

**Ministry of Higher Education and  
Scientific Research  
University of Babylon  
College of Material's Engineering  
Department of Metallurgical Engineering**



# **Production of Metal Powder by Electrochemical Method**

**This project is submitted to the University of Babylon / College of Material's  
Engineering Department of Metallurgical as a part of requirement for  
bachelor degree in Metallurgical Engineering**

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بِسْمِ اللَّهِ الرَّحْمَنِ  
الرَّحِيمِ

وَمَا تَوْفِيقِي إِلَّا بِاللَّهِ عَلَيْهِ  
تَوَكَّلْتُ وَإِلَيْهِ أُنِيبُ

صَدَقَ اللَّهُ الْعَلِيُّ الْعَظِيمُ

سورة هود / الآية (88)

*Dedication*

*To my parents whom made every effort to be here today*

## شكر وتقدير

أتقدم بخالص الشكر والتقدير إلى استاذتي الفاضلة **(سندس عباس)** التي كان لها دور كبير في إنجاح البحث. راجياً المولى القدير ان يمن عليها بوافر الصحة والتوفيق والمواصلة.

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# **Chapter One**

## **Theoretical Part**

## **(1-1) Introduction:**

**Powders of many minerals are produced by the electrochemical method or the so-called electrodeposition, and this technique is one of the commonly used methods, as there are approximately (30) minerals that are deposited in this way, and many of these minerals have extensive and important industrially important uses, including (copper, zinc, chromium, Gold, iron, silver, tin, nickel, cadmium, platinum)**

**The electrochemical precipitation leads to the occurrence of branches and branching of the sediment, which in turn leads to the emergence of sediment growth, which results in either geometric or dendritic shapes. In this method, the deposition products have one of the following aspects:**

- **A strong, brittle mass that is crushed and ground into a powder.**
- **A soft, spongy, and easily friable material.**
- **Direct precipitation of the powder through the electrolyte, depending on the conditions used.**

**In general, the metal powders are produced either by Physiochemical Methods Or Mechanical Methods.**

- **mechanical methods include: Machining, Crushing, Milling, Graining, Atomization and shooting.**
- **either Physiochemical methods include: Condensation, Thermal Decomposition, Reduction, Precipitation from Aqueous Solution, and Precipitation from Fused Salts , Gaseous Reduction process , Intergranular Corrosion, Oxidation and Decarbonization, electrodeposition .**

**Copper is among the metals that can be electrostatically deposited, and it has a very wide use and is extremely important industrially.**

**Copper is one of the strategic elements in the world, not only because it is involved in the production of important alloys, but also because of its distinctive properties when it is present in its pure state . Although copper is found in pure and free form in nature, it is found in union with many minerals such as gold, silver and iron.**

Copper is extracted from natural sulphide ores at a high rate despite the many byproducts that appear when processed, and these products are present with natural ores .

In the field of powder technology, which is known as the science concerned with producing powders in small granular sizes and manufacturing final parts of these powders, they can be in complex shapes, precise dimensions, and at low economic costs, so copper has gained great importance in this field .

Powder technology is widely used in engineering applications, as a large part of the electrolytic copper powder is used in the manufacture of self-lubricating bearings, and in the manufacture of automobile and aircraft engines, and the high electrical and thermal conductivity that characterizes the electrolytic copper powder has led to widespread use in the electrical and electronic industries. Through its use in electronic resistors, control switches, and electronic valves, copper powder also plays an important role in many chemical and pharmaceutical applications and in petrochemical industries with a specific granular size and high purity.

On the basis of the aforementioned importance of the electro-deposition method in the production of copper powder, and its importance in various industrial applications, many studies and researches aimed at raising the efficiency and development of the electrodeposition process and obtaining suitable powders.

As for the current research, it aims to study the electro-deposition process of copper powder produced from aqueous solutions and to show the effect of the sedimentation conditions used, represented by different current densities, on the quality of the produced powder, its purity and its physical properties, in order to obtain the quality of a powder that is most suitable in practical applications.

### **(1-2) Copper (or copper powder) properties**

Copper has many extremely useful properties, including:

- good electrical conductivity
- good thermal conductivity
- corrosion resistance, copper is low in the reactivity series. This means that it doesn't tend to corrode.



- easy to alloy, copper can be combined easily with other metals to make alloys.
- hygienic, copper is inherently hygienic, meaning it is hostile to bacteria, viruses and fungi that settle on its surface.
- easily joined, copper can be joined easily by soldering or brazing.
- ductile, This means that it can easily be shaped into pipes and drawn into wires.
- tough, The property of toughness is vital for copper and copper alloys in the modern world. They do not shatter when they are dropped or become brittle when cooled below 0°C.
- non-magnetic, copper is non-magnetic and non-sparking.
- attractive, copper and its alloys, such as brass, are used for jewellery and ornaments. They have an attractive golden colour.
- recyclable, copper can be recycled without any loss of quality.
- catalytic, copper can act as a catalyst – meaning a substance that can speed up a chemical reaction and improve its efficiency.

### **(1-3) Methods production of copper powder**

A number of methods of producing powders have been developed, as some of them can be produced in one method while other powders can be produced in several methods at the same time, but with a lesser degree of purity and with different shapes, sizes and minutes, so there is a close relationship between the method used to produce a particular powder and the specifications. The resulting powder: Practically any substance can be made into powder form using one or more of the powder production methods, and the choice of method depends on :

- The type of raw materials available.
- Required features.
- The structure and nature of use.
- The production process as a whole and the limitations of some powder technology methods.
- The market consuming the final product.

**In general, metal powders are produced by mechanical methods ,Mechanical Methods or Physiochemical Methods.**

**Granular copper powder can be produced by a number of methods, Each method yields a powder having certain inherent characteristics, These methods include:**

### **1-Atomization process**

**Typically, copper is melted and the liquid metal flows through an orifice where it is struck by a high velocity stream of gas or liquid, usually water, thus breaking the molten metal into particles which solidify rapidly. Particle size and shape are influenced particularly by the atomizing medium, the pressure and the flow rate. Controlled small additions of deoxidizing elements, such as phosphorus, also influence the particle size and shape. After atomization and annealing in a reducing atmosphere to decrease any surface oxide formed during atomization, the product is milled, classified and blended to achieve the particle size distribution required.**

**The purity of the product depends on that of the raw material since refining of the melt prior to atomization is generally not practiced. Purity is generally over 99%.**

### **2-Electrolysis process**

**Electrolytic copper powder is produced by following principles used in electroplating with the conditions changed to produce a loose powdery deposit rather than a smooth adherently solid layer. The formation of powder deposits that adhere loosely to the cathode is favored by low copper ion concentration in the electrolyte, high acid concentration and high cathode current density.**

**the copper powder obtained by electrolysis is high purity material, averaging more than 99% copper and it is dendritic in shape.**

### **3- Hydrometallurgy process**

**The hydrometallurgy process can be used to produce copper powder from cement copper, concentrates or scrap copper. The copper is leached from these materials with sulfuric acid or ammoniacal solutions and the pregnant solution is separated from the residue by filtration. The copper is precipitated from solution by reduction with hydrogen under pressure. The process yields a high purity powder, averaging more than 99% copper.**

### (1-4)Electrodeposition

electrodeposition is the deposition of a metal on a cathode during electrolysis; used as a method of purification.

The idea of electrical deposition is the opposite of the idea of electrochemical corrosion, as it relies on converting metal ions into atoms on the surface, while corrosion is the conversion of atoms on the surface of the metal into dissolved ions in the external environment, and the electro-deposition process includes the deposition of a high-purity powder from the metal. This is as a result of purifying the metal from impurities during electrolysis, which breaks down acids or bases and salts in aqueous solutions by electric current. The electrolytic solution contains salts of the metal to be deposited in a soluble form, and sometimes a salt is added to increase the ability of the solution to electrical conductivity .

The electrolytic cell is the basis of electrolysis in the process of electrolyte deposition and includes the anode, which is the electrode at which the oxidation process (loss of electrons), the negative electrode (Cathode), which is the electrode at which the reduction process (electrolyte) takes place, and the electrolyte It is the medium that conducts electricity, as the flow of current is accompanied by the movement of material . Figure (1 - 1) shows a diagram of the electrostatic precipitation cell .

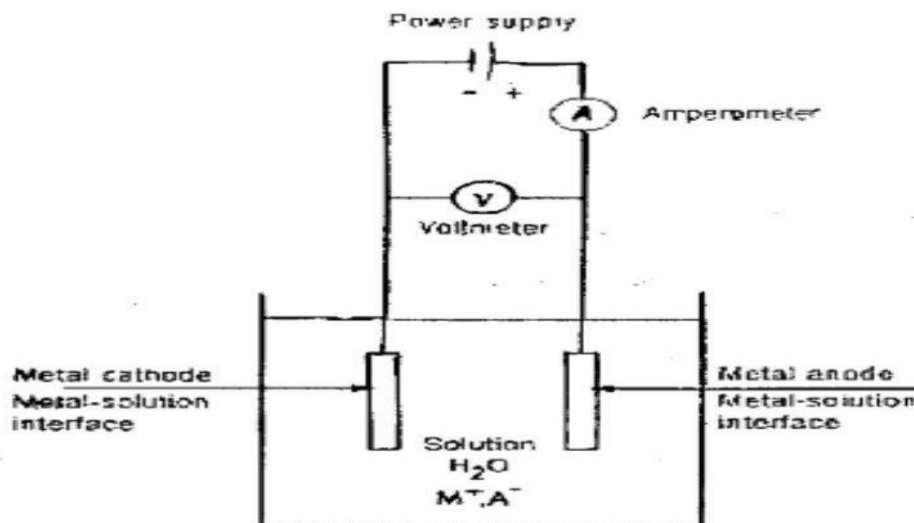


Figure (1 - 1) a diagram of the electrostatic precipitation cell

The anode and the cathode are connected through a DC power source, and through the application of an electrical voltage, the double electrical layers begin to appear on the interface, and the ionic types (cations, anions) move towards the cathode and the anode respectively, and oxidation and reduction processes occur. (Cations) represent the ions that carry the positive charge that is directed towards the negative electrode, while (anions) represent the ions that carry the negative charge towards the positive electrode in the electrolysis process, where the positive ions are consumed near the cathode to turn into metal atoms .

In the initial period and before any visible growth on the cathode, a decrease in the metal concentration occurs near the cathode, and an increase in the concentration of metal ions near the anode, as there is a concentration gradient between the anode and the cathode. This general unstable state produces a current flow, these currents lead to the consumption of (cations) that meet the electrons scattered on the interface to jump the metal atoms to the near vacuum location on the surface of the cathode. This electrochemical growth leads to the formation of dense branched mineral deposits or regular geometric shapes depending on the operating conditions used, and the completion rate of the electro-chemical deposition process also depends on the electrochemical properties of the metal ion, such as standard voltage, ion charge and electron transfer rate. Figure (1-2) illustrates the redox process at the electrode interface.

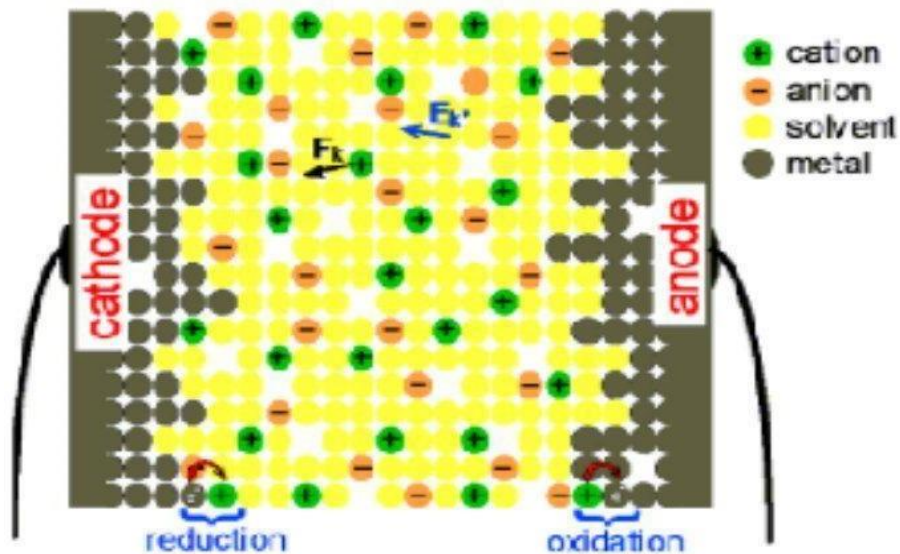


Figure (1 - 2) illustrates the redox process at the electrode interface.

The electrochemical chain is a basic rule in electrodeposition, as it includes standard electrode voltages for metals that precipitate more easily. This means that positive (noble) reducing voltage metals have preference in deposition over minerals of negative (effective) value.

The metal corrodes according to the equation .



Upon precipitation, an electrochemical reduction occurs:



### **(1-5) Main benefits of the electrodeposition process**

The electrodeposition technique has several advantages and they include:

- low cost, simplicity, scalability and manufacturability
- Electrodeposition process is the economical method to produce powders compared with other methods.
- electrodeposition can be used to grow a wider range of materials.
- Homogeneity of the characteristics of powders with high purity .
- Possibility of obtaining a wide range types of powders through the chemical composition of the deposition basin.

### **(1-6)The basic properties of powder**

1-chemical composition and purity:

The chemical composition is one of the important properties as it shows the type and percentage of impurities present in the material. As the effect of impurities is not only limited to the mechanical properties of the powder, but also on the chemical and electrical properties and on the subsequent compression and sintering processes.

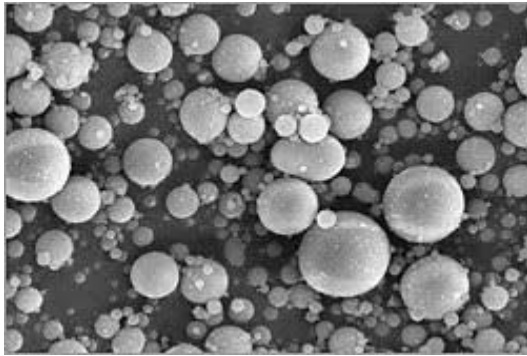
2- particle size :

Powder particle size is of great importance in powder technology due to its effect on many properties, including porosity, Flow ability, and caking properties. As the particle size of ultra fine powder has low apparent density,

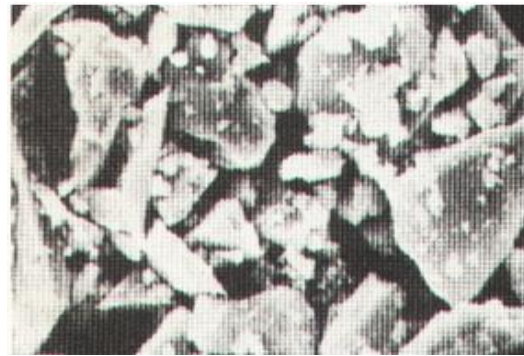
low flow rate, high friction between particles and gives good sintering compared to coarse particles.

### 3- particle shape:

The shape of the powder particles has an important influence on the mechanical properties of the product and on the pressing and sintering processes. There are many forms of mineral powders Spherical, Rounder or Droplet , Angular, Acicular, Dendritic, Flake, Porous, Irregular.



**Spherical**



**Irregular**

**Figure (1-3) Some shapes of powders**

### 4- particle microstructure:

The microstructure of powder particles is where the particle size is determined, their volumetric distribution, minute shape, internal distances and phases determined.

### 5- specific surface:

The specific surface represents the total surface area per unit weight. The specific surface depends on the shape, size and surface conditions of the minutes. Soft minutes have a large surface area, while coarse particles have less surface area.

### 6-apparent density :

The bulk density represents the mass per unit volume of the powder. There are many variables that have a strong influence on the bulk density, including electrolyte composition, electrolyte rotation rate, and temperature. The volume distribution is the main factor responsible for changing bulk density.

#### **7- flow rate:**

**Define flow rate as the rate at which the mineral powder will flow under the influence of gravity from a container through an aperture. The flow rate is influenced by the size of powder particles, their volumetric distribution and shape, and the coefficient of friction between the minutes. Therefore, the fine particles have a low flow rate while the coarse particles have a high flow rate**

#### **8- porosity:**

**Porosity is defined as the ratio of the pores in the powder to the Bulk Volume of the powder, and it is one of the important characteristics in consideration of Packing Characteristics.**

### **(1-7) Electrodeposition solutions**

**To avoid the rapid deficiency of metal ions in the sedimentation basin on the surface of the cathode, very concentrated solutions are used. This requires the use of highly soluble salts.**

- **The metal nitrate has a high solubility, but it is reduced to (0.7V), and therefore it is difficult to use in most basins. Therefore, you must choose salts containing a negative root that cannot be reduced to the range of voltages used in the solutions, because the negative root affects the ionic activity in the Sedimentation basin .**
- **The perchlorate salts have good solubility, but are expensive and liable to explode.**
- **The carbonates adjust the pH and facilitate the control process, as well as reduce the polarization of the anode, and the high concentration reduces the current density.**
- **The use of chlorides in the sedimentation basin has two main effects, as it helps in dissolving the anode, increases the diffusion coefficient, and increases the ion throttle power as a result of increasing the cathode efficiency and the conductivity of the solution.**
- **Sulfate is a relatively inexpensive and commercially available substance that has a stable negative root ( $SO_4^{-2}$ ) that is not reduced to the cathode, does not oxidize at the anode, and has good solubility in water.**

In general, the negative root effect in salts appears through its control over the solubility of the salt, and thus the concentration and activity of the metal ion in the ponds, as well as through its co-ordination with the metal ion, and its effect on the sedimentation process itself .

### **(1-8) Copper Electrodeposition**

Copper is one of the most important industrial metals, and one of the most used metals, and copper is a metal with a bright reddish color and comes after gold in its distinctive color. It is characterized by its high electrical and thermal conductivity and is only superior to the elements in it except silver, and it is a good resistance to oxidation even at high temperatures, and it has high resistance to most solutions of salts, and copper is a soft metal that is malleable and withdrawable and facilitates its rolling to thin sheets and pulling it to wires. Its largest source is in minerals such as calcite and calcite. Copper is obtained from these ores and minerals by smelting, filtering and electrolysis. The main copper producing countries are Chile, Peru and China. and Table (1-1) between the physical properties of copper.

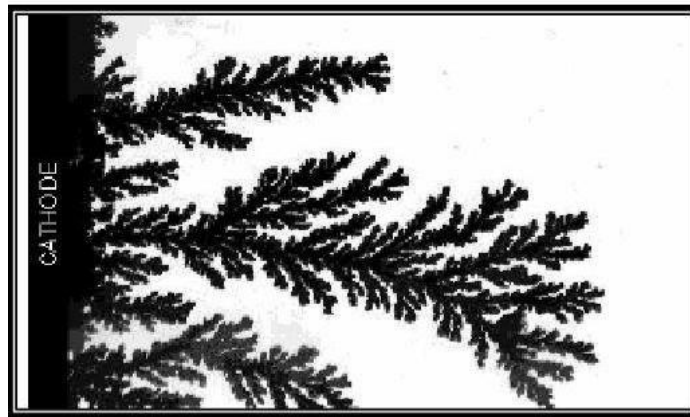
<b>Properties</b>	<b>Numerical value</b>
<b>Melting point</b>	<b>1083°C</b>
<b>Density at 25 °C</b>	<b>8.93g / cm<sup>3</sup></b>
<b>Thermal expansion coefficient</b>	<b>17.7×10<sup>-6</sup> per °K</b>
<b>Thermal conductivity</b>	<b>305-355 W/(m. K )</b>
<b>Specific heat capacity</b>	<b>0.385 KJ/(kg.k)</b>
<b>Electrical conductivity</b>	<b>75-90 % IACS</b>
<b>Modulus of elasticity</b>	<b>117Gpa</b>
<b>Modulus of rigidity</b>	<b>44 Gpa</b>
<b>Atomic Number</b>	<b>29</b>



<b>Atomic Weight</b>	<b>63.54 g/mol</b>
<b>Boiling Point</b>	<b>2595°C</b>
<b>Crystal form</b>	<b>Face center cubic</b>
<b>Standard Electrode Potential</b>	<b>+0.337 V</b>

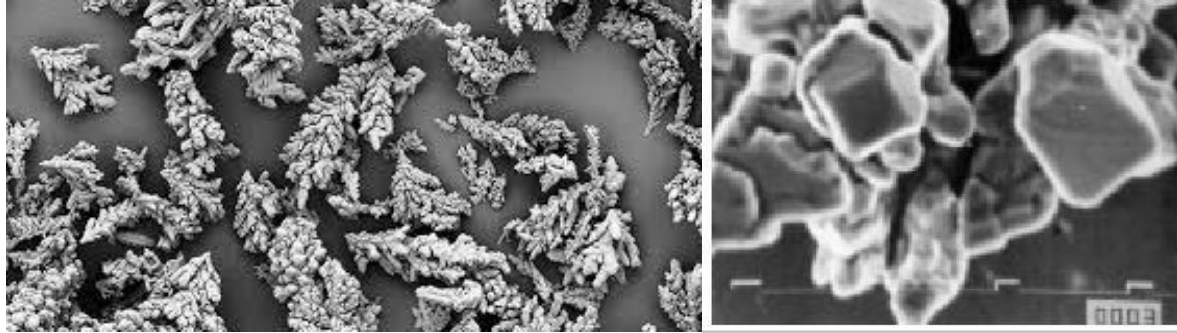
**Table (1-1) between the physical properties of copper**

The electrolytic copper powder production method by electrodeposition usually allows the production of highly pure powder with specifications that make it the most suitable for powder metallurgy processes. The difference in the shape of copper particles made a difference in the apparent densities of copper powder occurring under various conditions. And after the dendritic form is the most environmentally friendly for the copper powder minutes, which makes the bulk density of copper powder small, and the shape (1 -4) represents the electrical deposition of copper,



**Figure (1 - 4) represents the electrical deposition of copper**

The formation of copper powder particles of dendritic shape is strengthened by reducing the concentration of sediment ions, reducing the temperature, reducing the rotation rate of the solution, it is evident from this that the shape of the minutes can vary and change by electrodeposition depending on the operational conditions, and the higher the apparent density, the further away the shape of the dendritic copper powder minutes to a more systematic and engineering form. Figure (1-5) shows the shape of the dendritic and regular copper minutes.



( A ) dendritic powder

( B ) engineering form

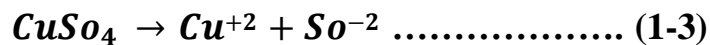
**Figure (1 - 5) Shapes of copper powder**

When electrically depositing copper, a number of reactions take place in the electrolytic cell, which are oxidation reactions, in which the metal atoms are converted into positive ions that are released to the ocean, and the electrons travel through a conductive body to the cathode where the reaction reactions occur on its surface, and ions are released In the form of gases or deposited on the surface of the cathode in the form of metal .

copper sulfate is in the electrolytic solution the main source of copper ions that are deposited on the surface of the cathode, as it is easily soluble in water and the main component of the sedimentation basin, while sulfuric acid increases The electrical conductivity of the solution, and the change in the concentration of sulfuric acid has a greater effect than the effect of the change in the concentration of copper sulfate on the conductivity of the solution .

When an electric potential (voltage) is deposited to precipitate the copper powder, a number of electrochemical reactions occur in the electrolytic cell, as follows :

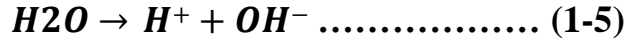
when the copper sulfate is dissolved in water it will decompose into its ions:



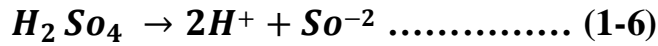
The positive copper electrode gradually dissolves, forming copper ions through the reaction:



The water also decomposes:



Sulfuric acid is also decomposed:



Positive copper ions (+ Cu) in the solution move toward the cathode and join with electrons at the surface of the cathode to precipitate copper powder:



Reducing hydrogen ions at the cathode electrode:



At the positive electrode, both the hydroxide ions( $OH^-$ ) and the sulfate root ( $SO_4^{2-}$ ) in the solution will be directed towards it.

Copper metal used as an electrode in the electrolytic cell is accompanied by a number of components and elements that are considered harmful impurities that must be isolated and disposed of because of its negative impact on the efficiency of this electrochemical process on the one hand and on the other hand, some elements impurities are extracted (and retrieved as elements of economic value for use in industrial processes .

The behavior of impurities in the anode metal depends on the value of its electrical potential in the chain of electrochemical efforts, whether it is higher or lower than the electrical voltage of the metal to be deposited and purified electrolytically. The metal impurities that are higher in their sequence dissolve at the anode and turn into charged ions in the electrolyte such as antimony, arsenic and bismuth, while those that are lower in their sequence do not dissolve but rather settle at the bottom of the tank to form an anode mud like gold and silver, and the basic value of the electrolytic throb comes From having these precious elements.

### **(1-9) Washing and drying processes of powders**

Washing and drying of powders is one of the important processes in electrodeposition technology, as oxidation of the precipitating powder does not occur during its formation in the acid electrolytes, while the oxides are most likely during the washing and drying process, thus the purity of the produced powder is affected by the way these operations are performed .

The precipitated powder should be washed with water to separate it from the electrolyte and then treat the moistened powder with a dilute solution of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) at a rate of (0.05%) to remove the traces of the acid. Sodium titrate (6g/L), which works to prevent oxidation during drying. Drying occurs at a temperature ( $80^\circ \text{C}$ ), then the powder is stored in a dry atmosphere.

Research recommends washing the wet powder several times with a large amount of mineral water so that the wet powder is free from the effects of the acid, after that the powder is raised quickly to prevent oxidation during drying, it is necessary to wash immediately, in order to avoid oxidation of the powder, and to prevent oxidation uses (Benzoic acid) As it is added (0.1%) to the water to wash the powder to protect it from subsequent oxidation, this substance is removed by additional washing. The powder is then dried in ovens in an atmosphere controlled by nitrogen at a temperature ( $110 - 120^\circ \text{C}$ ).

### **(1-10) The variables affecting Electrodeposition process**

There are many factors associated with the anode metal, the metal to be deposited on (the cathode) and the electrolytic environment, which are related to the physical and chemical conditions of the deposition process, the most important of which are:

- 1-electric current
- 2-temperature
- 3- polarization
- 4- PH
- 5- concentration of electrolyte
- 6- mixing

7-distance between electrode

8- standard electrode potential

9- limitation of deposition rate

### **(1-11)Main applications of copper powder**

And the powders produced by modern industry are in most cases non-toxic, non-radioactive, not explosive, nor even fire hazardous. Therefore, its scope of application is very wide. Most often, this product is used non-ferrous metals in powder metallurgy. Also, this material is widely used:

- In the paint industry
- In the chemical industry
- In conventional minerals.
- In the electric coal industry
- In microelectronics.
- In the automotive industry
- In the aviation industry
- In nanotechnology.
- In instrument making.

# **Chapter Two**

## **Experimental Part**

## **(2-1) Introduction**

Practical part includes, how are preparation and operation electrolytic, cell structures and concentration of chemicals materials, devices and equipment, method of work, for the production of copper powder.

## **(2-2) materials**

1-Distilled water.

2 -Electrodes made of copper to be used as a node and cathode in the electrolytic cell.

3-Copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ).

4-Sulfuric acid ( $\text{H}_2\text{SO}_4$ ).

5-Copper chloride ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ).

6-Sodium bicarbonate ( $\text{Na}_2\text{CO}_3$ ).

7-Sodium tartarate (  $\text{CuH}_4\text{Na}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$  ) .

8-Filter paper.

## **(2-3) Apparatus**

1-Power supply, type parmer, English-made maximum value of the current (5A) and the voltage (50 v) It contains magnetic strirrer.

2-Baker glass- heat resistant.

3-Sensitve balance (sartorius German- made accurately 10.0001(.

4-Electric drying oven type (PRODIT.S.a.s , 5g-10026 santenatorino- Italy( , upper limit of temperature up to 250 C".

5-Optical microscope to knowledge and determine shape of the powder particles (Ataly- Biolab – 1007).

**6-Tubes and glass beakers.**

**7-pH meter to measure PH of solution type (philips/pw 9421 ph meter ).**

**8-Mercuial thermometer to measure the temperature C''.**

**9- X- ray diffraction.**

## **(2-4) Experiment part**

### **1-Electrolytic cell**

To prepare an electrolytic cell and operations, baker glass heat-resistant cylindrical used. As it has been preparing of copper electrodes and working anode and cathode in cell. It has been cutting electrodes an dimension of equal (4cm) length, (4cm) width and(1cm) thickness. Electrolytic cell provided with crossbar of wood to put electrodes, where hole electrodes. Copper wires used to hang it and immersed with in solution to ensure moving location of each electrode inside the cell. The figure (2-1) shows electrolytic cell used in the electro position.



**Figure (2-1) electrolytic cell**



## 2-Aqueous Solution

The preparation of the electrolytic solution special to produce of copper powder. Dissolving copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) with distilled water, sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and copper chloride ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ) by dependence on the operating conditions.

## 2-Electrodeposition process

Electrodeposition process started with installs the distance between the electrode and hang of the electrodes in the specified place. Electric circuit connected to pass the electrical current at temperature ( $30^\circ\text{C}$ ). As well as move of the solution during the electrodeposition process to dispose of the bubbles of hydrogen gas which formed on the electrode surface and this is done using a magnetic stirrer.

Then note the electrodeposition process to produce copper powder to know shape of particle. After completing the experiment time, collects the powder deposited on the cathode by using a brush and glass baker.

## 3- Washing and drying powder

Washing powder, which was collected starting with distilled water and then treated with a solution of sodium bicarbonate (0.05%) and sodium tartarate (6g/L). After completion of the process of washing, powder filtered and dry inside the oven at temperature ( $80^\circ\text{C}$ ) then weighted powder.

### (2-5) Effect of current density

The effect of current density on electrodeposition process to produce copper powder by taking by taking multiple values of current density and noticing the effect of this under these conditions. The table (2-1) show operating Conditions for effecting current density.

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	$\text{H}_2\text{SO}_4$	$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$	Time	Distance	Current density
200 g/L	50g/L	100 mg/L	30min	2 cm	(0.12,0.14,0.16,0.18)A mp/cm <sup>2</sup>

Table (2-1) operating Conditions for effecting current density.

### **(2-6) Current efficiency**

Electrolysis laws put by faraday to show the relationship between amount of electric charge pass through solution and amount of deposition on metal surface. So calculate current efficiency from equation :-

$$\text{Current efficiency (\%)} = \frac{W}{W_{\text{theoretical}}} * 100$$

$$W = It M / nf$$

**W:** the weight of the metal deposition in experiment

**W:** the weight of the metal deposition in theory

**M:** Atomic weight of copper (63.54) g/mole

**I:** Current in ampere

**t:** time in second

**F:** Faraday constant (96484.coulombs per mole).

It was also current density calculation through the following law :

$$\text{Current density} = \frac{\text{current}}{\text{Area}}$$

### **(2-7) Particle shape**

A optical microscope used to know particle shape for copper powder. As it has been put amount of copper powder on a slide from glass under microscope to know particle shape and strong enlarge ( 200 X ).

### **(2-8 ) X-Ray Diffraction Test**

X-Ray diffraction was used to study the phases of copper powder with measuring condition as below : Target = cu, wave length 1.5406 Å°.

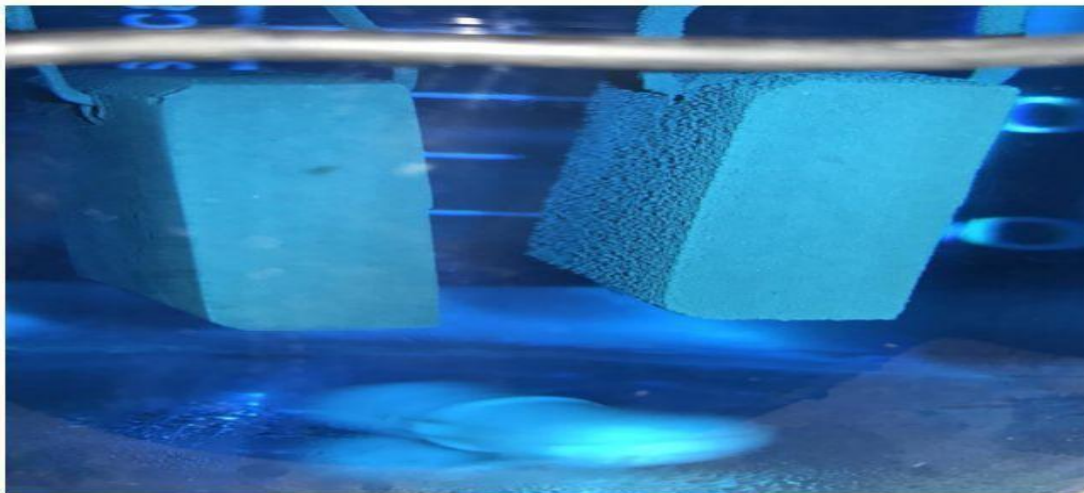
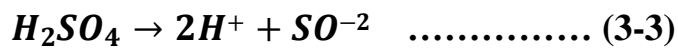
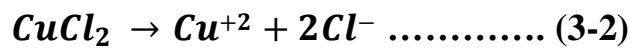
# **Chapter Three**

## **Results and discussion**

### **(3-1) Electrode position process**

In electrode position process used of acidic solution (PH=1.4), component of copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and copper chloride ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ). where deposition of copper powder practically by electrolysis.

Copper powder deposition on form of layer fine powder on the surface of cathode electrode. An indication of deposition of copper of high current efficiency, show fig(3-1),(3-2)



**Fig (3-1) Show deposited copper powder after electrode position process.**



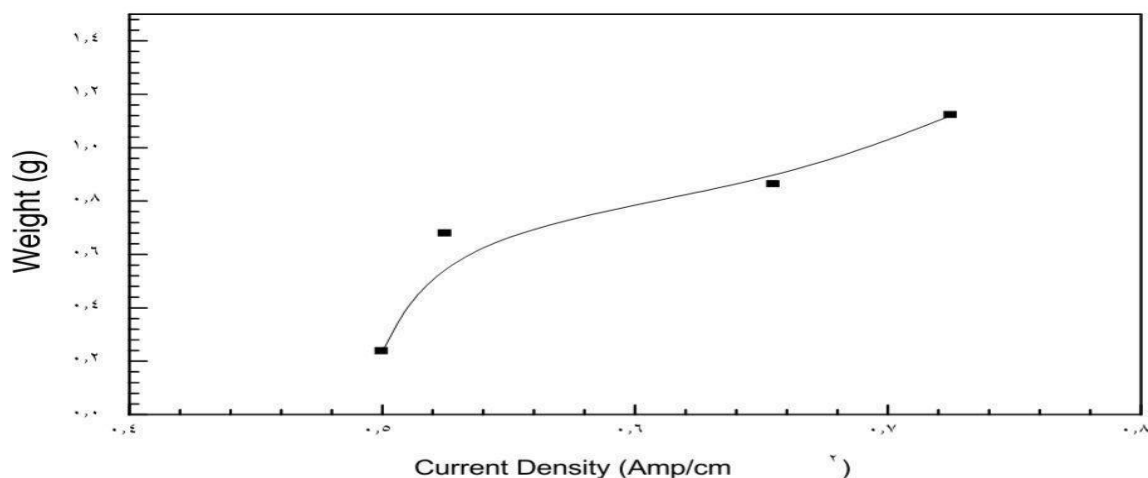
**Fig (3-2) a picture showing the shape of the copper powder after it is removal from the electrode.**

### **(3-2) Effect of current density**

To show effect of current densities in electrode position process of copper powder depending on the different values of the current density (0.12,0.14,0.16,0.18) Amp\cm<sup>2</sup> in electrolyte cell.

The results showed getting deposition of Copper Powder, note- increasing weight of the copper Powder deposited with increases value of the current density as its reaches current efficiency (65.2%) at value (0.18 Amp\cm<sup>2</sup>), while reach to the value (56.7%) at (0.16 Amp\cm<sup>2</sup>) and decrease to the value (49.9%) at (0.14 Amp\cm<sup>2</sup>) also reach at (19.9%) at value ( 0.12Amp\cm<sup>2</sup>).

The increase in the values at current densities make the solution more saturated of cons because increased rate of the current passing in electrolyte cell. which leads to the deposition of copper powder of the surface of the cathode electrode. Show fig (3-3).



**Fig (3-3) show the Effect of current density on weight**

### **(3-3) particle size**

After that was the production of copper Powder by electrode position process, calculated Particle size at all values current density the results in table (3-1) were exhibited the Particles size reduced with increased current density because of the electro - crystallization of capper powder has two stages: “nucleation and growth”. In production of the powder each nucleus was represent powder particle, therefore, the amount of the powder can be obtained by changing the relative rate of nucleating and crystal growth. Increasing current density leds to more disperse structure of the powder particles.

<b>Current density A\cm<sup>2</sup></b>	<b>Particle size μm</b>
<b>0.12</b>	<b>8.72</b>
<b>0.14</b>	<b>3.96</b>
<b>0.16</b>	<b>3.59</b>
<b>0.18</b>	<b>2.42</b>

**Table (3-1) particle size of copper powder**

### (3-4) particle shape

Optical microscope used to determine shape of particle of copper powder, fig (3-4) show shape of particles of copper powder as show dendritic shape of copper powder. Dendritic shape of Copper powder seems to be identical to the standard for copper powder shape in electrode position process. Copper powder formed dendritic shape because of the nucleation on the grains can occurred continuously. Those grains of copper powder and sub particles grain can be produced dendritic growth on nuclei Composed on the initial surface and grains themselves.

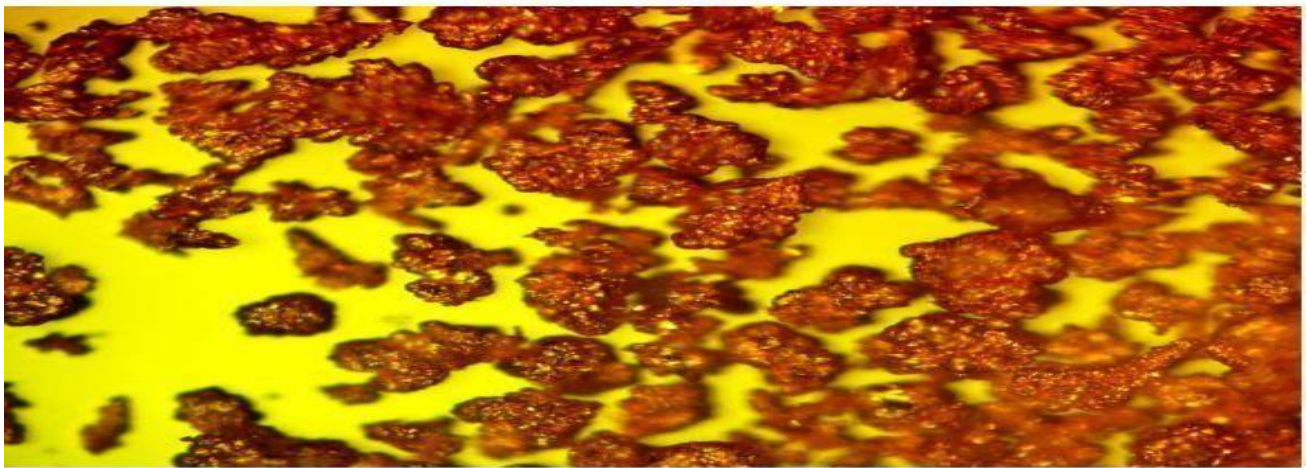


Fig (3-4) show dendritic shape of copper powder

### (3-5) X-Ray Diffraction

XRD patterns of copper powder prepared from aqueous solutions shown excellent crystal face centered cubic (FCC) structures of copper and no oxides or impurities could be found. show in fig (3-5)

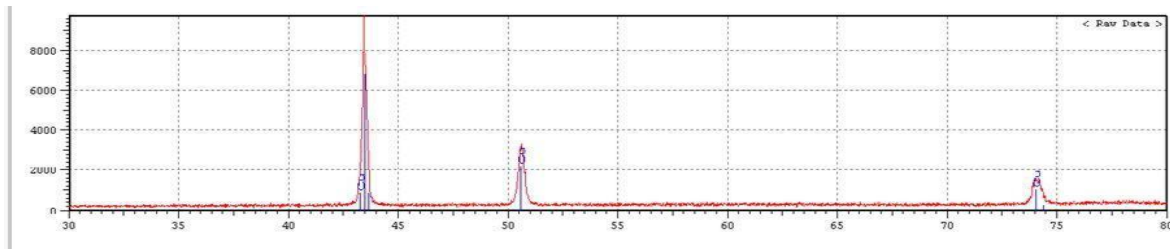


Fig (3-5) XRD patterns of copper powder

## **References:**

**1-Stephen K. Lower, "Electrochemistry: Chemical Reactions at an Electrode, Galvanic and Electrolytic Cells", on the web: [www.chme1.com/acad/webtext/virtualtextbook](http://www.chme1.com/acad/webtext/virtualtextbook), 2004. PP. (1-39).**

**2- Groover M.P, "Fundamentals of Modern Manufacturing", Inc, 1999, PP. (73 – 400).**

**3- M.G. pavlovic, LS.S pavlovic, V.M. Maksimovic, N.D. Nikolic and K.I. popov , characterization and morphology of copper powder particles as a function of different electrolytic regimes , university of Belgrade, in t.J.Electrochem. Sci , 5,1862-1878.**

**4- Wang Ming – Yong, Wang Zhi, Guo Zhan- cheng preparation of electrolytic copper powders with high current efficiency enhanced by super gravity field and its mechanism , Transactions of on. Ferrous metals society china, 20(2010), 1154-1160.**

**5- Lowenheim F.A, "Guide to the Selection and Used of Electroplated and Related Finishes", ASTM Special Technical Publication, 1982, PP. (11 – 17).**

**6- Gordon Dowson, "Powder Metallurgy", Adam Hilger, Bristol and New York, 1990, PP. (31 – 34).**

**7- Ali H,Abbar,” Electrolytic preparation of copper powder with partical size less than  $63\mu m$  “,mechanical engineering department ,college of engineering ,Al-Qadisiya university ,Vol.1,No.1,2008.**



**8- Sundus abbas Jasim,(2018), electrodeposition of copper powder from mixture sulphate –chloride acidic solution ,the Iraqi journal for mechanical and material engineering ,Vol.18,No.3.**

**9-S.G.Viswanth, M.M.Jachak, “Electrodeposition of copper powder from copper sulfate solution in presence of Glycerol and sulphric acid”, Association of metallurgical engineers of Serbia , india , UDC:669-337,2012.**

**10-Harry L. Swinng and John M. Huth, "Buoyancy and Electrically Driven Convection Models in Thin-Layer Electrodeposition", Vol. 59, No 2, American physical Society, 1999, PP. (2157 – 2167).**