metal structures is addressed by employing protective coatings, which act as a barrier between the metal surface and its surroundings.

Coatings are crucial for safeguarding irons and steel from severe weather conditions. Epoxy coating has been employed in this study due to its exceptional characteristics in safeguarding steel against corrosion. To improve the tribological characteristics of the pristine epoxy matrix, a variety of fillers are used to create composite coatings.

To begin, samples of low carbon steel 1008 underwent a shot peening process after heat treatment to prepare the surface by giving some roughness. Subsequently, these samples underwent cleaning and were coated with epoxy using the dip-coating method. The coating procedure included applying epoxy alone, epoxy blended with

1%wt of alumina powder, epoxy blended with 1% wt of silicon carbide powder, and finally, steel coated with epoxy mixed with both 0.5% wt of alumina and 0.5% wt of silicon carbide powders. Several analytical techniques such as Fourier Transform Infrared spectroscopy (FT-IR), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM), in addition to Energy Dispersive X-ray Spectroscopy (EDS), were employed to characterize the structure, composition, and morphology of the coatings. The performance of the coatings was evaluated using Tafel polarization curves the findings revealed that all additives contributed to prolonging the lifespan of the steel samples. However, the best results were observed in the sample coated with epoxy reinforced with alumina, showcasing superior corrosion resistance compared to other samples. Additionally, the sample coated with epoxy reinforced with silicon carbide exhibited increased hardness, by studying the mechanical characteristics for coatings using Vickers hardness and scratch tests, which indicated enhanced mechanical properties.

Keywords : Carbon Steel, Corrosion Resistance, Coating.

Science Code : 91510

ÖZET

Yüksek Lisans Tezi

KAPLAMALAR KULLANARAK DÜŞÜK KARBONLU ÇELİĞİN KOROZYON DİRENCİNİN ARTIRILMASI

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Endüstriyel ekipman ve metal yapılardaki korozyon sorunu, metal yüzey ile çevresi arasında bariyer görevi gören koruyucu kaplamalar kullanılarak giderilir.

Kaplamalar, demir ve çeliği zorlu hava koşullarından korumak için çok önemlidir. Çeliği korozyona karşı korumadaki olağanüstü özelliklerinden dolayı bu çalışmada epoksi kaplama kullanılmıştır. Bozulmamış epoksi matrisinin tribolojik özelliklerini geliştirmek amacıyla kompozit kaplamalar oluşturmak için çeşitli dolgu maddeleri kullanılır.

Öncelikle düşük karbonlu çelik 1008 numunelerine ısıtma işlemi sonrası bilyeli dövme işlemi uygulanarak yüzey pürüzlülük verilerek hazır hale getirildi. Daha sonra bu numuneler temizlendi ve daldırma kaplama yöntemiyle epoksi ile kaplandı. Kaplama prosedürü, tek başına epoksi, ağırlıkça %1 alümina tozuyla harmanlanmış epoksi, ağırlıkça %1 silisyum karbür tozuyla harmanlanmış epoksi ve son olarak hem ağırlıkça %0,5 alümina hem de ağırlıkça %0,5 silikonla karıştırılmış epoksi ile kaplanmış çeliğin uygulanmasını içeriyordu. karbür tozları. Yapıyı karakterize etmek için Enerji Dağıtıcı X-ışını Spektroskopisinin (EDS) yanı sıra Fourier Dönüşümü Kızılötesi spektroskopisi (FT-IR), X-ışını Kırınımı (XRD) ve Taramalı Elektron Mikroskobu (SEM) gibi çeşitli analitik teknikler kullanıldı. kaplamaların bileşimi ve morfolojisi. Kaplamaların performansı Tafel polarizasyon eğrileri kullanılarak değerlendirildi ve bulgular, tüm katkı maddelerinin çelik numunelerin ömrünü uzatmaya katkıda bulunduğunu ortaya çıkardı. Ancak en iyi sonuçlar, diğer numunelere göre üstün korozyon direnci sergileyen, alümina takviyeli epoksi ile kaplanmış numunede gözlemlendi. Ek olarak, silikon karbürle güçlendirilmiş epoksi ile kaplanmış numune, gelişmiş mekanik özellikleri gösteren Vickers sertlik ve çizilme testleri kullanılarak kaplamaların mekanik özellikleri incelenerek artan sertlik sergilemiştir.

Anahtar Kelimeler : Karbon Çeliği, Korozyon Direnci, Kaplama.

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PART 1

INTRODUCTION

1.1. INTRODUCTION

Metals' surfaces can change as a result of environmental factors interacting with them, endangering their structural integrity and functional qualities. This deterioration, sometimes referred to as corrosion, is the process by which metals or their alloys are consistently degraded by their surroundings—which may include exposure to components like oxygen, humidity, or different soil conditions—via chemical or electrochemical methods. More chemically stable oxides or other compounds are frequently produced as a result of this [1].

The most common steel used in engineering is carbon steel, which makes up around 85% of all steel produced worldwide. Despite its vulnerability to corrosion, it finds widespread application in everything from nuclear power plants to sea-based structures, and chemical processing facilities to the extraction and refinement of petroleum. It also has a big impact on infrastructure, including building, mining, pipelines, and machinery used in metal processing. Every year, the financial costs associated with metal corrosion exceed millions of dollars. Because carbon steels are used so widely, corrosion is a significant and expensive problem given their volume and related expenses. In an effort to either separate these metals from corrosive environments or modify the environments to prevent corrosion, this has led to the establishment of entire industries devoted to the provision of protective solutions for iron and steel products [2].

Many techniques can be used to prevent corrosion, one of the most common being coating technology. A good option to counteract corrosion is coatings. They serve as a defense against corrosive substances and can be strengthened by adding

compounds such as fillers and inhibitors. Epoxy coatings are especially prevalent for safeguarding steel, given their reliability and widespread availability. These coatings are employed in a host of industries, from marine to energy production, and from industrial plants to infrastructure like bridges and maritime vessels. Their protective function is primarily to impede the penetration of water and oxygen to the steel's surface. The properties of epoxy resins, such as high resistance to chemicals and corrosion, excellent thermal and mechanical attributes, strong adhesion to many materials, low curing shrinkage, superior electrical insulation, and the capacity for processing in different of situations, make them particularly advantageous. The selection of specific epoxy compositions is typically driven by the targeted physical and mechanical performance requirements.

As a result, the industrial application of epoxy coatings as a finish has become quite prevalent. Although the initial investment is potentially higher compared to other polymers, the enduring protection against corrosion, strong adhesive properties, and adaptability to a range of surfaces render epoxy coatings economically viable over time [2].

Market indicators suggest a growing trend for epoxy coatings within the paint and coating sector, propelled by their enhanced longevity and robustness. Data Bridge Market Research estimates the epoxy coating market's value at USD 36.32 billion as of 2021, anticipating an expansion to USD 54.15 billion by 2029. This projection is based on a forecasted compound annual growth rate (CAGR) of 5.12% from 2022 to 2029 [3] (refer to Figure 1.1).



Figure 1 1. Global epoxy coating market up to the year 2029

Figure (1.1) illustrates the Global Epoxy Coating Market, categorized by Technology (Waterborne, solvent-borne, Powder-Based) and Application (Construction, Adhesives, Transportation, Paints and Coatings, General Industrial, Electrical and Electronics, Others), with Industry Trends and forecasts up to the year 2029 [3].

1.2. AIM of THIS PROJECT

Low-carbon steel is a staple in a range of industrial applications due to its cost-effectiveness and reliable physical and mechanical characteristics. Industries such as automotive, construction, oil and gas, maritime transport, sea bridge construction, and chemical processing heavily rely on this material. The primary mode of degradation and failure in low-carbon steel is through corrosion, which leads to considerable economic costs and material wastage. Moreover, the ramifications of corrosion touch upon the safety of operations and the dependability of products, raising the risk of valuable asset loss, catastrophic events, and cessation of operations. As a result, corrosion has long-term effects on metal structures.

This research aims to improve the usefulness and endurance of metals while decreasing the problems brought on by corrosion, with a particular emphasis on low-carbon steel. This research suggests that epoxy coatings be used to protect surfaces.

These coatings are selected due to their great resistance to corrosive substances and strong connection with metal surfaces. The epoxy resin forms a protective coating and source of corrosion resistance, preventing corrosive ions from damaging the low-carbon steel.

Using protective coatings to stop corrosion has a lot of advantages. It improves the performance and longevity of metal components and helps in the creation of materials with improved functional properties. Industrial processes consequently become more efficient, and maintenance and replacement costs both fall less frequently. These coatings are also essential for reducing pollution in the environment and safeguarding limited natural resources.

PART 2

LITERATURE REVIEW

The first documented commercial bisphenol epoxy resins were created by Pierre Castan in the late 1930s while working for Ciba-Geigy, the company that eventually became Ciba. He was attempting to find a product that could be utilized for dentures, but this was not commercially successful. Around the same period, Sylvan Greelee, employed by the Devoe-Raynolds in the U.S., was reacting bisphenol-A with fatty acids to produce an air-drying coating. Several chemical companies dedicated their original research to this endeavor after the Second World War and released their first commercial-quality epoxies in the late of 1940s. Protectionally coated resins were among the first and still constitute the majority of the epoxies produced, they account for around 50% of all of the produced epoxies, however, other uses such as adhesive, casting, and flooring.

The application of coatings to metals has been researched in depth with regard to their corrosion protection and effectiveness for different structures. However, despite the significant advancements in coating technologies, issues still have to be addressed in order to preserve the long-term health of metals in adverse environments. One of the primary reasons for the limited number of high-performance coating systems is the complexity of the substrate system and the number of factors that affect the performance and lifespan of these protective coatings. Other factors, such as the composition of the coating, the solvents, fillers, and additives in the coating, as well as the type of substrate, the pretreatment of the substrate, the thickness of the coating, and the bond between the substrate and the coating, all contribute to the performance and durability of the coatings. For instance, a high-performance protective and long-lasting coating must have sufficient flexibility and toughness while resisting impact and cracking as well as

maintaining its properties when subjected to stress, physical damage, and weathering [4.5].

Polymer coatings are typically augmented with fillers that are either micro-sized or nano-sized, in order to enhance their tribological and mechanical properties.

Xianming Shi et al. 2009 reported that the anti-corrosive effects of the epoxy coating containing nanoparticles of SiO₂, Zn, Fe, O, and halloysite clay on steel in both 0.3 percent and 3.5 percent NaCl solutions over a period of 28 days. Potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) were employed to assess the effect of combining different nanoparticles with the corrosion resistance of epoxy-covered steel. The outcome demonstrated that the utilization of nanoparticles enhanced the corrosion resistance of coated steel, the greatest increase in corrosion resistance was achieved with Fe O, and halloysite clay. The SiO₂ nanoparticles enhanced the anti-corrosive capabilities and microstructure of the coating matrix [6].

Nikje et al.2012 created SiO₂-reinforced epoxy composite coatings using in situ polymerization, the silica particles were first treated before being incorporated into the resin matrix. Their findings demonstrated that relative to unaltered coatings, altered coatings had a higher thermal and mechanical properties. [7].

Islam et. al.2013 documented the effect of 5% silica nanoparticles of 20nm being added to the epoxy resin and observed an increase in the tensile strength, yield strength, and Young's modulus of the composites. A 20% increase in the ultimate stress and a 50% increase in the yield stress were observed for this 5 % --sized nano-silica added to a matrix of epoxy [8].

Conradi et al.2015 employed a modified SNP coating on a DSS 2205 dual-stainless steel type. He recorded a significant increase in the hardness of 30% and a 4% increase in the toughness of fractures in comparison to the pure epoxy sample [9].

Abdelkarim et al. measured the hardness of composites made of epoxy that contained micro-sized (<0.5 μ m) and/or nano-sized (<0.1 μ m) TiO₂ particles according to the

ASTMD 2240 standards using a digital hardness tester. Epoxy-based micro composites and nanocomposites with a weight of 0.1, 0.25, 0.5, 0.75, 1 and 3.5 were tested. % TiO₂ were made separately, while the composites that contain both micro and nano-sized TiO₂ were created at 0.1 and 0.75 percentages of loading. The toughness test was conducted on four different samples for each composition in order to ensure the results were as close as possible by minimizing the standard deviation. The results demonstrated that the initial hardness of the unfilled epoxy, which is 73.5, was increased by the addition of TiO₂ particles, while micro-composites had a higher hardness than nanocomposites. Among the entire micro composites, the greatest hardness was achieved at 0.5 weight percent. % loading whereas the nanocomposite with 3 wt. The highest hardness value of % TiO₂ was 84, followed by % PLA and % SiO₂ with a hardness of 78. This situation demonstrated that the incorporation of low concentrations of micro-sized TiO₂ particles into the epoxy resin increased the hardness of the material [10].

Bedaiwi's team 2017 and Wenhue et al.2018 stated that the altered coating of nano-ZrO, incorporated into a phenolic-epoxy resin, shields steel components from the acidic environment of an industry using electrochemical methods. The results showed that adding 1 wt% and 3 wt% of nano-ZrO to the coatings increased their corrosion resistance, however, adding 5 wt% of the same substance decreased the effectiveness of the coating [11,12].

In a study by Yongxing Zhang et al.2018, the corrosion resistance of composite coatings with silicon nitride was evaluated by electrochemical impedance spectroscopy (EIS), and the results showed that the modified coating had a good anti-corrosive performance [13].

Hongli Cheng et al. 2018 studied the synthesis and characterization of poly (O-Ethoxyaniline)/ silica (POEA/SiO₂) composite material by in situ polymerization that was prepared on the surface of the treated carbon steel. Additionally, it studied the anti-corrosive properties of the material in 3.5 percent NaCl solution via polarization curve and EIS. The results showed that coatings that contain POEA/SiO₂ have a decreased rate of corrosion of 0.02 mm/year and a protection efficacy of 98 %

against corrosion. The inclusion of POEA/SiO enhances the anti-corrosive properties of the epoxy coating [14].

Zhang et al. 2018 exhibited a decrease of 80% and 76% in the coefficient of friction and wear rate, respectively, for the coatings at room temperature when 4.0 weight percent graphene was incorporated as compared to the neat epoxy coatings [15].

Luo et al. 2019 described the successful incorporation of basalt flakes into an epoxy coating with 3% nanosilica. This coating exhibited a superior chemical and mechanical performance [16].

It's commonly understood that the addition of particles that enhance the neat epoxy's strength can increase its resistance to sliding wear. For example, Oliveira et al. (2019) investigated resins that were augmented with micro Al₂O₃ particles and demonstrated that the addition of these particles enhanced the bond strength, wear, and corrosion resistance of the resin. Al₂O₃ particles were between 5 and 15 µm in size, and 26% of the volume was made up of these particles. The amount of wear was determined with a Calowear abrasion tester, the degree to which the substrate was connected to it was measured with pull-off tests. The results showed that the wear resistance of ceramic-loaded epoxy increased by 30% (which is equivalent to the decrease in wear rate from 4.2 to 2.9 x 10.1 N), and the bond to the substrate increased by 50% (which is equivalent to the increase in adhesion strength from 20.4 to 30.2 N). Additionally, the effect of coating thickness on wear resistance was examined for thicknesses of 120 µm, 240 µm and 360 µm; it was observed that increasing the thickness increased the resistance.

U. E. Ozcan et al. (2019) incorporated different concentrations of graphene nanoplatelets (GNPs) and silica NPs (SiO₂) into the matrix of epoxy separately or together as supplements. The nanofillers had a composition of SiO₂ (1 wt%), GNPs (0.1 wt %), and SiO₂-GNPs (1 and 0.1 wt%, respectively) hybrid. It documented that the mechanical properties of the (polymer / graphene and silica NPs) were enhanced. The resistance of impact and flexibility of the coatings made of epoxy-GNPs increased by 157.1 and 8.3% respectively, while the resistance of impact and

flexibility of the neat epoxy coating decreased by 8.3%. With the addition of SiO₂-GNPs, the microhardness increased by 53.8% while the depth of scratching decreased by 29.7% as a result of their combined effect [17].

Yuan et al. 2020 documented that the addition of titanium nanoparticle-modified graphene oxide to the coating increased the corrosion and wear resistance of the epoxy [18].

M. Zhang et al. (2020) documented a simple method of producing graphene oxide flakes that were coated with (SiO₂). By utilizing mechanical agitation, a variety of GO/Zn-EP and SiO₂-GO/Zn-EP coatings with different concentrations (0.1, 0.5, 1, and 2 percent) were created and applied to Q235 steel panels. The results showed that all coatings with GO additions and SiO₂-GO were significantly enhanced by these additions. The maximum force used to adhere to the substrate (2.11 MPa) and the greatest increase in the water's contact angle from 56.8 to 85.1° was attained when the GO or SiO₂-GO content was 0.5 percent. According to the Tafel polarization law and EIS, the addition of 0.5 percent SiO₂-GO to the epoxy coating that was rich in zinc increased the anti-corrosion efficacy. The i_{corr} decreased from 16.750 to 0.561 $\mu A/cm$ for the GO/Zn-EP coating and had the greatest R_p value (78.031 $M\Omega cm^2$), with an increase in the corrosion protection effectiveness from 67.01 to 99.58%. [19].

B. D. Abbass et al. (2021), a blended resin composed of tungsten trioxide (WO₃) NPs, these were then used to protect brass via a dip coating method and the effectiveness of this technique was evaluated in the context of corrosion resistance under adverse conditions. The ceramic NPs on the metal's surface, based on the electrochemical polarization test results, demonstrated that the coated specimens had a high degree of protection against corrosion; this was attributed to the presence of epoxy/ WO₃. The coated samples exhibited a respectable amount of protection against corrosion. The corrosion resistance increased as the weight percentage of WO₃ NPs increased from 1-3%, the lowest corrosion rate was 4.92×10^{-4} mm/ year at 3% addition. [20].

F. Davoodi et al. (2022), the modified epoxy coating's capacity to withstand corrosion when applied to St37 steel using polysulfide and CeO, as well as NPs. The CeO₂ NPs addition of (0.5-6) percent into the epoxy that had 10 percent polysulfide as its sole component. The EIS and cupping test results showed that a 10 weight percentage of the total was added. The percentage of polysulfide to epoxy increased its corrosion resistance and toughness. After being submerged in the 3.5% NaCl solution for 180 days, its decrease in impedance was minimal by only 22.2% (from 10.58 to 8.23 cm²). The results showed that the addition of 1 percent CeO₂ NPs increased the strength of adhesion (approximately 11 MPa) and the resistance of the coating to corrosion (approximately 17 MPa). After being submerged for 180 days in the harsh medium, the polarization resistance of the material without and with CeO₂ NPs was 173.38 and 2,511.88 MN.cm², respectively [21].

The studies mentioned above indicate that Epoxy coating has become the most popular method of protecting metal. The fillers are employed as a supplement to enhance the mechanical and tribological properties of epoxies.

PART 3

THEORETICAL PART

3.1. CORROSION

The definition of corrosion is the degradation of materials due to reactive chemical reactions with external variables and it is a pervasive and impulsive phenomenon. When a metal surface interacts with an electrolyte, electrochemical reactions result in anodic and cathodic processes that happen simultaneously and at similar speeds [1].

Industrial equipment may prematurely deteriorate and fail as a result of corrosion, among other issues. Corrosion can have different chances, outcomes, and manageability depending on how it occurs and the characteristics of the surface that is impacted. Various forms of corrosion can be observed, such as uniform surface wear, localized pitting, crevice formation, galvanic corrosion, combined erosion-corrosion, grain boundary corrosion, and cracking induced by stress. For example, stress corrosion cracking appears under the combined effects of tensile stress and corrosive environments, resulting in fine fractures on the material's surface [22]. In contrast, erosion-corrosion tends to accelerate when corrosive fluids move against a metal surface. Crevice corrosion is a localized form that occurs in confined spaces on metal surfaces. Uniform corrosion, as illustrated by the rust depicted in Figure 3.1 [23] is identified by a consistent loss of metal mass.



Figure 3 1. Rust the most familiar example of corrosion

Here, the iron component acts as the anode and is oxidized, while oxygen reduction occurs at the cathode, forming a water figure3.2 [24]. The reactions can be summarized by the following chemical equation:

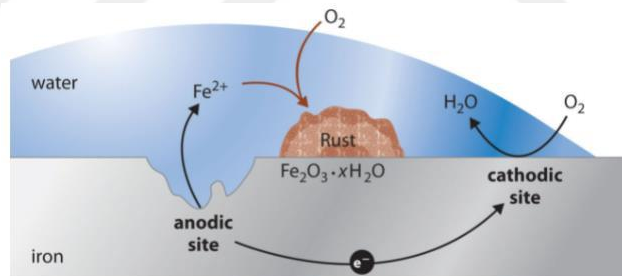
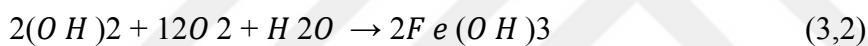


Figure 3 2. The result of corrosion of metallic iron.

Despite widespread awareness of corrosion and its various manifestations, the broader societal and economic repercussions of this naturally occurring degradation process are substantial and often underappreciated. These repercussions fuel the motivation behind ongoing research in corrosion science, which also has the potential to enrich a variety of other scientific and engineering fields [25].

Although advancements in managing corrosion have been notable over the last several years, the fundamental nature of corrosion as an unavoidable process persists; the objective is not to eliminate but to reduce its impact. The quest to lessen the

material and financial tolls of corrosion spans numerous industries, including automotive, potable water infrastructure, household appliances, pipeline networks, bridges, and public edifices. Thwarting the expensive and dangerous effects of corrosion is a critical concern. Tactics such as case hardening, diffusion treatments, conversion coatings, cathodic and anodic protection have all been deployed in combatting corrosion, yet there is a continuous need for more advanced and improved strategies to effectively manage and decelerate this process and to foster the adoption of best practices. In this vein, one of the leading and most cost-efficient approaches to tackling corrosion is through the application of protective coatings [26]. There is an expectation that by advancing protective coatings that inhibit changes to the surface properties, the aforementioned losses can be significantly reduced. It is noteworthy that the direct costs attributed to corrosion management in the United States have been estimated at \$121 billion, which represents 1.38% of the national GDP, with organic coatings comprising 88.3% of these expenditures [27]. This data highlights the critical role of corrosion prevention strategies in diminishing maintenance expenses and prolonging the service life of assets. Out of the myriad of options available, corrosion protective coatings stand out for their economical and efficient performance [28].

3.2. COATINGS

3.2.1. Development of Coatings History

The origins of painting stretch back to more than 100 millennia, with early human expressions found in cave art [29,30]. Since these primitive beginnings, paint technology has undergone a remarkable evolution [31,32]. For example, around 2000 B.C., Egyptians broadened their color palette by pulverizing baked potter clay to extract red, green, and blue pigments. In the Chou era of ancient China (1122-222 B.C.), the use of lacquers became popular in embellishing chariots and armaments, favoring more resinous compositions over a diversity of pigments. By roughly 200 A.D., linseed oil gained prominence in paint formulations for its drying and cross-linking properties upon contact with air, maintaining its status in paint recipes well

into the 11th century. Fast forward to the 17th century, lead-based paints were introduced and became common for the preservation of wooden constructions [29].

Today, the production of paint involves a staggering array of formulations each year. Specifically, in the United States, paint usage in 1987 was recorded at about 3.8 billion cubic meters, valued at nearly \$10 billion, underscoring the paint industry's significant contribution to contemporary society. Nevertheless, the evolution of paint technology presses forward, spurred by the continuous pursuit to fulfill emerging performance demands, environmental concerns, and economic factors with innovative paint compositions.

3.2.2. Coating Industry

Coatings are a pivotal defense against the degradation of metal materials due to corrosion [33]. Their significance is recognized in the prevention of both localized and widespread corrosion phenomena. Coatings can be broadly divided into four main groups: organic, inorganic, conversion, and metallic coatings [34]. Fundamentally, a coating is a substance, typically in a liquid state, that is applied onto a base material that hardens to form a protective layer [35]. The essential function of these coatings is to delay or alter the corrosion process. They are most commonly placed on the external part of objects to protect against the corrosive effects of the environment and contaminants. Coatings also defend against incidental contact with corrosive substances, which requires a protective layer on the metal surface [34].

The composition of coatings is intricate, involving a mix of components that must be precisely blended and applied to the base for optimal performance. Effective drying of the coating is crucial for its efficacy. While the specific makeup and application techniques can differ, an exemplary coating is characterized by several attributes [36,37]:

- a) It should form a continuous and uniform film on all surfaces.

- b) It should exhibit impermeability to various components and display low reactivity with the environment.
- c) It should withstand mechanical and physical stresses.
- d) It should fulfill the desired function, such as meeting aesthetic expectations or providing corrosion resistance.
- e) It should be cost-effective and compatible with the product.
- f) It should minimize energy loss in the environment.
- g) It should maintain stability over extended periods.

3.2.3. Organic Coatings

Thin layers of organic coatings are placed to metal surfaces to improve the material's surface properties and provide protection from external factors. These strata serve as a barrier to prevent the entry of substances such as water, oxygen, and different ions.

These coatings extend the life of metallic objects and offer protection from a range of physical and chemical factors, including moisture, wear and tear, exposure to the environment, and resilience. In addition to providing protection, organic coatings improve the appearance of metal surfaces. The performance of organic coatings depends on a range of aspects, such as the mechanical strength of the coating system, the type and amount of corrosion inhibitors integrated within the coating, the surface preparation before application, and how well the coating adheres to the metal substrate [34,36].

Coatings can be broadly classified into two categories based on how they are applied: thermoplastic and thermosetting coatings [38,39].

A. Thermoplastic Coatings

Thermoplastic coatings are made up of polymers that are not chemically crosslinked. These coatings give the necessary mechanical strength by utilizing one or more high molecular-weight polymers. and do not need additional polymerization. The benefits of thermoplastic coatings include their simple drying process through the loss of solvent and their re-solubility in a corresponding solvent. Nevertheless, their heat

sensitivity is a drawback; they tend to soften and become tacky when subjected to high temperatures [40].

B. Thermosetting Coatings

On the other hand, thermosetting coatings begin as lower molecular weight polymers that undergo further polymerization as they dry, creating a durable film characterized by a three-dimensional cross-linked molecular structure. This structure endows them with an enhanced resistance to both solvents and heat. This group includes a variety of coatings, from traditional oil-based paints that cure over time through exposure to air, to advanced epoxy-polyester powder coatings that form cross-links quickly while it is heated in the oven [41].

3.2.4. Organic Coating Materials

Coating materials are described in Fig. 3.3 [42]:

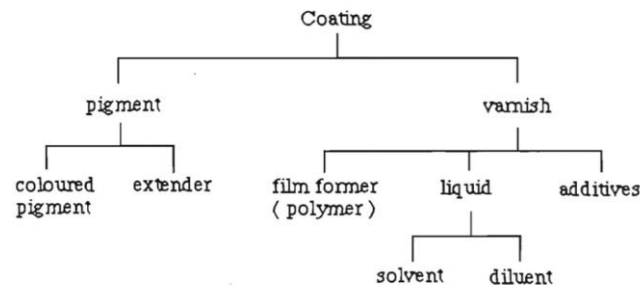


Figure 3 3. The composition of the coating material

A. Pigments

According to ASTM D16-47[43], pigments are defined as fine, insoluble solid particles within a paint medium. They are essential in establishing the definitive attributes of coatings. The selection, concentration, and granularity of these pigments influence various coating characteristics, such as sheen, structural integrity, thickness, and ease of application. Certain pigments additionally serve to prevent corrosion.

The size, form, and even spread of these pigment particles are pivotal in their functionality [44,45]. As paint dries, these pigment particles have a tendency to clump together. To achieve uniform dispersion in coatings, manufacturers must effectively break up these clumps into discrete particles. This is typically achieved through a milling process, which can increase the vibrancy and intensity of the color at the cost of a thicker viscosity [46].

Coatings incorporate three primary classes of pigments: white, colored, and non-reactive pigments. White pigments are optimized to minimally absorb visible light and are adept at dispersing light throughout the paint film, a process contingent on the size and density of the pigment particles.

Colored pigments offer a diverse palette for coatings, selectively absorbing particular wavelengths of light. The criteria for choosing colored pigments include their hue, affordability, translucency, longevity, tolerance to heat and chemicals, and their ability to resist color run. Finer pigment particles tend to have a more potent light-absorbing effect, yet particles that are too fine may negatively influence other aspects of the pigment's performance. The production of pigments typically aims for an optimal particle size that balances color intensity with other desirable traits.

Non-reactive pigments, in contrast, neither significantly absorb nor scatter light in coatings. Their main role is to bulk out the coating, altering its flow characteristics and the qualities of the dried film. While it's possible to use larger amounts of white or colored pigments to achieve similar effects, non-reactive pigments are a more economical alternative.

B. Additives

A variety of additives are incorporated into polymers, such as stabilizers, fillers, and plasticizers, and these are often found in coatings as well [47,48]. However, coatings benefit from certain specialized additives designed for their unique requirements, such as anti-skinning agents, pigment dispersion retainers, flattening agents, Antilivering agents, and leveling agents.

- a) Anti-skinning agents are added to coatings to inhibit surface film formation during the drying stage [49].
- b) Pigment dispersion retainers ensure the even spread of pigments within the coating, preventing the uneven coloration or ‘flooding’ that results from pigment settling [50].
- c) Flatting agents serve to diminish the shine of coatings, Commonly used substances include fumed silica gel and surfactants [51].
- d) Antilivering agents mitigate gelling caused by interactions between the pigments and the resin (livering).’ To stabilize the mixture, compounds such as esterified fatty acid glycol ethers and certain amines are introduced [52].
- e) Leveling agents work to smooth out application marks, such as those left by brushes, contributing to a more uniform finish.

C. Solvents

Solvents play an indispensable role in moderating the thickness of paints that use high molecular-weight polymers by decreasing their viscosity. This reduction is vital for ensuring even application and proper adherence to the underlying material. As the paint sets or cures, solvents dissipate, aiding in the solidification of the paint layer.

A wide array of solvents is used, ranging from water to various organic compounds like aliphatic hydrocarbon mixtures, aromatic hydrocarbons, alcohols, esters, ketones, and ethers, as well as specialized substances like ether-alcohols, nitroparaffins, and chlorinated paraffin [53]. Critical characteristics of solvents for use in coatings include their dissolving power, thickness, boiling range, rate of evaporation, ignition point, chemical composition, harmfulness, smell, and economic value. The proportion and viscosity of the solvent are key factors affecting the fluidity of the polymer solution. Furthermore, the final coating’s thickness can dictate how much solvent remains; thicker layers may trap solvents longer, slowing evaporation. This could result in solvent remnants within the dried coating, potentially diminishing its quality and performance.

D. Resins

A diverse array of resins are employed as binders in coating compositions, which include alkyd, amino, acrylic, polyester, and epoxy resins [54].

Alkyd resins are economical and suitable for both air-drying and forced-drying applications, known for their cost efficiency and straightforward application due to their lower surface tension. However, they may become brittle over time, change color as they age, and exhibit limited resistance to saponification.

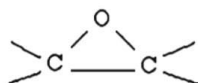
In the realm of amino resins for coatings, melamine-formaldehyde, and urea-formaldehyde are predominant, but various other amino resins are utilized as well. Urea-formaldehyde is a common choice for wood furniture finishes due to its aesthetic quality, and certain durable resins are selected for appliance coatings, like those on home laundry appliances, where resistance to detergents is critical.

Acrylic resins are segmented into thermoplastic and thermosetting types and are broadly used across industries for products such as automotive finishes, including clear coats, as well as for architectural and enamel coatings. Acrylics are recognized for their resistance to becoming brittle and for maintaining their integrity in outdoor settings.

Polyester resins, which are essentially low molecular weight polyesters with hydroxy- or sometimes carboxylic acid end-groups and no oil content, have become prominent, particularly in melamine-formaldehyde cured baking enamels, as an alternative to alkyd resins. When it comes to adhering directly to metallic surfaces, polyesters are often preferred over acrylics.

3.3. EPOXY COATINGS

Epoxy resins are characterized by the presence of at least two epoxy groups per macromolecule. These epoxy groups, also known as epoxide, oxirane, or ethoxyline groups, have a ring structure with three members.



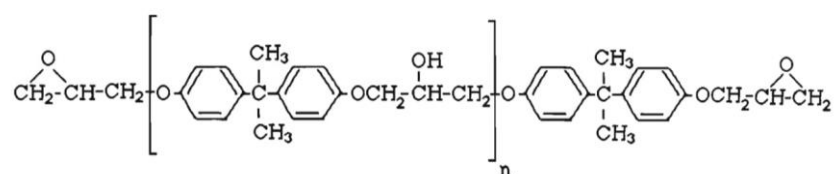
Epoxy resins available in the market can be based on aliphatic, cycloaliphatic, or aromatic structures. Their adaptability is derived from the reactivity of the epoxy group with a range of functional groups. The transformation of these resins into an inflexible, insoluble thermoset polymer occurs upon curing with specific agents that facilitate the crosslinking of the epoxy molecules. By altering the resin composition, hardening agents, or integrating various additives, the properties of epoxy resins can be tailored to a broad spectrum of uses, such as in adhesives, protective coatings, and as the matrix material in composite products.

The primary attributes of epoxy resins are detailed below [55]:

- a) **Adhesion:** One of the stellar characteristics of epoxy resins is their remarkable adhesion, especially to substrates like metals, glass, and ceramics. This adhesion attribute emanates from their epoxy and hydroxyl groups which facilitate a strong bond. Moreover, the typically low viscosity of these resins aids in enhancing wetting, spreading, and penetration capabilities, further bolstering their adhesive potential.
- b) **Cohesion:** Upon undergoing a proper curing process, epoxy resins manifest a strong cohesive strength which essentially is the internal bond strength of the resin. Coupled with their high adhesion to other materials, under stressful conditions, failure is more often witnessed in one of the adherents rather than within the epoxy resin itself or at the interface, showcasing the robustness of epoxy's cohesive and adhesive strengths.
- c) **Resistance to Solvents and Moisture:** Epoxy resins are more resistant to moisture than other resins, which enhances their usability and longevity in moist settings. Additionally, they function as a barrier against chemical attacks and are more appropriate for a variety of coating applications due to their exceptional solvent resistance.

- d) Low Creep: One special advantage of cured epoxies is their minimal creep and steadfast ability to maintain their shape under prolonged stress. In contrast, phenolics and a few other sticky resins might show observable creep. For the material's long-term stability, epoxy resins cure in a way that stops water or other condensation byproducts from escaping.
- e) Temperature Resistance: Because of their formulation flexibility, epoxy resins can be tailored to give cured solids that can withstand high temperatures-often as high as 200°C. This temperature resilience increases their value in circumstances when resistance to high temperatures is required.
- f) Low Shrinkage: The propensity of epoxy resin to have low shrinkage sets them apart from other kinds of resin. The main explanation for this property is that epoxies cure without releasing water or causing any other kind of reaction. byproducts, a characteristic that is commonly seen in phenolics and other types of resins. This minimal shrinkage property enhances the cured epoxy's performance in a variety of applications by maintaining the material dimensions and integrity.

Phenoxy resins, multifunctional epoxies, cycloaliphatic epoxies, liquid bisphenol A epichlorohydrin epoxies, and epoxidized natural oils are only a few of the several varieties of epoxy resins available on the market. Specifically, liquid bisphenol A epichlorohydrin epoxies are frequently selected due to their excellent performance in adhesives and coatings. This resin can be described using the structural formula below [56] :



$$n = 0, 1, 2$$

liquid bisphenol A epichlorohydrin resin

These resins' molecular chains can be ended by up to two epoxy groups. The polymer backbone has varying numbers of hydroxyl groups. These hydroxyl groups are vital for creating strong adhesion to polar substrates by means of Van der Waals forces and/or acid-base interactions at the interface. The polymer backbone's carbon-carbon and ether bonds confer these resins with impressive chemical resistance.

Recent research has turned to polymer coatings for their straightforward manufacturing process, superior tribological behavior, and improved substrate adhesion. As Table 1 illustrates, epoxy resins outperform many polymers in shear strength. These resins are known for their minimal shrinkage during curing, enhanced thermal stability, and exceptional mechanical robustness in comparison with other resin types as seen in Table 1. Their resistance to chemical wear and corrosion further positions epoxy resins as preferable materials for protective coating applications.

Table 3 1. Common Polymer Resin Properties:[57,58]

Property	Polyester	Vinylester	Epoxy
Specific gravity	1.2	1.2	1.25
compression or Tensile strength (MPa)	55	75	75
Young modulus in tension (MPa)]	3350	3350	3100
In-plane shear modulus (MPa)]	1350	1400	1500
Shear strength (MPa)]	approx. 50	approx. 65	approx. 80
Expansion coefficient (10 ⁻⁶ K ⁻¹)	50–120	50–75	45–65
Cure shrinkage (%)	5–12	5–10	1–5

3.3.1. Modification of Epoxy Resins for Improved Properties

In the realm of material science, there has been an increasing interest in augmenting unaltered epoxy systems by integrating micro and nano-sized fillers, giving rise to either standalone or composite micro/nanocomposite coatings. These innovations are directed at overcoming the inherent drawbacks of unmodified epoxy coatings. Epoxy

coatings can be broadly divided into two categories: those based on liquid formulations and those known as fusion bonded epoxy (FBE) coatings, which are based on powder.

With liquid-based epoxy (LBE) coatings, the base resin is in liquid form. A variety of fillers have been widely explored to enhance the original frictional properties, corrosion resistance, and mechanical support capabilities of the epoxy resins. Figure 3.4 depicts a selection of fillers that are frequently incorporated to enhance the properties of liquid epoxy resin coatings [57].

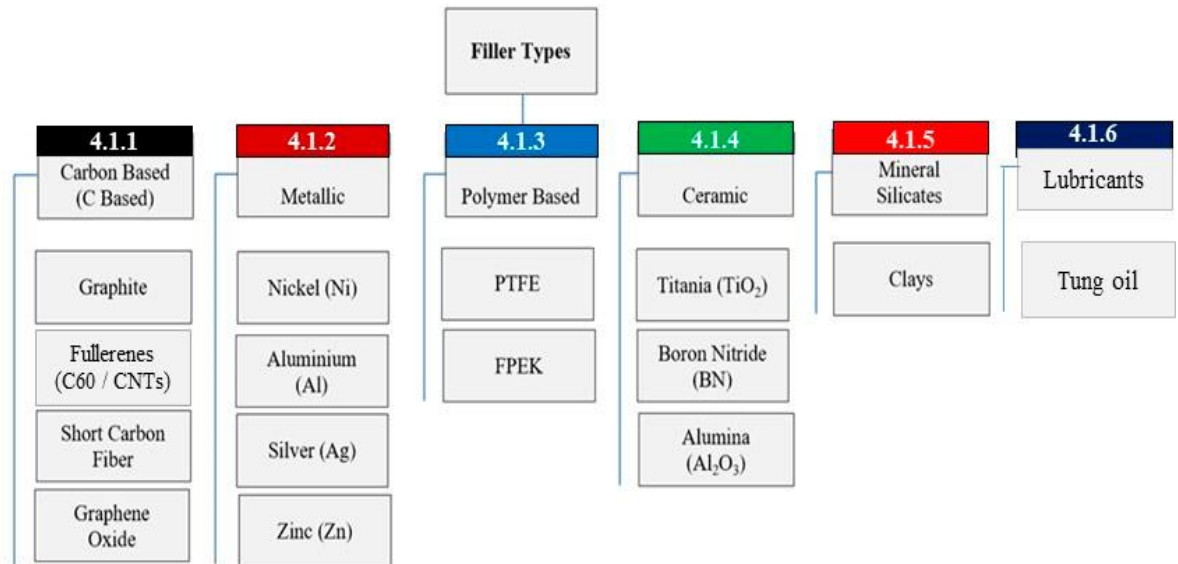


Figure 3 4. Different fillers Classification used for epoxy matrix reinforcement.

Fillers are typically categorized into several groups, such as carbon-based, metal-based, polymer, ceramic, mineral silicates, and lubricative varieties. These can be embedded into an epoxy matrix singularly or mixed to create composite or hybrid systems. Such hybrid configurations are known to capitalize on the distinct attributes of each filler type, discovered through methodical testing. The employment of these fillers, either singularly or as part of a hybrid mix, has been proven to substantially enhance tribological performance by diminishing friction and improving corrosion resistance.

The current research incorporates alumina and silicon carbide as fillers within an epoxy blend to bolster the characteristics of the resultant epoxy coatings.

Alumina or aluminum oxide (Al_2O_3) is predominantly known for its use in producing aluminum [59], a metal anticipated to see increased demand with the shift toward a low-carbon future. Alumina exhibits a multitude of advantageous features, such as excellent thermal conductivity, robustness, abrasion resistance, chemical stability, and resistance to wear and corrosion. These attributes not only contribute to aluminum's resistance to corrosion but also render alumina a robust candidate for cost-effective industrial applications [60].

Silicon carbide, also known as carborundum, is a robust chemical compound formed from silicon and carbon in a crystalline formation. Its versatility has led to widespread application across numerous sectors. Since its initial use as an abrasive in the late 19th century [61], SiC has expanded its applications to include the semiconductor industry. Its outstanding qualities include maintaining structural integrity at elevated temperatures, formidable high-temperature oxidation resistance, superior wear resistance, minimal thermal expansion, high thermal conductivity, exceptional resistance to thermal shock, extreme hardness, a lower density contributing to lightness, and reliable corrosion resistance [62].

3.4. COATING ADHESION

3.4.1. Significance of Coating Adhesion

Coatings hold a pivotal role across a spectrum of applications spanning varied fields, serving multiple purposes that align with the specific needs of those applications. The utility and performance of coatings are often tested under various stresses, contingent on the distinct conditions under which they are utilized. A prevailing issue that can surface is inadequate adhesion, which can culminate in the delamination of the coating from the substrate, thereby undermining its efficacy and longevity. Hence, a universally desirable trait in coatings, irrespective of their designated

function be it functional, decorative, or protective robust adhesion to the substrate. The importance of this adhesion is exemplified by the following instances [63] :

- a) In instances where coatings are manifested as thin film coatings (with a thickness of less than $1\text{ }\mu\text{m}$), they are intrinsically fragile and necessitate the support of a more robust substrate to maintain their structural integrity. The extent to which these thin films can capitalize on the support and strength offered by the substrate is intimately tethered to the level of adhesion established between them.
- b) The overarching attributes of durability, longevity, and wear resistance embodied by coatings are heavily influenced by the degree of adhesion they share with the substrate. A solid bond between the two ensures the coating remains intact and operational over an extended period, thus augmenting its overall lifespan.
- c) Strong adhesion is essential in a range of applications where coatings act as shields against environmental harm. When corrosive substances and moisture are present in the environment, weak adhesion can cause the underlying material to deteriorate quickly.
- d) 4. To increase their gas permeability, polymer substrates can sometimes be metallized. Good metal-to-polymer adhesion is crucial to the success of this improvement, highlighting the importance of adhesion in such technical operations.
- e) Adhesion influences the formation of the film and its subsequent physical qualities in thin-film technology, such as that utilized in vapor deposition techniques, which are critical to the film's functionality. When applied on base metals, adhesion affects a number of properties of metal coatings, including the grain structure, integrity, and uniformity.
- f) The foundation of overall performance and structural integrity in the field of multilayer construction is the adhesion between individual layers. Strong adhesion makes sure that the layers work together harmoniously, which maximizes the multilayer construct's performance and robustness.
- g) In microelectronics, the integrity of circuits depends on the adherence of thick-film ($>1\text{ }\mu\text{m}$) conductors, particularly when using techniques like

reactive and frit bonding. The dependability and effectiveness of the microcircuitry are determined by the adherence of these conductors to other components, highlighting the importance of adhesion in such complex production procedures.

The previously listed instances clearly demonstrate how important adhesion is to coatings technology. It's an essential aspect that significantly affects the functionality, robustness, and general performance of coatings in a wide range of applications, therefore careful consideration and optimization are required to guarantee the achievement of the intended goals.

3.4.2. Mechanisms of Adhesion

Understanding how surface chemistry and physical topography affect metal adhesive bonding has garnered a great deal of scholarly attention throughout the last 20 years.

When a coating adheres to a substrate, it typically does so through a combination of two principal bonding mechanisms: chemical and physical. Chemical bonds are established through molecular interactions between the substances on the substrate's surface and the coating's molecules. Physical bonds may develop from either the coating getting mechanically entangled with the substrate or through van der Waals forces – a form of physical adsorption. Physical bonds may also form when the coating's molecules diffuse into the substrate. Broadly speaking, these bonding processes fall into four main categories [64,65].

A. Mechanical Interlocking

Mechanical interlocking transpires when the surface of the substrate has pores or projections that the adhesive can fill or wrap around, respectively [66, 67]. Once the adhesive hardens, it forms a mechanical bond, which can ensure strong joints even if the natural affinity between the adhesive and the substrate is minimal. This kind of bonding is especially relevant for porous and fibrous materials like wood, fabrics, or paper. To enhance this effect, surfaces of many metals and plastics are typically

etched before the bonding process to improve the penetration and interlocking of the adhesive. However, this bonding mechanism is less effective for bonding smooth, non-porous materials such as glass or certain types of metals.

B. Interdiffusion

Interdiffusion occurs when a liquid adhesive dissolves and permeates the substrate material. This process is influenced by the compatibility between the adhesive and substrate molecules. Typically composed of polymers, adhesive molecules only partially mix with substrate molecules to form a narrow interdiffusion zone that spans 0.5 to 10 nanometers. However, this interdiffusion zone can grow to thicknesses of up to 10 micrometers when materials are highly compatible. In contrast to mechanical interlocking, which involves the adhesive filling or wrapping around larger physical features of the substrate, this phenomenon includes the mutual penetration of adhesive and substrate molecules at a molecular scale.

C. Adsorption and Surface Reaction

Organic adhesive chemicals cannot penetrate thick, nonporous surfaces like metals or ceramics when attached to them. In these situations, adhesion is promoted by the attachment of adhesive molecules to the solid's surface, frequently in response to surface reactions.

The affinity of adhesive molecules for certain spots on the solid's surface defines the adsorption process. This attraction may result from particular donor-acceptor interactions or from generic van der Waals interactions, which are greater when molecules have higher electrical polarity. Furthermore, the adhesive and the substrate may engage in chemisorption, which is the formation of a chemical bond at the surface interface, thereby enhancing adhesion.

D. Electrostatic Attraction

It is suggested that when materials with dissimilar electronic band structures come into contact, electrostatic forces may emerge at their juncture. These forces are a consequence of electron migration across the interface, resulting in the generation of opposite charges that draw together. Take, for example, the interaction between an organic polymer and a metal; electrons tend to move from the metal to the polymer, thereby establishing an electrostatic double layer [68]. The relative strength of these electrostatic forces compared to van der Waals forces has been a point of contention. Some researchers maintain that the contribution of the electrostatic double layer to overall adhesion is minimal, whereas others believe that these forces are a predominant factor [69].

3.4.3. The Factors that Affect Adhesion

The robustness of adhesion is shaped by an array of elements. These include characteristics of the coating such as its viscosity and surface tension, attributes of the substrate like its texture and cleanliness, qualities at the interface including stress levels and chemical connectivity, as well as external variables like humidity, heat, and atmospheric pressure [63]. Table 3.2 details the variables that impact the initial strength of adhesion.

Table 3 2. The factors affecting the adhesion

The factor affection the initial adhesion	
Environment	Moisture
	Temperature
Coating Material	Coating Thickness
	Coating Composition
	Penetration Into Substrate
	Physical Properties
Substrate	Surface Energy
	Micro Surface Roughness
	Microstructure
Interfacial Properties	Chemical Bonds
	Wettability
	Internal Stress

3.4.4. The Methods to Improve Coating Adhesion

3.4.4.1. Surface Pretreatments of Substrates

The Surface Pretreatments of Substrates include:

A. Cleaning by Using Solvents

Surfaces of substrates repeatedly harbor impurities like oils and greases, which require removal to guarantee effective adhesion. Usual methods of pretreatment involve steam degreasing, cleansing the surface with cloths moistened in a solvent, or utilizing degreasing baths in liquid or vapor form, which may include the use of ultrasonic agitation. Solvent pretreatment is advised as the essential minimum step before applying a coating [65].

Subsequent to the degreasing stage, any remaining impurities are often removed using an alkaline cleaning solution that emulsifies these surface impurities, suspending them away from the surface.

B. Mechanical Abrasion

A range of techniques exists for mechanically abrading surfaces, including the use of wire brushes, abrasive cloths, scouring pads, and various forms of blasting with grit or shot. Among these, grit blasting is favored in industrial settings for its reliable outcomes when it is a practical option. Mechanical abrasion can enhance the surface texture of the substrate, which could contribute to better adhesion. This enhancement is due to multiple factors: the roughened texture's interlocking effect, the abrasion method's thorough cleansing effect, an increased surface area for the coating to adhere to, and frequently, an improvement in the coating's ability to spread over the surface.

C. Chemical Pretreatments

Chemical treatments for surface preparation include the use of particular inorganic or organic substances to change the surface's chemical makeup or increase its area. Although they can be costly and require careful administration, these techniques are known to significantly increase the bond strength.

Oxide layers that metals frequently form can impede the bonding process. These oxides can be removed physically using techniques like grinding or abrasive cleaning, but they quickly reappear. While removing oxide layers with basic or acidic solutions can be successful, but rinsing and drying the surface may result in the formation of new oxide layers. Interestingly, these newly generated layers might occasionally help with adhesion because they may include characteristics like porosity, thread-like extensions, or a tiny roughness that can provide mechanical support for coating adhesion by offering interlocking locations [70].

3.4.4.2. The Agents of Chemical Coupling

Agents of Chemical Coupling, which are low molecular weight compounds with multiple functional groups, serve as the ultimate pretreatment phase to create a chemical linkage between the metal substrate and the polymer coating. These agents

are generally deployed via a solution. Their role is crucial in establishing water-resistant covalent connections at the interface between metal and polymer, thus improving the bond's strength and the long-term performance of metal/epoxy coatings.

3.4.4.3. The Relieving of the Internal Stresses

Internal Stresses can be relieved by the following:

A. Fillers Addition

Incorporating fillers into epoxy mixtures can mitigate the level of internal stresses that arise during the curing process. By introducing fillers, The coefficient of thermal expansion of the epoxy coating is reduced, bringing it closer to that of the substrate and thus diminishing the thermal expansion disparity. Fillers, particularly those like aluminum or alumina, have been recognized for their role in bolstering the bond strength within metal/epoxy coating systems. The underlying reasons for this enhancement may include the alleviation of internal stresses or the elevation of the coating's fracture toughness due to the presence of the filler particles. Furthermore, the use of fillers can lead to benefits like cost savings and heightened resistance to wear [71, 72].

B. Addition of Flexibilizers

Additionally, the integration of flexibilizers into epoxy coatings can lessen internal stress. Monofunctional flexibilizers usually have extended, pliable chains ending with an epoxy functional group. During the curing process, where the reaction chiefly involves epoxy groups, incorporating monofunctional flexibilizers lowers the system's cross-link density, thereby infusing the epoxy matrix with long, unattached chains. These unanchored chains lead to a more elastic network with a greater capacity for segmental movement. This increased flexibility enables the material to more effectively dissipate internal stresses that develop throughout the curing phase.

3.4.4.4. Ways to Avoid Delamination Caused by Corrosion

The formation of hydroxides at the interface between the metal and the polymer coating requires the simultaneous presence of five elements: moisture, oxygen, electrons, cationic counterions, and a catalytically active oxide layer for the reduction process. The interruption of this cathodic corrosion reaction can be achieved by eliminating at least one of these five essential components from the interface area [73].

- a) Water: The presence of water is fundamental as it acts as a medium through which ions can travel. It facilitates the transport of other essential components to the interface, thereby playing a critical role in the progression of the corrosion process.
- b) Oxygen: Oxygen is another key player in the corrosion mechanism and participates in the electrochemical events that occur at the interface between the metal and the polymer, facilitating the oxidation process that leads to the creation of hydroxides.
- c) Electrons: Participating in the electrochemical reactions at the interface are electrons, which are charge carriers. The redox reactions that drive the corrosion process depend on their mobility and transmission.
- d) Cation Counterions: During electrochemical reactions, the presence of cation counterions is necessary to maintain charge neutrality. They participate in preserving the ionic equilibrium, which helps to keep the processes at the interface running.
- e) Catalytically Active Oxide Layer: In addition to acting as a barrier, the oxide layer on the metal surface catalyzes the reduction processes that are essential for the creation of hydroxides. Its catalytic activity accelerates the rate at which these reactions occur, thereby influencing the extent of corrosion.

A promising approach to mitigate the destructive cathodic corrosion reaction is to eliminate or restrict the presence of at least one of these essential components in the interfacial region. By doing so, the electrochemical pathway necessary for corrosion can be disrupted. For instance, creating a barrier that prevents water or oxygen access

to the interface could significantly retard the corrosion process. Similarly, modifying the oxide layer to be catalytically inactive could potentially halt the reduction reactions essential for hydroxide formation. Thereby enhancing the longevity and performance of metal/polymer interfaces in a plethora of applications [74].

A. Reduced water permeation via the Coating of epoxy

Organic polymer coatings, while exceptional in many protective and aesthetic applications, have a known susceptibility to water permeation to a certain degree. This permeation occurs due to the molecular structure of the polymers, which may allow water molecules to traverse through, especially over prolonged periods or under certain environmental conditions. One of the common types of these coatings is epoxy coatings, widely used due to their strong adhesive properties and resistance to harsh chemicals. However, their interaction with water remains a critical concern as water permeation can adversely affect the coating's integrity and the underlying substrate's protection, potentially leading to degradation or corrosion over time [75].

Incorporating fluorine into the structure of epoxy resins has been found to diminish the extent to which water can infiltrate these organic polymer coatings. When fluorine is introduced into the epoxy network, a fluorinated epoxy resin coating is created.

The underlying chemistry behind this modification is that fluorine atoms possess a high degree of electronegativity, which can potentially repel water molecules, thereby reducing the affinity of water toward the coating. Moreover, epoxy fluorination can result in a denser molecular structure, which might make it more difficult for water molecules to try to pass through the coating. Extensive gains in water resistance have been shown in studies investigating the efficacy of coatings made of fluorinated epoxy resin. The effectiveness of this modification has been demonstrated empirically by fluorinated epoxy resin coatings, which are reported to absorb up to 85% less water than their unmodified equivalents. This is in contrast to regular epoxy coatings. This substantial decrease in water absorption is a big step

toward guaranteeing improved toughness and extended protective performance of epoxy coatings, especially in damp conditions.

B. Decreased Oxide Layer Electrical Conductivity

When an oxide layer forms on a substrate, the oxide layer's electrical conductivity is a critical factor that affects the electrochemical reactions that take place at the substrate-coating content. Particularly, when the oxide layer showcases poor electrical conductivity and has a relatively substantial thickness, it creates an excessive potential barrier that impedes the flow of electrons generated from the anodic reaction. Consequently, the electrons seek alternative paths, and electron flow is confined to the flawed areas of the coating where defects or anomalies exist. These defects in the coating may be due to mechanical damage, poor adhesion, or any other factor that compromises the integrity of the protective barrier, and they create a portal where the oxide layer has been eradicated, thereby facilitating the flow of electrons.

In areas where the coating is intact, and the oxide layer remains beneath it, When electrons cannot navigate through the oxide layer to reach the coating-substrate interface in areas adjacent to the defect, the corrosive reactions that drive delamination are hindered.

C. Utilizing of Inhibitors

corrosion inhibitors that are based on adsorption function by adhering to the metal surface, thereby impeding the processes that lead to anodic and cathodic corrosion. These inhibitors are proficient in preventing the delamination of coatings [76]. It is a standard procedure to incorporate fillers with corrosion-inhibiting properties into coatings.

3.5. STRUCTURAL and MORPHOLOGICAL CHARACTERIZATION TESTS

3.5.1. X-Ray Diffraction (XRD) Test

X-ray diffraction is frequently employed to determine the composition or structural properties of a specimen. For larger crystals, such as molecules that contain a mixture of organic and inorganic components, this can be employed to deduce the composition of atoms in the sample. A crystal's phase, composition, and structure can all be seen if it is too small. Through it, X-ray radiation is used in this process. Instead of using a larger wavelength, which would not be impacted by the spacing between atoms, X-ray beams are chosen because their wavelength is similar to the spacing between atoms in the sample. This will have the same effect on the angle of diffraction as the spacing between atoms in the molecule. The x-rays then passed through the sample, "bouncing" off the atoms in the structure to change the beam's direction from the initial beam by a factor of 90, or theta. This is the direction of the diffraction angle's arrow. Some of the diffracted beams have a negative association with one another, but if the beams have the same wavelength, then constructive interference will take place. constructive interference is when multiple X-ray beams of the same frequency and magnitude are created. The larger amplitude of the wave results in a larger signal for this specific angle of diffraction. The angle of diffraction can then be employed to differentiate between the atomic planes using Bragg's law as shown in the equation: fig 3.5

$$\sin\Theta=n\lambda/2d \quad (4,1)$$

where d represents the distance between the atomic planes, n: integer, O: diffraction angle,

Then, one can use the gap between the atomic plates to distinguish between structural or compositional characteristics [77].

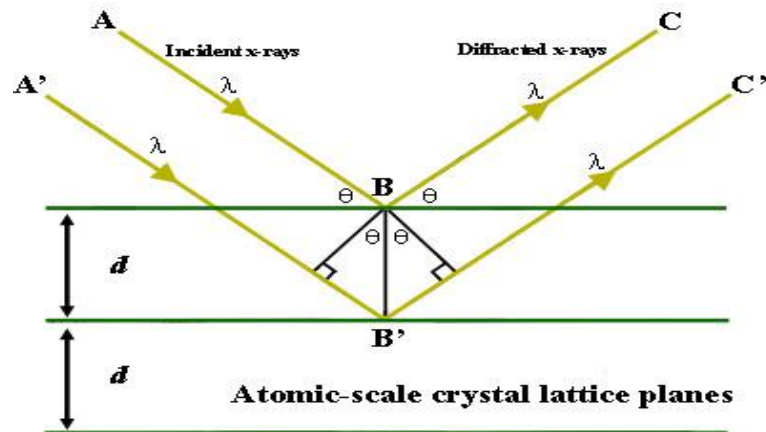


Figure 3 5. Bragg's Law reflected

When the distance between pathways ABC and A'B'C' differs by an integer number of wavelengths (λ), the diffracted X-rays show constructive interference.

3.5.1.1. Data interpretation of XRD

The result of the X-ray pattern's diffraction angles' outcome is the magnitude of the signal for each of the specific 2θ points in its diffraction pattern. The two theta positions are associated with a specific distance between the crystals or atoms in the sample, which is determined by the angle of diffraction from the X-ray beam that is incident on the sample. The amount of molecules in that phase or distance is closely correlated with the volume of the altitudes. The greater the peak's intensity, the more crystals or molecules of that particular composition there are [77].

The length of the peaks is dependent on the magnitude of the crystal. A higher elevation that is also less steep is associated with a larger crystal. A higher elevation that is more expansive suggests a smaller crystal, a flaw in the structural composition, or that the material is amorphous, a solid that lacks full crystallinity. For smaller samples, the patterns obtained via XRD analysis can be used to deduce the composition of the sample. A significant contributor to materials, substances, as well as minerals with the diffraction pattern connected to chemicals, elements, and minerals. When the pattern for an unknown compound matches the relative heights, widths, and locations of the diffraction patterns, it can be used to confirm the.

identity of an element by comparing it to values found in the literature and through experimentation. The figure 3.6 shows a diffraction pattern for silicon carbide.

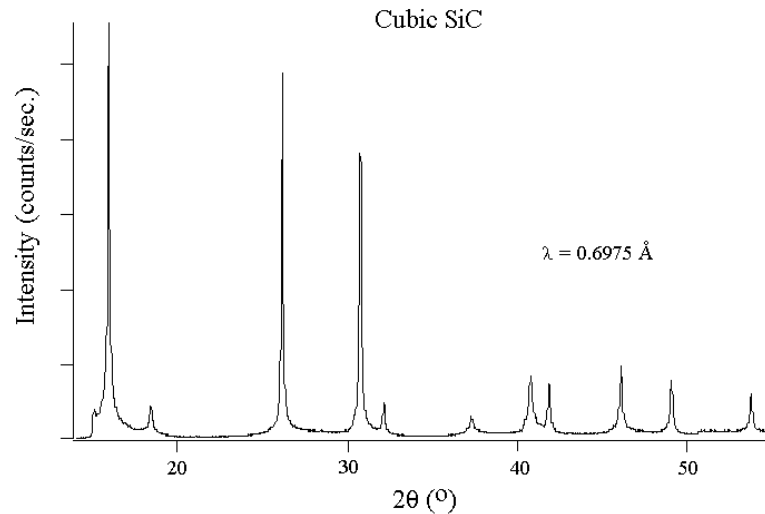


Figure 3 6. A diffraction pattern for Silicon Carbide (SiC)

3.5.1.2. Benefits and Uses of XRD.

XRD is a technique that is nondestructive and employs to [78]:

- a) Describe the characteristics of crystalline phases and their orientation.
- b) Determine the structural properties:
 - 1) Lattice parameters.
 - 2) Grain size
 - 3) strain
 - 4) Epitaxy
 - 5) Composition of phases
 - 6) Preferred direction
- c) Figure out how the atoms are organized

3.5.2. Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR is known as Fourier Transform Infrared Spectroscopy abbreviated as FTIR. The measurement of infrared spectrum is called FTIR, this analysis is concerned with

the measurement of radiation that is longer in wavelength and has a lower frequency than visible light. This spectrum is apparent in the sample's spectrum of infrared radiation (IR). The primary theory is that different atoms have different bond lengths, which results in different abilities to absorb light at different frequencies.

With the FTIR method, the light is measured by an infrared spectrometer that generates a spectrum of infrared light. The FTIR spectrum is a graph of the light absorption capacity of infrared radiation by the substance, which is represented by the frequency (wavelength) of the horizontal axis and the time of the vertical axis [79].

The analysis of FTIR is used to determine the length of the infrared spectrum that is possessed by a substance. This is accomplished by applying infrared radiation (IR) to material samples. The material's capacity to transform infrared radiation into other types of energy is assessed in order to ascertain the makeup and structure of the molecule.

Unknown chemicals are identified by observing the IR spectrum against a database that includes a broad spectrum of reference spectra. Materials can be identified using the technique of FTIR as long as a standard graph of the concentration of the component of interest can be created [80].

Fourier Transform Infrared Spectroscopy Analysis is capable of differentiating between different substances, components that are added to polymers, surface impurities, and other phenomena. The results of the tests can demonstrate the molecular composition and organization of the sample.

A common device that is typically used to identify samples is an interferometer, this device generates an optical signal that contains all of the infrared frequencies incorporated into it. The signal is recognizable easily [81].

Next, the signal is analyzed via a mathematical method of interpretation called Fourier analysis. This computer-created procedure then produces a correspondence

between spectrum data and its associated mapping. Figure 3.7 The resulting graph is the FTIR spectrum, which is then compared to libraries of references for identification.

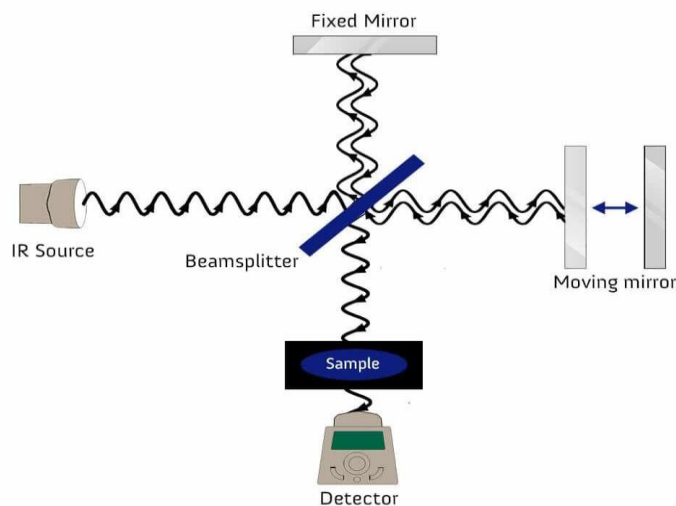


Figure 3 7. Principles of FTIR

It is possible to see samples as small as 20 micrometers with the microscope's attachment. This makes it easier to quickly and affordably identify unknown threads, films, or particles. Moreover, FTIR may detect additives or contaminants in substances, as well as the degree of oxidation of the polymer or the amount of polymer left in other materials.

3.5.2.1. Data Interpretation of FTIR

The infrared spectrum is shown by the x-axis, also known as the horizontal axis, which is used to indicate how intense an infrared spectrum is. The absorption bands are another name for the peaks, these are associated with the various vibrations of the sample's atoms when exposed to the infrared spectrum's region. For the middle range of IR, the wave number associated with the infrared spectrum is between 4,000 and 400 cm^{-1} . The y-axis is also called the vertical axis, this axis represents the amount of infrared light transmitted or absorbed by the sample material being examined.

The bands of absorption that are apparent when a substance is exposed to infrared light are typically partitioned into two categories: the group frequencies and the molecular frequencies associated with absorption [82].

The individual frequencies are dedicated to small clusters of atoms or groups of atoms that have a functional association like CH₂, OH, or C=O. These bands are typically observed at over 1,500 cm⁻¹ in the infrared spectrum, and they are typically associated with a particular functional group, this enables them to be used to recognize functional groups in a molecule.

For the fingerprint's frequency, this is specific to the molecule as a whole; it describes the internal composition of the molecule. These types of absorption are typically observed below 1500cm⁻¹ in the infrared spectrum; however, some functional groups will also absorb in this region. As such, this portion of the spectrum is less helpful in the identification process, but the lack of a specific band is often more significant than the presence of a specific band in this region.

3.5.2.2. The Advantage of FTIR

- a) Describe compounds that are smaller than 10-20 microns.
- b) Many instances involve the analysis of samples that do not have any adverse effects on the sample, nor do they alter the sample through analysis.
- c) In Infrared spectroscopy, the absorption bands in the middle of the spectrum are measured simultaneously, this provides a lot of analytical information quickly.
- d) The analysis itself can be accomplished in a relatively short time, too; users can complete expedited requests in as little as 24-48 hours.
- e) Helpful for studying a variety of organic and inorganic substances.
- f) Wavelength measuring is completely understood, which enables analytical techniques like spectral subtraction to be quick and highly accurate.

3.5.2.3. Usages of FTIR

FTIR spectroscopy is a recognized method of quality assessment in the evaluation of manufactured material this technique is often the first step in the process of analyzing a material. A change in the typical absorption pattern is indicative of a change in the composition of the material or the presence of contamination. If problems with the product are recognized by visual inspection, the origin is typically determined by analyzing the FTIR spectrum for micro-level changes. This approach is beneficial for studying the chemical composition of smaller particles, which are typically 10-50 micrometers in size, as well as larger regions on the surface [82, 83].

FTIR analysis is used to:

- a) Describe and identify materials that are unknown (e.g., films, solids, powders, or liquids)
- b) Recognize the presence of contamination in or on a material (e.g., fibers, particles, powders, or liquids)
- c) After obtaining the additives from a polymer's matrix, recognize them.
- d) Describe the process of recognizing oxidation, degradation, or uncured monomers in studies of failure.

3.5.3. Scanning Electron Microscopy (SEM) and Energy Dispersive Analysis (EDS)

Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) can study specific components of a surface's composition. These methods are typically used for the analysis of material surfaces, the investigation of design failures, the identification of contaminants, the analysis of solder joints, and other purposes.

Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) are both considered to be "upgraded" instruments for scientists that study substances. Since the commercialization of the method in the 1950s, several

significant advances in technology have occurred, however, the fundamental physics of the method has not been altered [84].

SEM is the imaging device associated with the technology. While an "ordinary" optical microscope employs light to image, a scanning electron microscope employs electrons that essentially transmit the interactions of electrons with light into an optical signal. While optical microscopy has benefits in certain instances, it also has limitations, the first of which is the light wavelength and the low focal depth. However, with SEM, the magnitude and resolution are primarily defined from the electron optics and the interaction of the sample, this increases the depth of focus.

3.5.3.1. The Principles of Operation of SEM

The physical mechanism associated with SEM is basic and resembles the glorified of a cathode ray tube. The electrons are released at the cathode by using a metallic wire to carry electricity. The electrons are followed by a high voltage that is used to accelerate them towards the anode. At the anode, the electron beam is restricted through a series of small openings, and the resulting magnetic fields that surround the column are generated. The electron beam is finally freed from the column and is directed towards the sample's surface. When the adjacent beam lands on the sample, several physical effects occur, however, we discuss primarily two of these physical interactions in regards to imaging: the production of secondary electrons and backscattered electrons.

- a) Other electrons are derived from the sample itself, these electrons are released by the beam's energy. The secondary electrons are "summoned" by a centered detector that employs the secondary electron to illuminate a scintillating material on the surface of the detector, similar to the way a cathode ray tube would utilize phosphorus to demonstrate light on a screen. The fluorescent material on the detector's surface is converted to a digital signal using a photomultiplier. The location of the signal is determined by "scanning" the sample with the electron beam and the detector's signal is connected to the beam's position.

- b) Backscattered electrons aren't derived from the sample. They're the electrons that result from the composition of the sample, these electrons then exit the sample.

Backscattered electrons have lower energy than secondary electrons, and we're primarily utilizing them to differentiate regions with different average atomic numbers, because of their capacity to show a difference in absorption between substances.

Because SEM employs electrons that are excited to visualize the object, the electron microscope must operate in a high vacuum environment that prevents the electrons from being absorbed by the atmospheric molecules as they travel to the object and the detector. As such, we must consider the behavior of the sample in high vacuum conditions, this includes the exclusion of biologics and liquids from the analysis. Additionally, the sample must have the capacity to complete an electrical circuit, this means that it must be conductive or designed to be conductive by sputter coating (the application of a "thin film" of material that is conductive). Many of the technological advances over the years have attempted to make the use of SEM in low vacuum conditions more accessible, with a decreased surface conductivity. The digitalization and computerization of image acquisition have also had a significant impact on the increasing capacity of SEM analysis [84].

3.5.3.2. The Energy Dispersive X-ray Spectroscopy (EDS)

At the surface of the sample, in addition to the electron interactions used for imaging, several physical processes occur. These interactions can be exploited to gain chemical knowledge. During the imaging process, electrons with an energy of around 12.8 kJ are created. As a result, the atom is ionized and additional electrons need to be added to it in order to return to the original state. According to the law of energy conservation, a photon is released that is associated with the shell transition that occurred and is "quantized" according to the rules of quantum mechanics. We consume the energy of the photons that are released, and "see" the different energy states that correspond to each element. The EDS detector is the device we utilize to gauge the energy of the photons that are released in the X-ray spectrum of electricity.

The detector is a solid-state chip or crystal that is temperature-suppressed to a superconducting state that has a high degree of quantum efficiency, it is displaced by 60 degrees from the beam's direction.

The detected X-rays are categorized by their interaction with the detector and the spectrum of energies associated with the detector is then assembled. With the spectrum, we can locate the greatest energy elevations and determine what chemical transition occurred, which element is associated with it. We can also utilize this information to enhance the SEM technique, this will produce X-ray images of the data that will be combined or displayed as a high concentration of individual components [85].

Technological developments in EDS analysis have resulted in the creation of superconductive detectors without requiring liquid nitrogen cooling. Additionally, more effective chips have been developed along with electronic processing that has caused a 1000-time increase in the typical detection threshold. A greater understanding of the X-ray intensity data has also led to accurate, numerical methods that are also applicable to other quantitative data. Ultimately, enhancements to software and computational abilities have contributed to an increase in the efficiency of EDS. Figure 3.8 demonstrates the SEM, an EDS device's simulation.

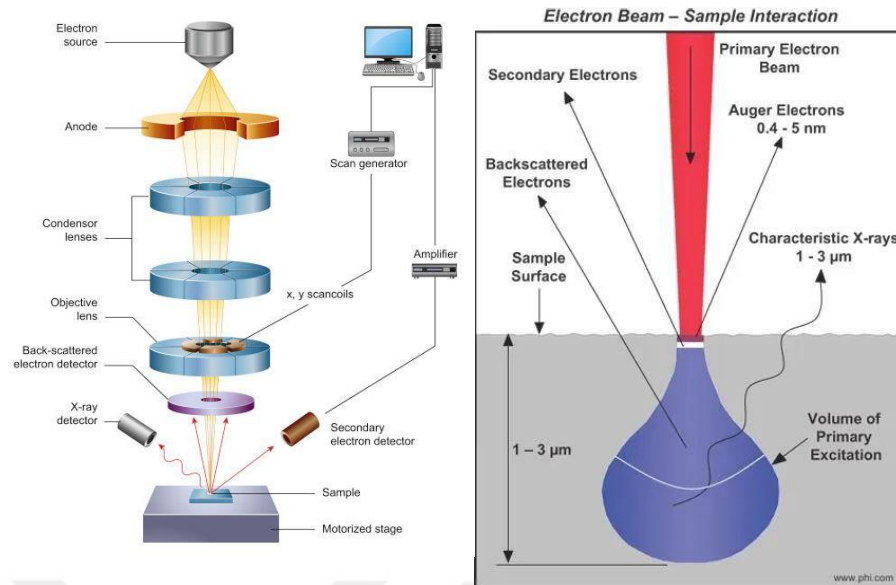


Figure 3.8. a. SEM, EDS device b. Interaction area of an incident beam.

3.5.3.3. The Utilization of Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)

SEM and EDS analysis are frequently utilized in a variety of locations that require a high-resolution, high-depth image as well as an elemental composition for a bulk material, or a specific region of the material. For example [85]:

- Failure analysis of components, metals, and plastics.
- The analysis of the surface features for micro-constituents or micro-inclusion content.
- The recognition and measurement of the surface characteristics of crystals about metal conversion coatings.
- The analysis of particle size and composition distribution.
- Contaminant identification in paint.
- The identification of the organic failure modes that lead to metal bonds.
- The identification of intermetallic compounds in the soldering interface (printed circuit boards).
- The characterization of the distribution of intermetallics at the interfaces of metallic coatings.
- Unknown material identification.

- j) The identification of alloys.
- k) Measuring the thickness and uniformity of coatings.

3.6. MECHANICAL PROPERTY TESTING

3.6.1. Vickers Hardness

The hardness test is a mechanical procedure for testing the properties of materials that is employed in the design of engineering, the analysis of structures, and the development of materials. The primary goal of the hardness test is to determine the appropriateness of a material for a particular application or treatment. The simple nature of the hardness test has led to its greatest popularity as a means of inspection for metals and alloys.

Hardness is the resistance of a material to permanent deformation that includes indentation, wear, abrasion, or scratch.

Vickers's hardness testing has been primarily utilized as a means of rudimentary and genuine material testing. The Vickers hardness test calculates the indentation hardness for a portion of metal, composites or ceramic. Ultimately, the toughness of a material is its capacity to withstand structural integrity and resistance to deformation following the application of constant pressure. As a result, the utilization of the Vickers hardness test.

Using a diamond indenter, pressure (sometimes referred to as a "load") is applied to the test material to create an indentation. The sample's hardness is directly correlated with the depth of the indentation on the material. Generally speaking, an object is harder the smaller the indentation Figure 3.9. Based on the amount of stress applied to the material being tested, hardness tests are divided into two categories: micro-indentation tests and macro-indentation tests. Loads of up to 200 gf are commonly used in micro-indentation tests, while loads of up to 1000 gf are used in macro-indentation tests. The most widely utilized is the Vickers Hardness test, which may be used for both macro- and micro-indentation [86].

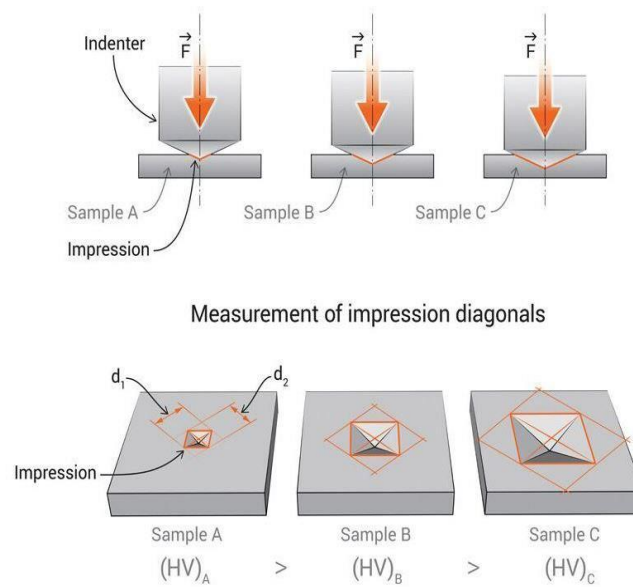


Figure 3 9. Vickers Hardness Test indentation examples – 3 materials of different hardness.

The process is initiated by putting the diamond indenter on the surface and pressing it into the intended area. The indenter is a pyramid and has a square base and a 136-degree angle between the adjacent sides. Typically, The full load is typically applied for 10 to 15 seconds. The length of each of the two diagonals on the test surface is measured as illustrated in Figure 3.10 , the average of these lengths is then used to calculate the Hardness, This is established by the formula F/A , where F is the force or load (in kgf) and A is the indentation's surface area (in sq. mm). Either the hardness value (H_v) or the DPH (usually a number without particular units) are reported. Microscopes that are integrated are used to take the measurements. Software is used by certain more sophisticated testing scales to produce faster and more accurate findings. The following formula is used to determine the Vickers hardness [86,87]:

$$HV = 1.8544 F/d^2 \quad (4,2)$$

F = Load in kgf

d = Arithmetic mean of the two diagonals, d_1 and d_2 in mm

HV = Vickers hardness

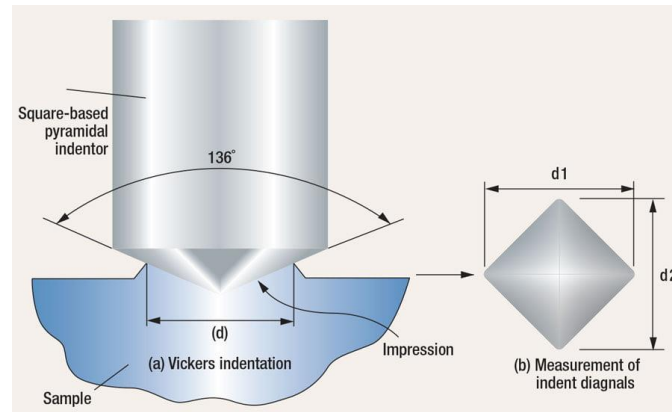


Figure 3 10. Vickers indenter and indent measurement

Several aspects need to be considered before the measurement process occurs. One of the conditions is having a flat, smooth surface that can be accurately measured and accurately indented. It must also be of a smaller size than the scale. Additionally, indentations must be large enough to achieve a high degree of accuracy in measurement. When all of these prerequisites are present, the testing process begins. Testing is accomplished by placing a sample on the tester, then the indenter makes contact with the sample's material, the indenter is squarer-based and has a pyramid-like shape. The load that is applied to the test sample presses into the test sample and has a dwell time of 10-15 seconds. When the dwell time is up, the load is excised, and the indenter is also released for the operator to observe via an optical method. The dimensions of indentation is determined. However, the smaller the indentation, the greater the probability of error, and the opposite is also true [88].

The Vickers hardness test is employed for the following reasons:

- a) Universal Indentation - Because of the universal nature of the indenters in the Vickers hardness test, the process of calibration is less time-consuming. The stretchiness of the diamond's shape also contributes to the fact that the indenters don't have to be replaced. Additionally, the indentations are identical for all tests, which eliminates the need for correctional factors in regards to particular materials.
- b) Non-destructive procedure – The material under test is not completely destroyed by the test, which allows it to be utilized for other purposes. Except

for the visible indentations, the Vickers hardness test is intended to minimize the wasted samples during testing.

- c) Useful range of application: The Vickers hardness test method is appropriate for measuring both hard substances, such as metal, as well as softer substances, such as plastic..

3.6.2. Scratch Test

Scratch resistance tests are an integral part of quality control processes, playing a vital role in assessing the ability of coatings and materials to withstand everyday wear and abrasion. Scratch resistance tests involve subjecting a coating or material to controlled scratching using different test methods. The primary reason is to evaluate the material's resistance to scratches and the effects of external forces, and these tests provide valuable insights into a material's mechanical properties, such as adhesion, hardness, and wear resistance. Moreover, they help identify potential weaknesses and enable the development of more resilient coatings. A few simple steps must be followed for scratch testing to be effective. Firstly, the sample surfaces must be visually inspected to ensure it is clean and free from contaminants, as any impurities or damages can impact the test results. Additionally, the sample should be representative of the material or coating under evaluation [89].

Once the sample is prepared, choose the appropriate scratch testator based on the method of testing desired and the material being tested. After that, the tester must be configured appropriately. This involves putting the chosen indenter into the testing device and adjusting it. Proper calibration ensures accurate and legitimate measurements during the testing.

Scratch testing is a quick and easy way to characterize coatings, but the results depend on many variables, such as the coating's thickness, the mechanical characteristics of the substrate, the bond between the substrate and the coating, and the testing environment, which includes the indenter's tip size, the load applied, and the rate at which scratches occur.

In this instance, the object is held motionless, and as shown in Figure 3.11, a Rockwell diamond tool with a 200-micrometer tip diameter is usually used to scratch the object for a predetermined length using a predetermined normal force [90].

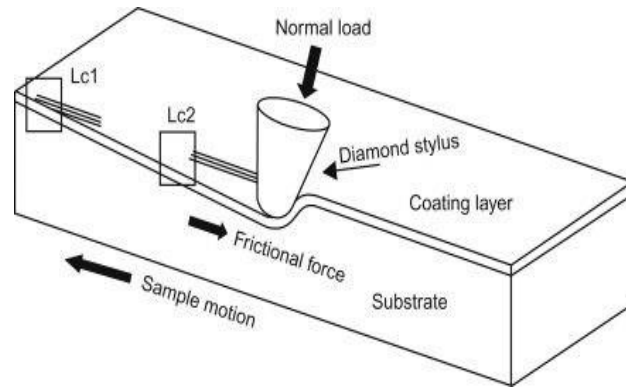


Figure 3 11. Diagram illustrating the workpiece being scratched by an indenter.

Lc1: Lc2: critical load for the initial crack and the critical load for severe cracking.

The load's capacity can be increased or altered in steps, depending on what is necessary. The acoustic emission, permeation depth, and tangential force of abrasion are used to quantify the output. The pressure generated by the frictional contact at the indenter's tip is measured using transducers. As seen in Figure 3.12, the load that contributes to the coating breaking is known as the critical load, and it is linked to both the increase in the coefficient of friction and the acoustic emission that is used to evaluate the coating's adhesion to the substrate. The cohesive, surface, conformal, and brittle chipping processes, among others, may be explained by the form of the scratch mark and the transverse direction cracks. The "lower critical load" is a measure of the coating's binding strength and is the load at which the first signs of failure show. The phrase "upper critical load" refers to the load at which the entire coating is peeling off the substrate and is used to assess the coating's strength to the substrate [91, 92].

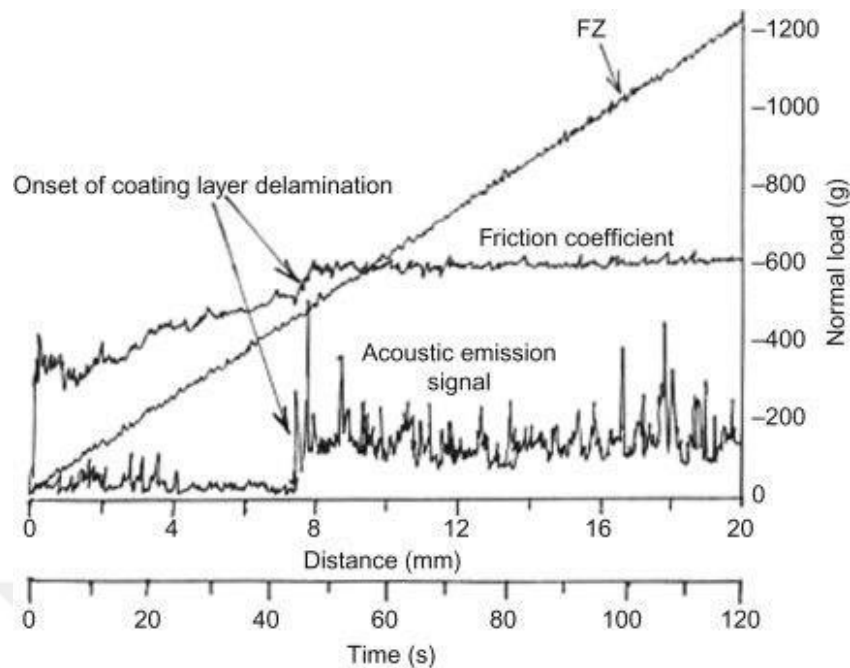


Figure 3 12. Typical scratch test data plots.

3.7. ELECTROCHEMICAL MEASUREMENT TESTS

3.7.1. Polarization Method (Tafel Extrapolation)

The term "polarization" is used to describe the open-circuit potential (corrosion potential) E , or a shift in potential with respect to the equilibrium potential of half cells. The polarization tendency is associated with the kinematic properties of the electrochemical reactions. Techniques for measuring the potential and current of electrodes are necessary to study this phenomenon, as well as the theoretical and experimental connections between them and the development of these connections through time. The electrochemical corrosion method is employed to estimate the corrosion rate of metals using a potentiostat, the method's purpose is to adjust the difference of potential between the applied and the detected currents, and to estimate the degree to which a metal is corroded. The potentiostat is linked to the corrosion cell as illustrated in Fig3.13 The corrosion cell was composed of the working electrode, which was formed from the coated sample, the SEC as the reference electrode, and the platinum as an auxiliary electrode. The three electrodes are placed in the corrosion test cell within a particular solution (corrosion medium) that is

connected to the potentiostat via its wires. An ammeter is positioned between the device and the auxiliary electrode in order to automatically measure the current level. Additionally, a voltage meter is connected between the reference electrode and the working electrode in order to measure the potential of metal [93,94].

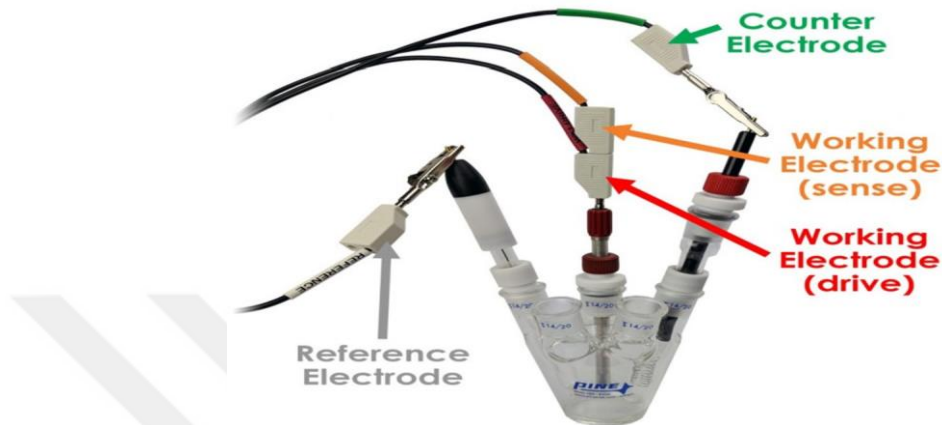


Figure 3 13. Corrosion cell

At the start of the test, the metal's potential is recorded, and a stable state is called the (Open circuit potential), then the metal's voltage is shifted to the anode and cathode positions by 200 mV from the stability voltage to illustrate the oxidation and redaction reaction.

A current flows from the potentiostat's apparatus to the solution, then to the working electrode via the auxiliary electrode in order to change the potential. When the voltage and current associated with the test are documented, several values are used to create a diagram known as the polarization curves for Tafel. The (i_{corr}) is deduced from the Tafel slope by taking two direct lines through the cathode and anode curves as illustrated in Fig.3.14 [95].

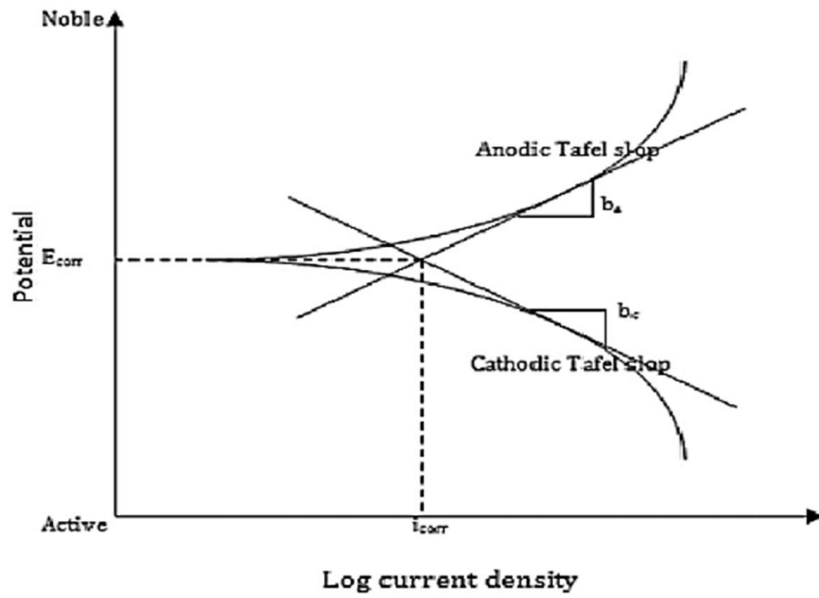


Figure 3 14. The polarization curves for Tafel

The R_p also was calculated using the equation [96].

$$R_p = \frac{B}{i_{cor}} \quad (4,3)$$

$$B = \frac{\beta_a \beta_c}{2.3 (\beta_a + \beta_c)} \quad (4,4)$$

Where:

And the corrosion rate (C.R) was calculated using equation (2.10) [97].

$$C.R = \frac{I_{corr} K EW}{d A} \quad (4,5)$$

Where K: a constant that defines the units for the corrosion rate = 3272 for (mm/year), EW: is the equivalent weight in grams/equivalent, d: is density in grams/cm³, A= surface area of the sample in cm² The inhibition efficiency percentage (IE%) was determined using the formula (2.11) [98]

$$IE\% = \frac{i^o_{cor} - i^f_{cor}}{i^o_{cor}} \times 100 \quad (4,6)$$

Where i^o_{cor} The corrosion current density of the uncoated Carbon steel specimen and i^f_{cor} is the corrosion current density of the coated specimen.

PART 4

METHODOLOGY AND EXPERIMENT

4.1. MATERIALS

In this study, the primary objective was to investigate the properties and characteristics of alloy 1008, which is classified as a low-carbon steel. The alloy used in the study was sourced from the Kardemir factory, situated in the Karabuk province.

To conduct the experiment, a comprehensive methodology was followed. Firstly, the alloy samples were carefully selected and prepared for analysis. The samples were obtained in a controlled manner to ensure their representativeness and reliability.

Next, a series of tests and analyses were performed on the alloy samples. These tests aimed to determine the composition and elemental makeup of the alloy. Table 4.1 presented in the study, provides detailed information about the elements that were found to be present in the alloy.

The analysis techniques employed in this study included but were not limited to spectroscopy, microscopy, and elemental analysis. These techniques allowed for a thorough examination of the alloy's chemical composition and structural properties.

Furthermore, the experimental setup and conditions were meticulously controlled to ensure accurate and reproducible results. The testing procedures were conducted in accordance with established industry standards and protocols.

The collected data from the experiments were then carefully analyzed and interpreted. Statistical methods and data visualization techniques were employed to derive meaningful insights and draw conclusions about the alloy's characteristics.

Table 4 1. The chemical composition of low carbon steel specimen

Fe	C	Mn	P	S
99.4%	0.10 %	0.50 %	0.040 %	0.050 %

4.2. HEAT TREATMENT

Heat treatment was carried out using a furnace of type "Protherm Furnaces" as illustrated in Fig 4.1, for low-carbon steel alloy 1008 at a temperature of 250 degrees Celsius, for two hours per sample. The primary objective of the heat treatment was to achieve homogeneity in the crystalline structure of the alloy. This goal is crucial for enhancing the material's internal composition, leading to improvements in its mechanical and structural properties. Fig 4.1 depicts the furnace utilized in this process.



Figure 4 1. Heat treatment furnace

4.3. COLD BAKELITE

Bakelite was employed for microstructure testing, with Cold Bakelite utilized, consisting of resin, color, and hardener. The process involved pouring the sample mixture into the mold and allowing it to set for 24 hours, as depicted in Fig. 4.2. Subsequently, the samples were removed from the mold after the designated drying period.



Figure 4 2. Sample after the cold bakelite process

This method utilizing Cold Bakelite, which includes a combination of resin, color, and hardener, ensures the formation of a stable and representative microstructure for testing purposes. The 24-hour setting time allows for the material to solidify and take the desired form in the mold, facilitating accurate and reliable microstructure examinations.

4.4. SHOOT PEENING

Before the coating process, the samples were subjected to shoot peening using a shoot peening device, as depicted in Fig 4.3. The primary objective of the shoot peening operation was to ensure stronger adhesion of the coating to the metal surface.

by giving the surface some roughness before coating. The results after the shoot peening process are evident in the image, illustrating the effect of the technique on the surface, enhancing effective coating adhesion. This surface preparation is crucial to ensure improved coating adhesion, thereby enhancing the mechanical protection and long-term durability of the metal.



Figure 4 3. Sample surface after Shoot peening process

4.5. COATING

Four cases were used to coat the samples with epoxy, namely epoxy with Al_2O_3 powder, epoxy with SIC powder, epoxy with Al_2O_3 + SIC powder, and epoxy. with 1% wt concentration of Al_2O_3 , 1%wt concentration of SIC, and 0.5% wt of concentration of Al_2O_3 +0.5% wt of concentration of SIC.

Different epoxy coating systems were prepared by combining different percentages using the rule of mixture equation as indicated in Table 4.2 the table illustrates the ratios of materials used for each coating process. The objective of employing these diverse coating materials was to assess their individual and combined effects on the performance and properties of the coated surfaces.

Al_2O_3 and SIC coatings were applied independently, each showcasing unique characteristics. Additionally, a combination of Al_2O_3 and SIC was used to explore potential synergies between these materials. Lastly, epoxy coating was introduced as a separated groups, providing a different perspective on protective coatings.

This varied approach allowed for a comprehensive evaluation of the coatings' effectiveness in enhancing durability, corrosion resistance, and overall performance. The specified ratios of materials in each coating process play a crucial role in determining the final properties of the coated surfaces.

Table 4 2. The amount of materials used in the coating systems

Epoxy with Al₂O₃	Epoxy with SIC	Epoxy with (Al₂O₃+ SIC)	epoxy
Al ₂ O ₃ =0.15 g	SIC=0.15 g	Al ₂ O ₃ =0.075g SIC=0.075 g	Epoxy=14.84 g
Epoxy=14.84 g	Epoxy=14.84 g	Epoxy=14.92 g	
hardener =4.95g	hardener =4.95g	hardener =4.97g	hardener =4.97g

The coating process for the metal was conducted by the dip-coating method. After preparing the coating mixture, the samples were immersed in the coating solution for fifteen seconds. This process was repeated five times to ensure optimal adhesion of the coating to the surface. Subsequently, the samples were left at room temperature to air dry, with each repetition enhancing the coating's bond to the metal surface. Allowing the samples to air dry at room temperature ensures a gradual and thorough curing of the coating, contributing to its durability and overall effectiveness in protecting the metal.

4.6. MICROSTRUCTURE CHARACTERIZATION

4.6.1. Optical Microstructure

Fig 4.4 depicts the device of the Optical Microstructure test conducted using a specific type (ZEISS) of the device in the Materials and Metallurgy laboratory. A detector of type "nitric" was utilized to identify the components of the materials present in the sample. Optical Microstructure is a valuable tool for analyzing and studying materials at a microscopic level, enabling the observation of small details and fine structures. This type of analysis helps evaluate the physical and chemical properties of materials and is widely used in scientific research and industrial applications.



Figure 4 4. Optical Microstructure device.

4.6.2. Scanning Electron Microscopy (SEM) and Energy Dispersive Analysis (EDX)

The SEM and EDX tests were conducted using a Carl Zeiss Plus Gemini apparatus at the Institute of Iron and Steel at Karabuk University. Figure 4.5 illustrates the scanning electron microscope (SEM) image used in the experiment.



Figure 4 5. SEM device

The gold coating device depicted in Figure 4.6 was employed to coat the samples after testing. This procedure aids in enhancing the samples' electron tolerance and increases contrast during analyses conducted by SEM and EDX.



Figure 4 6. Gold coating device

4.7. X-RAY DIFFRACTION (XRD)

XRD test was conducted using a device of type (Rigaku Ultima IV -X-Ray) as depicted in Figure 4.7 X-ray Diffraction (XRD) is a powerful technique for analyzing the crystalline structure of materials. The device used in this test provides signals representing the interaction between incident X-rays and crystals in the sample, enabling examination of the diffraction patterns produced by the crystals.



Figure 4 7. XRD device.

XRD technology allows for determining the arrangement of atoms in the crystal lattice of materials, thus providing deeper insights into their structural properties. This analysis can be utilized to identify the crystalline nature of materials and determine the crystal structures present in the sample.

4.8. FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

The FTIR test was conducted using a device of type (BRUKER ALPHA) available at the Institute of Iron and Steel at Karabuk University. Figure 4.8 illustrates the device used in this test.

FTIR (Fourier Transform Infrared Spectroscopy) measures the absorption of materials when exposed to infrared radiation. The FTIR device used in this test is instrumental in analyzing the composition and concentration of chemical compounds in materials. This type of analysis is employed to detect chemical bonds and functional groups in materials, providing a deeper understanding of their composition and structure.



Figure 4 8. FTIR device

4.9. POTENTIODYNAMIC POLARIZATION TEST (TAFEL EXTRAPOLATION)

The prepared specimens underwent a wrapping process using copper wire and were subsequently coated with epoxy resin. Following this, adhesive tape featuring a circular empty area of 0.25 cm² at its midpoint was precisely centered and affixed to the sample surface. This meticulous application ensured that corrosion tests for all specimens were conducted within a standardized area. Moreover, this method served to mitigate any potential detrimental effects arising from epoxy joints.

Potentiodynamic polarization tests were executed at a temperature of 25 °C in both 3.5% NaCl and simulated body fluid (SBF) environments. The tests were conducted using a computer-controlled DC105 corrosion analysis system, employing a Gamry model PC4/300 mA potentiostat/galvanostat. The electrochemical cell setup comprised a graphite rod as the counter electrode, a saturated calomel electrode (SCE) as the reference electrode, and a classical three-electrode cell with the sample surface serving as the working electrode Fig 4.9.

Polarization curves were generated by scanning within the range of 0.25 V (vs. open circuit potential, E_{oc}) to +0.25 V (vs. E_{oc}) at a scanning rate of 1 mV.s^{-1} . This comprehensive testing methodology provided a thorough evaluation of the corrosion behavior of the specimens under controlled conditions.

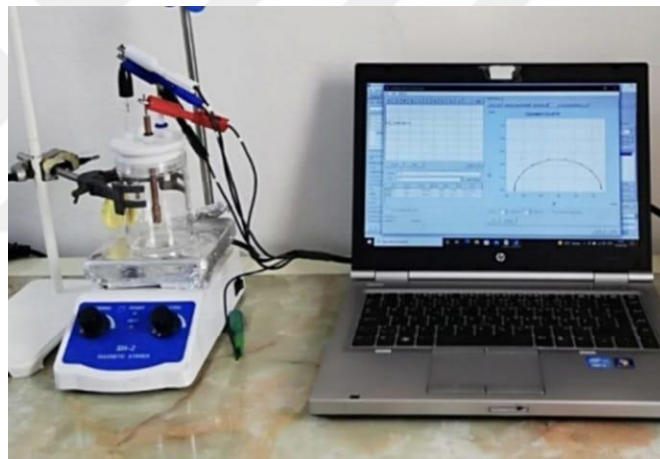


Figure 4.9. Potentiodynamic device

4.10. MECHANICAL PROPERTIES

4.10.1. Hardness Testing

Hardness testing was conducted using a Vickers hardness testing machine, as depicted in Figure 4.10, at the Research Institute of Karabuk University. Hardness testing is a crucial procedure in materials engineering, measuring a material's resistance to deformation under external forces.



Figure 4 10. Hardness testing device.

The Vickers technique for hardness measurement relies on the penetration of a diamond or sapphire indenter into the surface of the test material, followed by measuring the resulting indentation. This test can be valuable in evaluating material resistance and determining the effect of processes like hardening or heat treatment on its hardness.

4.10.2. Scratch Testing

Scratch testing was conducted in the laboratories of Ortadoğu University, utilizing a device of type (Nano-Mechanical Test) as depicted in Figure 4.11 Scratch testing is a significant assessment of material hardness, utilized to determine the material's resistance to scratching.



Figure 4 11. Scratch testing device.

The test typically involves using a material with a known hardness, such as diamond or tungsten carbide, to induce scratches on the surface of the tested material. The scratch is then measured based on the damage caused by the material used for scratching.



PART 5

RESULTS AND DISCUSSION

5.1. INTRODUCTION:

In this part, the characterization of low-carbon steel alloy 1008 coated with epoxy and reinforced with Al₂O₃ and SIC will be discussed. Different concentrations of alloy elements will be observed using optical microscopy and analyzed through SEM/EDS. The electrochemical behavior was evaluated, along with hardness measurements for the coated samples. XRD and FTIR tests were conducted, and scratch tests on the coated samples were performed to demonstrate the suitability of this alloy for various applications.

5.2. MICROSTRUCTURE CHARACTERIZATION

5.2.1. Optical Microstructure (OM) Analysis

The investigation of Alloy 1008 involved a thorough examination utilizing an optical microscope to delve into its microstructural features. The shapes and characteristics of the alloy were meticulously observed and documented to gain insights into its internal composition and morphology.

Fig. 5.1 meticulously details the diverse Figures and configurations that were discerned when the alloy was subjected to optical microscope analysis. This figure serves as a comprehensive reference, providing a visual representation of the alloy's microstructure.

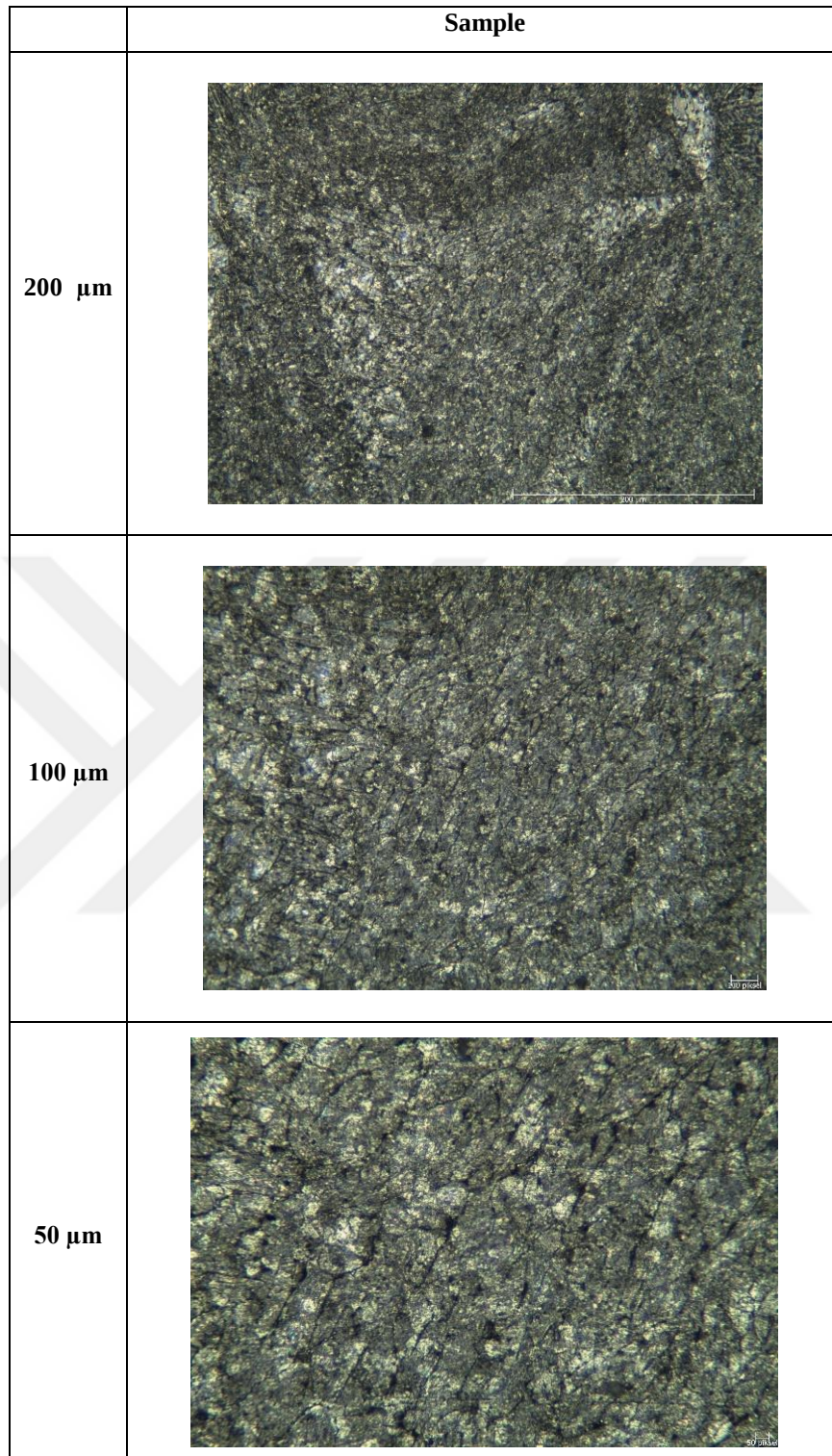


Figure 5.1 ASTM 1008 steel images by optical microscopy

The results discussed for the microscopic analysis of alloy 1008 indicate a specific metal structure. The findings reveal that the structure primarily comprises pearlite

and ferrite grains, predominantly located along the boundaries of deformed prior austenite grains.

In Fig. 5.1 pearlite and ferrite grains are observed as the main constituents of the structure, particularly along the boundaries of the deformed prior austenite grains. The presence of bainite in the space between the ferrite and pearlite structure suggests a diversity in the composition, indicating the presence of bainite grains.

From these results, it can be concluded that the composition of alloy 1008 demonstrates a complex interweaving of pearlite and ferrite grains, with the presence of bainite in the voids between them. The existence of ferrite on the boundaries of prior austenite grains and the presence of bainite with sharp ferrite indicate diversity in the composition, forming various substructures within the alloy [99].

5.2.2. Scanning Electron Microscopy (SEM) and Energy Dispersive Analysis (EDX)

In Figure 5.2 SEM images of the 1008 alloy coated with epoxy reinforced with different amounts of Al₂O₃ and SiC powders are showcased, and Figure 5.3 depicts the EDX analysis of samples. This combined SEM and EDX analysis offers a comprehensive understanding of the microstructure and elemental composition of the coated 1008 alloy.

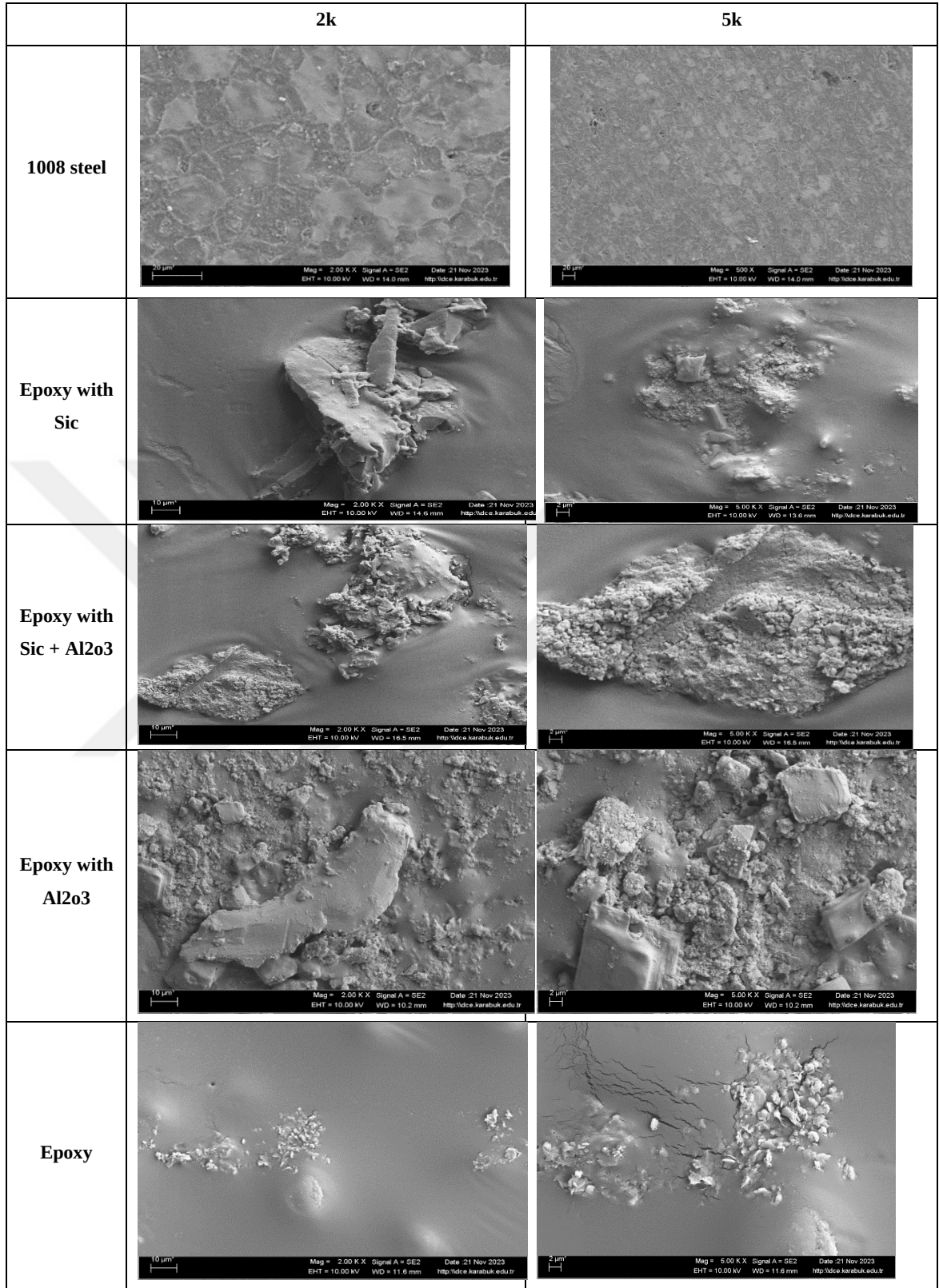
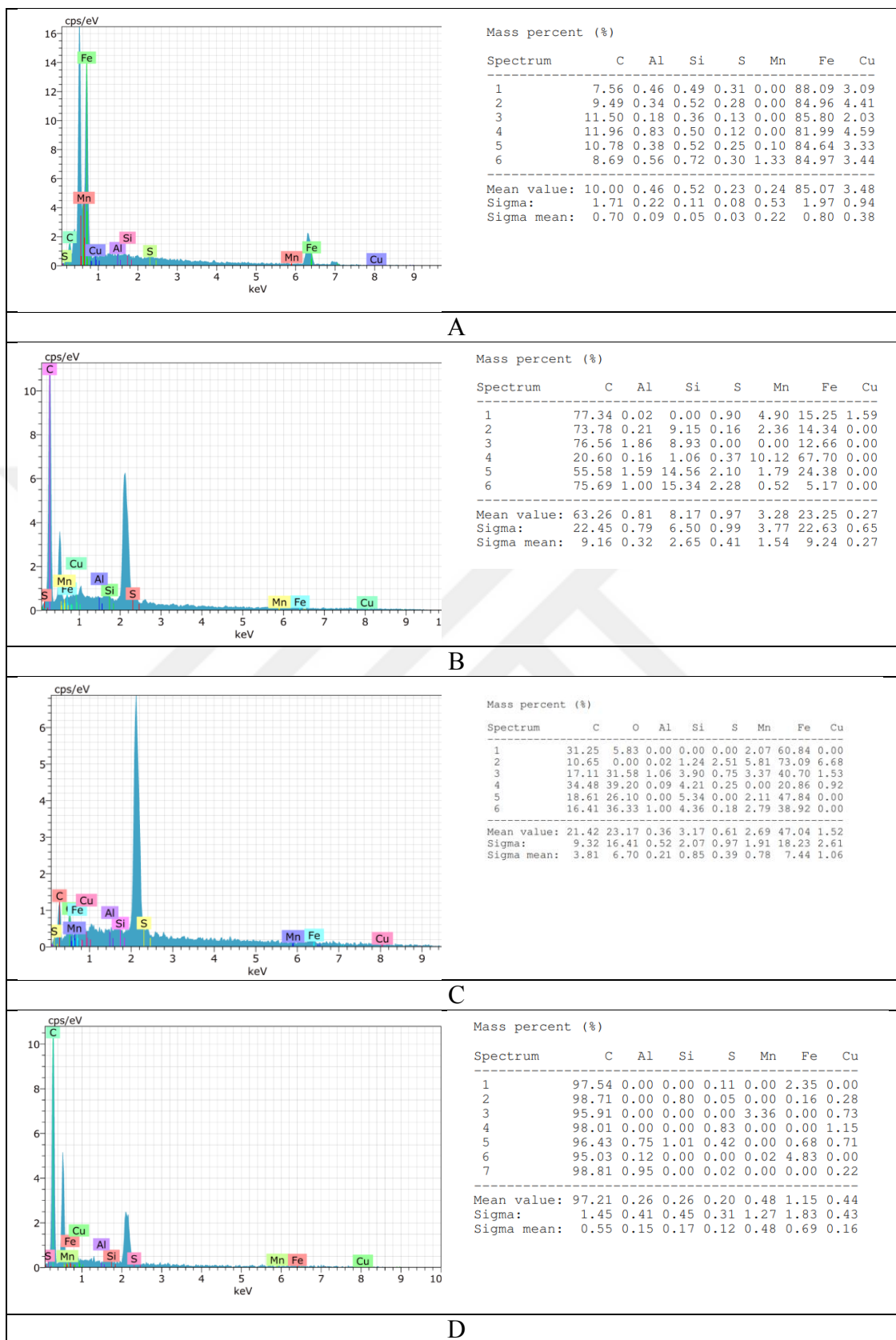


Figure 5 2. SEM images of samples



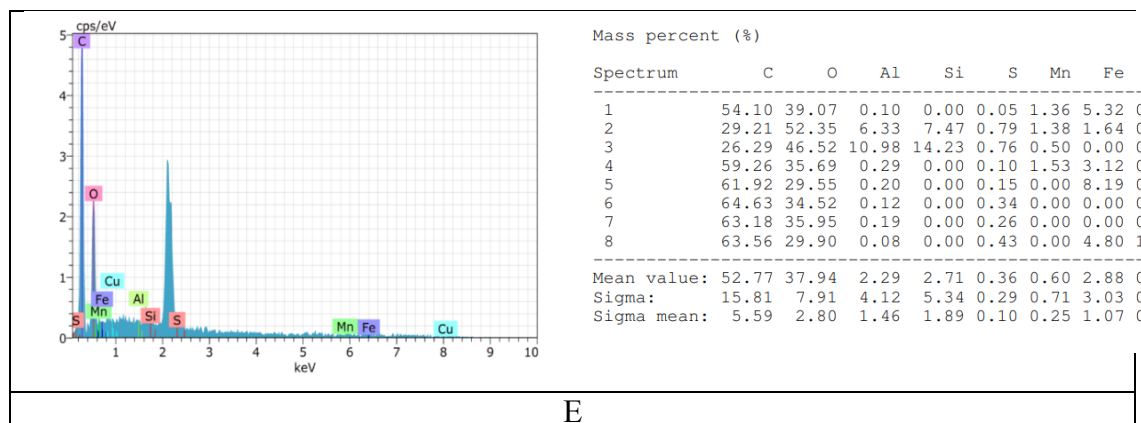
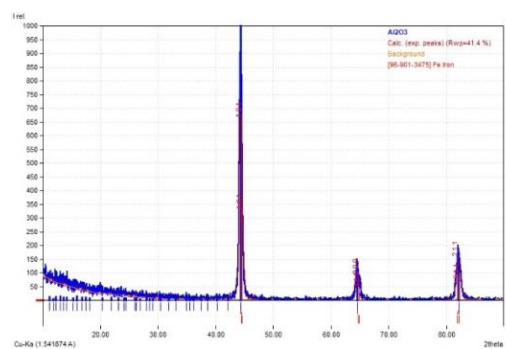


Figure 5 3. EDX analysis (a) ASTM 1008 steel (b) epoxy (c) epoxy with Al₂O₃ (d) epoxy with SiC (e) epoxy with (SiC + Al₂O₃).

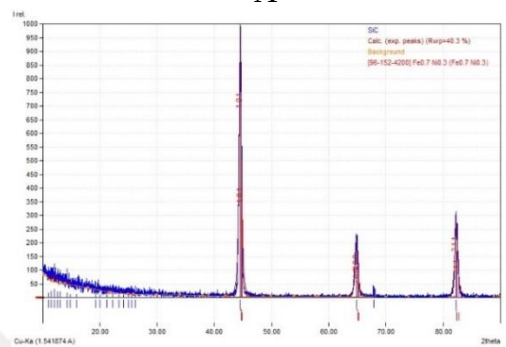
SEM testing was conducted on samples uncoated and coated with epoxy, epoxy with SiC, epoxy with Al₂O₃, and epoxy with SiC+Al₂O₃ for alloy 1008. The resulting images showed clear variations in surface structures, and the images exhibited a homogeneous distribution of Al₂O₃ and SiC particles in the epoxy matrix, This test indicates the influence of additives on the surface structure and the potential enhancement of mechanical properties for the studied alloy, Figure 5.3 shows the proportions of the elements through analysis using the EDX device [84,85].and it gives an evidence to the existence of all content elements in the composite coating.

5.3. X-RAY DIFFRACTION (XRD)

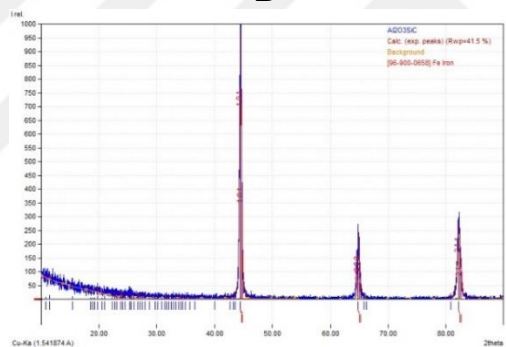
The Match software was utilized to analyze the X-ray diffraction (XRD) results for samples of epoxy-coated 1008 alloy, epoxy with Al₂O₃, epoxy with SiC, and epoxy with Al₂O₃+SiC. Figure 5.4 illustrates detailed analysis models of the crystalline phases and resulting peaks from these interactions. Changes in peak positions and crystalline phase formation allow for inferences about chemical reactions and structural transformations. This analysis enhances our understanding of the crystalline and chemical properties of the materials, contributing to the overall improvement of the coated 1008 alloy performance.



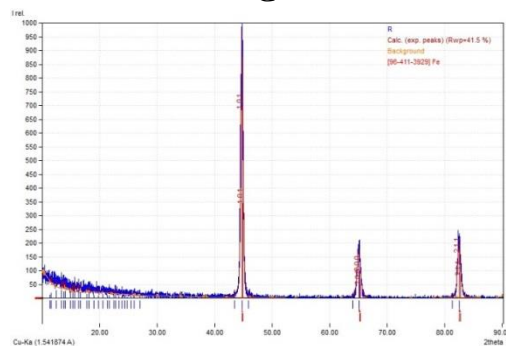
A



B



C



D

Figure 5 4. XRD analysis (a) epoxy with Al₂O₃ (b) epoxy with SIC (c) epoxy with (Al₂O₃+SIC) (d) Epoxy.

5.4. FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

The OMNIC Software Suite was utilized to analyze the FTIR test results for coated alloy samples labeled as 1008. The detailed Figures (from Figure 5.5 to Figure 5.8) reveal the acquired results, exposing intricate details of molecular composition and chemical reactions. These findings serve as a foundation for a deep understanding of the chemical behavior of the alloy and the effects of the coating [83]. AS the table 5.1 shows the FT-IR frequency values of ligands and complexes.

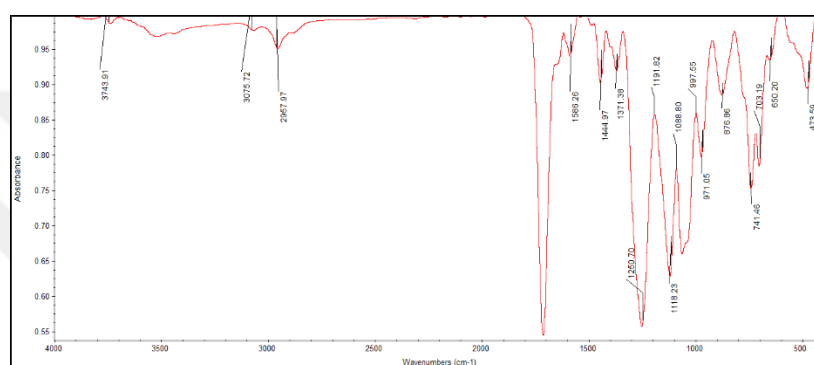


Figure 5 5. FTIR analysis of epoxy with AL₂O₃

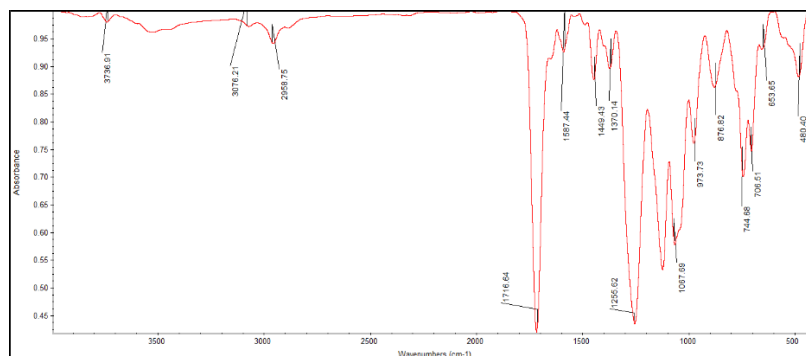


Figure 5 6. FTIR analysis of epoxy with SIC

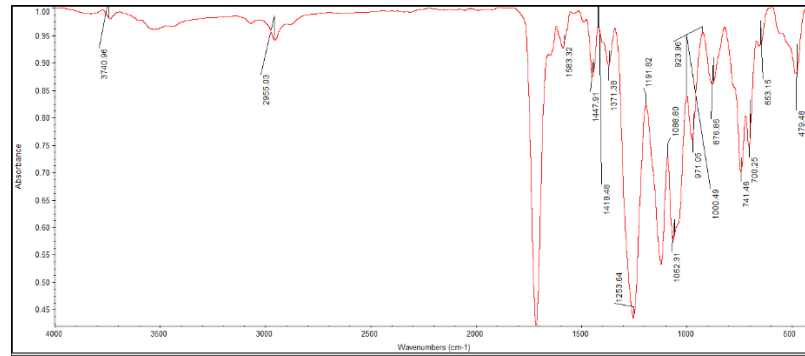


Figure 5 7. FTIR analysis of epoxy with (AL203+SIC)

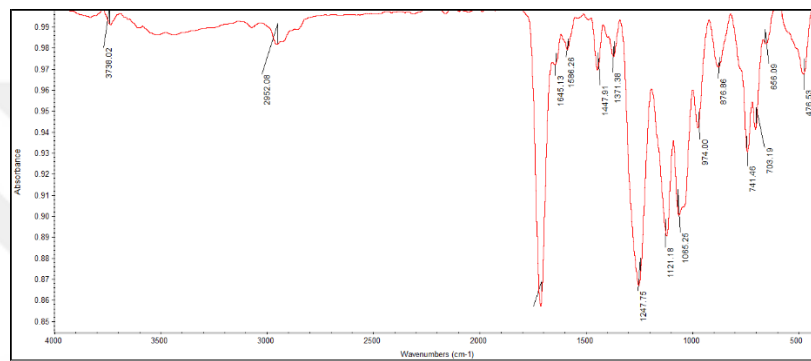


Figure 5 8. FTIR analysis of epoxy

Table 5 1. FT-IR frequency values of ligands and complexes.

		C-H	C-C	C-O	Si-C	Al-O	Fe
Epoxy		3740	1583	1371	1191	741	479
with(Al ₂ O ₃ +SiC)		2955	1447	1253	1088	700	
			1418		1063	653	
					923		
					971		
Epoxy	with	3743	1586	1371	1191	7041	473
Al ₂ O ₃		3075	1444		1088	403	
		2957				650	
Epoxy with SiC		3736	1716	1370	1067	703	480
		3076	1687	1255	973	650	
		2958	1449		876		
Epoxy		3780	1645	1371	1065	741	476
		2952	1447	1121	974	703	
					876	650	

5.5. ELECTROCHEMICAL ANALYSIS

5.5.1. Potentiodynamic Polarization (Tafel Extrapolation)

Corrosion experiments were conducted on coated 1008 samples using a 3.5% sodium chloride (NaCl) saline solution. Figure 5.9 illustrates corrosion plots in the saline water, while Table 5.2 provides values for I_{corr} (total current) and E_{corr} (total voltage) for the coated 1008 samples.

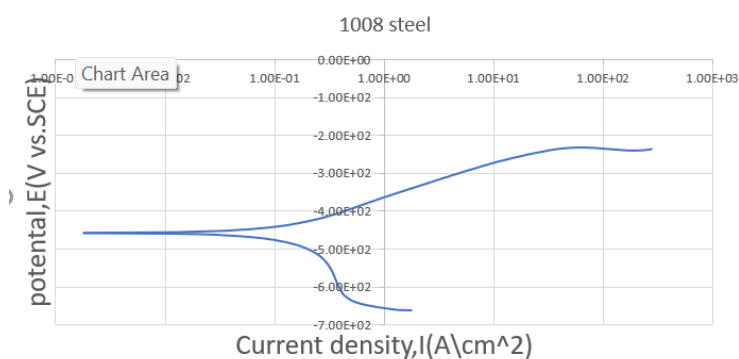


Table 5 2. Value $\dot{I}_{corr}$ - E_{corr} . of samples

Sample	$\dot{I}_{corr}(\mu A/cm^2) \cdot e-6$	$E_{corr} (mV)$
Steel 1008	142	-457
Epoxy	12.90	-496
Epoxy with sic	13,06	-492
Epoxy with Al2O3+sic	11.80	-481
Epoxy with Al2O3	3,29	-125

A corrosion test was performed on alloy 1008 samples coated to evaluate their corrosion resistance and analyze their performance compared to the base sample. The test results showed differences in corrosion behavior between the different samples. An improvement in corrosion resistance was observed for the epoxy-coated sample due to the protective barrier provided by the coating. This shows the effectiveness of the epoxy resin in reducing the harmful effects of environmental conditions on the surface. When examining the sample treated with Sic additive, a positive influence on corrosion resistance was observed. This is attributed to the greater corrosion resistance due to the presence of silicon carbide particles. A super improvement in corrosion resistance was observed in the sample treated with the addition of Al2O3, and the corrosion resistance of alumina suggests that it can withstand aggressive corrosive environments. In the sample treated with Sic+Al2O3, a mutually reinforcing effect of Sic and Al2O3 particles on corrosion resistance was observed. This shows the positive synergy between the properties of the reinforcing materials. In general, the results indicate that the improvement processes, either by coating or adding reinforcing materials, contributed to improving the corrosion resistance of the 1008 alloy [93].

5.6. MECHANICAL PROPERTIES

5.6.1. Vickers Hardness

The Vickers hardness test was employed to measure the hardness of the samples, and the Vickers hardness equation was utilized for hardness calculations.

$$H_v = (F/d^2) * 1.854$$

Table 5.3 presents the hardness test results for the treated samples alongside the base sample.

Table 5 3. Hardness test results for samples

Sample	Hv
Epoxy with Sic	120.0
Epoxy with (Sic + Al ₂ O ₃)	108.28
Epoxy with Al ₂ O ₃	104.35
Epoxy	99.13
Steel 1008	97.6

Hardness tests were carried out on alloy 1008 samples coated with epoxy, epoxy with Sic, epoxy with Al₂O₃, and epoxy with (Sic+Al₂O₃). The test aimed to evaluate the influence of the improvement processes on the hardness of the alloy and the improvement of its mechanical properties. The test results showed a significant improvement in the hardness of the treated samples compared to the base sample. When considering the sample treated with epoxy and Sic, the hardness was higher due to the uniform distribution of silicon carbide particles on the surface. This indicates an improvement in mechanical properties such as surface hardness. For the Al₂O₃ epoxy-coated sample, the presence of uniformly distributed aluminum oxide particles was responsible for the improvement in hardness. For the sample treated with epoxy and SIC+Al₂O₃, the result showed a homogeneous distribution of SIC and Al₂O₃ particles, which enhanced the mutual effect of the reinforcing materials and helped to achieve a balance between surface hardness and corrosion resistance [87,88].

5.6.2. Scratch Test

The Scratch test was conducted to assess the adhesion of the coating and its impact on surface depth. Figure 5.10 clearly illustrates the frictional force and coefficient of friction, providing specialists with insight into the coating's resistance to external

influences and frictional forces. This aspect of the test is crucial in determining the coating's ability to withstand environmental conditions and its anticipated applications.

Similarly, Figure 5.11 presents details related to the depth of penetration and the remaining depth after the test. This figure highlights the coating's capacity to resist penetration and corrosion, allowing analysts to measure the impact of stress or loading on the coating's surface and estimate its efficiency in maintaining its adhesion and original depth.

The Scratch test is considered an effective method for examining the quality and hardness of coatings, guiding the selection for specific purposes, whether industrial or commercial applications. Consequently, this test provides valuable results that enhance our understanding of how coatings interact with environmental factors and their effectiveness in preserving their quality and performance over time [89, 92].

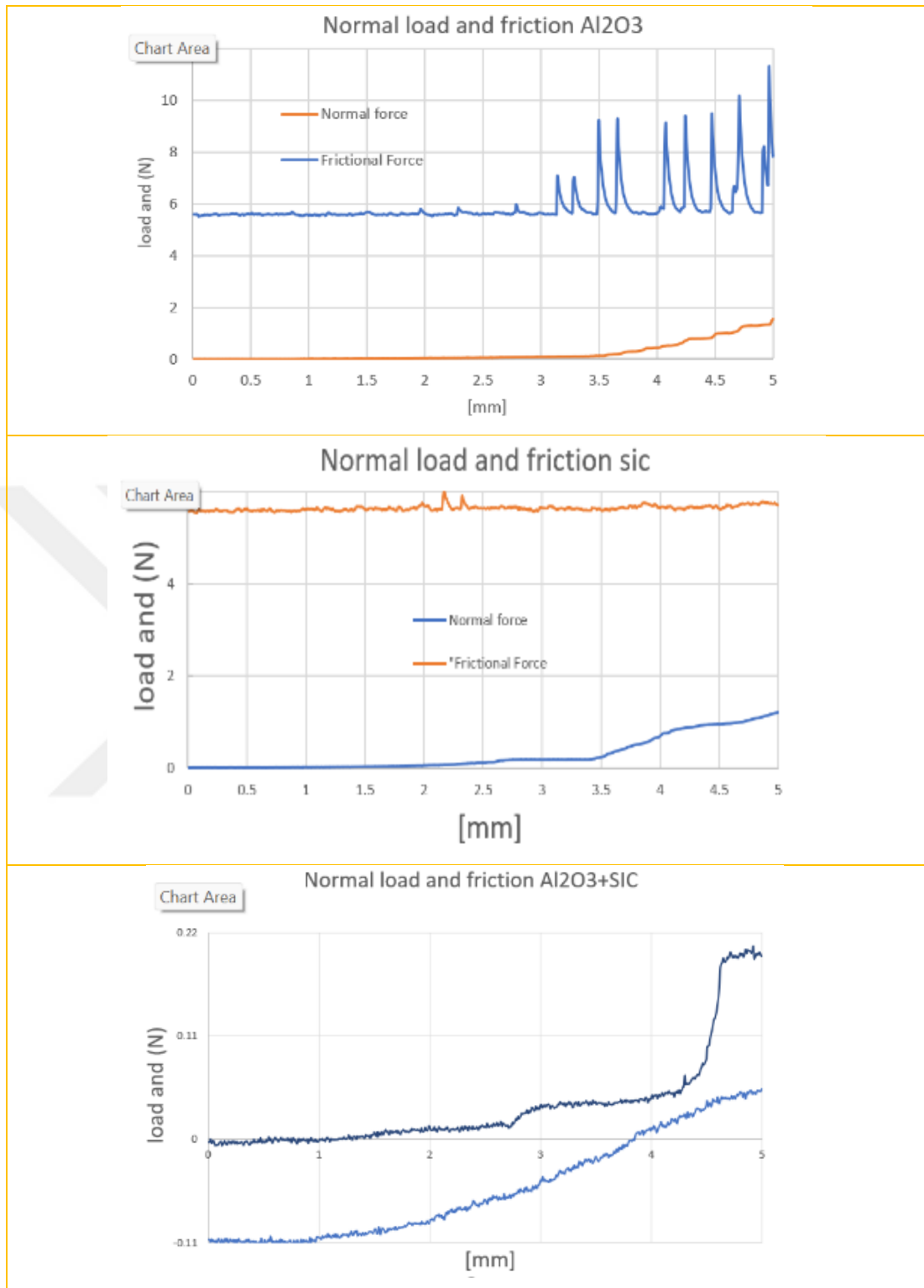


Figure 5 10. Scratch testing of samples

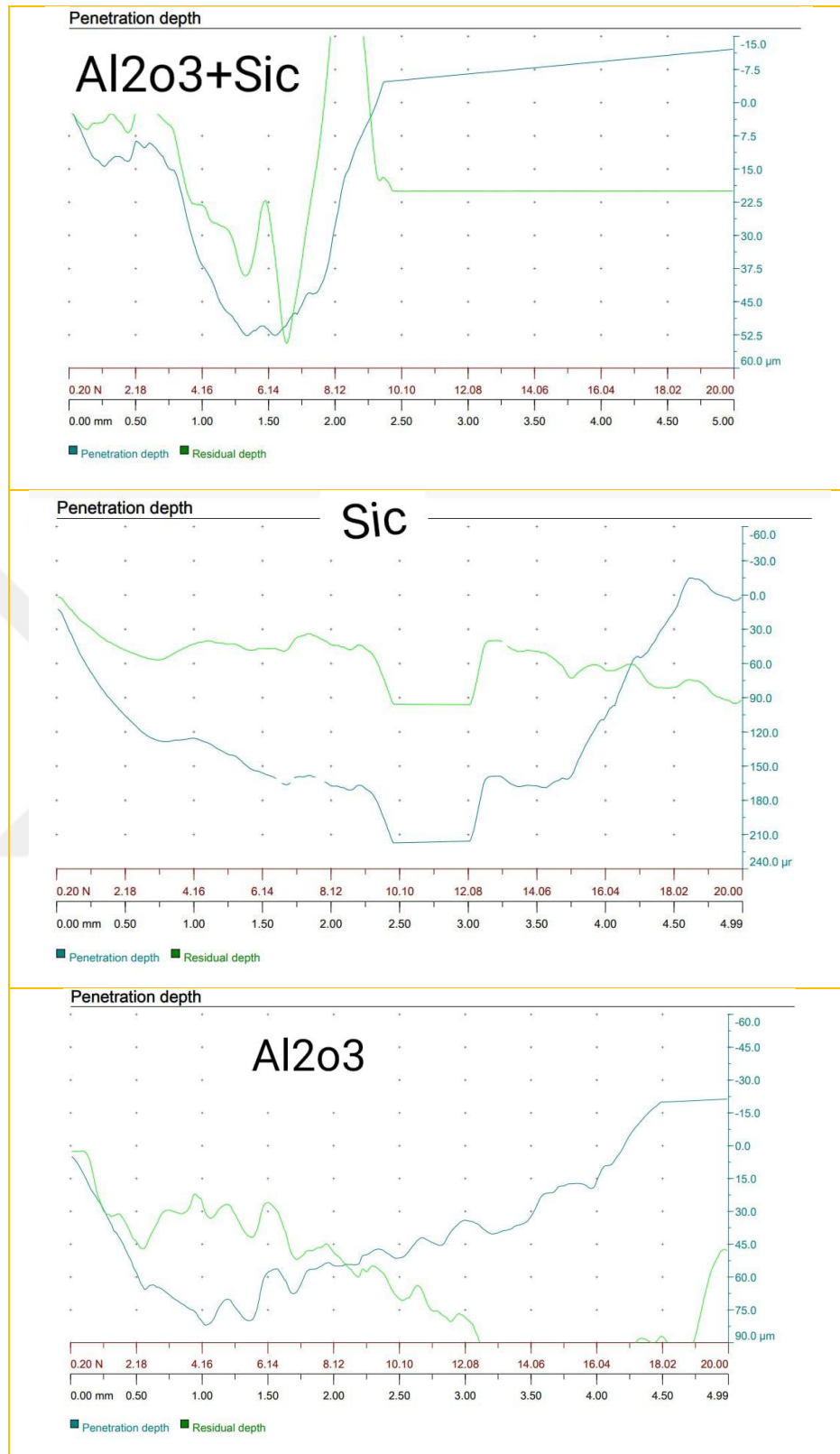


Figure 5 11. Scratch testing penetration of samples

From Figures 5.10 and 5.11, it can be inferred that the low-carbon steel alloy sample coated with SIC material exhibited greater resistance to penetration. This can be attributed to its resistance to abrasion and corrosion, as indicated by the scratch test results, showcasing the coating's adhesion strength and effectiveness.

Figure 5.10 provides a clear insight into the frictional force and coefficient of friction for this coated sample. Understanding these values helps determine the coating's resistance to environmental conditions and frictional forces. Furthermore, Figure 5.11 highlights details regarding the depth of penetration and the remaining depth after the test, reflecting the coating's ability to resist penetration, and corrosion, and maintain the original depth level.

We have been provided the normal force and frictional force at the highest point for the sample coated with epoxy and Al_2O_3 (49.9-1.2), (4.961-11.2)N, sample coated with epoxy and SIC (4.99-1.2), (2.18-2.185)N, and the sample coated with epoxy and (Al_2O_3 +SIC) (4.99-0.05), (4.92-0.2)N consecutively. The observation from Figure 5.11 indicates that the remaining depth was better in the epoxy-sic coated steel alloy, where the remaining depth was 96 μm and the sequential depth was 207 μm .

Conclusively, based on the presented results, the alloy that is coated with epoxy and is reinforced SIC emerges as an effective option when it comes to the coating's resistance to penetration and its ability to preserve its quality and performance amidst environmental and mechanical challenges.

PART 6

GENERAL RESULTS

In this study, low-carbon steel alloy 1008 was coated with epoxy in various configurations, yielding the following results:

- a) Through hardness and scratch tests, it was observed that the sample coated with epoxy reinforced with SIC exhibited significantly higher hardness compared to other samples.
- b) Corrosion testing revealed that the sample coated with epoxy reinforced with Al₂O₃ demonstrated greater corrosion resistance.
- c) FTIR and XRD analyses were conducted to study the distribution of crystalline and molecular elements, as well as the crystalline properties of the materials.
- d) Optical microscopy indicated a complex interweaving of ferrite and pearlite particles in the alloy, with the presence of bainite in the gaps between them.
- e) SEM and EDX analyses were performed to analyze the elemental ratios present in the samples.

These results reflect the effectiveness of improving hardness and corrosion resistance using epoxy coating with strengthening elements. Additionally, they elucidate the structural and mechanical properties of the studied alloys.

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RESUME

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