

SURFACE ANALYSIS OF CHEMICAL CONVERSION AND  
NICKEL CHROME COATINGS ON ASTM-A516 GRADE-70



Author

**Raza Ali**

00000330436

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Supervisor

**Dr. Rashid Naseer**

DEPARTMENT OF MECHANICAL ENGINEERING  
COLLEGE OF ELECTRICAL & MECHANICAL ENGINEERING  
NATIONAL UNIVERSITY OF SCIENCES AND TECHNOLOGY  
ISLAMABAD

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Signature: \_\_\_\_\_  
Name of Supervisor: Dr. Rashid Naseer  
Date: 01-11-2024

Signature (HOD): \_\_\_\_\_  
Name: Dr. Imran Akhter  
Date: 01 Nov 2024

Signature (Dean): \_\_\_\_\_  
Name: Dr. Nasir Rashid  
Dated: 01 Nov 2024

## **DEDICATION**

*To my dear family and my wonderful parents, who made all this possible with their endless love, encouragement, and patience. Their unwavering support has been my guiding light throughout this journey and inspired me to strive for excellence.*

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First and foremost, I am extremely thankful for the special blessing of Allah Almighty upon me throughout my journey without which, I would never be able to achieve and accomplish what I have today. I would like to thank my advisors Dr. Rashid Naseer and Dr. Muhammad Ali Khan, for their immense guidance, help, and consistent support. I would not have been here today without their encouragement throughout all the stages of this research. Moreover, I would like to express my gratitude to Dr. Muhammad Rehan Khan, and Dr. Usama Bin Perwez for being on my thesis guidance and evaluation committee. I would also like to thank Dr. Imran Akhtar and Dr. Nasir Rashid for their support which made my thesis defense possible within short timeline. Finally, I would like to thank my parents, family members, family friends, and many other friends who have always been an incessant source of veneration, support, and motivation for me.

## ABSTRACT

In this experimental based research study, Chemical conversion coating, Chromium (Cr) and Nickel (Ni) electroplating techniques are being used to deposit anti-corrosion protective layers on ASTM-A516 Grade 17 Steel, which is used in construction and manufacturing industries most commonly in fabrication of large storage tanks. During the process of chemical conversion coating which is also known as passivation, Sodium hydroxide (NaOH) and Sodium nitrate (NaNO<sub>3</sub>) solution is used as source material to prepare hot alkaline oxide bath solution. After this process outermost surface of sample is converted into magnetite (Fe<sub>3</sub>O<sub>4</sub>). This magnetite layer prevents substrate surface from any corrosive attack. ASTM-A516 Grade 17 Steel is electroplated with Ni and Cr in which substrate is taken as cathode and metal to be coated are taken as anode. Chromium tri-oxide and Nickel sulfate are used to prepare electrolytic bath solution for Cr and Ni electroplating respectively. After depositing the coatings, Salt spray test is performed on coated and uncoated samples for weight loss analysis, to evaluate corrosion trends of coated and uncoated samples. The salt spray test serves as a benchmark for comparative analysis of various samples. In this test, samples are placed in a controlled environment with specific temperature and humidity levels and exposed to an atomized 5% NaCl solution. This atmosphere is highly corrosive, leading to surface damage and accelerating the corrosion process. The results exhibit minimum weight loss in dual coated (Ni-Cr) samples which indicates high resistance against corrosion. Coated samples micro structure and surface morphology are studied to further validate result and deduce conclusion. This is done through X-ray diffraction (XRD), Scanning electron microscope (SEM) along with energy dispersive spectrometer (EDS) respectively.

### Keywords

Corrosion, Chemical conversion coating, Electroplating, Passivation, Salt spray test, XRD, SEM, EDS, ASTM-A516 Grade 17 Steel

# TABLE OF CONTENTS

<b>ACKNOWLEDGMENT .....</b>	<b>I</b>
<b>ABSTRACT.....</b>	<b>II</b>
<b>CHAPTER 1: INTRODUCTION.....</b>	<b>1</b>
1.1 Background.....	1
1.2 Motivation .....	2
1.3 Impact of Corrosion on Global GDP.....	3
1.4 Aims of the Research.....	4
1.5 Thesis Outline.....	5
<b>CHAPTER 2: LITERATURE REVIEW .....</b>	<b>6</b>
2.1 Corrosion .....	6
2.1.1 Corrosion factors.....	6
2.1.2 Corrosion Chemical Reactions: .....	7
2.1.3 Alakaline Anoxic Corrosion: .....	7
2.2 Corrosion Control:.....	8
2.2.1 Surface Treatment.....	9
2.2.2 Protective Nano-coatings Types as per Deposition Techniques.....	9
2.3 Electrodeposition / Electroplating .....	11
2.3.1 Passivation Coating.....	12
2.4 Analysis of Surface Treatment for ASTM A516 Grade 70.....	13
2.5 Tri-sandwich Cu-Ni-Cr coating.....	13
2.6 Mechanical and corrosion behavior of Cu, Cr, Ni and Zn electroplating .....	14
2.7 Corrosion protection by Passive Oxide film.....	16
2.8 Impact of Benzotriazole on Phosphating.....	16
2.9 Organic Compounds addition into Phosphating .....	17
2.10 Subsequent Application of Anodizing and Annealing .....	17
2.11 Chromium-Carbon Coating .....	18
2.12 Effect of Microstructure and Composition variation.....	19
2.13 Impact of Surface finishing on Carbon Steel.....	20
<b>CHAPTER 3: EXPERIMENTAL WORK.....</b>	<b>22</b>
3.1 Design of experiment: .....	22

3.2	Material Technical Specifications .....	23
3.3	Material Arrangement: .....	24
3.4	Samples Cutting (Process-1) .....	24
3.5	Passivation (Process-2).....	25
3.6	Surface Grinding and Polishing (Process-3) .....	26
3.7	Nickel Electroplating (Process-4).....	26
3.7.1	Acid Pickling .....	26
3.7.2	Fresh Water Rinsing .....	26
3.7.3	Neutralizing.....	26
3.7.4	Fresh Water Rinsing .....	27
3.8	Nickel electroplating.....	27
3.9	Nickel-Chrome Dual Electroplating:.....	27
<b>CHAPTER 4: TESTING, RESULTS AND DISCUSSIONS.....</b>		<b>29</b>
4.1	Initiation.....	29
4.2	Salt Spray Test.....	29
4.3	Weight Loss Analysis .....	31
4.4	Surface Roughness Analysis .....	32
4.5	X-Ray Diffractometry (XRD) Analysis .....	34
4.6	Scanning Electron Microscopy and EDS Analysis: .....	36
4.6.1	Scanning electron microscopy (SEM) Results .....	37
4.6.2	Energy Dispersive Spectrometry Results.....	39
<b>CHAPTER 5: CONCLUSION AND FUTURE WORK.....</b>		<b>41</b>
5.1	Conclusion .....	41
5.2	Limitations.....	41
5.3	Future work.....	41

## LIST OF FIGURES

FIGURE -1: EXAMPLE OF CORROSION EFFECTS ON BOLTS, VALVES AND PIPES [ ].....	1
FIGURE -2: CORROSION COST OF FIVE DIFFERENT COUNTRIES PER ECONOMIC SECTOR [3].....	4
FIGURE -3: THIN FILM DEPOSITION METHODS [8] .....	11
FIGURE -4: BASE MATERIAL CUTTING SPECIMENS (SAMPLE TYPE-1) .....	25
FIGURE -5: PASSIVATED SPECIMENS (SAMPLE TYPE-2) .....	25
FIGURE -6: POLISHED SURFACE SPECIMENS (SAMPLE TYPE-3).....	26
FIGURE-7: NICKEL ELECTROPLATED SPECIMENS (SAMPLE TYPE-4).....	27
FIGURE -8: DUAL COATED (NI-CR) SPECIMENS (SAMPLE TYPE-5).....	27
FIGURE- 9: PROCESS QUALITY CONTROL TABLE FOR ELECTROPLATING PROCESS .....	28
FIGURE -10: SALT SPRAY TESTING CHAMBER .....	30
FIGURE -11: MANUFACTURING OPERATION STANDARD FOR SALT SPRAY TESTING MACHINE..	30
FIGURE -12: SARTORIUS MODEL TE313S-DS ELECTRONIC ANALYTICAL AND PRECISION METAL SCALE.....	31
FIGURE -13: WEIGHT LOSS ANALYSIS FOR COATED AND UNCOATED ASTM-A516 GRADE 70 STEEL .....	32
FIGURE -14: IMAGE OBTAINED AT 280X RESOLUTION OF PASSIVATED SPECIMEN .....	33
FIGURE -15: IMAGE OBTAINED AT 280X RESOLUTION OF NI- COATED SPECIMEN.....	34
FIGURE -16: IMAGE OBTAINED AT 280X OF NI-CR COATED SPECIMEN .....	34
FIGURE -17: XRD ANALYSIS OF PASSIVATED SPECIMEN .....	35
FIGURE -18: XRD ANALYSIS OF NI- COATED SPECIMEN.....	36
FIGURE -19: XRD ANALYSIS OF NI-CR COATED SPECIMEN .....	36
FIGURE -20: SEM IMAGE AT X2430 RESOLUTION OF PASSIVATED SPECIMEN.....	37
FIGURE -21: SEM IMAGE AT X2430 RESOLUTION OF NI-CR COATED SPECIMEN .....	38
FIGURE -22: SEM IMAGE AT X2430 RESOLUTION OF NICKEL COATED SPECIMEN.....	38
FIGURE -23: EDS ANALYSIS OF PASSIVATED SPECIMEN .....	39
FIGURE -24: EDS ANALYSIS OF NI-ELECTROPLATED SPECIMEN.....	39
FIGURE -25: EDS ANALYSIS OF NI-CR ELECTROPLATED SPECIMEN .....	40



## LIST OF TABLES

TABLE -1: ELECTROPLATING CHARACTERISTICS AND APPLICATIONS [9] .....	12
TABLE -2: PROCESS FLOW FOR SAMPLES PREPARATION .....	22
TABLE -3: CHEMICAL COMPOSITION (WT %) OF ASTM A516 GRADE 70 .....	23
TABLE -4: MECHANICAL PROPERTIES OF ASTM A516 GRADE 70 .....	23
TABLE -5: CHEMICAL COMPOSITION (WT %) OF TEST SPECIMEN.....	24
TABLE- 6: MECHANICAL PROPERTIES OF TEST SPECIMEN .....	24
TABLE -7: SALT SPRAY TEST RESULTS OF SAMPLES.....	31
TABLE -8: SURFACE ROUGHNESS ANALYSIS OF COATED SAMPLES .....	33

# CHAPTER 1: INTRODUCTION

## 1.1 Background

Corrosion is a chemical process that leads to the degradation of materials through interactions with their environment. This involves a combination of oxidation and reduction processes, known collectively as redox reactions. Given its degrading nature, various physical and chemical methods are used to enhance the corrosion resistance of materials. As a degrading phenomenon, various physical and chemical methods are employed to make materials resistant to corrosion. In some cases, the outer surface is treated to resist corrosion, while in others, a protective layer is applied to the original surface. The significance of safeguarding valuable and essential equipment from corrosion cannot be overstated [1].



**Figure -1:** Example of corrosion effects on bolts, valves and pipes [2]

## 1.2 Motivation

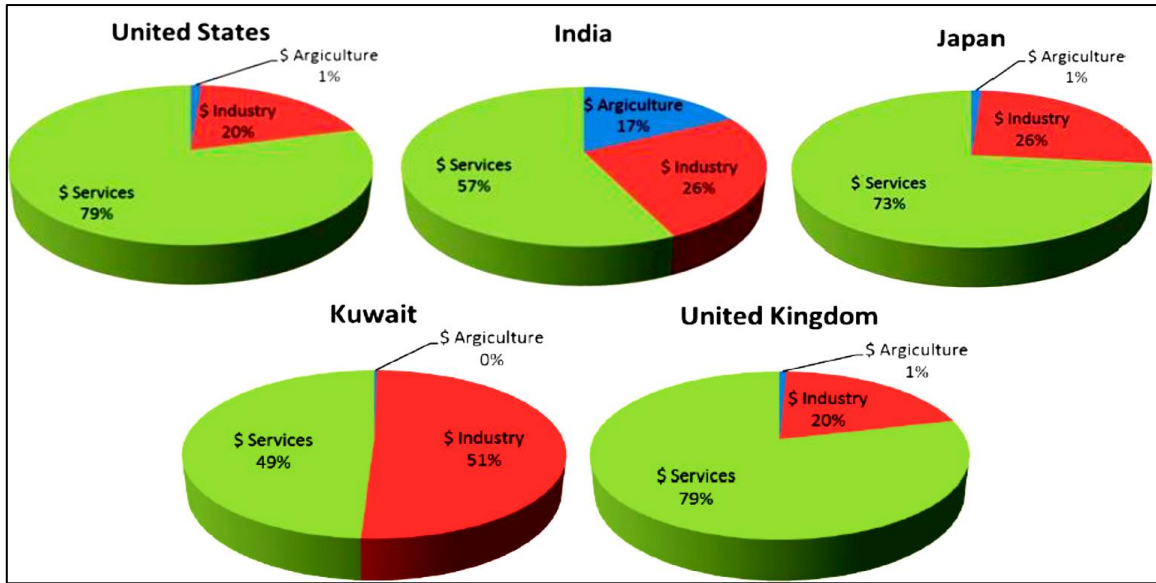
Corrosion has been a significant field of inquiry for over 150 years, as it is recognized as a critical issue that leads to degradation, failure, and severe accidents in various industrial processes and domestic systems [2]. It refers to the deterioration of metals due to reactions with corrosive elements in their surroundings, including chlorine, fluorine, carbon dioxide, and oxygen. The financial impacts of corrosion are considerable, encompassing repair and maintenance expenses, material loss, equipment damage, diminished efficiency, and a reduced useful life. Furthermore, corrosion poses societal challenges, such as safety risks (including fires, explosions, and the release of toxic substances), health threats (like personal injuries and pollution from toxic contamination), and the depletion of resources. Corrosion is fundamentally an undesirable process that leads to the deterioration of materials, particularly metals, through chemical reactions with their environment. This phenomenon can significantly compromise the integrity and functionality of equipment, making it a critical concern across various industries. In structural and mechanical applications, the implications of corrosion are particularly pronounced. These systems often operate under demanding conditions, such as exposure to moisture, extreme temperatures, and abrasive substances. When corrosion occurs in these contexts, it can lead to serious structural failures, reduced load-bearing capacity, and a shortened lifespan of the equipment. This is especially problematic in industries such as construction, transportation, and manufacturing, where safety and reliability are paramount. The adverse effects of corrosion extend beyond mere aesthetic issues. It can lead to a loss of strength and durability, compromising the overall serviceability of materials. For instance, in bridges and buildings, even minor corrosion can escalate into significant structural damage, resulting in costly repairs and potential safety hazards. Additionally, equipment failure due to corrosion can lead to production downtimes and increased maintenance costs, which are critical considerations for businesses. Given these challenges, the preservation of metals from corrosion is of utmost importance. This necessity drives the research focus on developing effective corrosion prevention methods, such as protective coatings, inhibitors, and material selection strategies. By understanding the mechanisms of corrosion and exploring innovative solutions, this research aims to enhance the longevity and performance of metallic components in various applications, ultimately contributing to improved safety and efficiency in industrial operations.

### 1.3 Impact of Corrosion on Global GDP

A study conducted by the National Association of Corrosion Engineers (NACE) estimated that the global cost of corrosion amounts to approximately \$255 billion USD, which is equivalent to 3.4% of the total global gross domestic product (GDP) [3]. In the United States alone, the direct and indirect annual costs associated with corrosion are estimated to reach \$552 billion, representing 6% of the U.S. GDP. These costs encompass a wide range of direct effects, including expenses for controlling corrosion in various infrastructures and appliances. Key sectors impacted by corrosion include:

- **Transportation Infrastructure:** This includes highway bridges, roads, and rail systems that require regular maintenance to prevent deterioration and ensure safety.
- **Automotive and Aerospace Industries:** Corrosion can lead to significant maintenance costs and potential failures in vehicles and aircraft, making it crucial to implement effective protective measures.
- **Industrial Plants:** Facilities involved in energy production and distribution, petrochemical processing, desalination, and pharmaceuticals face substantial costs related to corrosion management.

In addition to direct costs, indirect costs associated with corrosion can be equally substantial. These include productivity losses due to operational delays, equipment failures, and outages, which can disrupt workflows and impact overall economic efficiency. Additionally, there are costs related to taxes and overhead expenses that are tied to corrosion management efforts. An analysis of corrosion costs across five different regions reveals interesting patterns. As shown in (Figure -2), the United States, United Kingdom, and Japan exhibit similar corrosion costs, which can be attributed to their advanced industrial and service-based economies. In contrast, countries like India and Kuwait show significant contributions to corrosion costs from their agricultural and oil industries, respectively. This difference underscores the varying economic structures and dependencies of different regions on specific sectors. Given the substantial financial implications of corrosion, effective strategies for prevention and management are essential. Implementing comprehensive corrosion prevention measures, including regular monitoring and maintenance, can lead to significant cost savings. Estimates suggest that proper practices can save between 15-35% of the losses caused by corrosion. This emphasizes the importance of adhering to safety standards and best practices across various industries to mitigate the effects of corrosion and enhance the longevity of infrastructure and equipment.



**Figure -2:** Corrosion Cost of Five different Countries per Economic Sector [3]

#### 1.4 Aims of the Research

Research focused on electroplating typically aims to enhance corrosion resistance by investigating how various techniques and materials can improve coated surfaces. Another key goal is to improve adhesion, studying methods to strengthen the bond between electroplated coatings and substrates to prevent issues like peeling or flaking during use. Additionally, optimizing coating properties is a significant aim, exploring the effects of different electroplating parameters—such as current density, temperature, and solution composition—on physical and mechanical characteristics like hardness, wear resistance, and thickness. Another important area of research is the development of eco-friendly electroplating processes that minimize toxic waste and utilize less harmful chemicals. Investigating the microstructural characteristics of electroplated layers is also crucial to understanding how these features influence performance in real-world applications. Cost-effectiveness is another consideration, as researchers assess the economic feasibility of various electroplating methods to identify the most cost-efficient approaches for industrial use. The functionality of different electroplated materials for specific applications—such as decorative finishes, electrical conductivity, or thermal stability—is another focus area. Furthermore, analyzing the long-term durability and performance of electroplated coatings under various environmental conditions and mechanical stresses is essential for practical applications. Characterizing surface morphology using techniques like scanning electron microscopy (SEM) or atomic force microscopy (AFM) allows for the correlation of surface features with performance characteristics. Finally,

conducting comparative studies between different electroplating methods or materials helps identify best practices for specific applications. These aims collectively guide research efforts and contribute to advancements in electroplating technology.

## **1.5 Thesis Outline**

This research examined various anti-corrosion techniques implemented globally and aimed to develop a viable and effective method for preventing corrosion. The process began with a thorough literature review, followed by an experimental and testing phase. After conducting the experiments, the results were analyzed based on the collected data, leading to the formulation of conclusions and recommendations.

## **CHAPTER 2: LITERATURE REVIEW**

### **2.1 Corrosion**

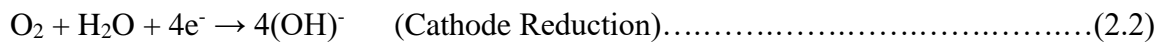
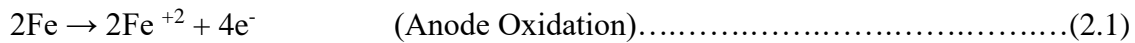
Metals elements exhibiting key characteristics such as excellent thermal and electrical conductivity [1]. These materials are typically opaque and shiny, and they possess unique properties like ductility, malleability, and fusibility. These attributes make metals highly versatile and valuable in a wide range of applications, from construction to electronics. However, metals are not stable in their elemental form; they readily undergo redox reactions with their environment, which can lead to changes in their structure and composition. This process, known as corrosion, involves the gradual degradation of the metal as it reacts with substances such as oxygen, moisture, and acids in its surroundings. Corrosion is an unwanted and naturally occurring phenomenon that compromises the integrity of metals, making them less effective and potentially unsafe for their intended applications. The impact of corrosion is significant, leading to enormous financial losses across various industries. It not only wastes valuable resources but also affects the performance and durability of both finished and unfinished materials. For instance, in the construction industry, corrosion can weaken structural components, leading to costly repairs and increased safety risks. In manufacturing and production, corrosion can degrade machinery and equipment, resulting in downtime and decreased efficiency. Overall, understanding corrosion and its mechanisms is crucial for developing effective prevention and mitigation strategies. By addressing this pervasive issue, industries can protect their investments, enhance the longevity of materials, and ensure the safety and reliability of their products. Research into anti-corrosion techniques and coatings is vital to combat these challenges and minimize the detrimental effects of corrosion on metals.

#### **2.1.1 Corrosion factors**

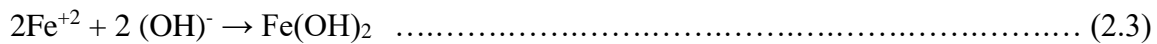
Several factors influence the initiation, propagation, and transformation of corrosion, with some having a greater impact than others [4]. Some of these factors serve as essential components for the corrosion process, while others act as catalysts. The reactivity of a metal, often referred to as its oxidation state, determines the speed of redox reactions. The presence of water is essential for corrosion to occur, and the concentration of electrolytes in the water directly affects the corrosion rate, with higher electrolyte levels leading to increased corrosion. Additionally, the rate of corrosion tends to rise with increasing temperature. Similarly, the amount of dissolved oxygen in the water is also positively correlated with the corrosion rate, further highlighting the complexity of the factors involved in this process.

### 2.1.2 Corrosion Chemical Reactions:

Corrosion fundamentally involves a series of oxidation-reduction reactions, known as redox reactions [5] In this process, the base metal functions as the anode, primarily oxidizing due to the presence of oxygen in the air. This oxidation generates metal ions and releases free electrons, which then reduce oxygen to form hydroxide ions in the presence of water. For corrosion of iron, or rusting, the following reactions take place:



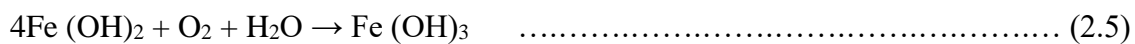
Resulting ions reacts as



Overall reaction can be exposed as,



Further oxidation occurs,



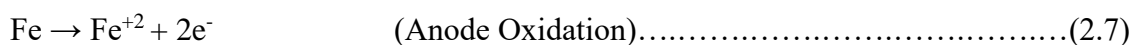
Then, Dehydration process taken place,



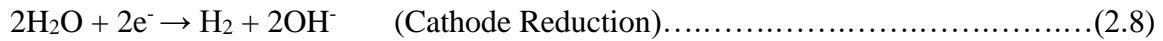
Iron oxide ( $\text{Fe}_2\text{O}_3$ ), referred to as rust, is the ultimate product of iron corrosion. This substance is highly unstable and readily breaks down, resulting in pitting and adversely affecting the mechanical integrity of the material.

### 2.1.3 Alkaline Anoxic Corrosion:

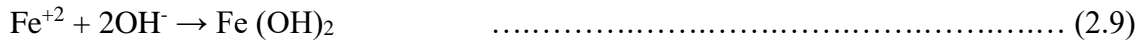
Alkaline anoxic corrosion is utilized to produce a thermodynamically more stable iron oxide,  $\text{Fe}_3\text{O}_4$ , known as magnetite, on the outer surface of corroding iron or its alloys [6]. This process involves an aqueous solution containing an alkaline element along with an oxidizing anoxic source. The purpose of the alkaline element is to promote the reduction of water, releasing hydrogen gas and hydroxide ions. This reaction is crucial for the precipitation of the slightly soluble  $\text{Fe}(\text{OH})_2$ . The following reactions take place during this procedure:





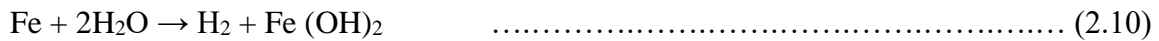


Resulting ions would react,

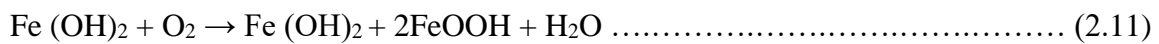


(Yellow brown)

Redox Reaction can be summed up as

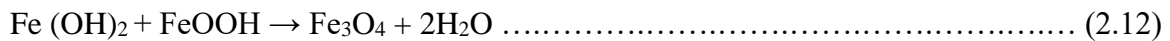


Further Oxidation takes places



(Reddish brown)

Finally,



As a consequence of these reactions, the outer layer of iron or its alloys converts into iron oxide, Fe<sub>3</sub>O<sub>4</sub>. This magnetite produced from the alkaline anoxic corrosion process functions as an effective barrier against corrosion, shielding the underlying metal.

## 2.2 Corrosion Control:

Corrosion protection is a critical aspect of coating applications, playing a vital role in maintaining the integrity and longevity of materials. On average, the economic impact of corrosion damage can reach around 4% of a country's GDP, underscoring the significance of effective corrosion management strategies across various industries [7]. The process of corrosion prevention typically encompasses three phases, although two of these phases are usually mandatory. The first phase focuses on preparing the substrate surface to ensure optimal adhesion and performance of the coating. This preparation may involve cleaning the surface to remove contaminants such as dirt, grease, or rust, as well as roughening it to enhance mechanical bonding. The second phase is the application of a suitable coating material to the prepared surface. This coating serves as a protective barrier, shielding the substrate from corrosive elements in the environment, such as moisture, oxygen, and pollutants. The choice of coating material is crucial and can vary widely based on the specific application, environmental conditions, and the type of substrate. Common options include paints, polymers,

metallic coatings, and specialized corrosion inhibitors. In some cases, a third phase may be introduced, where the surface to be coated undergoes additional treatments, either thermochemical or mechanical. Thermochemical treatments might involve processes like heat treatment or chemical baths to enhance the surface properties, while mechanical treatments could include processes such as shot blasting or grinding to achieve the desired surface finish and texture. Together, these phases form a comprehensive approach to corrosion prevention, aimed at maximizing the effectiveness of coatings and prolonging the life of the materials they protect. By investing in proper surface preparation and selecting appropriate coating materials, industries can significantly reduce the risks and costs associated with corrosion damage. A comprehensive study conducted by Martin Fenker focused on the anti-corrosion properties of hard coatings on steel surfaces [6]. The research delved into aspects such as film growth, microstructure, and defects. It was concluded that a single-layered conducting nitride coating is only effective if its thickness exceeds 10 $\mu$ m or if energetic deposition methods are employed. Additionally, recent findings highlighted that mapping TiN coatings using a confocal microscope reveals that open porosities and growth defects at both micro and macro scales significantly influence the anti-corrosive performance of hard coatings on steel.

### **2.2.1 Surface Treatment**

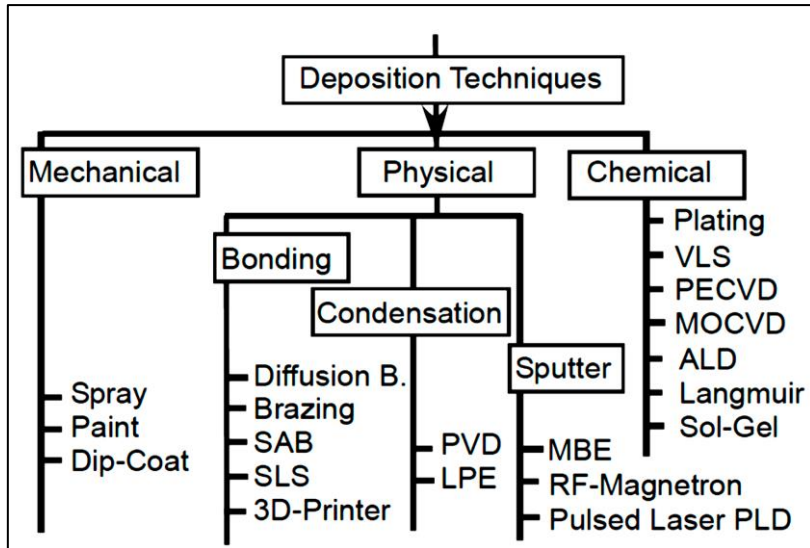
It has been scientifically established that surface treatment significantly impacts the subsequent anti-corrosion properties of coatings [8]. In some instances, surface treatment can modify the mechanical structure of the surface geometry, leading to changes in properties such as thermal and electrical conductivities, which in turn affect the overall coating performance. Nearly all material failures originate at the surface, so enhancing surface properties can effectively mitigate these issues and improve the overall behavior of the material.

### **2.2.2 Protective Nano-coatings Types as per Deposition Techniques**

Nano-coatings are advanced surface treatments that can be produced through three main deposition methods: mechanical, physical, and chemical deposition, as illustrated in (Figure - 3)[9]. Each of these methods offers distinct advantages and applications, allowing for the customization of nano-coatings for various purposes.

- 1) **Mechanical Deposition:** Mechanical deposition is recognized as the most cost-effective method for applying nano-coatings. This approach can be implemented through several techniques, including:

- a) **Spraying:** Involves dispersing coating material onto a substrate using a spray nozzle, allowing for uniform coverage over large areas.
  - b) **Painting:** A straightforward method where the coating material is applied directly with brushes or rollers, often used for protective coatings in construction and manufacturing.
  - c) **Spin-Coating:** A technique where a small amount of liquid coating is placed on a substrate, which is then rapidly spun to spread the material evenly. This method is particularly effective for achieving thin, uniform coatings.
  - d) **Dip-Coating:** Involves immersing the substrate in a liquid coating solution, allowing the material to adhere as the substrate is withdrawn. This technique is often used for complex geometries.
- 2) **Physical Deposition:** Physical deposition methods utilize physical processes to create coatings. These include:
- a) **Bonding:** This category encompasses various techniques, including:
    - i) **Physical Diffusion Bonding:** Moderate pressure and temperature are applied to achieve a strong bond between surfaces. This method is beneficial for materials that require high strength.
    - ii) **Brazing Bonding:** Lubricants are introduced under elevated temperatures, allowing for the joining of dissimilar metals or alloys with good mechanical properties.
    - iii) **Surface-Activated Bonding (SAB):** This technique is performed at lower temperatures and pressures, suitable for surfaces that are cleaned and atomically flat-polished, ensuring strong adhesion.
    - iv) **Selective Laser Sintering (SLS):** A cutting-edge three-dimensional (3D) printing technique where a material is built layer-by-layer using an additive manufacturing process. This method allows for intricate designs and customized coatings that traditional methods cannot achieve.
    - v) **Condensation:** This process typically occurs under vacuum conditions, such as in Physical Vapor Deposition (PVD), which allows for the deposition of thin films. It can also occur at normal pressure, exemplified by Liquid Phase Epitaxy (LPE), where layers of material are grown from a liquid solution.
    - vi) **Sputtering:** Although often more expensive due to lower growth rates, sputtering techniques provide excellent epitaxial growth and robust bonding characteristics. Sputtering can be performed using methods like Molecular Beam Epitaxy (MBE), which allows for precise control over the deposited material's thickness and composition.



**Figure -3:** Thin film deposition methods [8]

In conclusion, the choice of deposition method for nano-coatings depends on various factors, including cost, desired properties, and application requirements. Mechanical methods offer simplicity and affordability, while physical methods provide advanced techniques for achieving high-quality coatings with specific characteristics. Understanding these methods enables researchers and manufacturers to tailor nano-coatings for a wide range of applications, from enhancing corrosion resistance to improving mechanical properties in various industries.

### 2.3 Electrodeposition / Electroplating

Electroplating is a process that modifies the surface properties of a metal component by applying a metal coating through the action of electric current; this method is also referred to as "electrodeposition" [10]. In this process, the object to be coated is given a negative charge and submerged in a solution containing a salt of the metal intended for deposition. The metallic ions in the salt carry a positive charge, causing them to be attracted to the negatively charged object. Various metals are utilized in electroplating, including aluminum, brass, bronze, cadmium, copper, chromium, iron, lead, nickel, tin, and zinc, as well as precious metals like gold, platinum, and silver. By adjusting parameters such as voltage, amperage, temperature, residence time, and the purity of the bath solutions, different types of coatings can be achieved. Electroplating has numerous applications due to its cost-effectiveness and simplicity. It is widely employed across various industries, including electronics, optics, and automotive manufacturing. For instance, chrome plating is commonly used to improve the corrosion resistance of metal components. Below is a table that shows the diversity of electroplating metals and the associated applications:

<b>Metal Coating</b>	<b>Characteristics</b>	<b>Typical Applications</b>
Zinc	Excellent corrosion protection for steel; economic & safe	Connecting elements, auto and construction industry, plant engineering
Alloys – zinc, copper, and precious metal	Excellent corrosion protection, decorative finish, wear resistant	Extreme corrosion resistance combined with high thermal stress, e.g., car exhausts, engine elements, and electronics parts
Copper, Nickel, Chrome	Good corrosion protection, decorative finish	Motor vehicles, plumbing fittings, steel furniture, shop fittings
Hard Chrome	Extreme hardness, resistance to wear, corrosion protection, improved lubricating properties	Hydraulics, mold making, vehicles, shafts, and bearings
Silver, silver alloys	Good conductivity for heat and electricity, antibacterial, decorative finish with enhanced value.	Jewelry, electrical and electronic parts, household goods
Gold	Flexible, non- reactive with other elements, good electrical conductivity, resistance to corrosion, enhanced value	Jewelry, electrical and electronic parts, household goods
Bronze	Resistance to wear and corrosion, flexible	Bearing shells
Tin	Softness, flexible, solderable	Household applicanes, electronics, printed circuits, food industry

**Table -1:** Electroplating Characteristics and Applications [9]

### 2.3.1 Passivation Coating

The passivation process, often referred to as conversion coating, is a globally recognized technique that improves the corrosion resistance of materials by treating their outer surfaces. In some cases, this process occurs naturally, converting an initially unstable material into a more stable state [11]. Whether it happens through natural or artificial means, this procedure involves both physical and chemical alterations in the surface layer, extending to a certain depth that depends on various influencing factors. Once altered, the material resists any further modifications, effectively serving as a barrier to the underlying core, thereby maintaining its original properties. This process is beneficial across numerous industries globally. One notable example is the oxide passivation layer formed on firearms. This process has various variants, referred to as blackening, and others, depending on the specific method used. Passivation can be performed either physically or chemically, depending on the sample's composition and the required level of protection. Occasionally, this process is combined with surface finishing techniques that may be utilized.

## **2.4 Analysis of Surface Treatment for ASTM A516 Grade 70**

Khan, M. A. [12] done comprehensive analysis on Conversion coating which is an effective method for protecting materials against corrosion by creating a protective surface layer that inhibits corrosive attack. This study focuses on surface engineering applied to ASTM A 516 Grade 70, a steel widely utilized in construction. In this research, a passivation process was employed in which the samples were immersed in a hot alkaline oxide bath containing sodium hydroxide and sodium nitrate. This treatment helps to enhance the corrosion resistance of the steel by forming a protective oxide layer on its surface. To evaluate the effectiveness of the conversion coating, advanced testing procedures were conducted. A salt spray test was utilized as a benchmark for comparative analysis among different sample groups, including unpassivated samples, passivated samples, and passivated samples that had undergone prior surface treatment. The results indicated that mechanical surface treatment can improve the lifespan of the material by 3%. However, when passivation is applied following mechanical surface treatment, the lifespan is significantly enhanced, increasing by 18%. These findings underscore the importance of both surface preparation and passivation in extending the durability and performance of ASTM A 516 Grade 70 steel in corrosive environments. Overall, this study demonstrates that conversion coatings, particularly when combined with effective surface treatments, can significantly enhance the corrosion resistance and longevity of construction materials, making them more suitable for demanding applications.

## **2.5 Tri-sandwich Cu-Ni-Cr coating**

Raj, D. J [13] developed Tri-Sandwich Cu-Ni-Cr Coating over Weathering Steel in his research work. Weathering steel, classified as high-strength low-alloy (HSLA) steel, contains elements such as chromium, nickel, copper, silicon, and phosphorus. The presence of silicon and phosphorus enhances the corrosion resistance of this material. Corten A588 Grade weathering steel is commonly used in applications like the construction of towers, bridges, walls, and roofs, as well as various architectural designs. However, factors such as continuous dry and wet cycles, prolonged water exposure, and water accumulation in large weathering steel structures can limit its usability. To address these challenges, surface modification is crucial for improving both the corrosion resistance and mechanical properties of the steel. One effective method is electroplating, a cost-effective deposition technique widely used in industrial applications. This research focuses on developing a three-layered sandwich coating using copper (Cu), chromium (Cr), and nickel (Ni) through an electrodeposition process to enhance

the material's corrosion and mechanical properties. The tri-sandwich Cu-Ni-Cr coating is characterized through various analyses, including microhardness testing, electrochemical impedance spectroscopy (EIS), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDAX). The micro Vickers hardness of the tri-coated Cu-Ni-Cr layer on Corten A588 weathering steel, achieved through the electroplating process, measures  $1908.4 \pm 0.03$  HV. This represents a substantial increase from the uncoated Corten A588 steel, which has a hardness of  $323.63 \pm 0.02$  HV. This dramatic enhancement in hardness is attributed to the densely packed chromium and chromium oxide layers. The tri-coated Cu-Ni-Cr layer exhibits a cross-sectional thickness of  $11 \mu\text{m}$ , evenly distributed across the weathering steel. Specifically, the thickness of the first electroplated Cu layer is  $1 \mu\text{m}$ , while the second Ni layer measures  $9 \mu\text{m}$ , and the top Cr layer is also  $1 \mu\text{m}$  thick. The weight percentage composition of the Cu-Ni-Cr layer on Corten A588 weathering steel includes Iron (30.20%), Nickel (63.90%), Copper (5.74%), and Chromium (0.16%), along with trace amounts of nitrogen and oxygen. The first layer of copper, along with copper oxides like CuO and Cu<sub>2</sub>O, facilitates the adhesion of the sandwich coating to the weathering steel. The top layer consists of chromium and chromium oxides (CrO, Cr<sub>2</sub>O), which significantly enhance the mechanical and corrosion resistance of the steel. Electrochemical tests reveal that the tri-coated Cu-Ni-Cr sandwich-coated weathering steel exhibits a corrosion potential ( $E_{\text{corr}}$ ) of  $-1.916$  V and a low corrosion current density ( $I_{\text{corr}}$ ) of  $0.30$  mA/cm<sup>2</sup>. This performance is attributed to the stable, tightly packed chromium layer on the surface, supported by the underlying Cu and Ni layers. The robust chromium oxide layer formed at the electrolyte-material interface effectively prevents chromium dissolution from the sample's surface. Furthermore, the charge transfer resistance ( $R_{\text{ct}}$ ) for the tri-coated Cu-Ni-Cr sandwich-coated weathering steel is measured at  $1553.983$  Ω/cm<sup>2</sup>. This increase in  $R_{\text{ct}}$  value indicates the presence of a strong passive chromium oxide layer that acts as a protective barrier, enhancing corrosion resistance. Overall, the findings of this research demonstrate that the tri-coated Cu-Ni-Cr sandwich layer significantly improves the corrosion resistance of Corten A588 weathering steel. This Cu-Ni-Cr coating can withstand harsh corrosive environments and continuous wet-dry conditions, thereby broadening the potential applications of weathering steel in various structural and architectural projects.

## **2.6 Mechanical and corrosion behavior of Cu, Cr, Ni and Zn electroplating**

Deepak, J. R., and Raja [14] investigated the mechanical and corrosion properties of electroplated copper, chromium, nickel, and zinc on Corten A588 steel to assess potential enhancements for construction applications. In this study, the researchers focused on the

deposition of copper (Cu), chromium (Cr), nickel (Ni), and zinc (Zn) onto Corten A588 grade steel through an electroplating technique. This method is commonly used in various industrial applications due to its ability to enhance the surface properties of metals. After the electroplating process, several analyses were conducted to evaluate the properties of the coated steel. The microstructure of the coatings was examined using X-ray diffraction (XRD), a technique that helps identify the crystalline structure of materials. This analysis, along with energy-dispersive spectroscopy (EDS), confirmed the presence of Cu, Cr, Ni, and Zn elements in the respective coatings, indicating successful deposition of these metals. To assess the hardness of the coatings, Vickers hardness testing was performed. The results showed that the chromium-coated Corten A588 substrate exhibited the highest microhardness, measured at 1918.1 HVN. This elevated hardness level suggests that chromium not only provides a protective layer but also enhances the mechanical strength of the substrate. Surface morphology was analyzed using field emission scanning electron microscopy (FESEM). This technique provided detailed images of the surface structure, allowing for a better understanding of how the electroplated layers interact with the underlying steel and the environment. To evaluate the corrosion resistance of the coatings, electrochemical impedance spectroscopy (EIS) was utilized. EIS is a powerful technique for analyzing the electrochemical behavior of materials in corrosive environments. The results revealed that the chromium electroplated steel exhibited exceptional corrosion resistance, with an open circuit potential ( $E_{corr}$ ) of -1.742 V and a corrosion current density ( $I_{corr}$ ) of 0.334 mA. These values indicate that the chromium-coated samples were significantly more resistant to corrosion compared to the Cu, Ni, and Zn coatings, as well as the uncoated Corten A588 substrate. Additionally, weight loss analysis was performed to further investigate the corrosion behavior of the samples. Coated and uncoated samples were exposed to a 3.5% sodium chloride (NaCl) solution for one week, simulating a corrosive environment. The results showed that the chromium-coated Corten A588 samples experienced minimal weight loss, underscoring the effectiveness of the chromium coating in protecting the underlying steel from corrosion. The findings of this study indicate that coatings of Cu, Cr, Ni, and Zn on Corten A588 steel enhance its properties significantly, with chromium coatings providing the best protection against corrosion. The combination of high microhardness and superior corrosion resistance makes chromium-coated Corten A588 samples particularly suitable for construction applications, where materials are often exposed to harsh environmental conditions. In summary, the research highlights the importance of surface coatings in improving the durability and longevity of structural materials, particularly in outdoor and industrial applications. The insights gained from this study can inform future



efforts to optimize coating processes and material selections for enhanced performance in various engineering contexts.

## **2.7 Corrosion protection by Passive Oxide film**

Maurice [15] in his research paper studied Current developments of nanoscale insight into corrosion protection by passive oxide films. Oxide passive films play a crucial role in the durability of metal and alloy components, making them a significant focus in corrosion science and engineering. This discussion explores recent advancements in our understanding of the barrier and adsorption properties of passive oxide films at the nanometer and sub-nanometer scales, informed by the latest experimental and theoretical studies. The homogeneity and enrichment of chromium at the nanoscale within passive films formed on chromium-containing alloys, such as stainless steel. An examination of the corrosion characteristics at grain boundaries during the initial stages of intergranular corrosion, prior to the penetration and propagation within the grain boundary network. The interaction of organic inhibitor molecules with metallic surfaces that are incompletely passivated.

## **2.8 Impact of Benzotriazole on Phosphating**

Phosphating is a widely recognized process utilized across various industries for surface treatment and preparation for subsequent painting, thereby enhancing corrosion resistance. Cheng-Hong Ye et al. [16] conducted a study that highlights the significant changes in the physicochemical properties of both ferrous and non-ferrous metals upon the deposition of a phosphate layer. This phosphate coating serves a dual purpose: it acts as an electrical insulator, which is crucial for preventing unwanted reactions on the metal surface, and it enhances oil absorption, thereby improving the metal's ability to retain lubrication. In their research, phosphating was specifically applied to modify the WE43 magnesium alloy, a material known for its lightweight and high strength, making it ideal for various applications, including in the biomedical field. To accelerate the phosphating process, sodium m-nitrobenzene sulfate was introduced as an acceleration agent, which helps speed up the formation of the phosphate layer. Furthermore, the study evaluated the biocompatibility and microstructural characteristics of the treated alloy. Biocompatibility was assessed using hemolysis tests, which measure the potential of materials to cause hemolysis (destruction of red blood cells), an important factor for materials used in medical applications. The microstructure of the coated alloy was analyzed through scanning electron microscopy, allowing for a detailed examination of the surface morphology and the effectiveness of the phosphate coating. The findings of this research

conclusively demonstrate that phosphate coatings not only enhance corrosion protection for the WE43 alloy but also improve its biocompatibility. This dual benefit is crucial for extending the application of WE43 magnesium alloys in environments where both durability and compatibility with biological systems are essential, such as in medical implants or devices.

## **2.9 Organic Compounds addition into Phosphating**

Organic compounds can be introduced into the phosphating process to enhance corrosion protection by effectively reducing the porosity of the phosphate layer. A notable example, as highlighted by E.P. Banczek et al. [17], is benzotriazole, which can be incorporated into a zinc phosphating bath to improve the corrosion resistance of carbon steel significantly. In their study, two samples were analyzed: one treated with a standard zinc phosphating bath and the other with the addition of benzotriazole (referred to as BTAH). To evaluate the effectiveness of this addition, the researchers employed techniques such as electrochemical impedance spectroscopy and anodic polarization curves. These methods provide insights into the electrochemical behavior of the treated surfaces, allowing for a detailed comparison of their protective qualities. The findings revealed that the mass of the phosphate layer deposited in the sample treated with PZN+BTAH was considerably higher than that of the sample with the standard zinc phosphating bath. This increase in mass indicates a thicker and more robust phosphate layer, which is advantageous for corrosion resistance. Interestingly, despite the variation in mass, the overall composition of the phosphate layers in both samples remained consistent, suggesting that the chemical makeup of the coating was not adversely affected by the presence of benzotriazole. One of the key advantages of adding BTAH is its effect on the size of phosphate nucleation particles. The presence of benzotriazole promotes the formation of smaller nucleation sites during the phosphating process. This leads to a denser and more compact phosphate layer, enhancing the layer's protective qualities. A more compact coating can better shield the underlying metal from corrosive elements, thereby improving the overall durability of the carbon steel. In summary, the incorporation of benzotriazole into the zinc phosphating process not only increases the mass of the deposited layer but also results in a finer, more compact structure. This modification significantly enhances corrosion protection, making it a valuable addition for applications requiring durable surface treatment.

## **2.10 Subsequent Application of Anodizing and Annealing**

In the study conducted by Yong-Wook Choi et al. [18], the impact of anodizing, annealing, and their combination on the corrosion resistance of iron oxide was investigated using

electrochemical methods. The research revealed that corrosion protection achieved through single annealing resulted in an amorphous phase, which fell short of effectiveness standards. When anodization and annealing were applied together, a positive shift in the Tafel plot indicated an improvement in corrosion potential. This combination led to the formation of a thicker oxide layer following the annealing process. The oxide films produced by this dual approach exhibited a very low rate of charge transfer, suggesting enhanced protective properties. The optimal annealing temperature was identified as 500°C. However, exceeding this temperature resulted in negative effects, such as a shift in the Tafel plot due to film cracking and the formation of hematite, which undermined corrosion resistance. This highlights the necessity of precisely controlling the annealing temperature to achieve optimal corrosion protection.

### **2.11 Chromium-Carbon Coating**

S. Ghaziof et al. [19] undertook a comprehensive study focused on understanding how various substrates and their surface preparations impact the corrosion protection provided by chromium-carbon coatings. This research is particularly relevant in the context of trivalent chromium plating, which offers numerous health, technical, and environmental advantages compared to traditional hexavalent chromium (Cr(VI)) coatings, which are known to pose significant health risks and environmental concerns. The plating solution used in the study contains Cr(III) salts, such as chromium chloride ( $\text{CrCl}_3$ ) or chromium sulfate ( $\text{Cr}_2(\text{SO}_4)_3$ ), alongside several other components, including a complexing agent to stabilize the chromium ions, a conducting salt to facilitate the electrochemical deposition process, pH buffers to maintain an optimal acidity level, wetting agents to enhance the coating's adherence, and brighteners to improve the aesthetic quality of the coating. To mitigate undesirable reactions that could occur during the plating process, carboxylic acids are typically added. These acids facilitate the release of carbon, which is essential for the formation of a durable Cr-C (chromium-carbon) layer capable of withstanding temperatures as high as 600°C. The coatings were electro-deposited from a trivalent chromium bath, and the researchers selected two distinct substrates: copper and low carbon steel. For the low carbon steel samples, three different surface preparation techniques were applied: mechanical polishing, electro-polishing, and anodic etching. Each of these methods aims to optimize the substrate's surface characteristics, thereby enhancing the quality and performance of the resulting coatings. The study's findings indicated that the coatings produced through these methods displayed an amorphous cracked structure, accompanied by a consistent granular morphology. This

structural characteristic is significant as it affects the coating's overall performance in terms of corrosion resistance. Notably, the low carbon steel samples exhibited active anodic behavior, which indicates a propensity to oxidize, while the copper substrates demonstrated passivation behavior, meaning they formed a protective oxide layer that mitigated corrosion. Importantly, the researchers observed that when coatings were applied to surfaces that had undergone pre-anodic etching, the resulting layers were free from cracks and exhibited significantly lower porosity. This reduction in porosity is critical because it leads to decreased dissolution of the underlying steel substrate, thereby enhancing the overall corrosion resistance of the coated materials. The ability to achieve crack-free, low-porosity coatings through specific surface preparations highlights the importance of substrate treatment in the efficacy of corrosion protection strategies. This study provides valuable insights for industries seeking to improve the durability and longevity of coated components in various applications.

## **2.12 Effect of Microstructure and Composition variation**

Her-Hsiung et al. [20] conducted a detailed investigation into the impact of varying chemical composition and microstructure on the electrochemical behavior of weld metal and the heat-affected zone (HAZ) using ASTM A516 (Grade 70). This particular grade of steel is widely used in pressure vessel fabrication, making it relevant for studies related to welding and corrosion resistance. In their experiments, the researchers utilized ASTM A516 (Grade 70) with specific microstructural features, namely pearlite and ferrite structures. These structures were selected due to their significance in influencing mechanical properties and corrosion resistance. The study involved varying welding heat conditions and two distinct welding processes: submerged arc welding and thermal simulation welding. This multifaceted approach allowed for a comprehensive analysis of how different welding parameters affect the resultant electrochemical properties. Three sets of samples were tested using potentiodynamic polarization tests and electrochemical impedance spectroscopy (EIS). These methods are widely employed to evaluate the corrosion resistance of materials by analyzing their electrochemical behavior. The polarization tests revealed distinct electrochemical response curves for the base metal, weld metal, and HAZ at a specific welding heat of 30 kJ/cm. The differences in these curves were attributed to the variations in composition and microstructure of the materials involved. For instance, the presence of different phases and their respective distributions within the microstructure significantly influenced the corrosion behavior. As the welding heat input varied across three different conditions, the electrochemical behavior of the base metals and HAZ also exhibited notable differences. A key observation was that an increase

in welding heat resulted in a decrease in cathodic current density. This suggests that higher welding heat may enhance the stability of the passive layer on the metal surface, thereby reducing corrosion susceptibility. The researchers compared the polarization curves for bare metal, weld material, and HAZ as a function of welding heat input. The results indicated that, irrespective of the welding heat conditions, the weld metal consistently demonstrated the highest polarization resistance, which was directly proportional to the welding heat. This implies that the weld metal's microstructure, likely influenced by the welding process, contributes significantly to its enhanced corrosion resistance. In the case of the HAZ prepared by a Gleeble machine, a similar trend was observed. Below a welding heat input of 30 kJ/cm<sup>2</sup>, the polarization resistance of the HAZ was lower than that of the base metal, indicating that the microstructural changes occurring in the HAZ may render it more susceptible to corrosion under certain conditions. EIS measurements were performed under the same variable conditions as the polarization tests. The results showed that as the welding heat input increased, the impedance loops for both the weld metal and HAZ expanded. Among the samples tested, the weld metal exhibited the largest loop, highlighting its superior electrochemical stability and corrosion resistance compared to the other materials. Furthermore, the polarization resistance of the HAZ was found to increase with higher welding heat input, reinforcing the notion that thermal treatments during welding can enhance the protective qualities of the coating or passive film on the surface. In summary, the findings of Her-Hsiung et al. underscore the significant influence of microstructure and chemical composition on the electrochemical behavior of weld materials and HAZ. The study demonstrates that variations in welding heat input and processing techniques can lead to substantial differences in corrosion resistance, which is critical for applications involving welded structures. By understanding these relationships, the research contributes valuable insights into optimizing welding practices to enhance the longevity and durability of steel components in demanding environments.

### **2.13 Impact of Surface finishing on Carbon Steel**

Yuan Li et al. [21] conducted a comprehensive study to explore the early stages of corrosion on carbon steel, focusing on how different surface roughness levels—achieved through various surface finishing treatments affect corrosion behavior. The researchers employed atomic force microscopy (AFM) alongside electrochemical measurements to analyze the surface characteristics and corrosion rates, with surface roughness quantified on a nanometer scale. The study revealed a clear relationship between surface roughness and the likelihood of corrosion. As the roughness of the surface increased, the probability of corrosion also rose.

This correlation is significant because it suggests that surface texture plays a critical role in determining the durability and longevity of carbon steel in corrosive environments. In the first phase, a rapid dissolution of material occurred as surface roughness increased. This phase is marked by heightened susceptibility to corrosion due to the increased surface area exposed to corrosive agents, which accelerates the dissolution process. Rough surfaces tend to trap moisture and contaminants more effectively, creating localized areas that are prone to corrosion. The second phase of corrosion is characterized by an equilibrium state where the corrosion rate stabilizes, and the surface roughness remains relatively constant. Interestingly, specimens with finer surface finishes required a longer duration to reach this second stage. This delay indicates that smoother surfaces initially resist corrosion better than rougher surfaces, highlighting the protective effect of fine finishing. The study's findings strongly suggest that achieving a finer surface finish can significantly enhance the corrosion resistance of carbon steel. The smoother the surface, the more effective it is at preventing localized corrosion processes. This protective mechanism can be attributed to the reduced number of surface irregularities that can act as initiation sites for corrosion. Moreover, the presence of surface irregularities was found to promote localized preferential corrosion. These irregularities can create conditions where corrosive agents accumulate, leading to deep and wide scratches on the surface. Such localized corrosion not only compromises the structural integrity of the material but also can lead to premature failure in applications where carbon steel is exposed to harsh environments. In conclusion, the research by Yuan Li et al. underscores the critical role of surface roughness in the early stages of corrosion on carbon steel. By demonstrating that finer surface finishes provide better protection against corrosion, the study highlights the importance of surface finishing treatments in extending the lifespan of carbon steel components. This knowledge can reform industrial practices and material selection, ultimately leading to improved performance and reliability in various applications, particularly those exposed to corrosive conditions.

## CHAPTER 3: EXPERIMENTAL WORK

### 3.1 Design of experiment:

The goal of this experiment was to develop a protective measure to counteract the effects of corrosion. A material of broad significance was chosen, with particular focus on its public and commercial applications to ensure that it could benefit the largest number of people. The material went through surface treatment and different types of coatings processes. Five different types of samples were created which are Base material, Passivated, Surface polished, Ni-Coated and dual coated (Ni & Cr) respectively to perform a comparative analysis. In sample-1 only base material cut to desired size and in sample-2 base material cut and goes through passivation process only. For sample-3 preparation, base material cut and only goes through surface grinding and polishing process and no protective coating is deposited on it. Sample-4 is prepared for nickel electroplating for which base material goes through cutting and surface polishing processes respectively. Sample-5 is prepared by electroplating sample-4 with chrome which makes sample-5 dually coated with Ni and Cr. Process flow for samples preparation is shown below in (Table -2)

Samples	Sample Type	Process Flow				
		Samples Cutting	Passivation	Surface Polish	Nickel Plating	Chrome Plating
Sample-1	Base Material	✓				
Sample-2	Passivated	✓	✓			
Sample-3	Polished	✓		✓		
Sample-4	Ni-Coated	✓		✓	✓	
Sample-5	Dual Coated	✓		✓	✓	✓

**Table -2:** Process flow for Samples Preparation

### 3.2 Material Technical Specifications

The primary step was selecting the appropriate material. Thorough consideration was taken to choose a material suitable for various applications. The necessity for corrosion protection, which is fundamental to this study, guided this choice. The potential short-term and long-term impacts of the expected outcomes were anticipated, which could greatly enhance the effectiveness of this project. ASTM A516 grade 70 was chosen for testing and experimentation, as it is commonly used in construction and structural applications where strength and durability are essential. To ensure durability, this material requires effective corrosion protection. This material is widely used in industrial applications for manufacturing pressure vessels and boilers due to its durability. It is capable of withstanding a wide range of temperatures. Its chemical composition and mechanical properties are given in (Table -3) and (Table -4) respectively.

Element Name	Element Symbol	Weightage %
Carbon	C	0.10~0.28
Manganese	Mn	0.85~1.2
Phosphorus	P	0.01~0.035
Sulphur	S	0.01~0.036
Silicon	Si	0.15~0.40
Iron	Fe	Remaining %

**Table -3:** Chemical Composition (Wt %) of ASTM A516 Grade 70

Yield Strength	> 260 N/mm <sup>2</sup>
Tensile Strength	485 – 620 N/mm <sup>2</sup>
Elongation	> 21 %

**Table -4:** Mechanical Properties of ASTM A516 Grade 70



### 3.3 Material Arrangement:

ASTM A516 grade 70 was obtained in sheets that are 3 mm thick, measuring 1000 mm in length and 990 mm in width. The sheet specimen was analyzed using an optical emission spectrometer to verify that it meets the required chemical composition and mechanical properties of the ASTM A516 grade 70 standard. (Table -5)and (Table- 6) contains the test result data showing that procured specimen conforms to the standard specification.

Element Name	Element Symbol	Weightage %	Result
Carbon	C	0.15	Satisfactory
Manganese	Mn	1.0	Satisfactory
Phosphorus	P	0.015	Satisfactory
Sulphur	S	0.015	Satisfactory
Silicon	Si	0.33	Satisfactory

**Table -5:** Chemical Composition (Wt %) of Test Specimen

Properties	Test Readings	Result
Yield Strength	424 N/mm <sup>2</sup>	OK
Tensile Strength	511 N/mm <sup>2</sup>	OK
Elongation	24 %	OK

**Table- 6:** Mechanical Properties of Test Specimen

### 3.4 Samples Cutting (Process-1)

The sheet was cut into coins shape having diameter of 20mm as necessary for experiments and testing Shown in (Figure -4). Special care was taken during the cutting process to ensure that the original properties of the sheet were uniformly preserved throughout all dimensions. For cutting purpose CNC sheet metal laser cut machine was used.



**Figure -4:** Base material cutting specimens (Sample type-1)

### **3.5 Passivation (Process-2)**

The samples shown in (Figure -5) were prepared by treating the original specimen in a hot alkaline oxide solution for a specified duration at a certain temperature. This solution consisted of an aqueous mixture of sodium nitrate ( $\text{NaNO}_3$ ) and sodium hydroxide ( $\text{NaOH}$ ) in a 1:3 ratio. For every liter of water, 200 grams of sodium nitrate and 600 grams of sodium hydroxide were added. The samples were immersed in the solution for 60 minutes at a temperature of  $130^\circ\text{C}$ . As a result, the outer surface of the samples was blackened, chemically transforming into magnetite ( $\text{Fe}_3\text{O}_4$ ). This oxide layer serves as a protective barrier against corrosion. The aim of creating these samples was to evaluate the corrosion resistance provided by the  $\text{Fe}_3\text{O}_4$  surface oxide layer.



**Figure -5:** Passivated specimens (Sample type-2)

### **3.6 Surface Grinding and Polishing (Process-3)**

To prepare surface treated samples as shown in (Figure -6) SiC emery paper was used and to achieve required surface finish substrate is grinded and polished with different grades of silicon carbide (SiC) emery papers by increasing grit size in 03 steps from 300 $\mu$ m, 600 $\mu$ m and 1200 $\mu$ m respectively. After grinding process to obtain mirror finish silica suspension is used which gives shinny mirror like finish to substrate surface.



**Figure -6:** Polished Surface specimens (Sample type-3)

### **3.7 Nickel Electroplating (Process-4)**

For Nickel electroplating ASTM-516 grade 17 steel as shown in (Figure-7) polished sample is taken as cathode and metal to be deposited is taken as anode which in this case is Ni. Electroplating process is carried out as per standard followed by Automobile OEMs which is currently being used for passenger vehicle head rests and motorbikes fenders and rims Ni & Cr electroplating. Sample goes through following processes to get ready for electroplating process.

#### **3.7.1 Acid Pickling**

To remove oil, rust and dust residue from substrate surface, polished samples dipped in Hydrochloric acid (HCl) concentrated solution for 5-10 minutes at room temperature.

#### **3.7.2 Fresh Water Rinsing**

After acid pickling process samples dipped in fresh water tank for 5-10 minutes to remove chemicals residue deposited on substrate surface due to acid pickling.

#### **3.7.3 Neutralizing**

Water rinsed samples dipped in low concentrated (10%) sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) solution for 3 minutes at room temperature to remove any rust present at surface.

### 3.7.4 Fresh Water Rinsing

After neutralizing, samples dipped in fresh water tank for 5-10 minutes to remove chemicals residue deposited on substrate surface due to chemical reaction.

### 3.8 Nickel electroplating

Now samples are ready for electroplating process. Polished surfaced sample dipped into nickel sulfate solution (pH value: 4.2-4.6) for 25-30 minutes at temperature 50-55°C in a controlled environment.



**Figure-7:** Nickel electroplated Specimens (Sample type-4)

### 3.9 Nickel-Chrome Dual Electroplating:

To develop dual coated samples (Figure -8), Nickel coated samples are being used for chrome electroplating process. Ni-coated samples are taken as cathode and Cr is taken as anode. For Cr-electroplating, chromium trioxide is used to prepare chromium bath. Samples dipped into solution of chromium trioxide for 4-5 minutes at 33-37°C temperature.

Voltage and current maintained at 4V and 250 A respectively. After electroplating, sampled rinsed with fresh water to remove any chemical residue.



**Figure -8:** Dual coated (Ni-Cr) Specimens (Sample type-5)

Process Quality Control Table (II)

PURCHASE & V.D. DEPARTMENT		VENDOR		ADDRESS		MODEL		PART NAME: HEAD REST FRAME COMP RR					
Process Order	Critical Item Failure Mode	Quality Characteristics			Production Condition Control			Schematic Drawing					
		Process Name/Equipment Name	Specification	Person in charge (PIC)	Control Item	Control Value	Person in charge (PIC)	Check Method	Check Frequency	Data Format			
<b>PART NAME: HEAD REST FRAME COMP RR</b>													
20	Cleaning	20.1	Parts should be Properly Clean with nothing should be left on the surface	Associate	Appearance	Visual	100%	NI	NI	Visual	Once day	Check sheet	
30	Polishing	30.1	Polish disc	Associate	Appearance	Visual	100%	NI	NI	Visual	Once day	Check sheet	
40	Acid Pickling	40.1	No Oil/Rust & Dust	Associate	Appearance	Visual	100%	NI	NI	Wall Clock	Once day	Check sheet	
50	Degreasing	50.1	Rust, damage, crack and deformations are not allowed	Associate	Appearance	Visual	100%	NI	NI	HCl Concentration	Once day	Check sheet	
60	Fresh Water Rinsing	60.1	No Effect of Chemicals	Associate	Removal of Chemical	Visual	100%	NI	NI	Temperature	Once day	Check sheet	
70	Neutralizing	70.1	No Rust	Associate	Anti Rust surface	Visual	100%	NI	NI	Dipping Time	Once day	Check sheet	
80	Fresh Water Rinsing	80.1	No Effect of Chemicals	Associate	Removal of Chemical	Visual	100%	NI	NI	Residence Time	Once day	Check sheet	
90	Nickel Plating	90.1	Nickel Flow & Peel off not Allowed. Surface cracks, blisters, peeling, holes or gloss and color changes not Allowed.	Associate	Appearance	Visual	100%	NI	NI	Dipping Time	Once day	Check sheet	
100	Fresh Water Rinsing	100.1	No Effect of Chemicals	Associate	Removal of Chemical	Visual	100%	NI	NI	Residence Time	Once day	Check sheet	
110	Chrome Plating	110.1	Chrome Flow & Peel off not Allowed. Surface cracks, blisters, peeling, holes or gloss and color changes not Allowed.	Associate	Appearance	Visual	100%	NI	NI	Dipping Time	Once day	Check sheet	
120	Fresh Water Rinsing	120.1	No Effect of Chemicals	Associate	Removal of Chemical	Visual	100%	NI	NI	Residence Time	Once day	Check sheet	
130	Buffing	130.1	Buffing Wheel should be clean and properly working	Associate	Appearance	Visual	100%	NI	NI	Buffing wheel	Once day	Check sheet	
140	Final Inspection	140.1	Thickness = 20 micron Chrome Flow & Peel off not Allowed. Surface cracks, blisters, peeling, holes or gloss and color changes not Allowed.	Associate	Appearance	Visual	100%	NI	NI	Check Sheet	Once day	Check sheet	

Figure- 9: Process Quality Control Table for Electroplating Process

## **CHAPTER 4: TESTING, RESULTS AND DISCUSSIONS**

### **4.1 Initiation**

To assess the performance of the various prepared samples, they were subjected to a series of basic and advanced tests at different research and technology labs available in Pakistan. These facilities were equipped with cutting-edge equipment and resources to conduct the tests effectively. The results were meticulously gathered and compared to draw conclusive findings.

### **4.2 Salt Spray Test**

One of the most fundamental and widely accepted methods for measuring corrosion protection is the salt spray test. In this procedure, the samples are placed in a controlled environment where temperature and humidity are regulated, and they are exposed to an atomized solution having 500 g NaCl per 10 liter of distilled water. This environment is highly corrosive, as it aggressively attacks the surface and accelerates the corrosion process. The samples are stored in a closed chamber for a specified duration, during which they are regularly monitored. Observations are made on the initiation and progression of corrosion, and a comparative analysis is conducted by evaluating the results across all samples. The results include weight changes due to corrosion and visual assessments to determine the percentage of the total area that has corroded. The samples were placed in the salt spray chamber as shown in (Figure -10), set at a lab temperature of 50°C and pressure bucket temperature set at 60°C. Solution composition for lab is prepared by using distilled water and acetic acid. Solution pH is set to 3-3.2. For pressure bucket composition 7.8 gm cupric chloride per 03 liter of distilled water is used They were exposed to this environment for a total of 96 hours, with visual inspections carried out every 24 hours to check for the onset and rate of corrosion. Initially, all samples designated for testing were carefully weighed and then positioned inside the chamber on two holding arms. They were subjected to the specified testing conditions for 24 hours. After this period, the chamber lid was opened for a visual inspection. This procedure was repeated every 24 hours, concluding after a total testing duration of 96 hours. Once the test was complete, the samples were weighed again. It was observed that all samples had gained weight due to the accumulation of corrosion products formed chemically. The samples were then washed to remove these corrosion products and weighed once more. After cleaning, it was noted that all

samples weighed less than their original weights, as the corroded portions had been washed away.



Figure -10: Salt spray testing Chamber

Manufacturing Operation Standard							
PROCESS NAME		CASS TEST MACHINE					
ILLUSTRATION	No. of Critical Steps	OPERATION	CHECK POINT	STANDARD	FREQUENCY	METHOD	COMMENTS
   	1	SWITCH ON THE MACHINE AND SET THE PARAMETERS.  مین کے سچاؤ آگ کر لیں اور ریٹرن کر دیتے ہیں۔	LAB TEMPERATURE	50 °C 150 کرنی	AT START	METER	
			PRESSURE BUCKET TEMPERATURE	65 °C 145 کرنی	AT START	METER	
			AIR PRESSURE	180/CM 140 کرنی	AT START	PRESSURE GAUGE	
			TIME CONTROL	FENCER & REM - 16 HOURS 16 گھنٹے	AT START	TIMER	
			LAB COMPOSITION	DISTILLED WATER UP TO OVERFLOW	AT START	VISUAL	
	2	MAKE THE RIGHT COMPOSITION AND PUT IN THE RIGHT PLACE.  کپڑے تیار کر کے اور احتیاط سے رکھیں۔	LAB COMPOSITION	ACETIC ACID UP TO PH 3-3.2	AT START	P.H PAPER	کون کتنی تیار کیا گیا ہے
			PRESSURE BUCKET COMPOSITION	7.8 gm CHLORINE IN 10 L WATER	AT START	WEIGHT SCALE	
			REAGENT ENTRANCE COMPOSITION	500 gm SODIUM CHLORIDE IN 10 L WATER	AT START	WEIGHT SCALE	
	3	PLACE THE PARTS IN MACHINE & AFTER COMPLETION OF GIVEN TIME, PRESS DEFOG BUTTEN.  پارٹس کو مشین کے ڈور میں رکھیں اور ٹائم وقت پورا کرنے کے بعد ڈی فگ بٹن دبا لیں۔	DEFOG	DO NOT OPEN THE MACHINE TILL ALL THE GAZE EXITE.	AFTER GIVEN TIME	VISUAL	
			TAKE OUT THE PARTS FROM MACHINE AND CLEAN.  مشین سے پارٹس کو باہر لائیں اور صاف کریں۔	HANDLING	CAREFULLY TAKE OUT THE PARTS FROM MACHINE.	AT END	MANUAL

Figure -11: Manufacturing Operation Standard for Salt Spray Testing machine

### 4.3 Weight Loss Analysis

For weight loss analysis Sartorius Model TE313S-DS Electronic analytical and Precision Metal Scale as shown in (Figure -12) is used which has least count of 0.001gm.

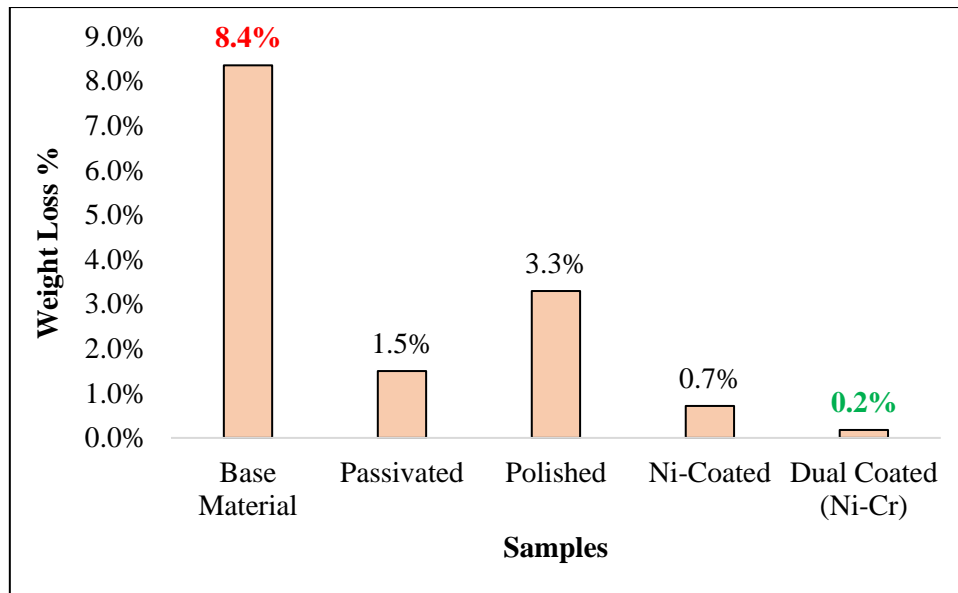


**Figure -12:** Sartorius Model TE313S-DS Electronic Analytical and Precision Metal Scale

Samples	Sample Type	Weight (g)						Corrosion Rating
		Initial (A)	With corrosion (B)	Diff C=(B-A)	After washing (D)	Diff E=(D-A)	% Diff =(E)/(A)	
Sample-1	Base Material	8.1620	8.1840	0.0220	7.4800	-0.6820	-8.4%	5
Sample-2	Passivated	8.0830	8.1030	0.0200	7.9620	-0.1210	-1.5%	3
Sample-3	Polished	7.4400	7.4470	0.0070	7.1950	-0.2450	-3.3%	4
Sample-4	Ni-Coated	7.1460	7.1700	0.0240	7.0950	-0.0510	-0.7%	2
Sample-5	Dual Coated (Ni-Cr)	7.2680	7.2920	0.0240	7.2550	-0.0130	-0.2%	1

**Table -7:** Salt spray test Results of Samples





**Figure -13:** Weight loss analysis for coated and uncoated ASTM-A516 Grade 70 Steel

Base material and polished samples had the more weight loss other than the rest of the samples as shown in (Figure -13). The reason here is the absence of any corrosion protective layer which was the main corrosion protection on the remaining three samples. Polished sample had comparatively reduce less weight as compared to base material due to the surface grinding has given some corrosion protection. Passivated sample shows good resistance against corrosion because of magnetite protective layer deposited on surface. Base material specimen had neither surface treatment nor oxide layer so it loss the more weight because of accelerated corrosion. Out of the three samples with corrosion protective layers, dual coated (Ni-Cr) displayed the highest resistance to corrosion because of its dual protection. Ni coated samples showed the second highest protection whereas passivated sample came third in weight loss analysis. It is evident from weight loss analysis, dual coated samples exhibit excellent performance against corrosion attack by reducing only 0.2% of initial weight. This clearly shows that the rich, smooth layer of chromium and chromium oxide on the surface acts as a barrier, enhancing resistance to corrosion. Whereas Ni-coated samples also performed very well by just reducing 0.7% weight which shows that Ni coating is as good as Cr coating in case of corrosion resistance applications.

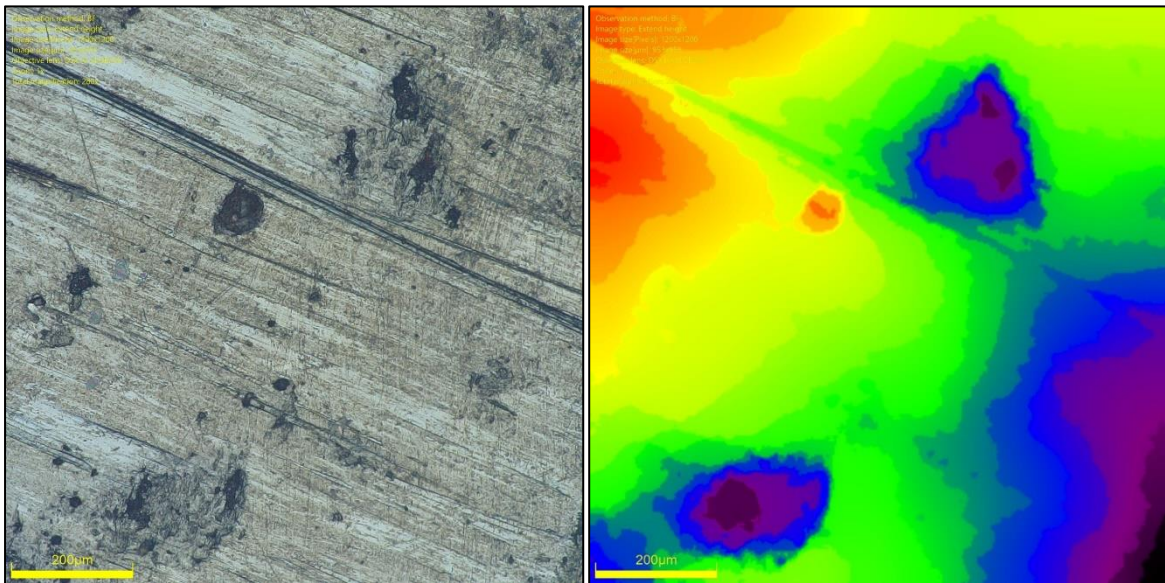
#### 4.4 Surface Roughness Analysis

Surface roughness analysis was conducted on each coated sample to assess the coating's surface condition, using the Olympus Digital Microscope DSX1000, which offers a magnification capacity of up to 9637X.

Sample Type	Surface Roughness 'Ra'						Ranking
	μm						
	X-axis			Y-axis			
	Min	Max	Average	Min	Max	Average	
Passivated	0.163	0.225	0.194	0.142	0.228	0.185	3
Ni-Coated	0.072	0.097	0.0845	0.004	0.113	0.0585	2
Ni-Cr Coated	0.041	0.069	0.055	0.0058	0.104	0.0549	1

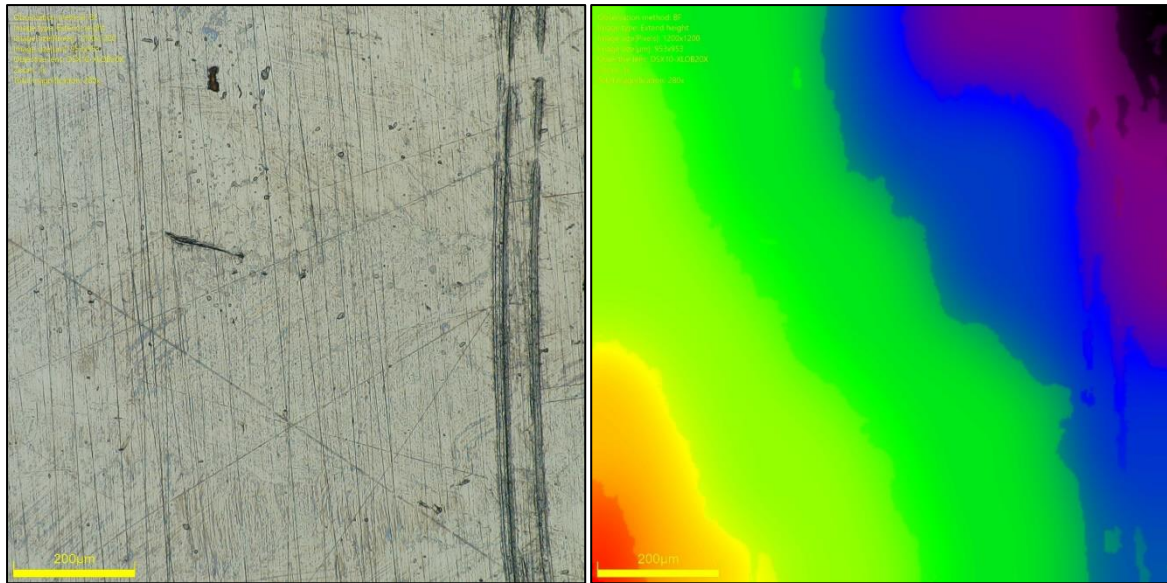
**Table -8:** Surface Roughness Analysis of Coated Samples

Surface analysis of coated samples (Table -8) clearly shows that the roughness value of the passivated samples is nearly twice that of the Ni-coated and Ni-Cr-coated samples, due to the absence of any surface treatment before passivation which is evident in image obtained at 280X magnification shown in (Figure -14) below:

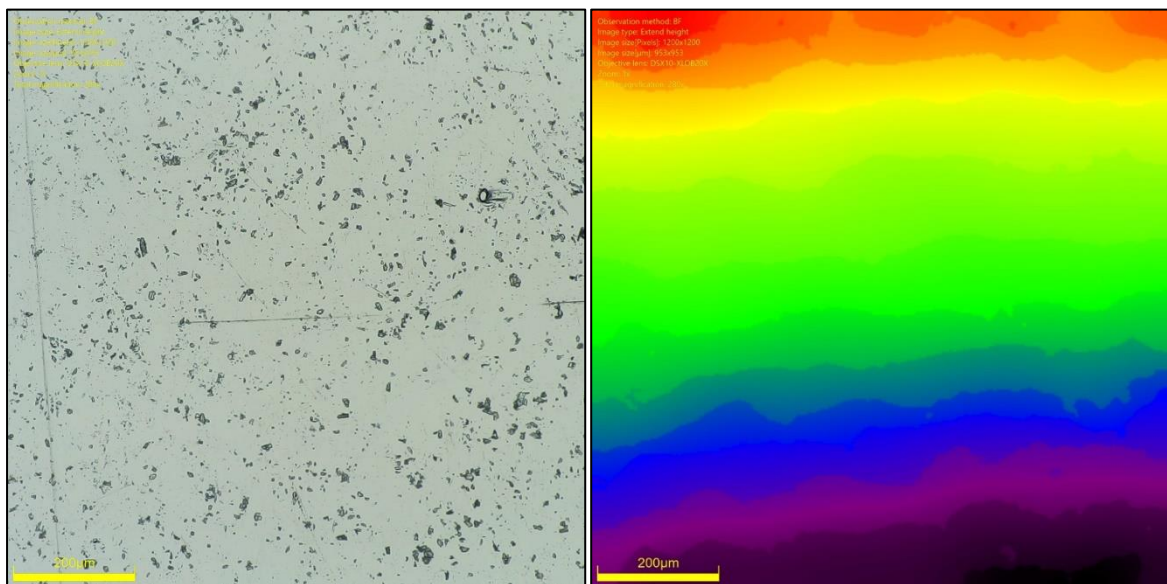


**Figure -14:** Image obtained at 280X resolution of Passivated Specimen

The Ni-coated and Ni-Cr-coated samples exhibited nearly identical surface finishes, as both underwent the same surface polishing process. This is evident from the (Figure -15) and (Figure -16) below, which display the surface conditions of the Ni-coated and Ni-Cr-coated samples respectively.



**Figure -15:** Image obtained at 280X resolution of Ni- Coated Specimen

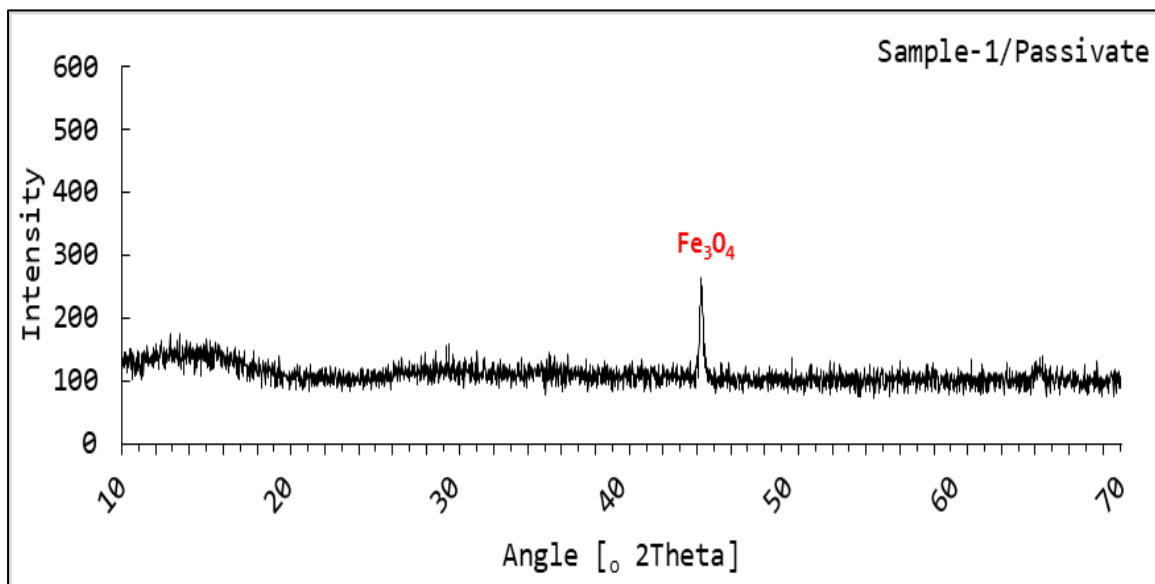


**Figure -16:** Image obtained at 280X of Ni-Cr Coated Specimen

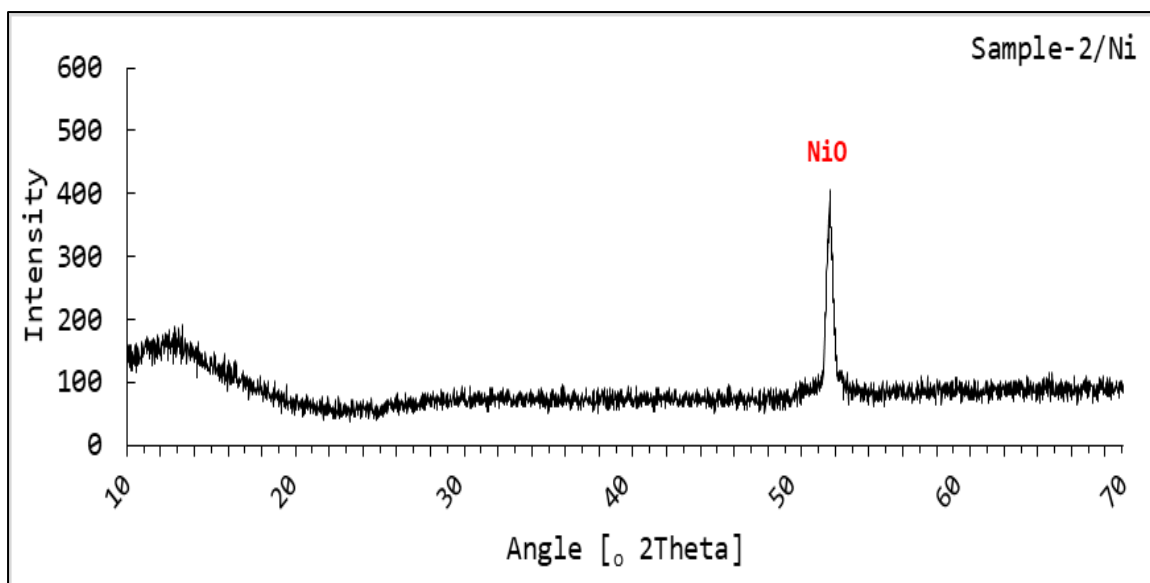
#### 4.5 X-Ray Diffractometry (XRD) Analysis

To examine the atomic and molecular crystalline structure, the coated samples passivated, Ni coated and Ni-Cr coated were analyzed using an X-Ray Diffractometer. Diffraction is a non-destructive analytical method that enables the identification and quantitative analysis of different crystalline forms, referred as phases, found in powdered and solid samples. The peak widths in a specific phase pattern indicate the average size of the crystallites: larger crystallites produce sharp peaks, whereas smaller crystallites result in broader peaks.

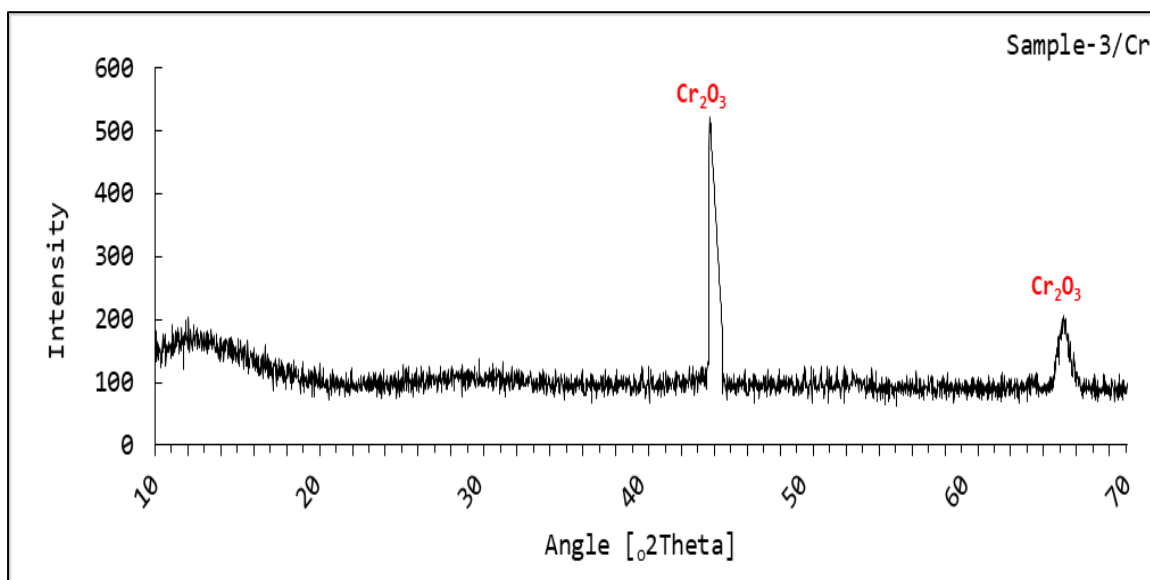
Figure -17), (Figure -18) and (Figure -19) present the crystallographic findings for the three samples, Passivated, Ni-Coated, Ni-Cr Coated respectively. Each sample exhibits a distinct diffraction pattern that graphs structure against count. Unique peaks represent different substances, and various forms of the same material are identified by separate peaks. Diffracted peaks of Cr compound chromium oxide  $\text{Cr}_2\text{O}_3$  is observed at  $44.24^\circ$  and  $66.09^\circ$  which corresponds to (1 1 0) and (2 0 0) crystallographic diffraction respectively. Its corundum-type structure,  $\text{Cr}_2\text{O}_3$  crystallizes in a rhombohedral lattice, which allows the identification of various crystallographic planes, including (110) and (200). These planes indicate specific arrangements of chromium and oxygen atoms within the crystal lattice. The most intense peak of Ni compound nickel oxide NiO is observed at  $52.37^\circ$  corresponds to (2 0 0) diffracted planes which matches with cubic structure of Ni. Diffracted peaks of Fe compound iron oxide  $\text{Fe}_3\text{O}_4$  which is also known as magnetite is observed at  $44.70^\circ$  which corresponds to (2 2 0) crystallographic structure. The presence of these peaks confirms the spinel structure of magnetite and helps in determining its phase purity and crystallinity.



**Figure -17:** XRD analysis of Passivated Specimen



**Figure -18:** XRD analysis of Ni- Coated Specimen



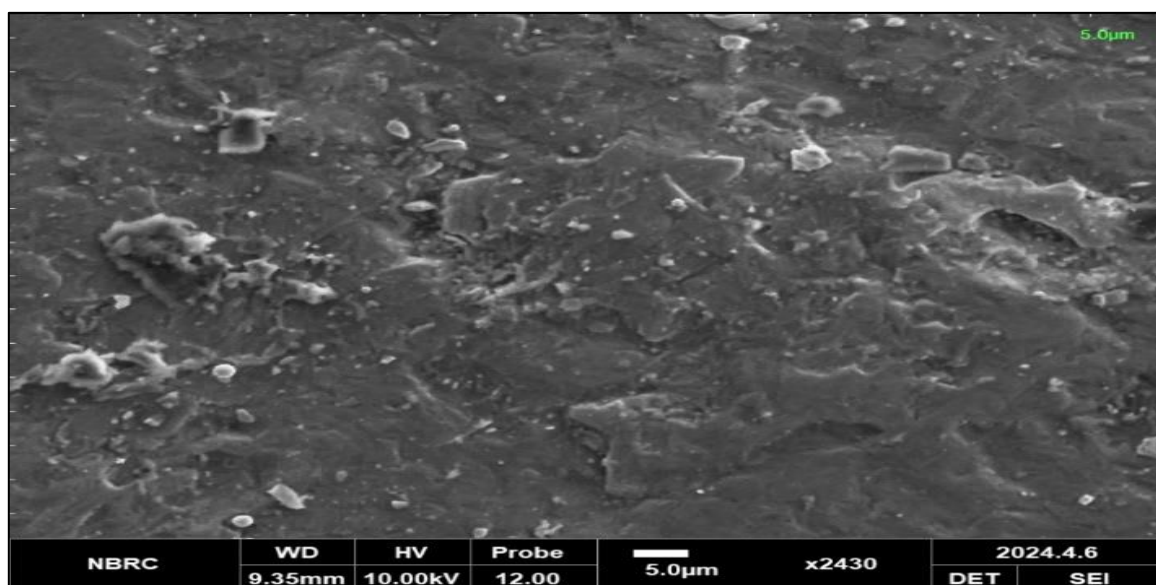
**Figure -19:** XRD Analysis of Ni-Cr coated Specimen

#### 4.6 Scanning Electron Microscopy and EDS Analysis:

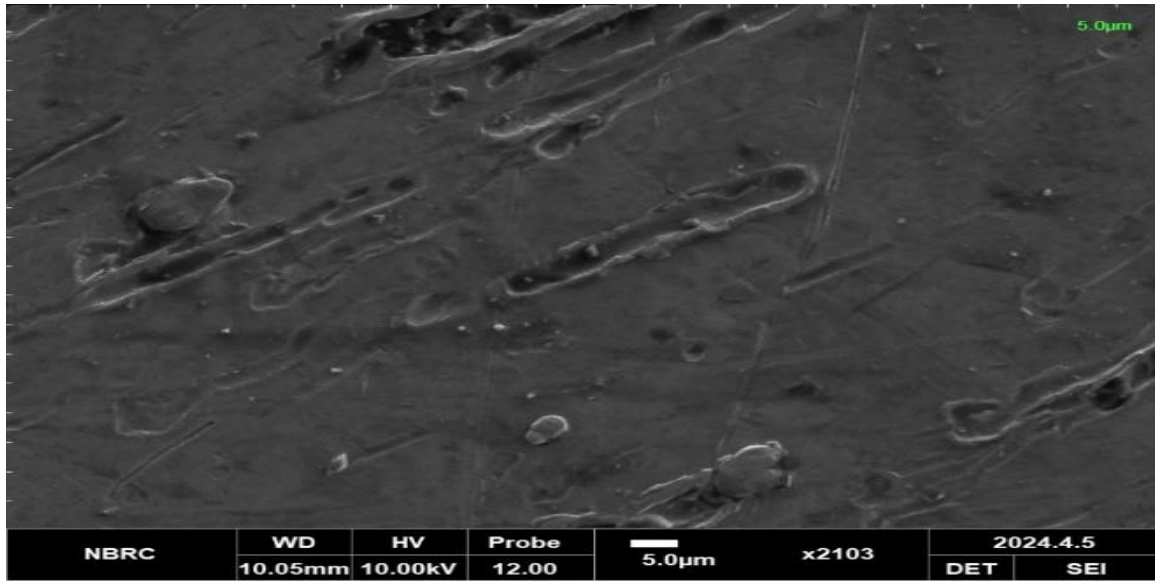
Scanning electron microscopy (SEM) is employed to investigate the morphology of materials at the nanoscale level. It provides the capability for high magnification imaging, allowing for detailed analysis of structure, porosity, and other related factors. The effectiveness of SEM is greatly improved when paired with energy dispersive spectrometry (EDS), which allows for accurate determination of the chemical composition of the specimen being examined. In this study, SEM and EDS analysis were performed only on coated samples to analyze coatings adhesion properties.

EDS analysis of dual coated Ni-Cr electroplated sample displayed in (Figure -25) shows a tight layer of Cr which is about 99.61% along with traces of iron. A dense layer of Cr and chromium oxides, such as CrO, Cr<sub>2</sub>O<sub>3</sub>, and FeOOH, forms on the surface. This layer significantly enhances corrosion resistance. The EDS analysis displayed in (Figure -24) reveals the elemental composition of the Ni electroplated on ASTM-A516 grade 70 steel. The passive top layer consists of nickel, nickel oxide (NiO), and nickel hydroxide (Ni(OH)<sub>2</sub>), resulting in a smooth, decorative nickel layer that contains 98.32% Ni, along with trace amounts of iron from the substrate. EDS analysis of passivated specimen as shown in (Figure -23) gives the spectrum of elements present in outer layer of substrate. The concentration of iron is significantly higher at 87.56% compared to other elements, which are present only in trace amounts. The top layer consists of various crystalline oxyhydroxides, including Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>·H<sub>2</sub>O, Fe<sub>3</sub>O<sub>4</sub>. Each of these compounds contributes to the overall properties of the layer, influencing factors such as corrosion resistance and stability. These oxyhydroxides can form under different conditions and play a significant role in the surface characteristics of the material. Passivated sample image obtained by SEM displayed in (Figure -20) shows rough oxide layer and irregular sub surface layers due to porosity. For nickel electroplated sample image obtained by SEM displayed in (Figure -22) shows uniform and smooth surface. Dual coated sample SEM image which is displayed in (Figure -21) shows low porosity and less defective sub surfaces layers which plays important role in corrosion protection.

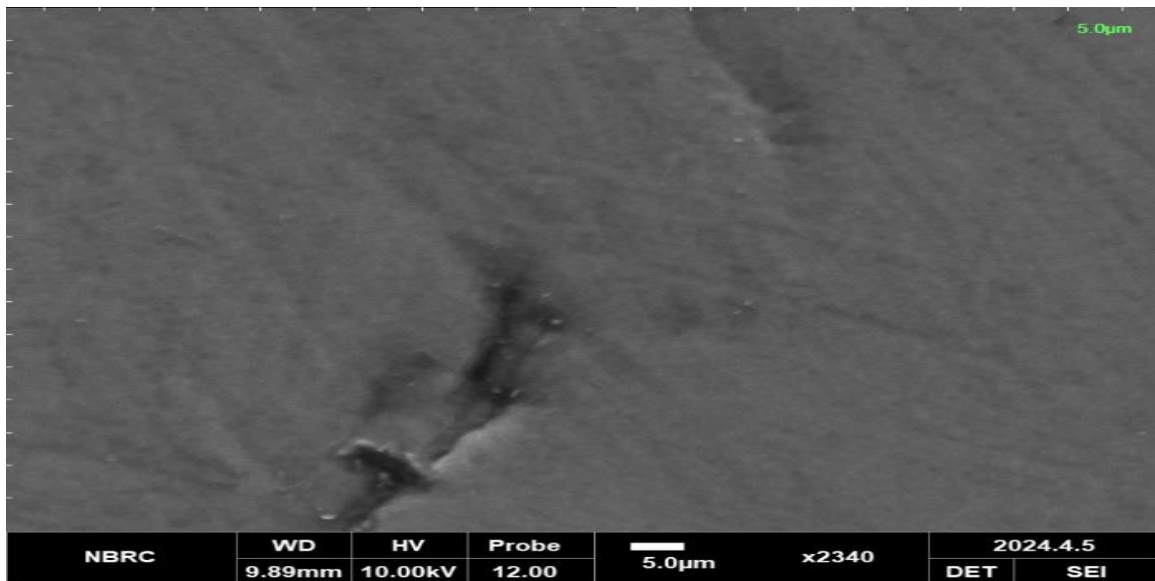
#### 4.6.1 Scanning electron microscopy (SEM) Results



**Figure -20:** SEM image at x2430 resolution of Passivated specimen



**Figure -21:** SEM image at x2430 resolution of Ni-Cr Coated specimen



**Figure -22:** SEM image at x2430 resolution of Nickel Coated specimen

#### 4.6.2 Energy Dispersive Spectrometry Results

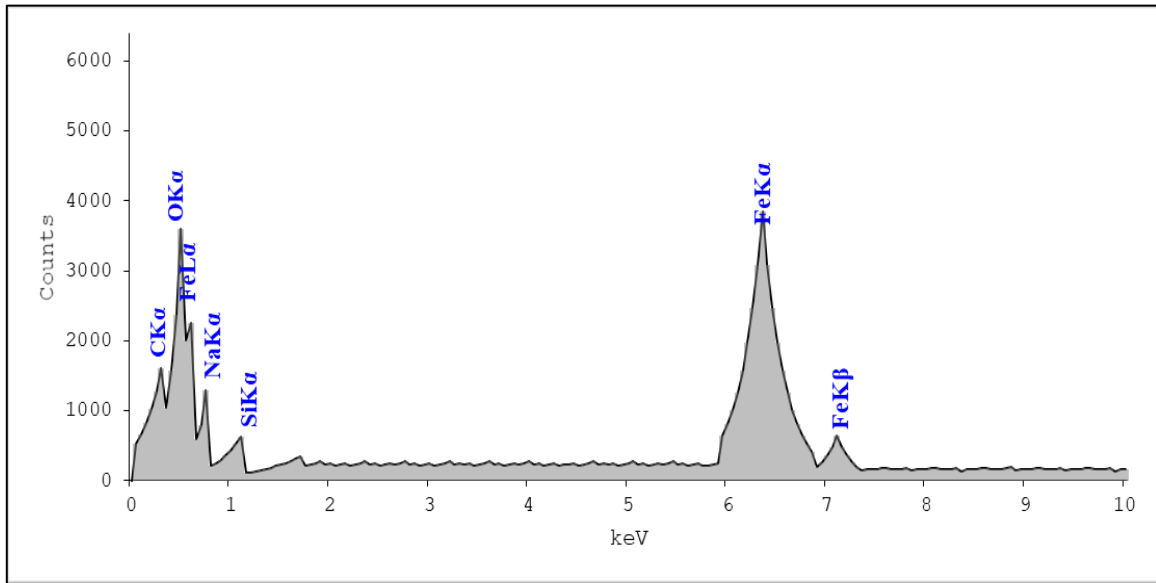


Figure -23: EDS analysis of Passivated Specimen

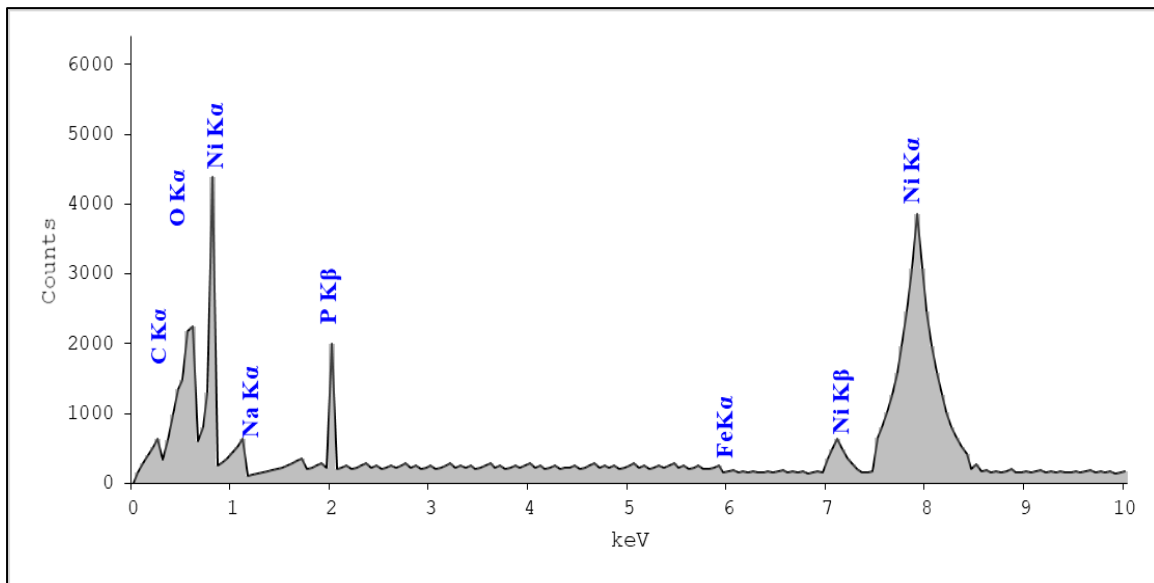
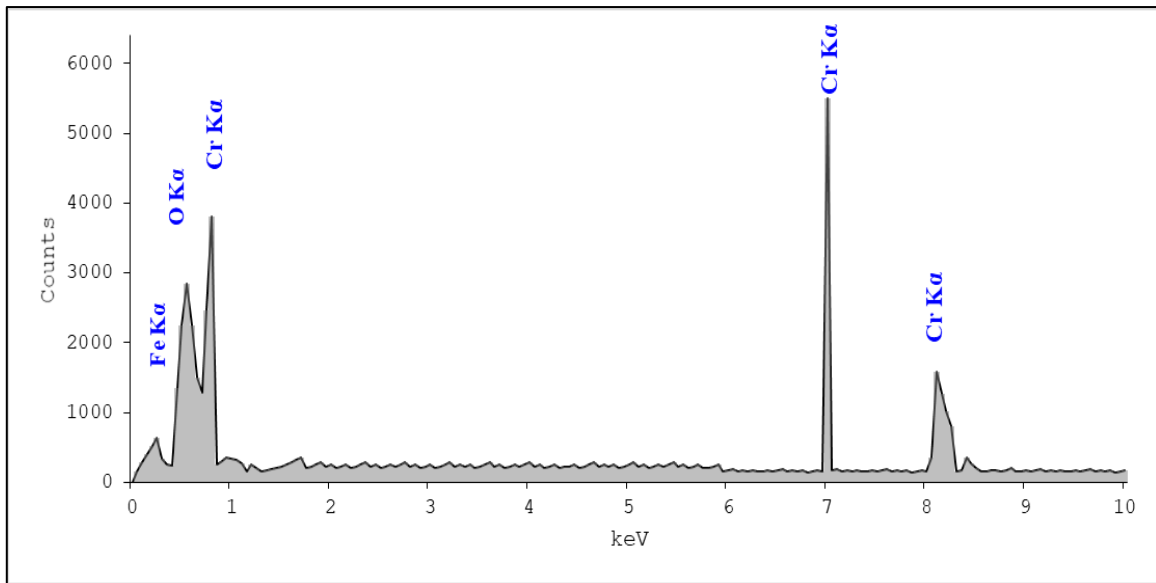


Figure -24: EDS Analysis of Ni-electroplated Specimen





**Figure -25:** EDS Analysis of Ni-Cr electroplated Specimen

# CHAPTER 5: CONCLUSION AND FUTURE WORK

## 5.1 Conclusion

Weight loss analysis indicates that the dual-coated samples demonstrate exceptional corrosion resistance, with only a 0.2% reduction in initial weight. This suggests that the dense, smooth layer of chromium and chromium oxide on the surface serves as an effective barrier against corrosion. In comparison, the nickel-coated samples also performed admirably, with a weight reduction of just 0.7%, indicating that nickel coating provides corrosion resistance comparable to that of chromium. The passivated sample demonstrates strong resistance to corrosion due to the magnetite protective layer formed on its surface. This research work can reform industrial practices and material selection, ultimately leading to improved performance and reliability in various applications, particularly those exposed to corrosive conditions.

## 5.2 Limitations

- Human intervention during experimental research can introduce errors when collecting data or conducting experiments, leading to variations in findings from one individual to another.
- Non-Calibrated tools, machines and testing equipment's can impact experimental results.

## 5.3 Future work

- Advanced methods, such as electrochemical impedance spectroscopy (EIS), can be used for more comprehensive assessment of corrosion behavior to determine rate of corrosion more effectively.
- A comparative analysis can be conducted on dual or triple coatings using other metals such as zinc and copper.
- Impact of coatings on mechanical properties such as surface hardness or tensile strength of specimen can be studied.

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