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Faculté des sciences et de la technologie  
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Le :    /    /2020

## **Effect of an organic additive on the behavior of an electrodeposited Ni-Co-SiC composite coatings**

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### **Jury :**

|     |                        |     |                      |            |
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# DEDICATIONS

*I modestly dedicate this work to:*

*For science*

*For my country and my family,*

*For my teachers and everyone who participated from near or far to  
conduct this scientific research, as well as all my friends.*

**MOHAMMED**

# Thanks

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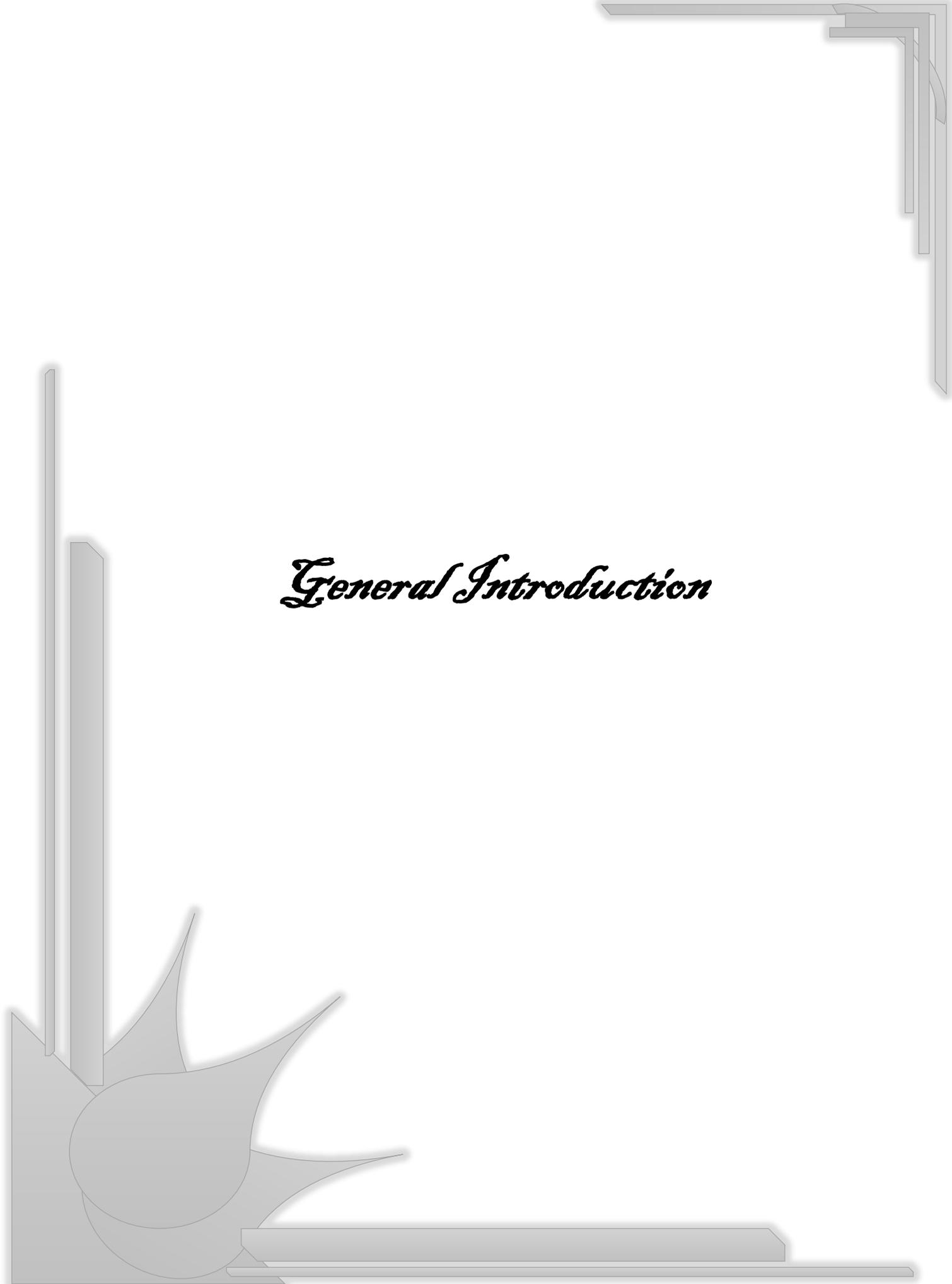
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**ملخص**

**Abstract**



# *General Introduction*

## **General Introduction**

The study of corrosion phenomena is a field of materials science which calls upon notions of chemistry as well as physics. Corrosion is an important industrial problem because it can be the cause of serious accidents following the rupture of a corroded part or structure. On the other hand, corrosion represents a very significant economic cost. In fact, it is estimated that every second, 5 tons of steel in the world are deteriorated by corrosion, which represents the equivalent of 2% of the global gross product [11].

Electroplating refers to a film growth process that involves the formation of a metallic coating on a base material occurring by electrochemical reduction of metal ions from an electrolyte.

The surface treatment is not limited to protection against wear or corrosion, but extends to many other areas, such as the development of luminescent, bactericidal, non-stick, self-cleaning, high performance surfaces thermal or mechanical, etc [14].

Consequently, the study of corrosion phenomena and the means of protection which at least make it possible to reduce its effects if it cannot be completely eliminated represents a certain interest for manufacturers both from the point of view of industrial safety and economic. Among the means which are frequently used to combat corrosive phenomena, mention may be made of the coatings of the parts to be protected. Composite coatings being the most effective. Indeed, composite coatings are experiencing increasing industrial development thanks to the particular properties conferred on them by the incorporation of solid particles. [11]

One of the most remarkable solutions which meets the requirements is the development of materials coated with thin layers. The properties of a deposited film may be better than that of the substrate.

Electrolytic deposits are obtained by the technique of electroplating. These coatings give the metal thus coated better mechanical, electrochemical and thermal properties.

The present work therefore consists in studying the effect of an organic additive on the behavior of an electrodeposited Ni-Co-SiC composite coating on previously treated steel substrates.

This memoir consists of three chapters:

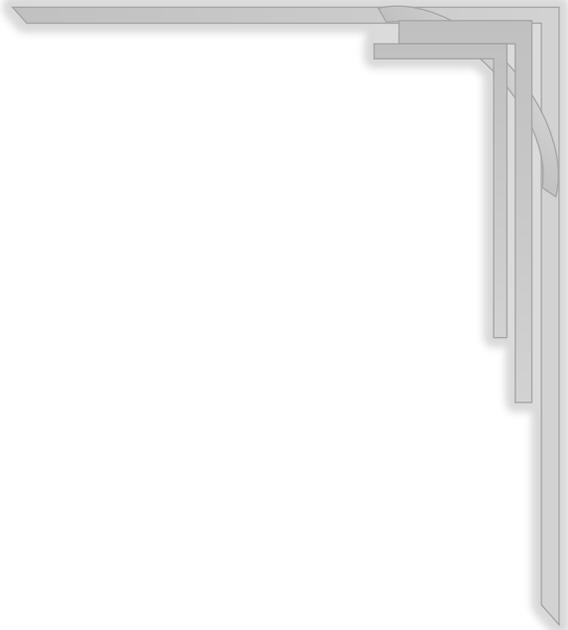
The first chapter is consecrated to the state of the art where we have presented, on the one hand, generalities on the phenomenon of corrosion, on the other hand, we have studied the electrodeposition process.

In the second chapter, we have detailed the different experimental devices for the different treatments and preparations of the substrates and then the development of the different Ni-Co-SiC composite coatings at different concentrations of solid SiC particles in the presence of organic additive in order to study its effects on behavior of elaborate coatings.

Several characterization tests were carried out to study the additive effect on the behavior of the coatings developed, namely: the adhesion test, the morphology by scanning electron microscopy (SEM), the resistance to corrosion in the corrosive environment, microhardness and X-ray diffraction (XRD).

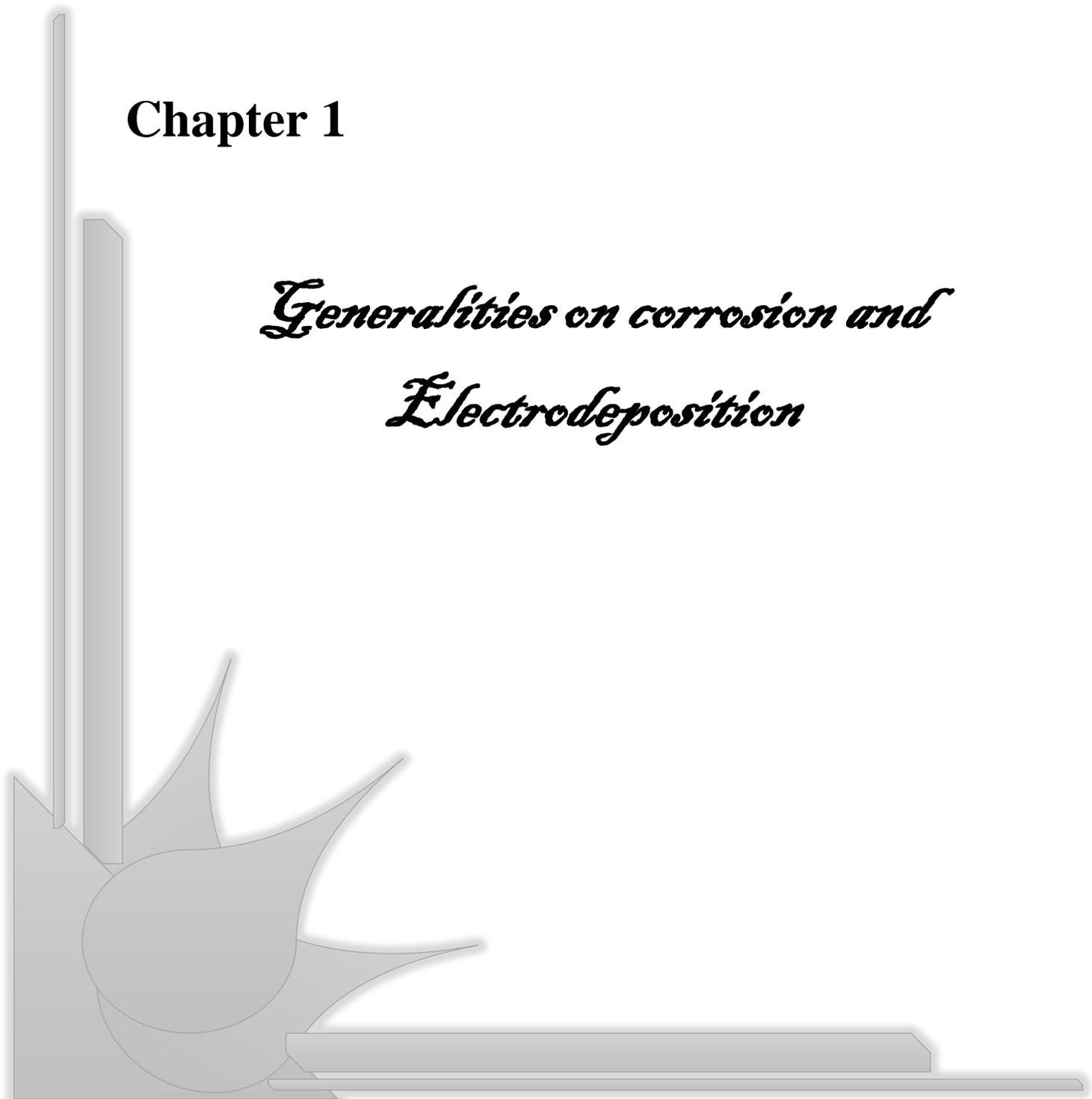
In the third chapter, we present the interpretation and the discussion of the different results obtained.

This study is completed by a general conclusion which recalls the different points described in the thesis and the main result.



# Chapter 1

## *Generalities on corrosion and Electrodeposition*



**I.1. Generalities on corrosion****I.1.1. Definition**

Corrosion is the deterioration or destruction of metals and alloys in the presence of an environment by chemical or electrochemical means. In simple terminology, corrosion processes involve reaction of metals with environmental species. As per IUPAC (International union of pur and applied chemistry), “Corrosion is an irreversible interfacial reaction of a material (metal, ceramic, polymer) with its environment which results in its consumption or dissolution into the material of a component of the environment. Often, but not necessarily, corrosion results in effects detrimental to the usage of the material considered. Exclusively physical or mechanical processes such as melting and evaporation, abrasion or mechanical fracture are not included in the term corrosion” With the knowledge of the role of various microorganisms present in soil and water bodies, the definition for corrosion need be further widened to include microbially-influenced factors [1].

**I.1.2. THE CONSEQUENCES OF CORROSION**

The consequences of corrosion are many and varied and the effects of these on the safe, reliable and efficient operation of equipment or structures are often more serious than the simple loss of a mass of metal. Failures of various kinds and the need for expensive replacements may occur even though the amount of metal destroyed is quite small. Some of the major harmful effects of corrosion can be summarized as follows:

1. Reduction of metal thickness leading to loss of mechanical strength and structural failure or breakdown. When the metal is lost in localized zones so as to give a cracked structure, very considerable weakening may result from quite a small amount of metal loss.
2. Hazards or injuries to people arising from structural failure or breakdown (e.g. bridges, cars, aircraft).
3. Loss of time in availability of profile-making industrial equipment.
4. Reduced value of goods due to deterioration of appearance.
5. Contamination of fluids in vessels and pipes (e.g. beer goes cloudy when small quantities of heavy metals are released by corrosion).

6. Perforation of vessels and pipes allowing escape of their contents and possible harm to the surroundings. For example, a leaky domestic radiator can cause expensive damage to carpets and decorations, while corrosive sea water may enter the boilers of a power station if the condenser tubes perforate.
7. Loss of technically important surface properties of a metallic component. These could include frictional and bearing properties, ease of fluid flow over a pipe surface, electrical conductivity of contacts, surface reflectivity or heat transfer across a surface.
8. Mechanical damage to valves, pumps, etc., or blockage of pipes by solid corrosion products.
9. Added complexity and expense of equipment which needs to be designed to withstand a certain amount of corrosion, and to allow corroded components to be conveniently replace [2].

### **I.2. Method of determining organic additives in an electroplating bath**

the present invention is directed to a method of analyzing organic additives in electroplating baths having improved reproducibility.

Besides the inorganic components of these plating solutions (nickel ions, sulfuric acid and small amounts of chloride ions) the baths contain one or more organic additives (brighteners, suppressors and levelers). In the proper concentrations, these organic additives give a bright, smooth deposit with excellent mechanical and electrical properties [6].

Organic additives such as addition agents, in low concentrations added to electrolytes, which make it possible to improve or modify the intrinsic properties of a coating: gloss, adhesion, good resistance to corrosion.

The addition of these additives in an electrolyte is fundamental because of their influences on the wearing properties of materials. Therefore, it means that the additives modify electrocrystallization by having a specific action during nucleation and / or crystal growth. These processes subsequently lead to changes in the physico-chemical properties of the coating. Apart from technical properties, economics such as reduction of production cost should be considered when choosing the additive.

A large number of compounds are used as additives, so it is not easy to choose the right additive

to make the coating. Therefore, researchers have taken the initiative to establish a classification of additives used for the preparation of electrolytic coating according to:

- Their chemical nature (organic or mineral compound).
- Interfacial activity (surfactants)
- The size of the particles (colloids)
- The mechanism of adsorption on the substrate.
- The effects on the coating (leveling, shine, refiner ...).

### **I.2.1. Principle of action of an additive**

Organic additives which can be functionalized molecules or polymers take on different roles within an electrolyte: brightener, refiner, leveler, antioxidant, corrosion inhibitor, etc. They are liable to adsorb to the surface of the coating and have the ability to modify the crystallization of the metal. Organic additives can also have significant effects on the efficiency of the reduction reaction of metal species by influencing the evolution of hydrogen as well as the rates of the electrodeposition steps.

These changes affect not only the collective organization of crystallites (texture, dendritic structure) but also the internal organization (microstructure, morphology) of each crystallite. Therefore, the intrinsic macroscopic properties of coatings are affected beyond the appearance of the surface. Despite the great diversity of additives and their roles, three types of additive action mechanisms can be distinguished [8]:

- Surface blocking
- Reactive adsorption
- Complexation in solution

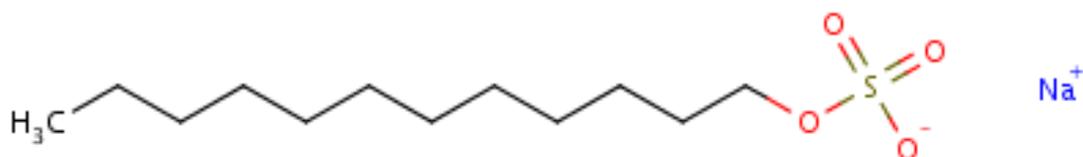
### **I.2.2. Sodium Lauryl Sulfate Profile**

Sodium lauryl sulfate (SLS) is a surfactant and emulsifier that is primarily used as a wetting agent and detergent. It is an alkyl sulfate salt, generally prepared by the sulfation of lauryl alcohol followed by neutralization with sodium carbonate. SLS is classified as an anionic surfactant alkyl sulfate detergent, and is one of the most studied and widely used surfactants. It has two moieties. One is a 12-carbon chain that is non-polar and lipophilic, and structurally similar to lauric acid. The other part of the molecule is a polar and hydrophilic (water soluble) negatively charged sulfate ion. The sodium ion increases the ability to go into aqueous solution over that of lauryl sulfate

SLS is a surfactant and emulsifier that is primarily used as a wetting agent and. The textile industry is a heavy user, using SLS to clean and prepare fabric for dyeing. It is used in separation of proteins and lipids by electrophoresis. SLS is a common ingredient in many household and personal care products, such as dishwashing and laundry detergents, toothpastes and shampoos [7].

### I.2.3. Chemical and Physical Properties

The molecular structure of SLS is presented in (Figure I.1).



(Figure I.1). Sodium Lauryl Sulfate molecular structure.

### I.2.4. Physical and Chemical Properties of Sodium Lauryl Sulfate

SLS lowers the surface tension of water. When heated to decomposition, it releases toxic sulfur oxides. Lauryl sulfate was compared with SLS in carrying out catalytic reactions on complex chemical structures. Both increased the rate of reaction, an effect that was decreased in the presence of various alcohols. SLS consistently had a higher degree of dissociation than lauryl sulfate [7].

The physical and chemical properties of SLS are presented in table (I.1).

**Table (I.1). Physical and Chemical Properties of Sodium Lauryl Sulfate**

| Property                        | Characteristic/Value  |
|---------------------------------|---|
| Molecular Formula:              | C <sub>12</sub> H <sub>25</sub> NaO <sub>4</sub> S                |
| Molecular Weight:               | 288.38  |
| Percent Composition:            | C: 49.98%; H: 8.74%; Na: 7.97%; O:22.19%; S: 11.12%               |
| Physical state at 25°C/1 Atm.   | Crystals or flakes  |
| Color                           | White or Cream  |
| Odor                            | Faint odor of fatty substances                                    |
| Density/Specific Gravity        | 0.6 g/cm <sup>3</sup>   |
| Melting point                   | 204°-207°C  |
| Boiling point                   | 588°C   |
| Solubility                      | 1 g/10 ml water   |
| Vapor pressure                  | 1.1 x 10 <sup>-12</sup> mm Hg at 25°C                             |
| pH                              | Neutral to alkali (8.5-11.0 in a 1% aqueous solution)             |
| Octanol/Water (Kow) coefficient | 1.6   |
| Viscosity                       | Not determined but expected to be close to the viscosity of water |
| Miscibility                     |   |
| Flammability                    | Does not contain combustible liquids                              |
| Storage stability               | Stable beyond one year  |
| Corrosion characteristics       |   |
| Air half life                   | 0.7 hr  |
| Soil half life                  | 77.5 hrs  |
| Water half life                 | 19.8 hrs  |
| Persistence                     | 595 hrs   |

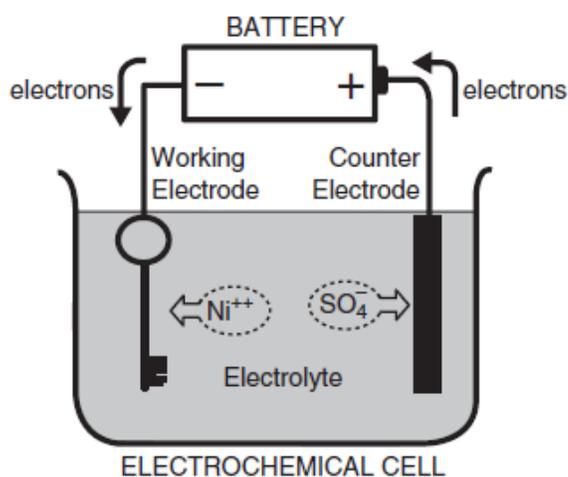
### I.3. Electrodeposition

#### I.3.1. Introduction

Most important concepts and techniques of an electrodeposition are introduced and described here in an easy-to-understand way. Examples of technological applications are given, with emphasis on the fabrication of nanostructures.

#### I.3.2. DEFINITIONS AND HISTORY

Electrodeposition is an electrochemical process that allows the preparation of solid deposits on the surface of conductive materials. It is a commercially highly relevant process, providing the basis for many industrial applications, such as electro-winning, refining, and metal plating. Metal plating is the process that has perhaps the closest contact with most people's everyday life, because we are surrounded by things that have a protective or decorative coating, such as watches, buttons, belt buckles, doorknobs, handlebars, etc. that consists of two conductive or semi-conducting electrodes immersed in an electrolyte. The electrodes are called the working electrode (cathode), consisting of the object where electrodeposition is planned, and the counter-electrode (anode), necessary to complete the electrical circuit. Electrolytes for electrodeposition are usually aqueous solutions containing positive and negative ions, prepared by dissolving metal salts an electrochemical cell with a battery is illustrated in Fig.I.2, where the motion of the ions toward the electrodes is also sketched.

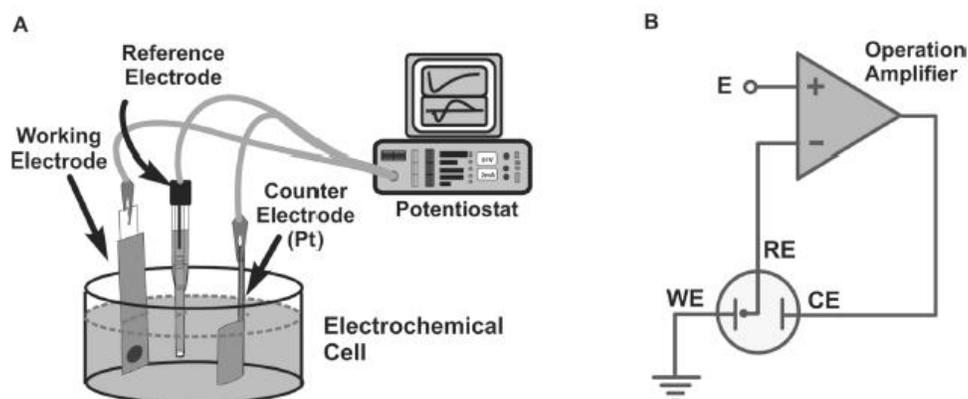


**Figure (I.2). Schematic diagram.**

Positively charged nickel ions in the electrolyte are attracted by the negatively charged key (working electrode). At the surface of the key they are reduced by gaining two electrons, and metal is deposited.

### I.3.3. ELECTRODEPOSITION APPARATUS AND CONCEPTS

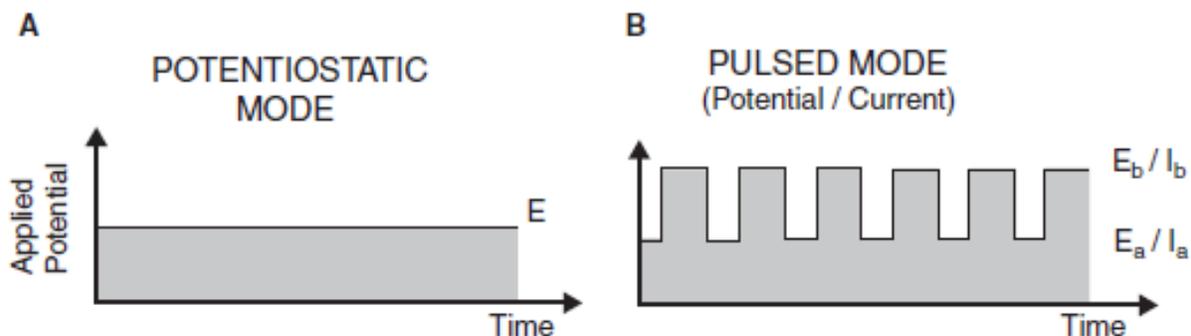
Electrodeposition on the industrial scale requires an electrochemical cell and a DC current power supply. This approach is relatively simple and inexpensive, and is known as galvanostatic plating system, because the current between the electrodes is controlled (maintained constant). Another important deposition mode is the potentiostatic one.



**Figure (I.3). (A) Electrochemical cell with three electrodes connected to a potentiostat (B) Electronic sketch illustrating the mode operation of a typical potentiostat.**

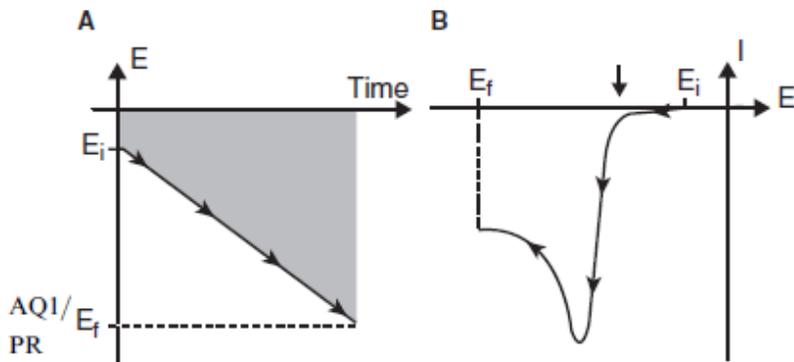
Fig.I.3 (A) shows an electrochemical cell with three electrodes (working-, reference-, and counter-electrodes) and a potentiostat. The potentiostat is an electronic apparatus that maintains the potential difference between the working- and reference electrodes by controlling the potential difference between the working- and counter-electrodes. Fig.I.3 (B) shows a block diagram of the electronic circuitry of a potentiostat with an operational amplifier that keeps the voltage between reference electrode (RE) and working electrode (W) equal to the applied voltage E at the positive terminal, by regulating the cell potential between W and counter-electrode (CE). By convention W is connected to ground. The three-electrode cell and the potentiostat enable the potentiostatic mode of deposition mentioned above. The potentiostatic mode means that the potential of the working electrode is kept constant during the experiment or deposition process.

As depicted in Fig.I.4 (A), an additional mode called pulsed deposition is also illustrated in Fig. I.4 (B). In this mode, for pulsed potential, the potentiostat switches electrode potential between two values in order to have the potential varying as a square wave. For pulsed current deposition, a current source with a square wave output is sufficient. The three-electrode cell and potentiostat is also a powerful experimental tool for electrochemical investigations, permitting the implementation of different techniques, such as voltammetry.



**Figure (I.4). Illustration of the potential as a function of time for potentiostatic and pulsed deposition modes.**

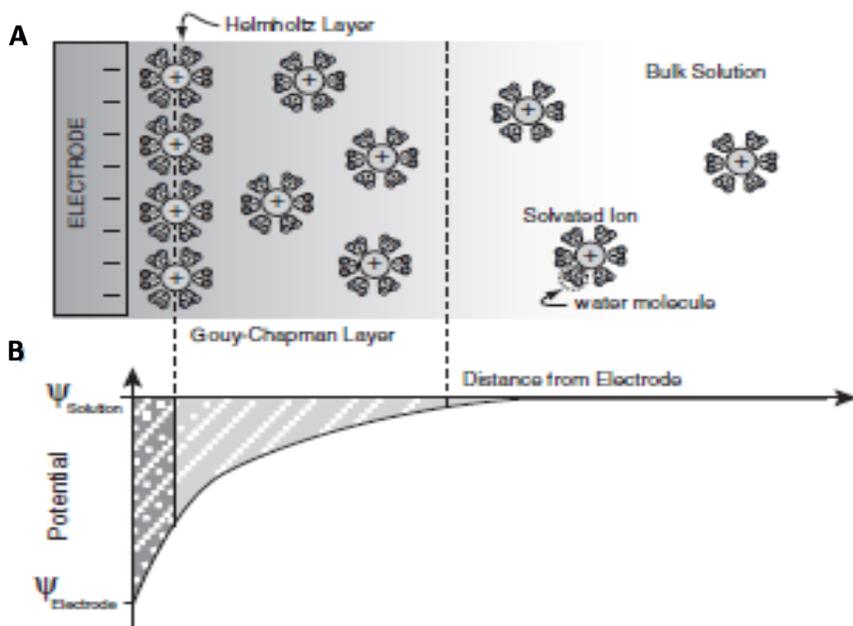
This technique consists of applying a potential ramp to the working electrode, which is achieved by applying a potential ramp to the positive terminal of the operational amplifier [of Fig.I.3 (B)], and measuring the resultant cell current. When the applied potential starts at a defined level and comes back to the same value after a period of time, the technique is called cyclic voltammetry. When the applied potential starts at a level 1 and goes to a level 2, the resulting plot of the current versus the potential, is called a polarization curve or simply voltammogram. Fig.I.5 (A) illustrates a potential ramp applied to the working electrode, and Fig.I.5 (B) the corresponding plot for the variation of the cathodic current as a function of the cathodic potential. To the maximum current at a given rate of change of potential (also known as the reduction peak).



**Figure (I.5). Illustration of the applied potential ramp (A) to obtain a typical polarization curve (voltammogram) of metal deposition on a metal working electrode.**

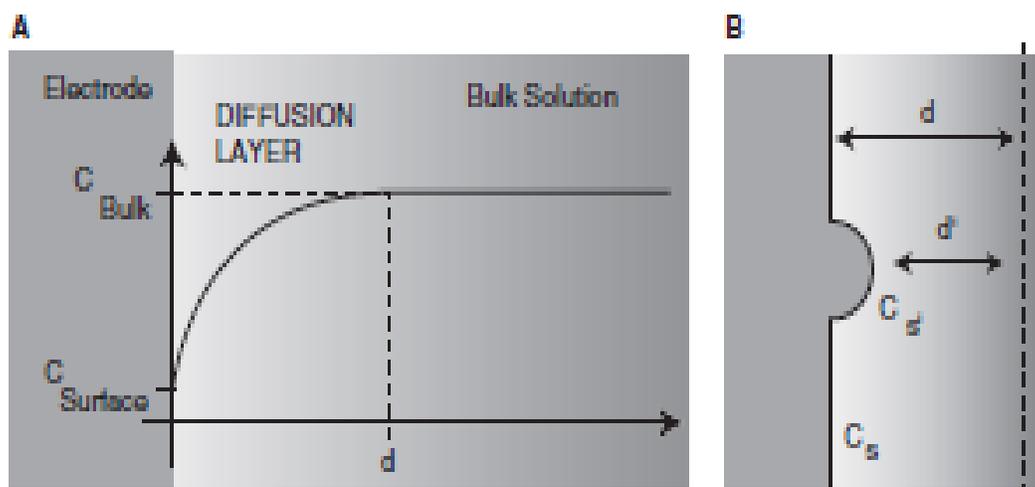
### I.3.4. Mass Transport

Electrodeposition has the ability to produce a relatively uniform distribution of metal upon a cathode of irregular shape. Though the uniformity depends on the distribution of electric fields inside the electrolyte toward the surface of the electrode, other important factors have to be considered. The addition of agents (additives) to the electrolyte, for example, can affect the microscopic mechanism of electrodeposition, reducing the roughness of the deposit and producing a visual effect known as brightening. Illustrated in Fig.I.6, will be formed Fig.I.6 (A) shows the double layer in greater detail. When the exchange process reaches equilibrium the double layer thickness depends on the physical and chemical properties of the electrode-electrolyte interface. In general, the description of the double layer considers the existence of two regions named the Helmholtz layer, a monolayer of ions on the surface of the electrode, and the Gouy-Chapman layer.



**Figure (I.6). Illustration of the electric double layer (A) and potential drop (B) near the surface of the electrode.**

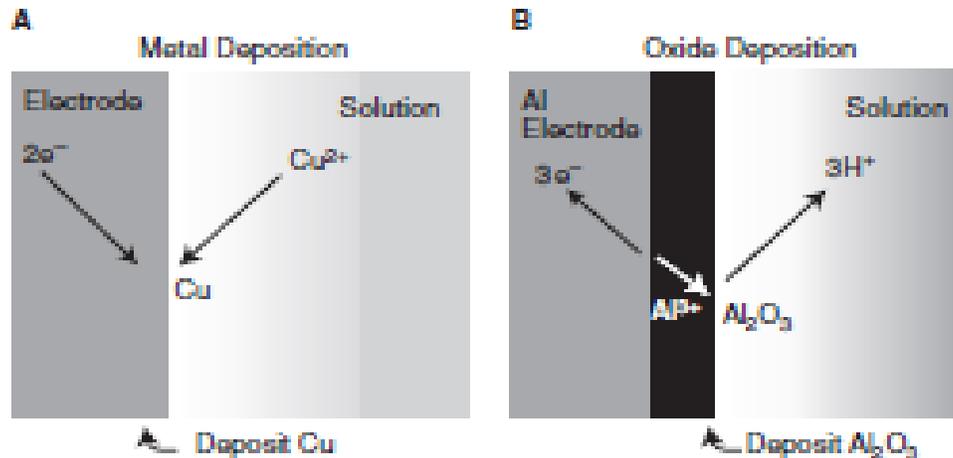
Fig.I.7 (A) is a plot of the concentration of an ionic species as a function of the distance from the surface of the electrode, showing the diffusion layer. The consumption of ions because of metal deposition generates a concentration gradient that, in steady-state conditions, is essentially determined by the redox reaction rate. If the consumption of ions arriving at the surface by diffusion is very high, the concentration of ions at the surface  $C_S$  is effectively zero, and the deposition process is controlled by diffusion. If the consumption is low, then the ion concentration at the surface is different from zero and the deposition is controlled by kinetics, i.e., by the velocity of the reaction. Electrodeposition in the diffusion-limited regime is very sensitive to asperities on the surface of the electrode, as shown in Fig.I.7 (B). These morphological structures reduce the size of the diffusion layer, thereby increasing the concentration gradient and hence the current.



**Figure (I.7).** (A) Idealized profile of the concentration gradient near the surface of the electrode and (B) an illustration of a deposition instability in the presence of an asperity.

### I.3.5. Electrode Reactions

The current that flows at the working electrode may be divided into two kinds: faradaic and non-faradaic. The faradaic processes are the ones where charges are transferred across the liquid–solid interface. These processes are called faradaic because they follow Faraday’s law. Two faradaic processes that are directly related to electrodeposition, where Fig.I.8 (A) represents simply the deposition of Cu by reduction of  $\text{Cu}^{2+}$  and Fig.I.8 (B) the growth of Al anodic oxide by oxidation of metallic aluminum, this being an example of anodic electrodeposition. Non-faradaic processes are structural changes of the electrode–solution interface, such as absorption and desorption of species that change the potential of the electrode and solution composition without charge transfer [4].



**Figure (I.8). Electrode reaction for (A) the cathodic electro-deposition of Cu and (B) the anodic electro-deposition.**

#### I.4. Phenomenon of co-deposition

Theoretically, the electro crystallization of alloys results from a co-deposition of at least two metals that can crystallize in one phase of the phase diagram. However, certain conditions of electro crystallization favor the solutions of famous metals insoluble. Electro crystallization makes possible the existence of phases outside their thermal stability as well as that of intermetallic compounds which are otherwise unknown. In some cases, the electrodeposited alloys can be obtained with grain fineness up to an amorphous structure. However, the electro crystallization of alloys is more complex to study than that of pure metals, co-deposition phenomena are more complex and cannot be found rigorous explanations. At present, there are very few fundamental studies concerning the mechanisms of co-deposition and the phenomena involved.

##### I.4.1. Different types of co-depositions

The baths themselves do not have any specific characteristics. It's the process that characterizes each type of co-deposition with the exception of code position equilibrium. There are five co-deposition modes that we classify next the influence of certain factors.

**I.4.1.1. Regular co- deposition**

It is characterized by a deposit under control of the diffusion of the cathodic reaction of the most noble metal.

**I.4.1.2. Irregular co-deposition**

It is also characterized by diffusion-controlled deposition and is symptomatic systems where the thermodynamic potentials of the elements of the alloy are very close together and where the metals form intermetallic compounds or solid solutions. In this case, the kinetic factors and especially the thermodynamic factors play a role.

**I.4.1.3. Co-deposition in equilibrium**

It is characterized by an identical ratio between the metallic elements in the solid phase and in the electrolyte.

**I.4.1.4. Abnormal co-deposition**

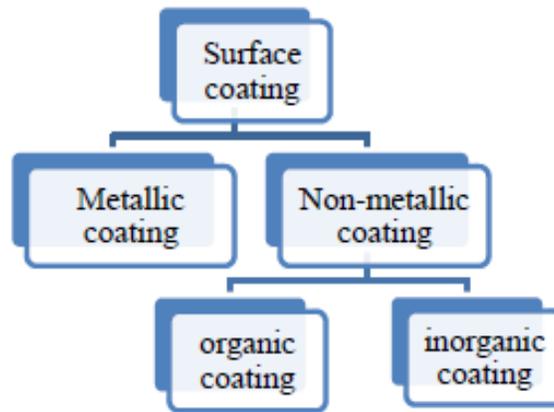
In the case of anomalous co-deposition, the least noble element is deposited preferentially. This type of co-deposition is rare and in most cases is related to code position a member of the iron group (iron, cobalt, nickel). This is the case of ZnNi alloys, ZnCo, CoFe ...etc.

**I.4.1.5. Co-deposition induced**

It takes place for certain metals that normally cannot be deposited alone aqueous solution such as germanium, molybdenum or tungsten. Co-testimony induced is characterized by the electroplating of its species as a component of an alloy (usually allied to an element of the iron group). This is the case of CoFeMo [5].

### I.5. Surface coating

There are two types of surface coating:



**Figure (I.9). Surface coating.**

#### I.5.1. Metallic coating

The structure is coated with a layer of other metal which may be nobler than the structure or less noble than it e.g. steel structures can be coated with copper which is nobler than steel or zinc which is less noble. In case of coating the structure with a more noble metal care should be taken that the coat is free from pores or cracks to avoid the formation of dissimilar metal corrosion cells which would lead to corrosion of the structure. [3]

#### I.5.2. Factors that must be considered in selection of a coating metal

1. The coating should be able to resist direct attack of the environment.
2. The coating should be nonporous and continuous (no cracks) to avoid acceleration of corrosion especially in case of a more noble metal (ex: coating of Fe by Cu).
3. The coating should be hard.

In case of coating the structure with a less noble metal the presence of pores and cracks in the coat is not dangerous because in this case the less noble metal will corrode by the formation of dissimilar metal cells while the structure will remain protected.

### I.5.3. In practice metallic coating is carried out by different methods such

- (1) Electroplating,
- (2) Hot dipping of the work piece in molten metal covered with a flux,
- (3) Spraying of the molten metal on the work piece.

#### I.5.3.1. Electroplating

- Electroplating is the method of coating one metal with another. It is most commonly used for decorative purposes, appearance and protection.
- Electroplated items include chrome bumpers, jewelry, electronics, circuit boards and airplane parts.

##### I.5.3.1.1. Factors affecting coating quality

The quality of coating depends on:

1. Average thickness of coating
2. Porosity and continuity
3. Uniformity of thickness

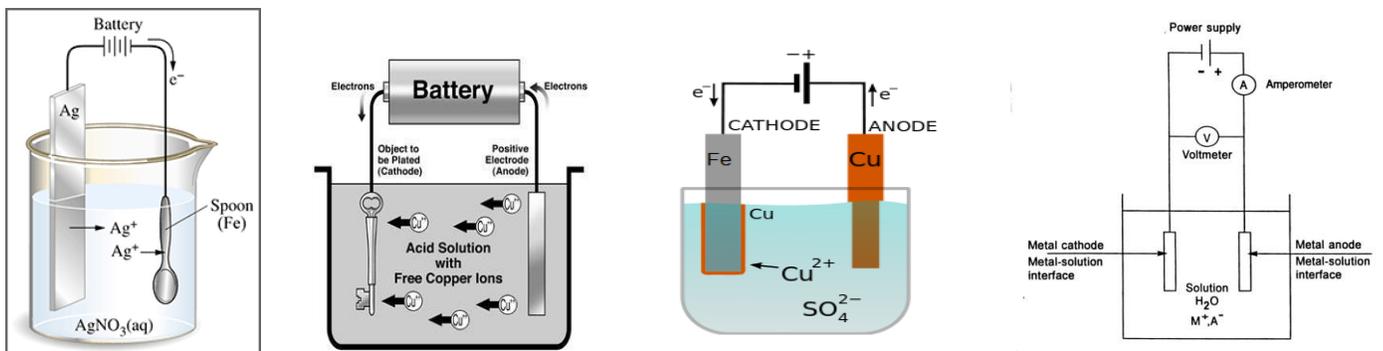


Figure (I.10). Some examples for electroplating process.

**I.5.1.2. Non-metallic coating**

There are two types of nonmetallic coating:

**I.5.1.2.1. Inorganic coating****i. Oxidation (passivation)**

Steel can be coated with an oxide film by a). Heating at high temperature, b). Chemical oxidation by treating steel with hot alkaline nitrate, or persulphate or perchlorate c). Anodic oxidation by making the steel structure an anode in electrolytic cell.

**ii. Phosphating**

Steel is coated with a layer of iron phosphate by dipping in a solution containing phosphoric acid and zinc phosphate. The iron phosphate film is not highly protective because it is porous so it usually covered with paint. The phosphate film improves the bond between the metal and the paint.

**iii. Enamels**

Enamels are glassy layer applied to the metal by dipping it in a suspension of powdered glass, and then the metal is heated in a stove (furnace) at high temperature where the glass powder melts and coat the metal.

**iv. Cement coating**

It is used to coat the inner side of steel pipelines carrying water or wastewater.

**I.5.1.2.2. Organic coating**

Organic coating such as the following:

**i. Paints**

Paint consists of :

1. a film forming substance such as linseed oil or a polymer (resin)
2. an organic solvent
3. a pigment (usually an inorganic oxide or metal powder)

Before applying paint to a steel surface, the metal surface should be cleaned of oxides by sand blasting or acid pickling. After cleaning, the metal surface is coated with a thin layer of primer.

**Note:**

**Primer:** A primer is a paint containing a pigment such as lead oxide ( $Pb_3O_4$  red lead) or zinc chromate which oxidizes the steel surface and inhibits its corrosion. Besides, the primer film increases the strength of the bond between steel and final paint film. When the primer film dries a thick film of the required paint is applied over the primer.

**ii. Lacquers**

A lacquer consists of a thermoplastic polymer dissolved in an organic solvent. Lacquers can be used to line steel tanks holding corrosive chemicals such as acids.

**iii. Coal tar**

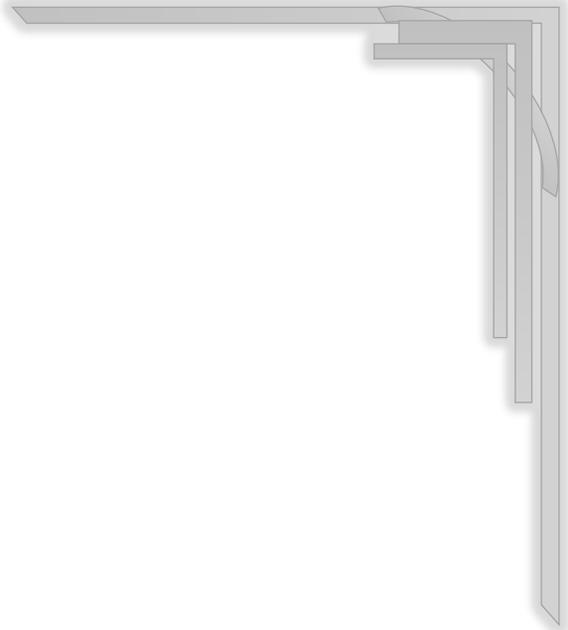
Coal tar is a brown or black liquid of extremely high viscosity. Coal tar is among the by-products when coal is carbonized to make coke or gasified to make coal gas. Coal tars are complex and variable mixtures of phenols, polycyclic aromatic hydrocarbons (PAHs), and heterocyclic compounds, about 200 substances in all.

**iv. Temporary coating**

It is used to protect metallic structures during shipping and storage by coating the structure with layer of lubricating oil which can be removed by an organic solvent when the structure is put to service [3].

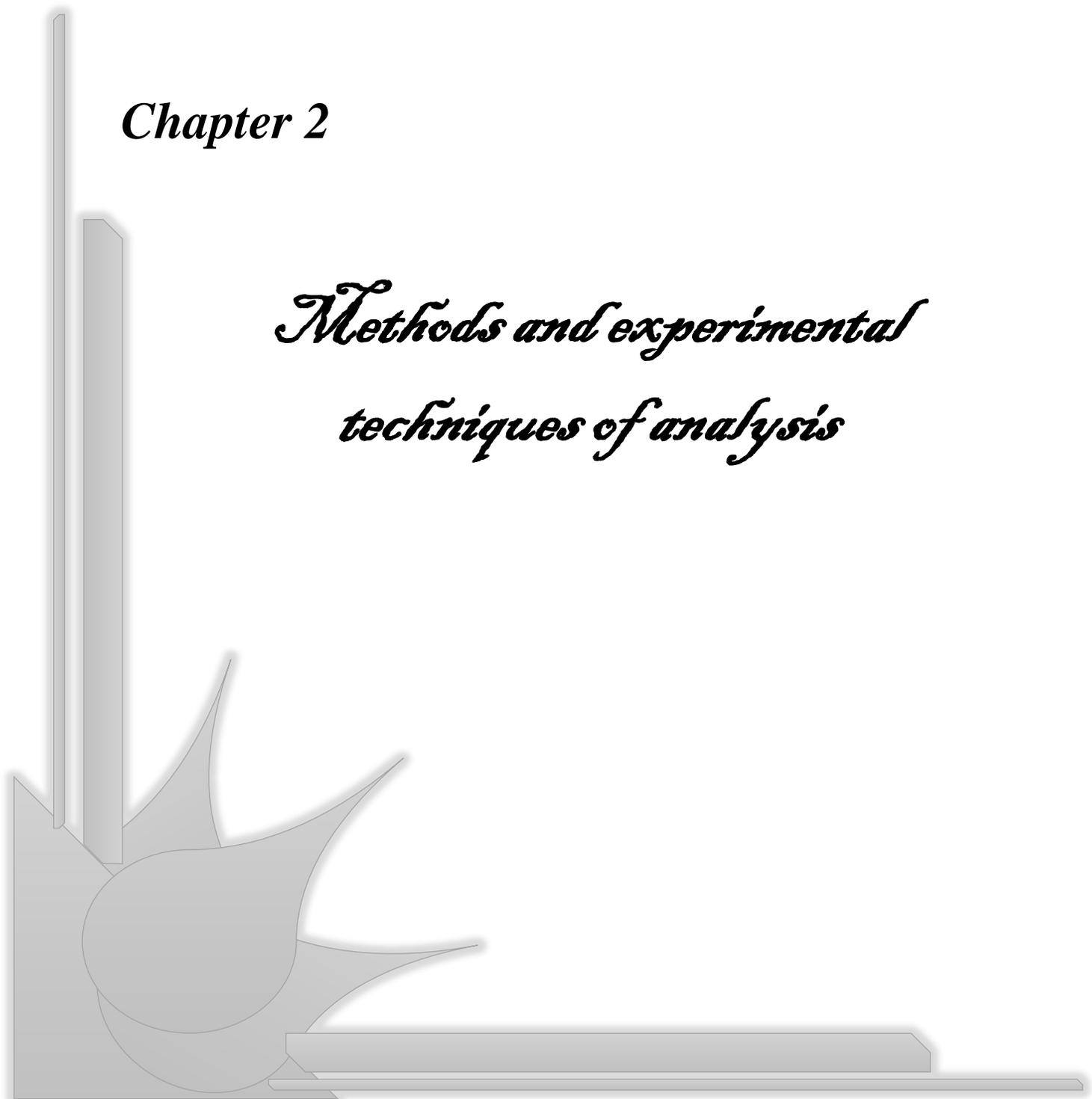
**1.6. CONCLUSIONS**

Electrodeposition is a process widely used in industry. In this entry, emphasis was given to fundamental aspects and to future potential applications of this technique [4].



*Chapter 2*

*Methods and experimental  
techniques of analysis*



## II. 1. Introduction

In this chapter we will present the use of the experimental device for sediment development and experimental procedures such as surface preparation, installation of the baths used, operating conditions, etc., in addition to use the different characterization techniques such as synthetic, morphological and electrochemical that we use to study the different coatings made.

## II.2. Experimental devices for preparing deposits

### II.2.3. Materials

#### II.2.3.1. Nickel plate

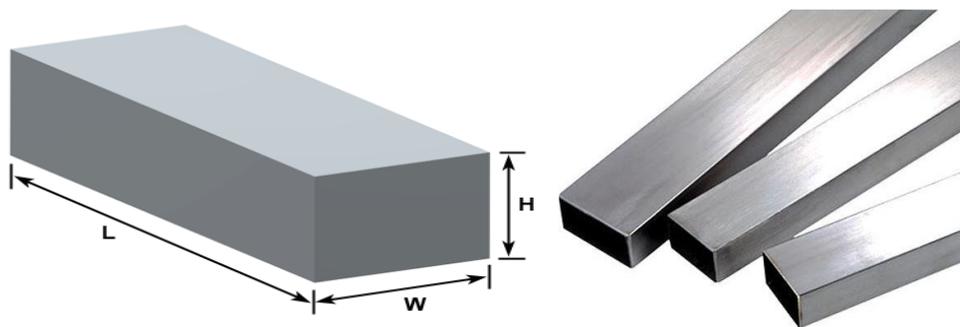
As anodes, we used two rectangular nickel plates on either side of the cathode, about 99 % purity [5].

#### II.2.3.2. Substrate used

As a substrate, we used rectangular plates of the steel type BS2 (0.19% C), The parts are rectangular in shape, It is 5 mm thick, 1 cm long and 5.8 mm wide. The following table gives the chemical composition of the substrate used (in percentage by mass).

**Table (II.1). Chemical compositions of the substrate.**

| Steel grade          |                       | C    | Si   | Mn   | P     | S     | Al    | N     | Nb   | Ti   |
|----------------------|-----------------------|------|------|------|-------|-------|-------|-------|------|------|
| Symbolic designation | Numerical designation | Max  | Max  | Min  | Max   | Min   | total | Min   | Max  | Max  |
| P265NB               | 1,0423                | 0,19 | 0,25 | 0,40 | 0,025 | 0,015 | 0,02  | 0,009 | 0,05 | 0,03 |



**Figure (II.1) Metal rectangular in shape.**

## II.2.2. Materials used

Table (II.2). Materials used

| Equipment  | Type – Model                             |
|--|--|
| <b>DC generator (Direct current generator)</b>     | Model GPS-18500.                         |
| <b>pH-meter</b>                                    | Model 903.                               |
| <b>Mechanical agitator with their magnetic bar</b> | DAIHAN LABTECH CO, LTD.                  |
| <b>Electronic balance</b>                          | Sartorius1219MP                          |
| <b>Adhesion of the deposited layer</b>             | ASTM.B571                                |
| <b>Bruker diffractometer Si</b>                    | D8 Advance model                         |
| <b>scanning electron microscope</b>                | JEOL-DAC microscope scanning electronics |
| <b>Microhardness</b>                               | Zwick/Roell Indentec                     |

## II.2.1. Chemical products

All the solutions used were prepared in distilled water with the following chemicals:

- 1 / Nickel sulfate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ )
- 2 / Cobalt sulfate ( $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ )
- 3 / Ammonium chloride ( $\text{NH}_4\text{Cl}$ )
- 4 / Boric acid ( $\text{H}_3\text{BO}_3$ )
- 5 / Sodium chloride ( $\text{NaCl}$ )
- 6 / Hydrochloric acid ( $\text{HCl}$ )

7 / Silicon carbide (SiC)

8 / Sodium dodecyl sulfate ( $C_{12}H_{25}NaO_4S$ )

9 / sodium hydroxide (NaOH)

10 / sodium carbonate ( $Na_2CO_3$ )

#### II.2.4. Procedures and experimental conditions

Before any deposition, the substrates have undergone a surface treatment. The solution is restless for 24 hours to ensure good dispersion of the particles. The elaboration conditions are determined based on the desired properties.

##### II.2.4.1. Samples preparation

Ni-Co-SiC coatings have been electrodeposited on pretreated steel substrates in order to guarantee a reproducible surface finish and to ensure good adhesion of the coating. The preparation consists of polishing, degreasing in an alkaline bath at room temperature for 3 min, then pickling in an acid solution for 2 min, these operations are always followed by rinsing with distilled water and then with acetone.

##### II.2.4.2. Polishing

The substrates have been polished using an abrasive paper of different particle sizes ranging from 120 to 1200, washed with distilled water and then stored in acetone to avoid contamination.

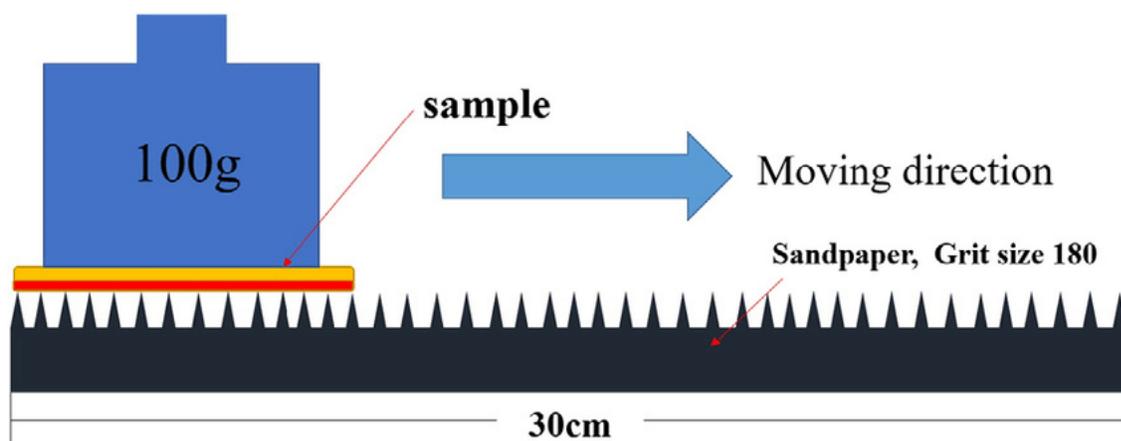


Figure (II.2) Diagram metal is polished using an abrasive paper.

### II.2.4.3. Electrolytic degreasing

For the degreasing operations, we used an alkaline solution containing a mixture of sodium hydroxide (NaOH) and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>). The operation is carried for 3 minutes at a current density of the order of 3 A / dm<sup>2</sup> and at room temperature.

**Table (II.3). Operating conditions and chemical composition of the degreasing bath.**

|                             |                                      |         |
|-----------------------------|--------------------------------------|---------|
| Chemical compositions (g/l) | NaOH                                 | 11.5    |
|                             | Na <sub>2</sub> CO <sub>3</sub>      | 53      |
| Operating conditions        | Current density (A/dm <sup>2</sup> ) | 3       |
|                             | Time (min)                           | 3 min   |
|                             | Temperature (°C)                     | Ambient |

### II.2.4.4. Chemical pickling

This operation was carried out in hydrochloric acid (10% by volume). The samples were completely immersed in the acid solution for 2 min.

### II.2.4.5. Rinsing

After performing the chemical pickling, the samples were immediately rinsed in distilled water, acetone and then rinsed with the plating bath solution.

### II.2.4.6. Plating baths

In this work the nickel bath used was prepared at the Laboratory of Thin Films Physics and Applications at the University of BISKRA [13]. For the Ni-Co/SiC co-deposition, the same nickel-cobalt bath conditions will be maintained with the addition of the quantities of silicon carbide (SiC) table (II.4).

**Table (II.4). Chemical compositions of the electroplating baths and operating conditions for obtaining deposits of pure nickel and of the alloy Ni-Co/SiC.**

|                             |                                      |    |
|-----------------------------|--------------------------------------|----|
| Chemical compositions (g/l) | NiSO <sub>4</sub> ,7H <sub>2</sub> O | 21 |
|                             | CoSO <sub>4</sub> ,7H <sub>2</sub> O | 5  |
|                             | NH <sub>4</sub> Cl                   | 21 |

|                             |  |            |
|-----------------------------|--|------------|
|                             | H <sub>3</sub> BO <sub>3</sub>                     | 20         |
|                             | NaCl   | 6          |
|                             | SiC  | 5,10,15,20 |
|                             | C <sub>12</sub> H <sub>25</sub> NaO <sub>4</sub> S |            |
| <b>Operating conditions</b> | Current density (A/dm <sup>2</sup> )               | 3          |
|                             | PH   | 4          |
|                             | Time (min)   | 60 min     |
|                             | Temperature (°C)                                   | 48         |

#### II.2.4.7. Plating device

The experimental device used for the electrolytic coating is composed of three essential parts Figure (II.3), namely:

- 1/ A current source (direct current generator).
- 2/ An electrolysis cell, 200 ml in volume which contains three electrodes, two of them constitute the anodes which are nickel in our case, and the third constitutes the cathode which is steel.
- 3/ A set of measuring devices (pH meter, thermometer)

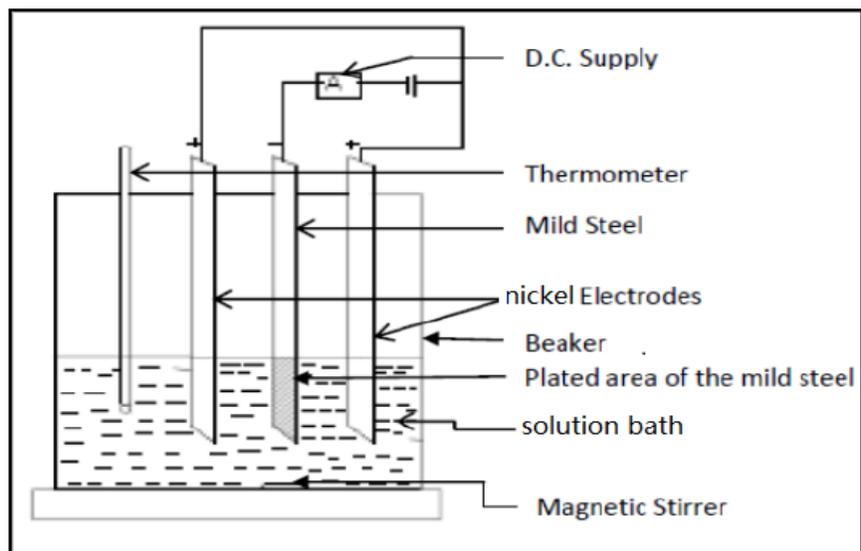


Figure (II.3). Diagram of the electroplating mounting device.

#### II.2.4.8. Addition of an organic additive

To the previous electroplating bath, we added the following additive: Sodium lauryl sulfate (sodium dodecyl sulfate). The addition of the additive at a concentration of 2 mmol/l is made after complete dissolution in distilled water by increasing the temperature.

### II.3. Characterization techniques

The different deposits obtained by electrodeposition are characterized by different techniques such as: adhesion quality, scanning electron microscopy (SEM) in order to observe the morphology of the various coatings produced, the diffraction of X rays (DRX) to identify the crystallographic structure of the phases obtained. The corrosion resistance of deposits produced in the following aggressive media: NaCl (3.5%) and Vickers microhardness measurement.

#### II.3.1. Adhesion of the deposited layer

The adhesion of the electrodeposited layers in this work was measured by the thermal shock method which is described by the Norm ASTM.B571. This test consists in placing the samples coated in an oven for one hour at a temperature which depends on the base metal (case of steel at 250°C). Then a quench fast in water at room temperature. The degree of grip is characterized by the size and whether or not there is flaking and cracks [12].

#### II.3.2. Surface analysis techniques

##### II.3.2.1. X-ray diffraction

X-ray diffraction is a method of identifying crystal phases. It gives information on the purity, the crystallinity and the value of the parameters crystallographic [14]. The principle of analysis is based on the diffraction of monochromatic X-rays by atomic planes of the crystals of the material studied (Figure II.4,5).

The diffraction will take place only in crystallized materials and when the Bragg relation is verified [13]:

$$2 d (hkl) \cdot \sin \theta = n \cdot \lambda$$

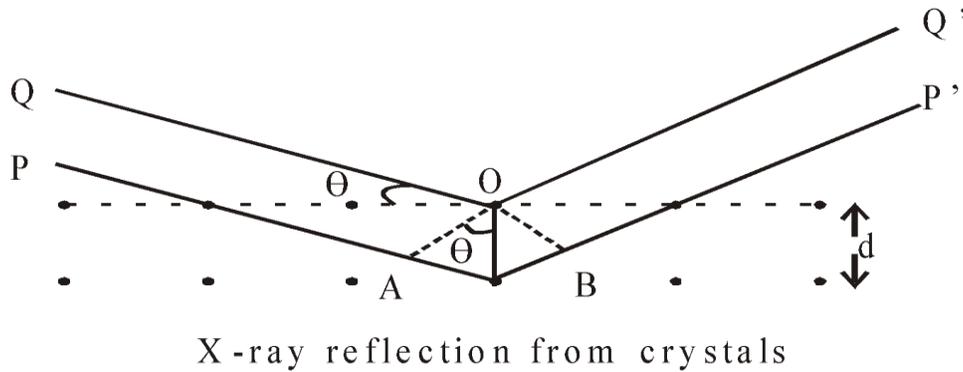
With:

$d$  (hkl): Inter-reticular distance from the corresponding crystallographic plane, that is the distance between the index planes (hkl),

$\theta$ : Angle of incidence of X-rays on the surface of the material studied,

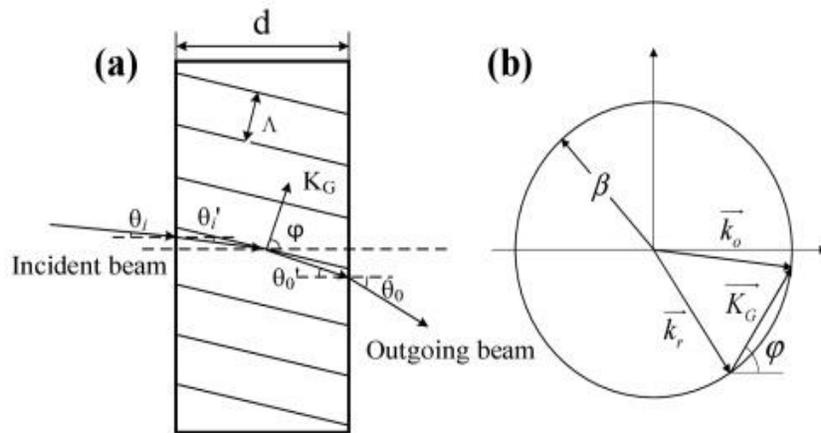
$n$ : Order of refraction,

$\lambda$ : Wavelength of the X-ray beam.



**Figure (II.4). Family of crystal planes in Bragg condition.**

All the diffraction spectra were carried out on a Bruker diffractometer Si (D8 Advance model). This diffractometer is characterized by: X-rays were produced from a source of CuK  $\alpha$ -radiation, having a wavelength of  $1.541838 \text{ \AA}$ , with a voltage acceleration of  $V = 40 \text{ kV}$  and a current of  $I = 40 \text{ mA}$ . The diagram of the apparatus is presented in figure (II.4).



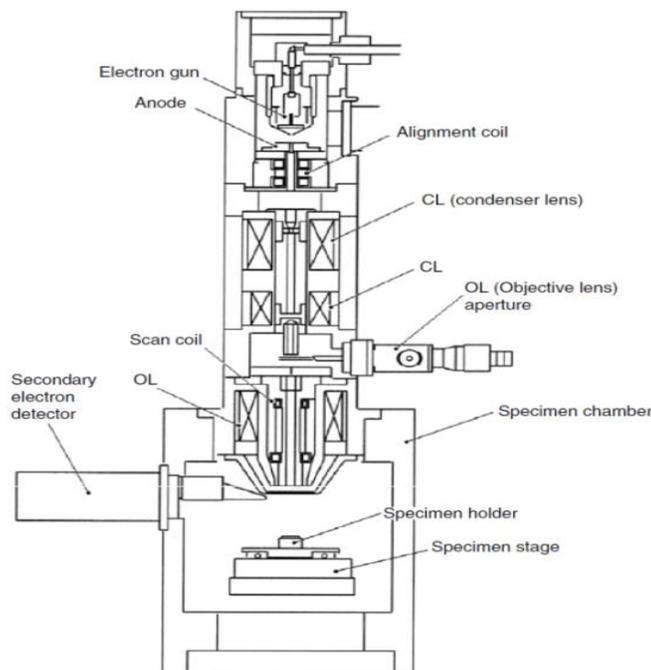
**Figure (II.5). Principle of a two-circle diffraction.**

### II.3.2.2. Scanning electron microscopy

Scanning electron microscope provides information in the form of images light, resulting from the interaction of an electron beam with a volume microscopic sample. The SEM consists: (see Figure II.6) • From an electron optical column;

- A vacuum circuit;
- An electronic part.

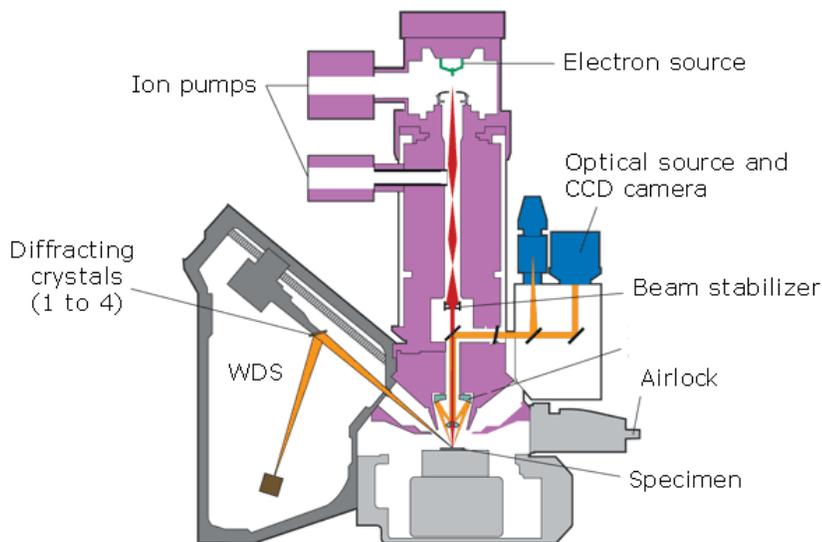
The column is remarkably short, since there are only three lenses for focus the electrons in a fine probe on the sample. And that there are no other lenses under the latter. On the other hand, the sample chamber is larger, because the SEM has no limit on the size of the sample, other than that of the chamber. The part electronics contains the scanning and display circuits. In addition, the power supplies lens and high voltage are significantly less bulky. The interaction of the electron beam and the micrometric volume of the sample gives rise to other electrons of different energies, such as secondary electrons, backscattered electrons, X-rays, etc. The collection of secondary electrons using a detector makes it possible to form an image giving the topography of the deposit obtained [15].



**Figure (II.6). Schematic diagram of a scanning electron microscope In our work, we used the device of type "JEOL-DAC microscope scanning electronics".**

### II.3.2.3. Energy dispersion analysis (EDS)

The energy dispersion analysis takes place in the enclosure of the electron microscope. This technique is based on the same principle as X-ray fluorescence except that the ionization of the material is this time obtained by electron bombardment. While X-ray fluorescence spectrometry gave the overall composition of the sample, EDS analysis can determine a local elemental composition at the surface [16].



**Figure (II.7) Energy dispersion analysis (EDS) diagram.**

### II.3.2.4. Corrosion study

Improving the resistance of electrodeposited deposits and co-deposited layers to electrochemical corrosion in aggressive environments is one of the most important objectives in electroplating. In order to confirm our study, the corrosion test was carried out using referent method (the lost mass method).

### II.3.2.5. Lost mass method

This characterization method consists in immersing in each beaker containing the aggressive middle (3.5 g/l NaCl) a sample of each bath for a period of 20 days, at room temperature. The bare part of each sample is coated with a layer of varnish so as not to be attacked by the aggressive middle. After this immersion period, the samples are removed from the solution, rinsed with distilled water and

acetone and then dried. Finally, the samples are weighed to determine the lost mass and the corrosion rate for each coating.

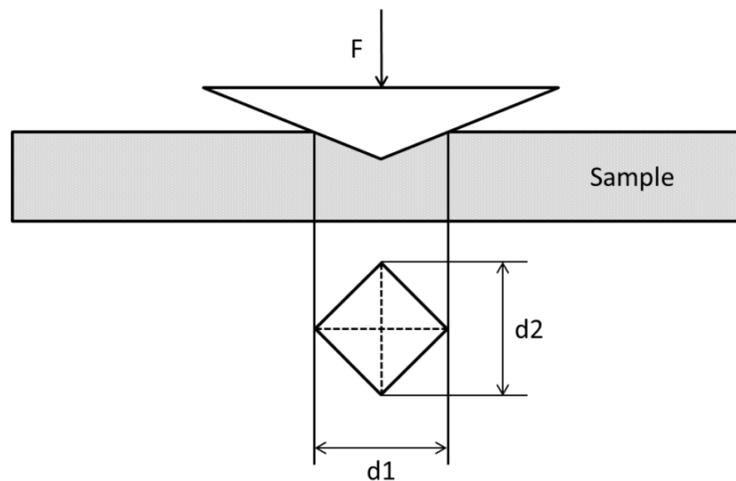
### II.3.2.6. Microhardness

Vickers hardness is the ratio of the applied load to the projected area of the indentation figure (II.8).

$$HV = F/S$$

Where F is the applied load in kgf, and S is the footprint area in mm<sup>2</sup>. The S value is obtained from the diagonal measurement d in mm.

Then:  $S = d^2 / 2 \cos 22^\circ$  and  $HV = 0.1891 F/d^2$



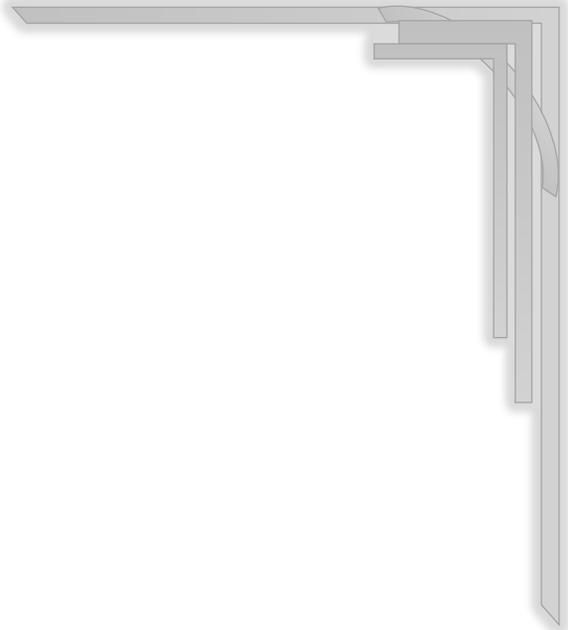
$$d = \frac{d_1 + d_2}{2}$$

$$HV = 0.102 * \frac{2 * F * \sin \frac{136^\circ}{2}}{d^2} = 0.1891 * \frac{F}{d^2}$$

Figure (II.8). The Vickers method of measuring hardness.

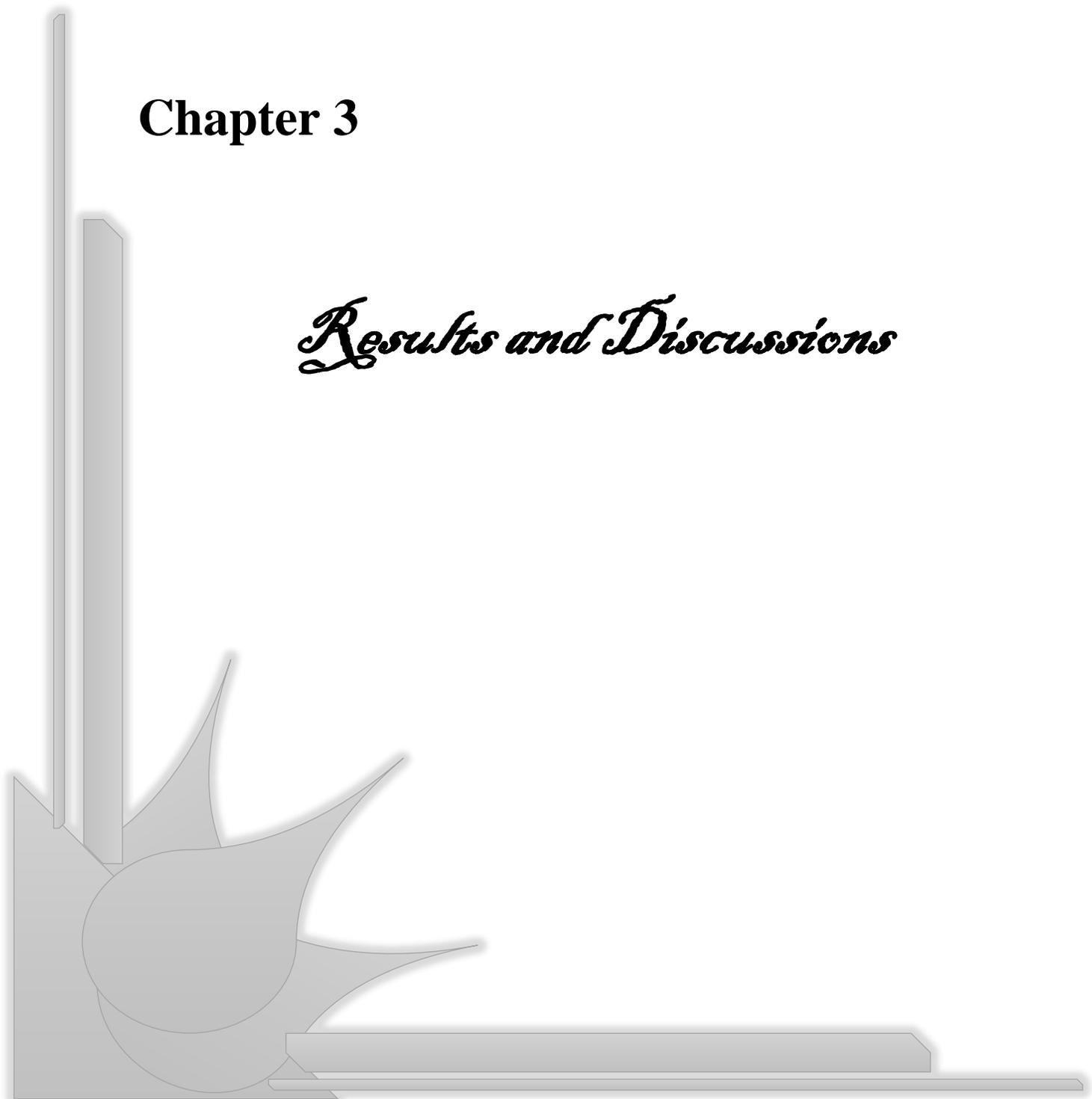
**II.4. Conclusion**

In this chapter, we have exposed the different experimental stages of preparation of composite Ni-Co-SiC + organic additive deposits. Several characterization techniques have been carried out such as: morphological by scanning electron microscopy (SEM), X-ray diffraction (XRD), micro-hardness and corrosion resistance in order to achieve the conditions that meet this objective



## Chapter 3

### *Results and Discussions*



### III.1. Introduction

In this chapter, we present the various experimental results relating to Ni-Co-SiC composite deposits in the presence of an organic additive with a concentration of 2 mmol / l at different concentrations of solid particles (SiC) elaborated on steel substrates beforehan treated.

The objective of this work consists in studying the effect of the addition of this organic additive (Sodium dodecyl sulfate ( $C_{12}H_{25}NaO_4S$ )) in the electroplating bath. To achieve our objective, several characterization tests were carried, namely: adhesion test, microhardness, corrosion resistance in aggressive media, X-ray diffraction (XRD) and morphology by scanning electron microscopy followed by energy dispersion analysis (EDS).

### III.2. Characterization of elaborate deposits

#### III.2.1. Adhesion test

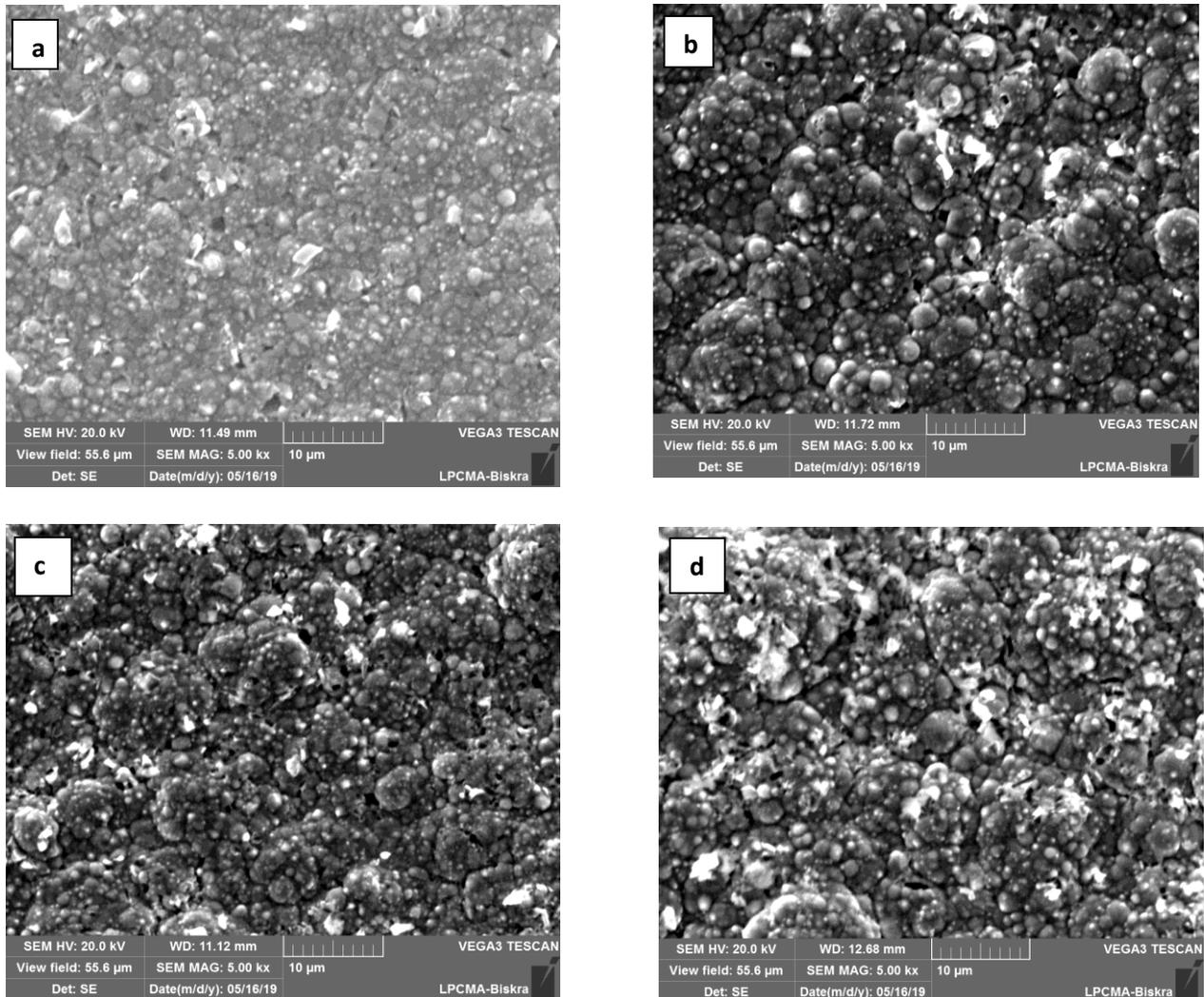
The thermal shock test for different layers leads us to conclude these coatings have very good adhesion.

#### III.2.2. Scanning electron microscopy of electrodeposited (SEM)

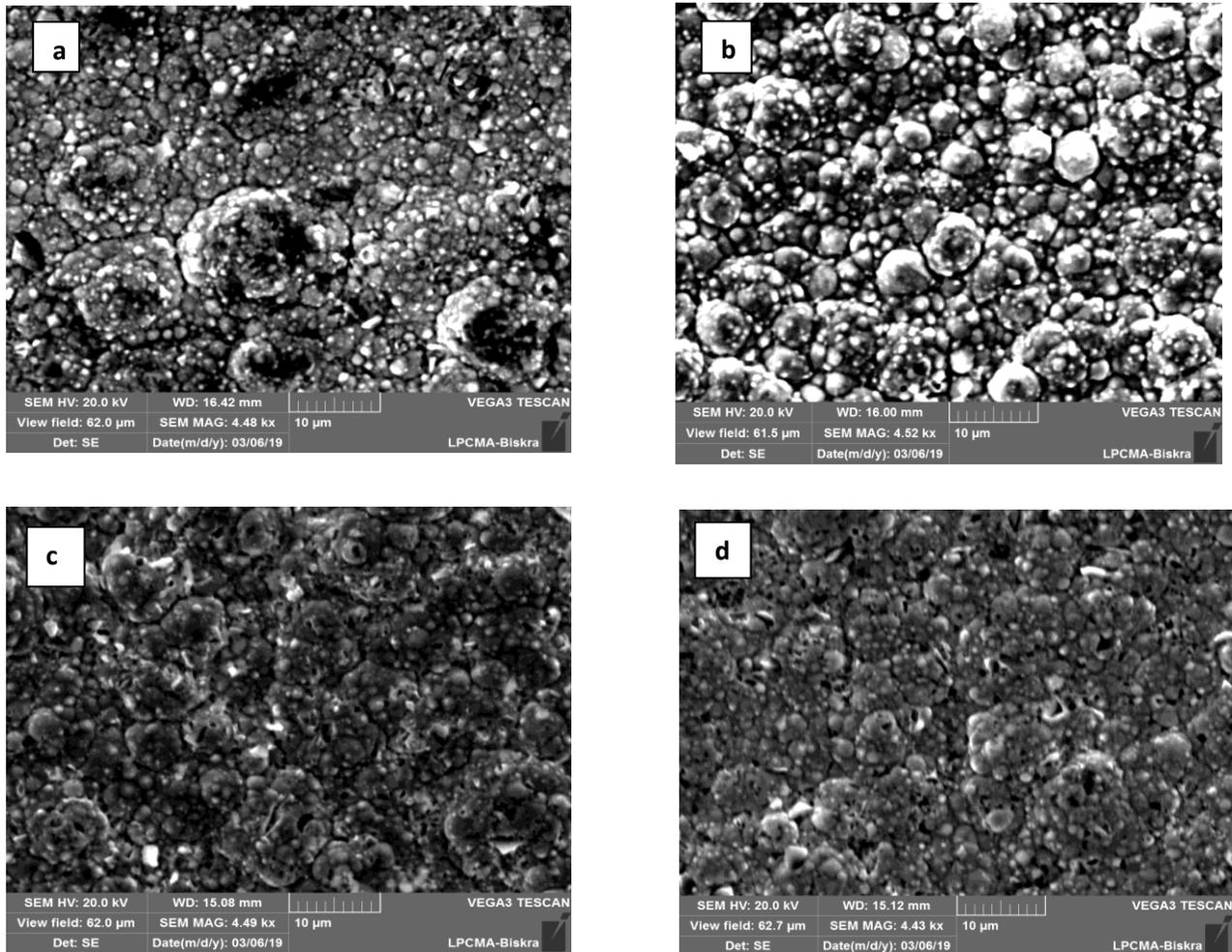
The figure (III.1) shows that the coatings obtained by addition of SiC have a high degree of co-deposition. The entire surface is covered by the deposit which is homogeneous and compact. The solid particles are incorporated into the Ni-Co matrix in a homogeneous manner. It is also noted that the degree of co-deposition is improved by increasing the quantity of solid particles because the increase in the concentration of solid particles (SiC) in the electroplating bath promotes the deposition of fine particles. Moreover, after the addition of sodium dodecyl sulfate ( $C_{12}H_{25}NaO_4S$ ), we see the release of hydrogen bubbles and therefore the reduction in the number of pores and cracks compared to the deposit of Ni-Co-SiC carried out without organic additive figure (III.2).

The solid particles take a positive charge by adsorption of metal ions and H ions. This promotes the transfer of particles to the surface of the cathode, by virtue to the electrostatic attraction leads to a rapid increase in the thickness of the layer determined by electron microscopy. Surfactants decrease tension and improve suspension stability of the suspension and

release the hydrogen bubbles trapped in the deposit. However, the presence of sodium dodecyl sulfate allows the deposition of fine particles which improves the properties of the deposits.



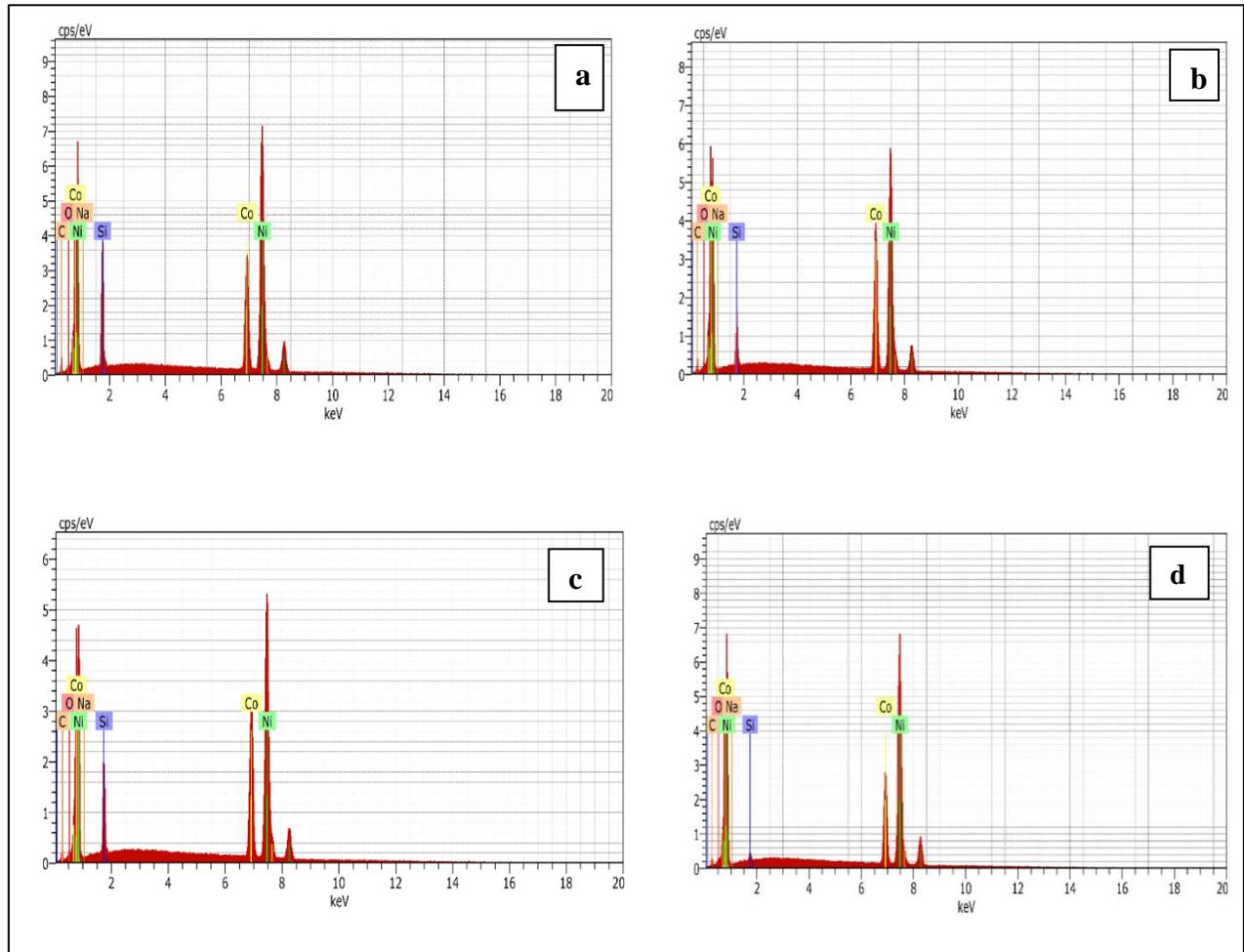
**Figure (III.1).** Morphological characterizations of alloy Ni-Co-SiC with organic additive coating by SEM: (a) 5 g/l, (b) 10 g/l, (c) 15 g/l, (d) 20 g/l of SiC.



**Figure (III.2). Morphological characterizations of Ni-Co-SiC alloy coatings by SEM:  
(a) 5 g/l, (b) 10 g/l, (c) 15 g/l, (d) 20 g/l of SiC without organic additive.**

### III.2.3 Energy dispersion analysis (EDS)

Microanalysis (EDS) is an analytical technique that allows obtaining the elementary and atomic percentage of the composition of a deposit (Figure III.3).



**Figure (III.3).** EDS spectrum of Ni-Co-SiC + organic additive alloy coatings: (a) 5 g /l, (b) 10 g /l, (c) 15 g /l, (d) 20 g /l of SiC.

On the one hand confirms that nickel and cobalt constitute the metallic matrix, while the solid particles (SiC) are incorporated in the matrix from where obtaining composite deposits. On the other hand, the rate of incorporated particles increases as the concentration of solid particles increases. This evolution of the rate of particles is well verified by the increase in the atomic and elementary percentage.

**Table (III.1). Percentage of the different elements of the composite coating.**

| Percentage of SiC | 5 g/l | 10 g/l | 15 g/l | 20 g/l |
|-------------------|-------|--------|--------|--------|
| %Ni               | 63.05 | 57.88  | 57.82  | 49.61  |
| %Co               | 26.85 | 24.43  | 12.04  | 15.55  |
| %Si               | 3.94  | 4.62   | 5.44   | 7.80   |
| %C                | 5.94  | 13.07  | 24.7   | 27.04  |

#### III.2.4. Microhardness method

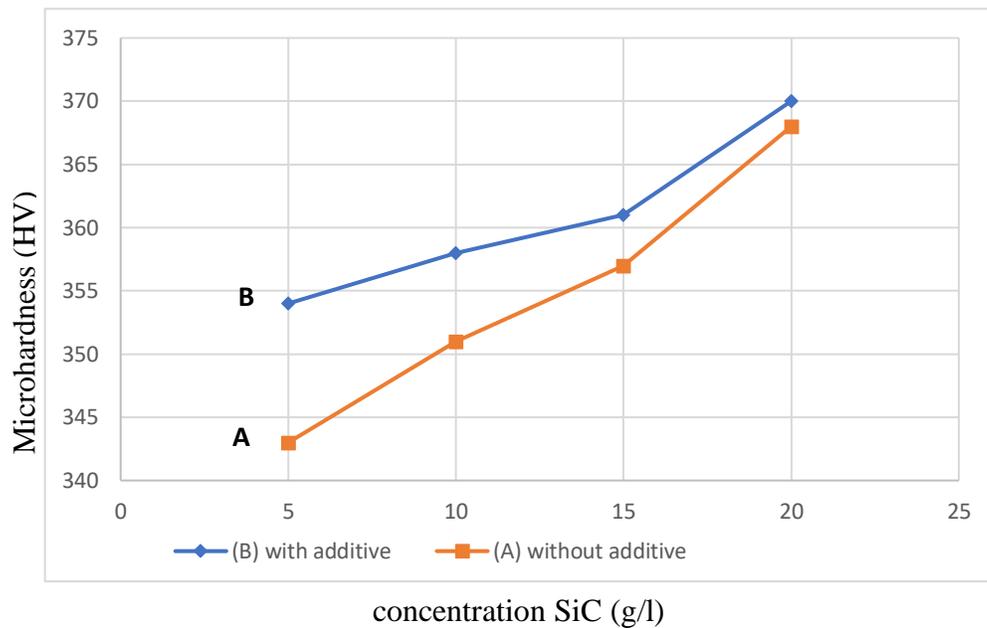
Measures to control the mechanical properties of nickel cobalt layers electrodeposited, in the presence of SiC particles, are summarized in the table following:

Table (III.2) groups the values of the micro-hardness into Vickers hardness (HV) of the different deposits electrodeposited.

**Table (III.2). Microhardness for different concentrations of SiC.**

| Sample                        | hardness (HV) with organic additive | hardness (HV) without organic additive |
|-------------------------------|-------------------------------------|--|
| Deposit of Ni-Co+5g/l de SiC  | 354                                 | 343                                    |
| Deposit of Ni-Co+10g/l de SiC | 358                                 | 351                                    |
| Deposit of Ni-Co+15g/l de SiC | 361                                 | 357                                    |
| Deposit of Ni-Co+20g/l de SiC | 370                                 | 368                                    |

The figure (III.4) shows that the coatings prepared in our bath have good hardness. It is also observed that the addition of solid particles (SiC) in a nickel-cobalt matrix increases the hardness. This is mainly due to the heterogeneity of the coating and the increase in the concentration of solid particles (SiC) which improves the deposition of fine particles making the layer very hard. The addition of the organic additive dislodges the hydrogen bubbles trapped inside the layer, which greatly improves this mechanical property.



**Figure (III.4). Variation of the micro hardness of the electro-deposited composite as a function of the concentration of solid particles SiC without organic additive (A) and with organic additive (B).**

### III.2.5. Corrosion test

#### III.2.5.1. Lost mass method

The corrosion test by the lost mass method examined in an aggressive medium (0.6 M NaCl) for 20 days at room temperature for the electrolytic deposition of nickel-cobalt-SiC, from the chosen bath shows that the corrosion rate is very low and which decreases considerably with the increase in the concentration of solid particles of SiC, this is due to the incorporation of the silicon carbide particles into the Ni-Co metal matrix and that these deposits present good resistance to corrosion, because these solid particles act well as a barrier diffusion between the corrosive medium and the substrate. The best result is obtained for a concentration of 20 g / l of SiC. It is clear that the addition of the organic additive in the electroplating bath further improves the resistance to corrosion in a corrosive medium as shown in figure (III.5).

**Table (III.3). Results of the corrosion test for the different coatings after 20 days without organic additive.**

| Sample               | S(dm <sup>2</sup> ) | m <sub>0</sub> (g) | m <sub>f</sub> (g) | Δm(g)  | Corrosion rate<br>(g. dm <sup>-2</sup> . month <sup>-1</sup> ) |
|----------------------|---------------------|--------------------|--------------------|--------|--|
| Ni+5g/l Co+5g/l SiC  | 0.0268              | 5.2256             | 4.3348             | 0.8908 | 49.830   |
| Ni+5g/l Co+10g/l SiC | 0.0263              | 4.9927             | 4.7896             | 0.2031 | 11.583   |
| Ni+5g/l Co+15g/l SiC | 0.0239              | 5.3251             | 4.1559             | 0.1692 | 10.619   |
| Ni+5g/l Co+20g/l SiC | 0.0269              | 5.3266             | 5.1747             | 0.1519 | 8.470  |

Table (III.4). Results of the corrosion test for the different coatings after 20 days with organic additive.

| Sample                                     | S(dm <sup>2</sup> ) | m <sub>0</sub> (g) | m <sub>f</sub> (g) | Δm(g)  | Corrosion rate (g. dm <sup>-2</sup> . month <sup>-1</sup> ) |
|--|---------------------|--------------------|--------------------|--------|---|
| Ni+5g/l Co+5g/l SiC<br>+ organic additive  | 0.0268              | 5.2346             | 4.5132             | 0.7214 | 40.376  |
| Ni+5g/l Co+10g/l SiC<br>+ organic additive | 0.0263              | 4.8967             | 4.8282             | 0.0685 | 3.906   |
| Ni+5g/l Co+15g/l SiC<br>+ organic additive | 0.0239              | 5.3341             | 5.3163             | 0.0178 | 1.117   |
| Ni+5g/l Co+20g/l SiC<br>+ organic additive | 0.0269              | 5.2996             | 5.2873             | 0.0123 | 0.685   |

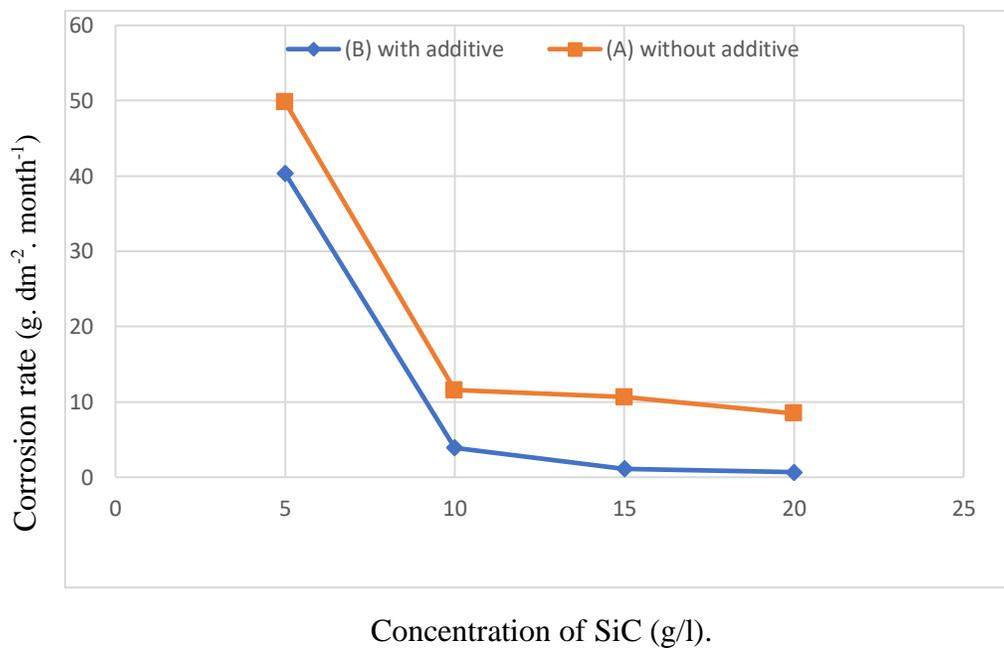


Figure (III.5). Variation in corrosion rate as a function of the concentration of solid particles (SiC) without organic additive (A) and with organic additive (B).

### III.2.6. Grain size

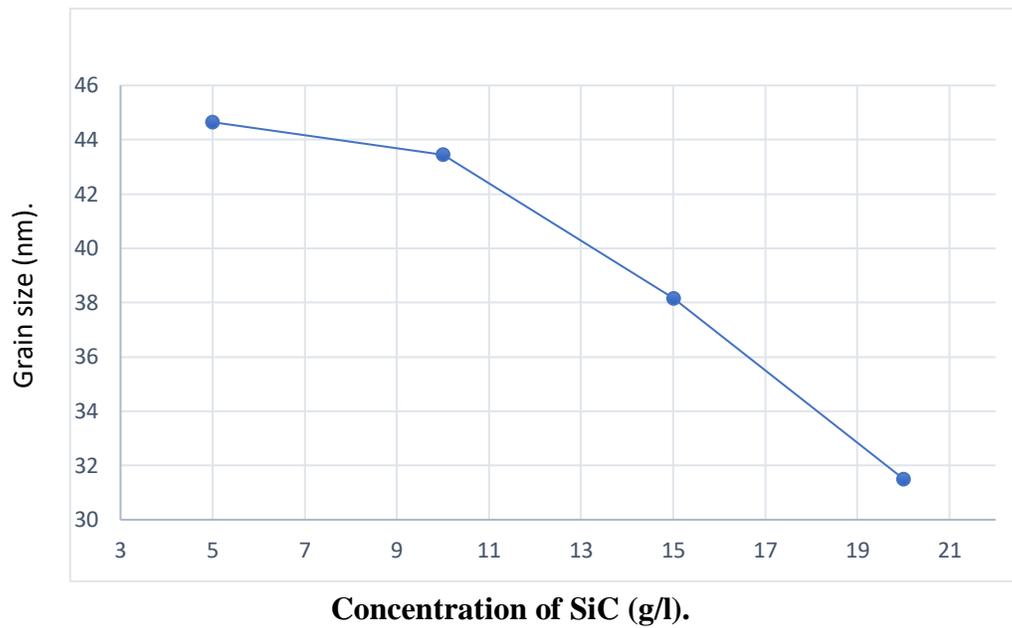
The grain size was determined from the width of the mid-height of the peak using the Scherrer equation modified by Warren and Biscoe [45]:

$$\tau = \frac{0.94\lambda}{\beta \cos\theta}$$

Where  $\theta$  is the position of the peak in the X-ray diffractogram,  $\beta$  is relative to the integral widening of the peak (in radians) which has been approximated to the width at half height (LMH),  $\lambda$  is the wavelength (in Å) and  $\tau$  is the grain size.

**Table (III.5): Values of the grain size of the different electrodeposited layers.**

| Samples                                | 2 $\theta$ (°) | cos $\theta$ | FWHM (°) | $\beta$ (rad) | $\tau$ (Å°) | $\tau$ (nm) |
|--|----------------|--------------|----------|---------------|-------------|-------------|
| Ni+5g/l Co+5g/l<br>SiC+ org. additive  | 44.4441        | 0.9257       | 0.2062   | 0.0035        | 446.971     | 44.6471     |
| Ni+5g/l Co+10g/l<br>SiC+ org. additive | 44.4168        | 0.9258       | 0.2066   | 0.0036        | 434.508     | 43.4508     |
| Ni+5g/l Co+15g/l<br>SiC+ org. additive | 44.4770        | 0.9256       | 0.2362   | 0.0041        | 381.609     | 38.1609     |
| Ni+5g/l Co+20g/l<br>SiC+ org. additive | 44.4327        | 0.9992       | 0.2657   | 0.0046        | 315.070     | 31.5070     |



**Fig. (III.6). Grain size of Ni-Co deposits as a function of the SiC concentration.**

Figure (III.6) shows that the grain size decreases by increasing the concentration of solid particles of silicon carbides. The addition of organic additive reduces the number of pores and therefore, leads to a compact deposit and improves the mechanical properties and corrosion resistance.

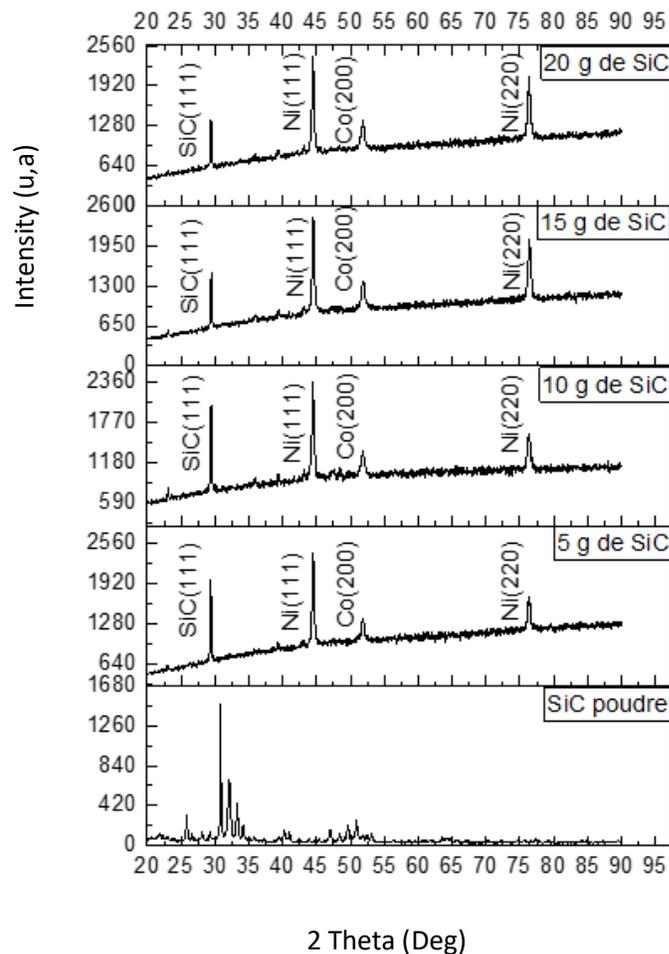
### III.2.7. X-ray diffraction analysis

X-ray diffraction analysis of the various deposits leads to the following results: In all diffractograms, the structure is clearly crystalline. Crystallization is perfect and all peaks are very well resolved.

Diffractogram analysis of Ni-Co-SiC substrates shows that the peaks at  $2\theta = 28-29^\circ$ ,  $44-45^\circ$ ,  $51-52^\circ$  and  $75-7^\circ$  correspond to the planes below: (1 1 1), (2 0 0) and (2 2 0) respectively.

The most intense peak is that indicated at  $44-45^\circ$  corresponding to the plane (1 1 1).

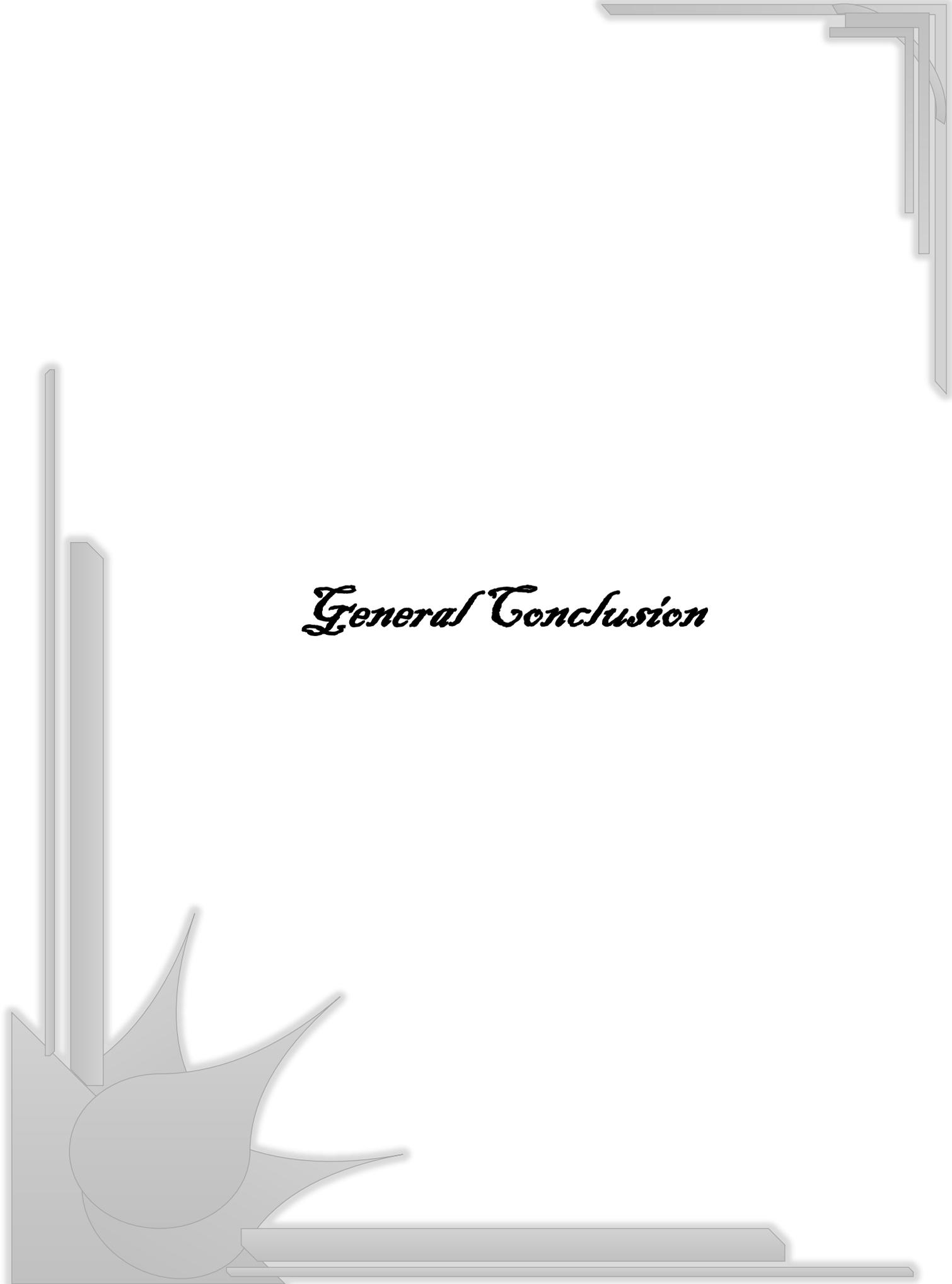
For the concentration of 5, 10, 15 and 20 g / l in SiC, we observe the SiC phase at  $2\theta$ :  $28-29^\circ$  correspond to plan (111).



**III.3. Conclusion**

The characterization of the different coatings by several techniques has shown that the deposits elaborated have perfect adhesion, present a good hardness, better corrosion resistance in the aggressive medium (NaCl 3.5%) and that the entire surface of the substrate is covered by a homogeneous and compact layer of Ni-Co-SiC, this is mainly due to the incorporation of solid particles (SiC) in the metal matrix.

Furthermore, the addition of organic additives removes the hydrogen bubbles formed inside the layer, resulting in a considerable improvement in the properties of the coating.

The page features decorative corner elements in the top-right and bottom-left corners. These elements consist of multiple overlapping, semi-transparent gray lines that form a stylized, layered L-shape, creating a sense of depth and architectural structure.

# *General Conclusion*

## **General Conclusion**

The aim of our work is the study of the effect of an organic additive on the behavior of a composite Ni-Co-SiC coating electrodeposited on steel pretreated at different concentration of solid particles (SiC).

Several characterization tests were carried out to study the effect of an organic additive on the properties of the coatings produced, namely: the adhesion test, morphology by scanning electron microscopy (SEM), corrosion resistance in the corrosive medium, microhardness and X-ray diffraction (XRD).

### ✓ **Adhesion test**

All composite coatings have very good adhesion to the substrate and have a good quality.

### ✓ **Morphologie**

Observation by scanning electron microscopy shows that the entire surface of the substrate is covered by the Ni-Co-SiC composite deposit which is very homogeneous and compact. It is clear that the addition of the organic additive to the electrolytic bath decreases the number of pores and the cracks and consequently improves the mechanical properties and the resistance to corrosion in the aggressive medium considered.

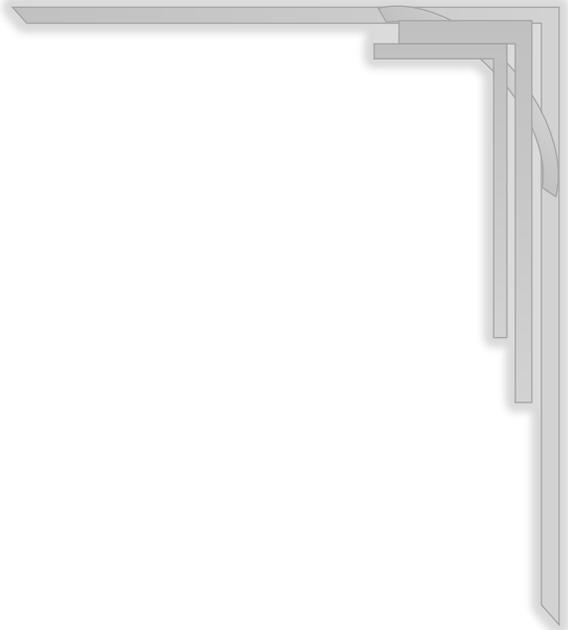
### ✓ **Microhardness**

The microhardness test shows that Ni-Co-SiC composite coatings have good hardness and the addition of organic additive improves this property well. This is due to the incorporation of solid particles into the Ni-Co metal matrix and the release of hydrogen bubbles. The increase in the SiC concentration increases the microhardness due to the small grain size.

### ✓ **Corrosion test**

The corrosion test by the lost mass method examined in an aggressive medium (3.5 g/l NaCl) for 20 days at room temperature for the different Ni-SiC-SiC composite coatings, shows that the corrosion rate is very low and which decreases considerably with the increase in the

concentration of solid particles of SiC, this is due to the incorporation of the particles of silicon carbide in the Ni-Co metal matrix. The addition of organic additive further improves the corrosion resistance of the corrosive medium.



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## ملخص

يهدف العمل الذي تم انجازه إلى دراسة تأثير مادة مضافة عضوية على خصائص مركب Ni-Co-SiC المتحصل عليه كهروكيميائياً على ركائز فولاذية مُعالجة مسبقاً. أُجريت عدة اختبارات لدراسة تأثير إضافة المضافات العضوية وهي: اختبار الالتصاق ، المجهر الماسح الإلكتروني (SEM)، مقاومة التآكل في وسط التآكل، الصلادة وانحراف الأشعة السينية (XRD). جميع الطلاءات المركبة لها التصاق جيد جداً بالركيزة وذات نوعية جيدة. تعمل إضافة المادة العضوية المضافة على تحسين مقاوم التآكل في الوسط الأكل. يُظهر اختبار الصلادة أن الطلاءات المركبة Ni-Co-SiC لها صلادة جيدة وإضافة المادة العضوية يحسن هذه الخاصية. يُظهر الفحص بالمجهر الماسح الإلكتروني أيضاً أن مركب Ni-Co-SiC متجانس ومضغوط للغاية وأن سطح الركيزة مغطى بالكامل بطلاء Ni-Co-SiC. تزيل إضافة الإضافات العضوية فقاعات الهيدروجين المتكونة داخل الطبقة، مما يحسن بشكل كبير من خصائص الطلاء.

**كلمات مفتاحية:** التوضع الكهروكيميائي، المضافات العضوية، مقاومة التآكل، المورفولوجيا، الصلادة.

## Abstract

The present work consists in studying the effect of an organic additive on the behavior of an electrodeposited Ni-Co-SiC composite coating on previously treated steel substrates. Several characterization tests were carried out to study the additive effect on the behavior of the coatings produced, such as: the adhesion test, morphology by scanning electron microscopy (SEM), corrosion test in corrosive medium.

All composite coatings have very good adhesion to the substrate and are of good quality. The addition of organic additive further improves corrosion resistance in the corrosive medium. The microhardness test shows that the Ni-Co-SiC composite coatings have good hardness and that the addition of organic additive improves this property well. The examination by scanning electron microscopy shows that the entire surface of the substrate is covered by the Ni-Co-SiC composite deposit which is very homogeneous and compact. The addition of organic additives removes the hydrogen bubbles formed inside the layer, which greatly improves the properties of the coating.

**Key words:** Electrodeposition; Organic additive; Corrosion; Morphology; Microhardness