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Fabrication of High Purity Copper Nanopowder via Wires Explosion Technique

Abstract - In this research a high purity copper powder was fabricated via wires explosion technique using copper wire with (99%) purity by the following dimensions (300 mm length, 0.2 mm diameter) on glass substrate inside vacuum chamber and under ambient argon gas, with the utilization of 2.2 kV of explosion voltage and 100 J of storage energy. The used wire purity and structural and morphology properties of the powder surface were diagnosed via X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Atomic Force Microscope (AFM). Experimental results showed that the average particle size of the prepared copper powder was in the range of (20-40 nm) and the samples have high purity and no impurities were observed which makes this nanopowder suitable for many applications especially as additives in lubricating oil for tribological characteristics improvement and suitable catalyst in the heat exchanger systems in the industrial installations.

Keywords: Wires explosion technique, copper nanopowder, nanopowder, nanoparticles characterization.


1. Introduction

Nanopowder is an agglomerate of ultrafine particles such as nanoparticles or nanoclusters produced on the nanoscale range [1]. The word “nano” itself refers to the length scale (one nanometer is one billionth of a meter) that is one thousand times smaller than the micro scale the scale that was traditionally associated with the electronics industry [2]. Basically, a reduction in powder size to the nanometer scale results in various interesting properties such as lower melting point compared to the bulk, lower sintering temperature, quantum size effects and the high surface area [3]. The fabrication methods for the metallic powders production have been developed in the last years and can be classified into three major classes: thermo-physical, mechano-chemical and chemical methods. The physical methods types include electro-explosion, high-energy ball-milling, gas condensation which using different vaporization techniques such as thermal evaporation, laser ablation and micro wave plasma processes [3,4]. The chemical methods types include chemical vapor deposition, sol-gel, hydro thermal techniques and molecular pyrolysis [3,5].

Electrical Explosion Wires technique (EEW) is a physical method, which used for production of nanopowders of conductive materials. Edward Nairne was discovered the first popular observation of fuses and exploding wire discharges and published it in the Royal Society [6]. Metal ultrafine nanopowders with reproducible properties were first obtained in the 1960s and 1970s, numerous works were carried out on metal nanopowder production by electrical explosion of wires [7]. When high-voltage (1–30 kV), power density (10^6–10^12 A/m^2) and impulse duration (10^4–10^7 sec) flows through a conductor wire (less than 1 mm diameter) which is usually produced by the discharge of a capacitor banks passes through a wire, the density of the energy in the wire may considerably exceed the binding energy because of a high rate of the energy injection and an expansion lag of the heated material. As a result, the material boils up in a burst, a bright light flashes and a mixture of superheated vapor and boiling droplets of the exploding wire material and a shock wave scatter to the ambient atmosphere so, nanoparticles will form when explosion products expand into the gaseous atmosphere. The main advantages of the EEW method over other physical methods of nanopowder production are: easily metallic nanoparticles production for different metals and alloys and flexible adjustment of process parameters, control of particle size distribution, structures modification, avoiding any unwanted products during chemical reaction, high efficiency of energy and production of nanopowders with effective nature in chemical processes [9-12]. Copper nanopowders were applicable in various industrial fields such as additives in lubricating oil, catalyst in the heat exchanger systems, high strength metals and...
alloys, high thermal conductive materials, conductive ink and paste for printed electronics, sintering additives, conductive coatings, biosensors and antimicrobial [13-17].

The aim of this work is to fabricating metal copper nanopowder by Electrical Explosion Wires (EEW) technique which is suitable additives in lubricating oil for tribological characteristics improvement for a long period of time.

2. Experimental Work

The work includes the following steps:

The First Step: Preparation of wire: A copper wire with high purity of (99%), (0.2 mm) diameter, (0.032) mm$^2$ cross section area and (300 mm) length was used and cleaned with pure ethanol alcohol.

The Second Step: Preparation of samples by cutting glass substrates to dimensions (1*1 ± 0.1 cm) and cleaned with distilled water then pure ethanol alcohol was used with ultrasonically for (15 min) to ensure good cleaning.

The Third Step: Wire explosion system set up which consist of vacuum chamber with spherical shape (12 cm diameter) was made from stainless steel and closed with flange, including a groove for vacuum sealing. The chamber was evacuated using a rotary pump (Varian D.S 602, Italy) connected to the chamber through PVC hose with a needle valve in order to get vacuum with ultimate pressure (10$^{-2}$ mbar) under ambient argon gas (2-3 bar) to avoiding oxidation of powder, with the utilization of 2.2 KV of explosion voltage and 100 J of storage energy. The bank capacitor was 40 µF and spark gap distance of (1 mm).

The Forth Step: Inspections which include:

1- Pulse Current Measurement Results

The current was equal to (~ 100 KA) and has been determined by current probe using equation (1) which taking from current probe data sheet. Peak voltage from Figure 1 equal to (4.24 V) and the peak pulse width equal to (14 µsec) as shown in Figure 2, while the Figure 3 shows the voltage breakdown of wire explosion.

\[
\text{Current transform ratio} = \left( \frac{\text{Peak Voltage (V)}}{0.01 (V/A)} \right) \times \text{No. of wire (1)}
\]

Figure 1: Current waveform of copper wire explosion (Peak voltage)

Figure 2: Current waveform of copper wire explosion (Peak pulse width)
II- X-Ray Fluorescence (XRF) Results
The sum concentration of Cu was (99%) and other elements were (1%) as illustrated in Table 1.

III- X-Ray Diffraction (XRD) Results
Figure 4 shows the XRD patterns of prepared copper nanopowder, which shows a matching with standard values in (JCPDS card No.04-0836). Table 2 shows the results of XRD of prepared copper nanopowder. This table was used to determine the, lattice constants according to the equations (2) and (3) as shown below [18]:

\[ 2d \sin \theta = n \lambda \]  
\[ \frac{1}{d^2} = \frac{h^2+k^2+l^2}{a^2} \]  
(Cubic)

The lattice constant was (a=3.614 Å), which was very close to card standard values (a=3.615 Å), and copper nanopowder has a polycrystalline with, a cubic system which highly oriented along (111) plane. No characteristic peaks for any other impurities are observed, suggesting that the sample has a high purity.

IV- Scanning Electron Microscopy (SEM) Results
Figure 5 shows the SEM micrograph image of prepared copper nanopowder, with magnification of 100 Kx, it can seem that the surface of the Cu nanopowder contains agglomeration of spherical grains microstructure with uniform distribution. The grains appear homogeneous, uniform and tightly packed suggesting that there is a uniform nucleation throughout the surface.

Table 1: XRF analysis elements of copper wire

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>99</td>
</tr>
<tr>
<td>Mg</td>
<td>0.030</td>
</tr>
<tr>
<td>Al</td>
<td>0.0814</td>
</tr>
<tr>
<td>Si</td>
<td>0.0023</td>
</tr>
<tr>
<td>Sn</td>
<td>0.0016</td>
</tr>
<tr>
<td>S</td>
<td>0.0020</td>
</tr>
<tr>
<td>Ag</td>
<td>0.0012</td>
</tr>
<tr>
<td>Cr</td>
<td>0.0562</td>
</tr>
<tr>
<td>Fe</td>
<td>0.0418</td>
</tr>
<tr>
<td>Co</td>
<td>0.0308</td>
</tr>
<tr>
<td>Ni</td>
<td>0.0052</td>
</tr>
<tr>
<td>W</td>
<td>0.0070</td>
</tr>
<tr>
<td>Mo</td>
<td>0.0101</td>
</tr>
</tbody>
</table>

Table 2: XRD data of Cu nanopowder

<table>
<thead>
<tr>
<th>Peak No.</th>
<th>$2\theta_{ST}$</th>
<th>$2\theta_M$</th>
<th>$I_{ST}$%</th>
<th>$I_M$%</th>
<th>(hkl)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>43.325</td>
<td>43.297</td>
<td>100</td>
<td>100</td>
<td>111</td>
</tr>
<tr>
<td>2</td>
<td>50.470</td>
<td>50.433</td>
<td>30</td>
<td>46</td>
<td>200</td>
</tr>
</tbody>
</table>

where: $2\theta_{ST}$: Standard angle (degree), $2\theta_M$: Measured angle (degree), $I_{ST}$%: Standard intensity of peaks (CPS), $I_M$%: Measured intensity of peaks (CPS), hkl: Miller indices
V. Atomic Force Microscopy (AFM) Results

Figure 6 (a,b) show surface morphology of the prepared copper nanopowder as observed from the (2-D) and (3-D) AFM micrograph, the surface shows agglomeration of small clusters of copper nanopowder and the concentration of these clusters is higher near the surface. The particle distribution was obtained from the multiple AFM image and is shown in Figure 7 and the average particle size in the range of (20-40 nm) were produced. The grain size distribution is nearly Gaussian type.

![AFM images of copper nanopowder](image)

**Figure 6: (2-D and 3-D) AFM images of copper nanopowder**

![Grain size distribution of copper nanopowder](image)

**Figure 7: Grain size distribution of copper nanopowder**

4. Conclusion

High purity copper nanopowder was fabricated using wires explosion technique, the surface of the Cu nanopowder contains spherical nanosized grains with uniform distribution. The grains appear homogeneous, uniform and tightly packed and the powder has a polycrystalline structure with a cubic system. The average particle size of the prepared copper powder was in the range of (20-40 nm) which makes this nanopowder suitable for many applications such as additives in lubricating and as catalyst.

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References


