

# 1. INTRODUCTION

## 1.1 Classification of Engineering Materials

The different types of engineering materials can be classified according to its composition into four types, metals, ceramics, polymers and composites as follows;

### 1.1.1 Metals

Metals are perhaps the most important engineering material in terms of production quantity, use, and economic impact. They have the exceptional mechanical properties of high strength and fracture toughness, which makes them useful in structural applications. In addition, their high electrical and thermal conductivities lead to applications in electronics and communications. Pure metals are most often used in applications that require exceptionally high electronic conductivity. For most other applications, a metal alloy with a composition tailored to provide specific properties, is used. Metal alloys are grouped into ferrous alloys (e.g., steel, cast iron) and non-ferrous alloys (e.g. aluminum alloys, copper alloys) in terms of their processing; the two most important general properties of metals are their melting points and their ability to undergo large amounts of uniform, plastic deformation. While the melting points of metallic elements range from below room temperature to in excess of 2000 °C, most metals can be melted at moderate temperatures (i.e., below 1600 °C). Entering into the molten state has two advantages: (1) alloys with uniform chemical composition can be prepared easily, and (2) the melt can be solidified by pouring and cooling in a mold. The second important of most metals is their ability to deform plastically. **Table 1.1** illustrates different fabrication methods of the several types of engineering materials.

### 1.1.2 Ceramic

Ceramics is inorganic monometallic products processed at high temperatures. Typically, but not exclusively a ceramic, a metallic oxide, nitride, boride, or carbide, or a mixture or compound of such materials. The wide ranges of materials that fall within this classification include clay minerals, cement, and glass. Ceramics are typically insulative to the passage of electricity and heat, and they are more resistant to high temperatures and harsh environments than metals and polymers.

### 1.1.3 Polymers

Polymers include plastic and rubber materials, are organic compounds that are chemically based on carbon, hydrogen, and other nonmetallic elements; furthermore, they have very large molecular structures. These

materials typically have low densities and may be extremely flexible. Polymers are classified as thermoplastics or thermosetting plastics.

### 1.1.4 Composites

A composite material is a combination of two or more chemically different materials, having a distinct interface between them, which act together to produce a desired (tailored) set of properties. A composite is desired to combine the best characteristics of each constituent material. Obviously this definition includes a vast number of materials, including materials that occur in nature. For example, wood consists of strong and flexible cellulose fibers that are surrounded and held together by a stiffer material called lignin.

**Table 1.1** classification of common processes in terms of the five categories

Category	Metal	Ceramic	Polymer
Melt	Sand casting Permanent mold casting Die casting	Float glass process Fusion draw process Blow molding	Extrusion Injection molding Reactive injection molding Blow molding Fused deposition modeling
Solid	Extrusion Forging Rolling Wire drawing	Rare	Thermoforming
Powder	Uniaxial and isostatic pressing Hot pressing Hot isostatic pressing Selective laser melting	Uniaxial and isostatic pressing Hot pressing Hot isostatic pressing Selective laser sintering 3D printing with binder	Rotational molding Selective laser sintering
Dispersion and Solution	Powder injection molding Coating	Slip casting Tape casting Extrusion Powder injection molding	Coating Stereolithography
Vapor	Sputtering Evaporation Chemical vapor deposition	Sputtering Evaporation Chemical vapor deposition	Rare

## **1.2 The Properties of Engineering Materials**

### **1.2.1 Mechanical Properties**

Many mechanical properties, such as toughness, are strongly dependent on the ease of formation or the presence of defects within a material when the system size decreases, the ability to support such defects becomes increasingly more difficult and subsequently the mechanical properties will be altered significantly. Nanostructures, which are quite different from bulk structures in terms of atomic structural arrangement, obviously show very different mechanical properties. For example, single- and multi-walled carbon nanotubes exhibit high mechanical strengths and high elastic limits that lead to excellent mechanical flexibility.

### **1.2.2 Electronic Properties**

The changes in electronic properties as the length scale is reduced are related mainly to the increasing influence of the wave-like property of the electrons and the scarcity of scattering centers. As the size of the system becomes comparable with the de Broglie wavelength of the electrons, the discrete nature of the energy states becomes apparent.

### **1.2.3 The chemical Properties**

The change in structure as a function of particle size is intrinsically related to the changes in electronic properties. Generally speaking, the ionization potential, the energy required to remove an electron, for small atomic clusters is higher than that for the corresponding bulk material. Meanwhile, it is found that the ionization potential may exhibit obvious fluctuations as a function of the cluster size. It appears that such effects are linked to chemical reactivity, such as the reaction of Fe<sub>n</sub> clusters with hydrogen gas.

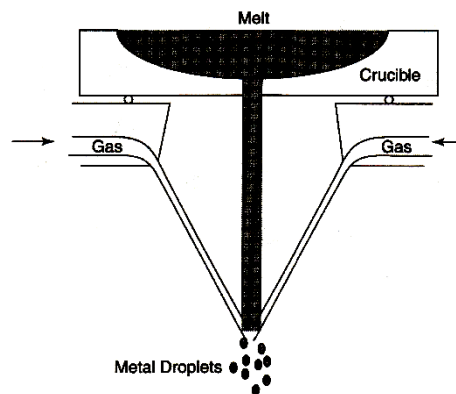
## **1.3 Metal Powder Processing**

In some several alloying applications the molten metals can not be obtained due to the high melting temperature of the metal such as tungsten (M.P. 3300°C) as well as the high reactivity of metals with the atmosphere like titanium react with air at high temperature. The fabrication of the metals in the powder form followed by consolidations at temperatures lower than the melting point give us the opportunity to avoid and solve these problems. The formation of a 3D shape by filling a die or mold with powder and applying pressure, uniaxially or isostatically, to come powder with or without concurrent heating. Parts formed by powder processes typically require post-processing (e.g., sintering) for densification. Additionally, several additive processes also involve powders. This method takes place by pyrolysis or hydrolysis reaction

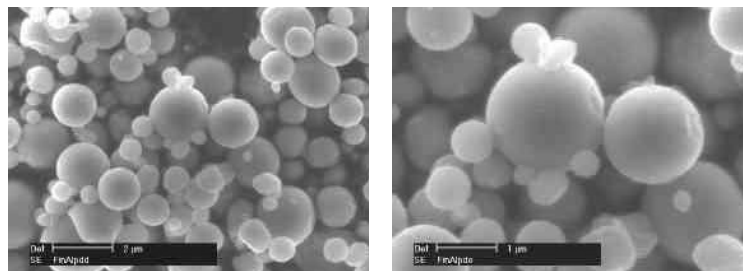
of an atomized droplet containing the precursor. It has a very important advantage which is the powder be produced has controlled surface morphology. There are different methods used for fabrication of metallic powders in the industry as follows;

### **1.3.1 Physical (Atomization) Technique**

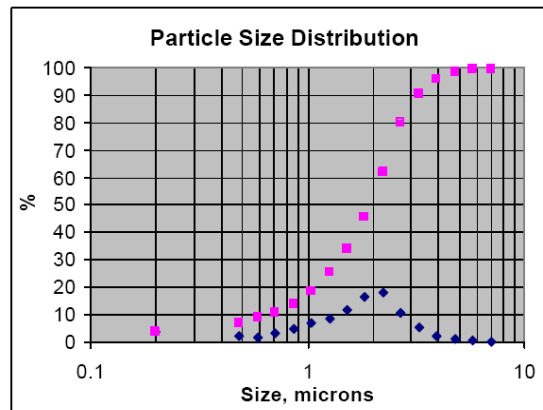
Reactive nanopowders as Aluminum nanopowder can be produced by gas atomization. A high velocity inert gas beam impacts a molten metal. The apparatus is illustrated in **Fig.1.1** A fine dispersion of metal droplets is formed when the metal is impacted by the gas, which transfers kinetic energy to the molten metal. This method can be used to produce large quantities of nanostructured powders, which are then subjected to hot consolidation to form bulk samples. **Fig.1.2** shows SEM images of a fine fraction of gas atomized powder which has been separated from a coarser, gas atomized distribution. The SEM images confirm that individual particles are effectively spherical and that there is a large size range from ~100 to 2000nm. Laser particle size analysis in **Fig. 1.3** shows a median size of ~ 1.9 $\mu$ m and that there is ~ 20% by mass which is sub-micron [1].



**Fig.1.1** Illustration of apparatus for making droplets of metal nanoparticles by gas atomization [1].



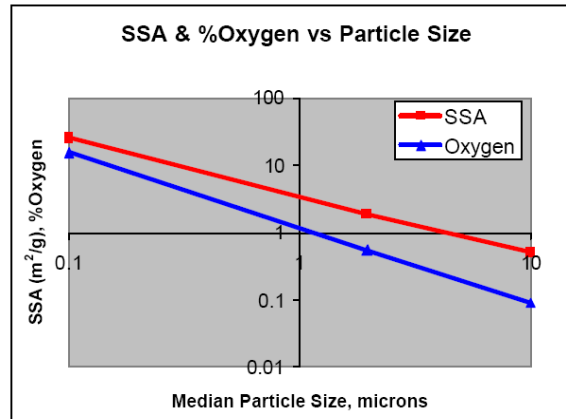
**Fig. 1.2** SEM images of 2 $\mu$ m median size gas atomized aluminum powder [2].



**Fig. 1.3** Size analysis of the aluminum powders produced by gas atomization technique [3].

The fact that atomization has been performed in inert gas and the particle size is relatively coarse results in a relatively pure powder product with relatively low oxide content. Analysis shows that there is 0.55% oxygen and 0.02% C [4]. Depending on the final application, this is potentially advantageous since it is known that adsorbed carbonaceous species can stifle surface activity of Al powders [5]. The specific surface area (SSA) of this sample was determined by BET (surface adsorption) analysis to be  $\sim 2\text{m}^2/\text{g}$ . This value is consistent with that predicted from an assumption of the oxide thickness and the measured oxygen content of the powder.

**Fig. 1.4** shows the relationship between median particle size and SSA and oxide content for selected gas atomized products. It appears that the gradient of the relationship between the SSA and median size is  $< 0.5$  (0.5 is the value expected of mono disperse spherical powders) while the gradient for % oxygen vs median size is  $> 0.5$ . The deviation from the expected gradient may indicate different size distributions and different oxide thicknesses on different particle sizes.

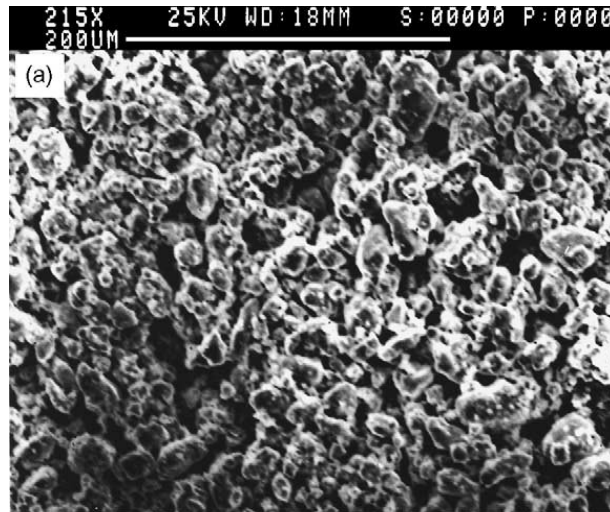


**Fig. 1.4** Relationships between median particle size and specific surface area (SSA), oxygen content [3, 6].

### **1.3.2 Mechanical Alloying**

Mechanical alloying/milling techniques have been used to produce large quantities for possible commercial use. Normal Mechanical alloying is a high-energy ball-milling process in which elemental blended powders are continuously welded and fractured to achieve alloying at the atomic level. Existing factors influencing the mechanical alloying/milling processes include milling time, charge ratio, milling environment, and the internal mechanics specific to each mill. A recent study of the microstructural evolution of nanocrystalline materials produced by mechanical milling indicates that milling environment and temperature strongly influence the deformation behavior of the powders.

An example for the cryogenic mechanical alloying is the formation of TiAl alloy prepared at  $-150\text{ }^{\circ}\text{C}$  for 5hr. SEM micrographs of milled powder mixtures are shown in **Fig. 1.5**. The particle size of these mixtures ranged from less than  $1\mu\text{m}$  up to  $60\mu\text{m}$ . The average particle size was estimated to be approximately  $10\mu\text{m}$  [7].



**Fig. 1.5** SEM micrographs of TiAl powder mixtures milled for 5 h at  $-150^{\circ}\text{C}$  [7].

### **1.3.3 Electrodeposition**

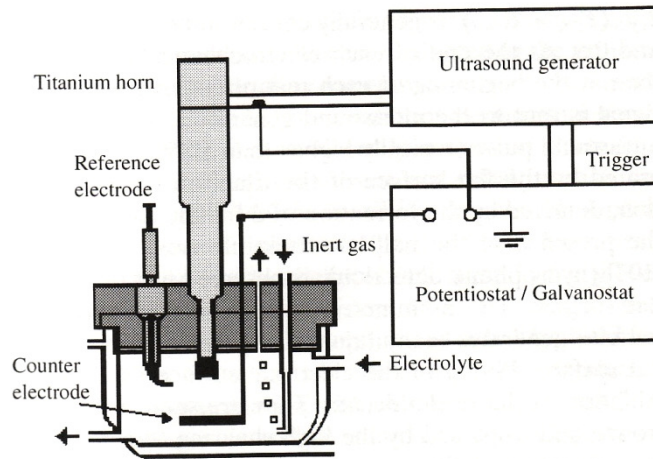
From the synthesis point of view, Electrodeposition is one of the important methods used to produce moderate reactivity nanometals such as Cu, Fe, Co, Ni .... etc., probably inadvertently in most cases. Consequently, there are numerous early reports in the literature describing electrodeposits with ultrafine structures. Review papers [8] describe the effects of various parameters. Such as current density, overvoltage, temperature, deposition time and electrolyte composition on the composition, grain size, and crystal structure of the deposited powder. The author in the following section will focus on the Electrodeposition of nanosized metal powders.

One of the advanced techniques for producing nanosized metal powders using the approach of Electrodeposition is the Electropulsed technique which combines high-current density electrochemistry with the mechanical effects of ultrasound power. The Electropulsed technique is perfectly suited to nanopowders production of pure metals, alloys, defined compounds (like semiconductors), electronic conducting oxides, or polymers. Aqueous or organic electrolytes may be used. The design could also be adapted to molten salt electrolysis. Accordingly, for any compound produced electrochemically in the literature, the parameters of the "Electropulsed" technique may be chosen to produce this compound as a nanopowder with selected values of the mean diameter.

The technique in brief explanation; the high-power ultrasounds are generated by a titanium horn with two piezoelectric ceramic sandwiches working at a frequency of about 20 kHz. This frequency produces the highest mechanical effects that can be generated by ultrasound waves. This horn is grounded and immersed in an electrochemical cell with a classical three-electrode design. The titanium horn is used as the working electrode of a potentiostat/galvanostat apparatus. The cylindrical end of the titanium tip is insulated, leaving only the flat surface in contact with the electrolyte. A reference electrode, chosen as a function of the electrolyte composition, and a counter-electrode, chosen as a function of the compound to be electrodeposited. **Fig. 1.6** shows a complete electrochemical design of the apparatus. The cell is thermostated to control the electrolyte temperature, and the electrolyte is usually de-aerated by continuously bubbling nitrogen or argon [9, 10].

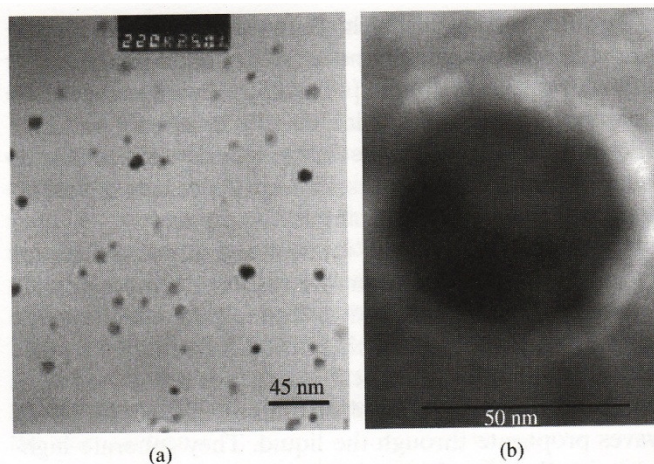
In the potentiostatic mode, the potentiostat is generating pulses of potential difference between the working and reference electrodes. In the galvanostatic mode, the galvanostat is generating pulses of current density between the working electrode and the counter-electrode. The amplitudes of the current and potential pulses in both modes are controlled by means of an oscilloscope. During each pulse, a large number of very small nuclei are formed on the surface of the titanium horn in contact with the electrolyte. The higher the overvoltage and/or the applied current density, the smaller the diameter of the nuclei. After each electrochemical pulse. Ultrasound pulse (typically higher than 50 W/cm<sup>2</sup>) is crated by the flat surface of the titanium horn [11]. The nanopowders are dispersed in the electrolyte. For the most reactive metals, a slow dissolution of the powders may occur, but the absence of dissolved oxygen and the high hydrogen evolution overvoltage on these metals usually limit the dissolution rate to a very low value. The powders are separated from the electrolyte by ultrafiltration or ultracentrifugation under a controlled atmosphere.





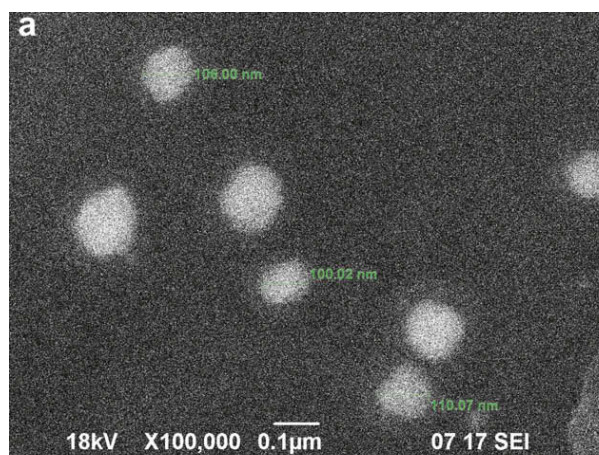
**Fig. 1.6** Schematic design of the cell and electronic equipment used in the electropulsed technique [9].

Copper nanopowders are produced from a pure copper sulfate/sulfuric acid bath [12-14]. Nanopowders with a narrow-diameter distribution range and variable mean diameters are produced without the addition of any inhibitor. **Fig. 1.7a** shows a scanning transmission electron microscope (STEM) picture of a copper powder with a mean diameter around 50 nm. The particles are round, but at high magnification as shown in **Fig. 1.7b** has facets particle shape.



**Fig. 1.7** (a) Copper Nanoparticles produced galvanostatically at  $2 \text{ A/cm}^2$  by the electropulse technique (b) high magnification of a single copper particle [14].

Finally, totally different morphology of the crystallites is obtained at stainless steel ultra-microelectrode (see **Fig. 1.8**). In this case separated almost ideally spherical particles (and nanoparticles are deposited). It may be assumed that surface diffusion of deposited copper ad-atoms is the fastest on stainless steel due to weak interaction between copper and stainless steel base **[15]**.



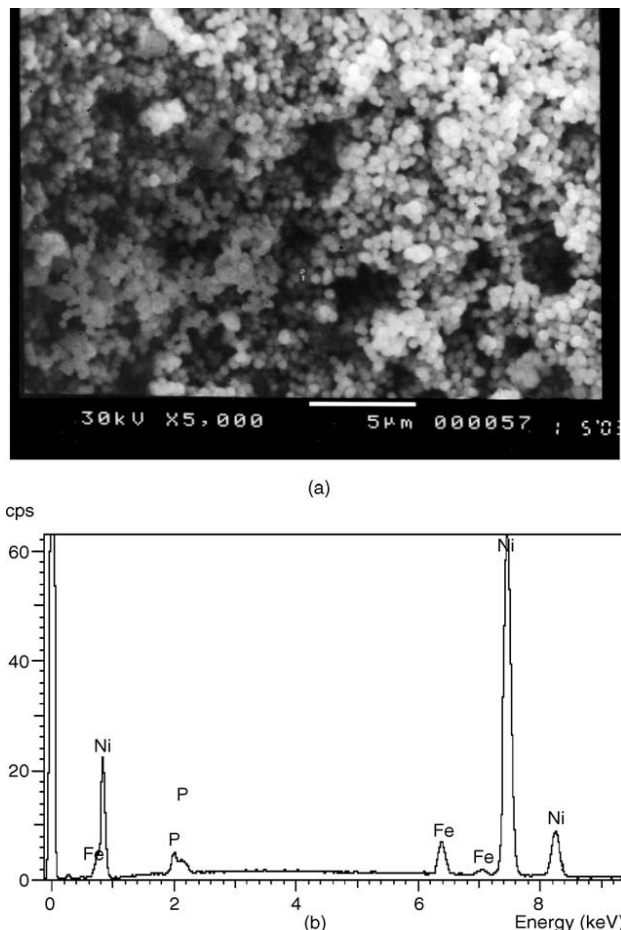
**Fig. 1.8** Copper nanoparticles (and particles) deposited on stainless steel ultra-microelectrode of diameter  $\varnothing$  25  $\mu\text{m}$ . Electrolysis conditions: E1 = 0.60 V, t = 0.10 s; E2 = -0.50 V, t = 0.005 s **[15]**.

### 1.3. 4 Electroless Deposition

In the case of electroless deposition methods, a number of metals can be reduced to the metallic state from solutions of their salts by chemical means. These are gold, silver, chromium, iron, nickel, cobalt, copper, antimony, arsenic and palladium in the elemental form or in the alloying state such as nickel–tungsten, cobalt iron, cobalt–nickel, iron–nickel and arsenic zinc. Although many reducing agents are available, those most commonly used are hypophosphite, borohydride, hydrazine and aldehydes. Many complexing agents were used as a chelating agent for the metals in the bath to prevent hydroxide precipitations as citrates, oxalates, acetates and tartarates **[16]**.

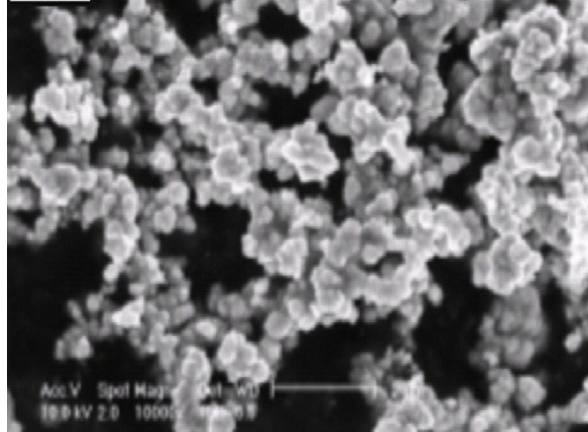
Electroless nickel–iron alloys are of interest as promising materials for computer storage and for coating of recording media. Nanocrystalline Permalloy (Fe20–Ni80) is a promising soft magnetic material that exhibits low coercive force and high permeability **[17, 18]**. In our previous work nano-sized Fe–80wt%Ni powders were prepared by the electroless deposition method

using alkaline tartarate bath.  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  and  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$  are used as the sources of iron and nickel, ammonium chloride is used as a buffering agent and hypophosphite as a reducing agent. By adjusting the pH above 9 with ammonia solution at temperature near  $92^\circ\text{C}$ . After the reaction was completed, the powder was washed by acetone, then filtered and finally dried at  $110^\circ\text{C}$ . **Fig. 1.9a** illustrates the precipitated FeNi powders as studied from the SEM investigations. One can notice that fine precipitates are formed of spherical-like particles of mean diameter particle size about 200 nm. From the EDAX point analysis illustrated in **Fig. 1.9b**, the phosphorus content of the investigated material was 2.4 wt% of the total alloy content showing that the alkaline tartarate bath giving an electroless precipitate has low phosphorus content [19].



**Fig. 1.9** SEM graph with EDAX for the prepared FeNi powder by electroless deposition method. (a) Scanning electron micrograph and (b) the EDAX point analysis for the investigated powder [19].

In a more recent work of our group, a nanosized copper powder was prepared by using alkaline tartarate bath and formaldehyde as a reducing agent. **Fig. 1.10** shows SEM micrograph of the electroless deposited copper powder has spheroid particle shape with size of 100–200 nm **[20]**.



**Fig. 1.10** SEM micrograph of nanosized copper powder prepared by electroless deposition in alkaline tartarate bath **[20]**.

## 2. EXPERIMENTAL

The experimental work is divided into two main works. The first one is the powder preparation and the second one is the powder characterizations.

### **2.1 Preparations Metal Powders**

Two methods were used to prepare the desired metallic powder as follows;

#### **2.1.1 Preparation of Copper Powder by chemical reduction in solutions**

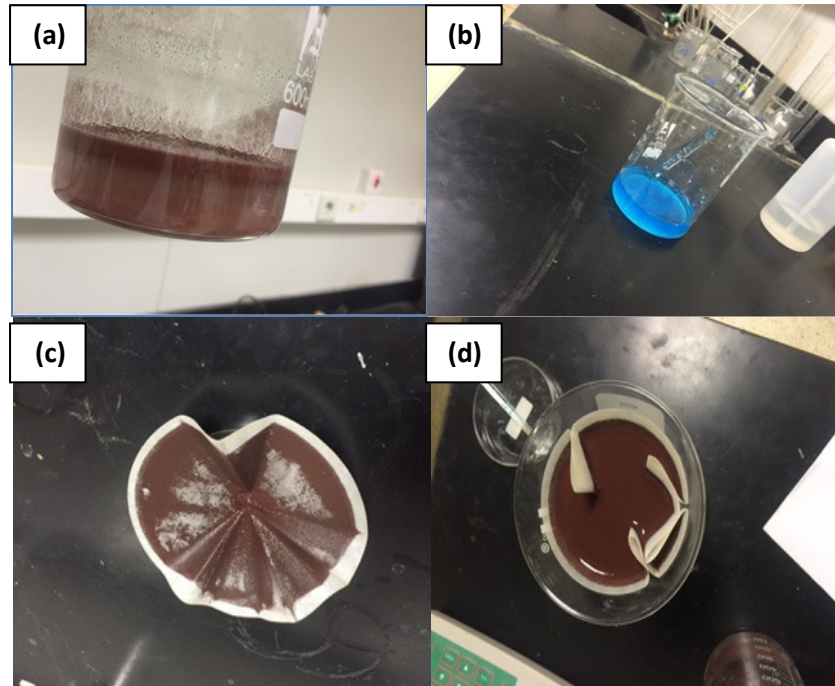
Copper powder was prepared by chemical reduction method in aqueous solution. The process of copper powders was carried out according to the following steps:

- 1- The chemical bath was composed of two chemical solutions A and B; and their compositions consisted of;

<u>Solution A</u>		<u>Solution B</u>
CuSO <sub>4</sub> .5H <sub>2</sub> O	35gm	37% formaldehyde
CH <sub>3</sub> COONa	170gm	
NaOH	50gm	
Distilled water	1000 CC	

Just before use, 5 parts of solution A were mixed with 1 part of solution B.

- 2- The pH of the solution was adjusted at 12.5 at room temperature.
- 3- The bath was stirred for few minutes
- 4- A fine pure copper powder was precipitated in about 10-15min.
- 5- The powder was filtrated and washed with distilled water, and then dried at 110°C.



**Fig. 2.1** the preparation steps of copper powder by chemical reduction of copper sulphate alkaline solution using formaldehyde as a reducing agent

### **2.1.2 Preparation of Powders by Replacement Reaction**

Copper as well as cadmium powder were prepared by replacement reaction of copper as well as cadmium from its nitrate salts using zinc as a reducing agent in aqueous solution. The process was carried out according to the following steps:

- 1- The chemical bath was composed of two chemical solutions A and B; and their compositions consisted of;

<u>Solution A</u>	<u>Solution B</u>
Cu (NO <sub>3</sub> ) <sub>2</sub> or Cd (NO <sub>3</sub> ) <sub>2</sub> (20~30) gm	10 gm Zinc granules
Distilled water	1000 CC

- 2- The bath was stirred for few minutes
- 3- A fine pure copper powder was precipitated in about 24-48 hr.
- 4- The powder was filtrated and washed with a dilute solution of hydrochloric acid to dissolve any excess of zinc granules followed by washing with distilled water, and then dried at 110°C.

## **2.2 .TESTING AND EXAMINATIONS:**

The produced powder of Copper and cadmium were examined and photographed using scanning electron microscope (JEOL, JSM-T20). The principals of the function of the SEM is depends on the interactions of electrons with the surface of the mater. Electrons are produced from an electron gun, and then accelerated through anode plate and focused by magnetic lens. The scanning coils force the electron beam to rapidly scan over an area of the specimen. Electrons emitted from a filament are reflected by the sample and images are formed using either secondary electrons or backscattered electrons. To investigate the nanometric scale, field emission microscope (FE-SEM) is required. FE microscopes could reach resolutions of the order of 1 nm using a cold cathode. If they are equipped with an energy dispersive spectrometer (EDS), the size distribution, shape and chemical composition of nanoparticles can be investigated by FE-SEM.

### 3. RESULTS AND DISCUSSION

#### **3.1 CHEMICAL PREPARATIONS OF POWDERS by REDUCTION IN SOLUTIONS**

Chemical precipitation of copper powders has been widely studied in relation to the solution composition. The minimum components of a solution are a metal salt and a reducing agent. The source of copper could be a simple copper salt, such as copper sulfate, chloride, or nitrate. Various common reducing agents have been also suggested for use in chemical precipitation copper baths, namely formaldehyde, hypophosphite, hydrazine, sugar, and dithionite. When chemical solution utilized formaldehyde as a reducing agent, the pH of the solution should be above 12.

Because simple copper salts are insoluble at pH above 4, the use of a complexing or chelating agent becomes necessary. The complexing agent could be one of the following groups of compounds:

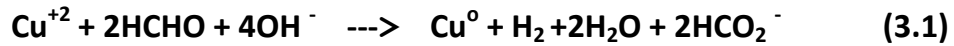
- Tartrate salts, Alkanol amines, or EDTA (ethylenediamine, tetra-acetic acid).

In the present investigation, a mixture of two chemical solutions A and B was used. The former was made by dissolving 35 gm  $\text{Cu SO}_4 \cdot 5\text{H}_2\text{O}$ , 170 gm  $\text{CH}_3\text{COONa}$ , 50 gm  $\text{NaOH}$ , in 500 ml distilled water and the solution was completed to one liter. The latter was 37 wt-% formaldehyde. As 5 parts of solution A were added to one part of solution B at ambient temperature the reaction started within 10 ~15 minutes, the precipitation reaction of copper powder was almost completed, and the solution color changed from blue to colorless. This solution composition is very close to that of electroless (autocatalytic) metal plating, however, it was found that this solution is not autocatalytic, and it is rather a chemical reduction process. The precipitation reaction depends mainly on the pH of the solution and the amount of



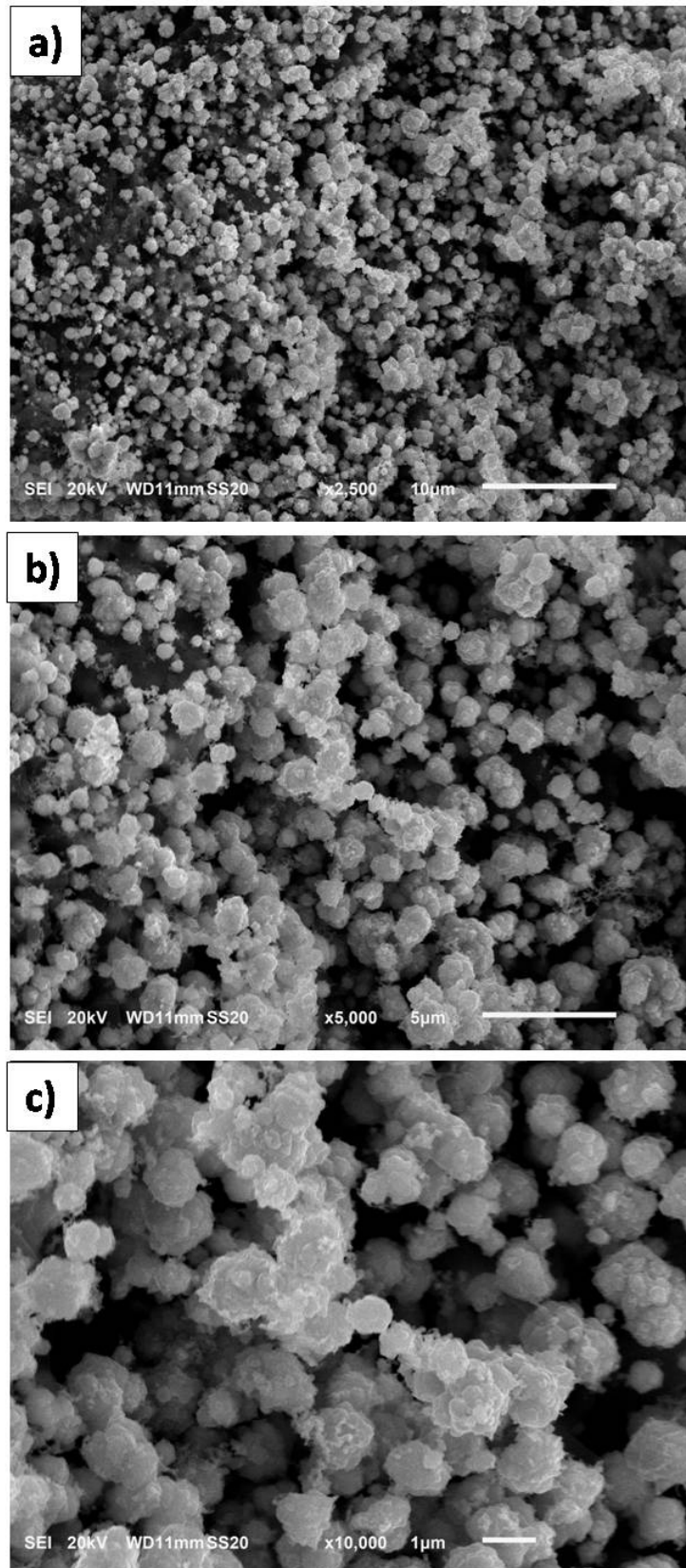
formaldehyde. Once the pH of the solution reaches 12.5, copper precipitates in powder form in the solution without any catalytic surface.

The Cu-precipitation is believed to proceed according to the following reaction:



By changing the pH of the bath, it was found that the rate of Cu-precipitation was increased with the increase of pH to a maximum at about pH 13 and then decreased further on.

The precipitation of Cu-powders was spherical with rough surface morphology. Ultra fine Cu-powders with round arms of sub-micron size can be seen also from **Fig. (3.1)**. These fine particles were formed due to the rapid reaction process as a consequence of multiple precipitations.



**Fig. 3.1** SEM images with different magnifications for the produced copper powder prepared by chemical reduction in alkaline solution using formaldehyde as a reducing agent.

## **4.2 CHEMICAL PREPARATIONS OF METAL POWDERS by REPLACEMENT REACTION**

Copper as well as cadmium powders were prepared by the replacement reaction between its salts with zinc granules as a reducing agent. The presence preparation method is depends basically on the difference in the chemical activities between the metals according to the electrochemical series as shown in Table 3.1.

Since the copper (II) ion has substantially greater reduction potential (+0.34V) than zinc ion (-0.76 V), it is readily reduced by zinc metal

The net ionic equation can be written as follows;



The standard potential of the above reaction can be calculated by the following equation

$$E^{\circ} = + 0.34\text{V} - (-0.76\text{V}) = + 1.10 \text{ V} \quad (3.3)$$

The positive voltage for  $E^{\circ}$  indicates that at standard conditions the reaction is spontaneous.

**Figure 3.3** shows a fine particles of copper were formed due to the replacement reaction process between the copper nitrate solution and zinc granules. The precipitation of Cu-powders was polygonal with smooth surface morphology. Ultra fine Cu-powers with micron size can be seen.

**Table 3.1** the electro chemical series of the elements

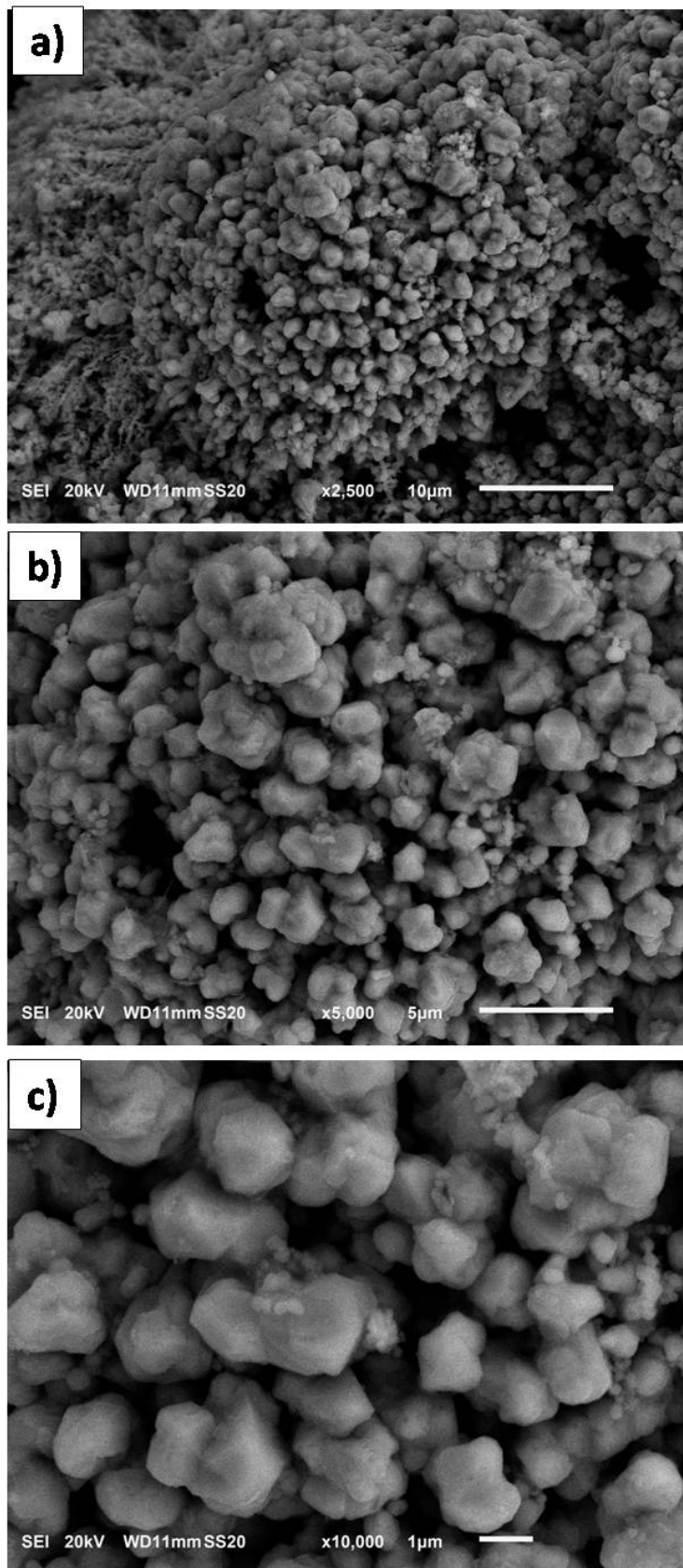
Elements	Electrode Reaction	$E_{red}^{\circ}$ (volts)
	<i>Oxidised Form + ne<sup>-</sup> → Reduced Form</i>	
Li	$Li^{+}(aq) + e^{-} \longrightarrow Li(s)$	- 3.05
K	$K^{+}(aq) + e^{-} \longrightarrow K(s)$	- 2.93
Ba	$Ba^{2+}(aq) + 2e^{-} \longrightarrow Ba(s)$	- 2.90
Ca	$Ca^{2+}(aq) + 2e^{-} \longrightarrow Ca(s)$	- 2.87
Na	$Na^{+}(aq) + e^{-} \longrightarrow Na(s)$	- 2.71
Mg	$Mg^{2+}(aq) + 2e^{-} \longrightarrow Mg(s)$	- 2.37
Al	$Al^{3+}(aq) + 3e^{-} \longrightarrow Al(s)$	- 1.66
Zn	$Zn^{2+}(aq) + 2e^{-} \longrightarrow Zn(s)$	- 0.76
Cr	$Cr^{3+}(aq) + 3e^{-} \longrightarrow Cr(s)$	- 0.74
Fe	$Fe^{2+}(aq) + 2e^{-} \longrightarrow Fe(s)$	- 0.44
	$H_2O(l) + e^{-} \longrightarrow \frac{1}{2} H_2(g) + OH^{-}(aq)$	- 0.41
Cd	$Cd^{2+}(aq) + 2e^{-} \longrightarrow Cd(s)$	- 0.40
Pb	$PbSO_4(s) + 2e^{-} \longrightarrow Pb(s) + SO_4^{2-}(aq)$	- 0.31
Co	$Co^{2+}(aq) + 2e^{-} \longrightarrow Co(s)$	- 0.28
Ni	$Ni^{2+}(aq) + 2e^{-} \longrightarrow Ni(s)$	- 0.25
Sn	$Sn^{2+}(aq) + 2e^{-} \longrightarrow Sn(s)$	- 0.14
Pb	$Pb^{2+}(aq) + 2e^{-} \longrightarrow Pb(s)$	- 0.13
<b>H<sub>2</sub></b>	<b><math>2H^{+} + 2e^{-} \longrightarrow H_2(g)</math> (standard electrode)</b>	<b>0.00</b>
Cu	$Cu^{2+}(aq) + 2e^{-} \longrightarrow Cu(s)$	+ 0.34
I <sub>2</sub>	$I_2(s) + 2e^{-} \longrightarrow 2I^{-}(aq)$	+ 0.54
Fe	$Fe^{3+}(aq) + e^{-} \longrightarrow Fe^{2+}(aq)$	+ 0.77
Hg	$Hg_2^{2+}(aq) + 2e^{-} \longrightarrow 2Hg(l)$	+ 0.79
Ag	$Ag^{+}(aq) + e^{-} \longrightarrow Ag(s)$	+ 0.80
Hg	$Hg^{2+}(aq) + 2e^{-} \longrightarrow Hg(l)$	+ 0.85
N <sub>2</sub>	$NO_3^{-} + 4H^{+} + 3e^{-} \longrightarrow NO(g) + 2H_2O$	+ 0.97
Br <sub>2</sub>	$Br_2(aq) + 2e^{-} \longrightarrow 2Br^{-}(aq)$	+ 1.08
O <sub>2</sub>	$O_2(g) + 2H_3O^{+}(aq) + 2e^{-} \longrightarrow 3H_2O$	+ 1.23
Cr	$Cr_2O_7^{2-} + 14H^{+} + 6e^{-} \longrightarrow 2Cr^{3+} + 7H_2O$	+ 1.33
Cl <sub>2</sub>	$Cl_2(g) + 2e^{-} \longrightarrow 2Cl^{-}(aq)$	+ 1.36
Au	$Au^{3+}(aq) + 3e^{-} \longrightarrow Au(s)$	+ 1.42
Mn	$MnO_4^{-}(aq) + 8H_3O^{+}(aq) + 5e^{-} \longrightarrow Mn^{2+}(aq) + 12H_2O(l)$	+ 1.51
F <sub>2</sub>	$F_2(g) + 2e^{-} \longrightarrow 2F^{-}(aq)$	+ 2.87

↑ Increase

(a) Tendency for oxidation to occur  
(b) Power as reducing agent

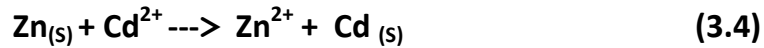
(a) Tendency for reduction to occur  
(b) Power as oxidising agent

↓ Increase



**Fig. 3.3** SEM images with different magnifications for the produced copper powder prepared by replacement reaction using zinc as a reducing agent of copper nitrate solution.

On the other hand in case of the replacement reaction of cadmium from its salts using zinc as a reducing agent the reaction takes place according to the net ionic equation as follows;

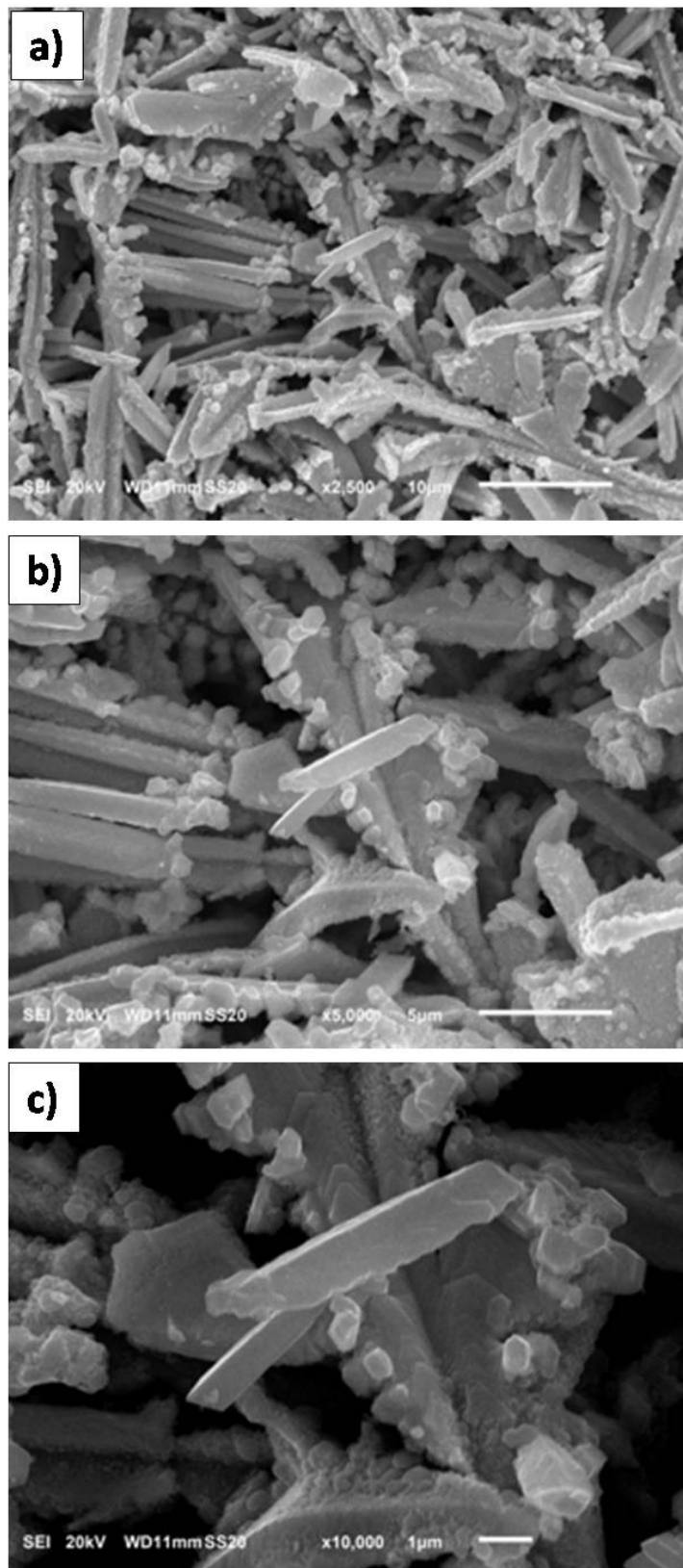


The standard potential of the above reaction can be calculated by the following equation

$$E^{\circ} = -0.4\text{V} - (-0.76\text{V}) = +0.36\text{V} \quad (3.5)$$

The small positive voltage for  $E^{\circ}$  indicates also that at standard conditions the reaction between the soluble cadmium salts and the zinc granules is spontaneous.

As **Figure 3.4** shows a fine flake like and needle shape with particle size of 1~10 microns particles of cadmium were formed due to the replacement reaction process between the cadmium nitrate solution and the zinc granules as a reducing agent.



**Fig. 3.4** SEM images with different magnifications for the produced cadmium powder prepared by replacement reaction using zinc as a reducing agent of cadmium nitrate solution.

#### **4. CONCLUSION**

This project provides an overview on a variety of processing techniques that have been developed for generating ultrafine metal powders. From the data obtained from the experimental work in this study, it was possible to arrive at the following conclusion; copper powders as well as cadmium powder were successfully precipitated using the replacement reaction by zinc as a reducing agent in aqueous solutions. On the other hand copper only can be prepared by the chemical reduction of copper salts using formaldehyde as a reducing agent in alkaline solution. In both methods an ultra-fine powders were produced.



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## المخلص العربي

### تحضير مواد فلزية فائقة النعومة باستخدام الاختزال الكيميائي فى المحاليل المائية

تم فى هذا المشروع تحضير مساحيق فائقة النعومة لبعض الفلزات مثل النحاس و الكاديوم. حيث تم تحضير هذه الفلزات من املاحها عن طريق استخدام طريقتى الاحلال ,والاختزال فى المحاليل المائية باستخدام عوامل مختزلة لايونات هذه الفلزات وتحويلها الى عناصرها النقية لتصلح كمواد معدنية يمكن استخدامها على هيئة المساحيق لتحضير سبائك معدنية عديدة او غيرها من المواد ذات الاستخدامات العديدة فى الصناعات الالكترونية والهندسية الاخرى.