ISSN: 2277-9655
Scientific Journal Impact Factor: 3.449
(ISRA), Impact Factor: 1.852

IJESRT

INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

Production of Nanocopper by Electrodeposition Methods

Sami Masadeh
Materials Engineering Department
Al-Balqa Applied University
AL-Salt Jordan
smasadeh@yahoo.com

Abstract

Nano copper powder was prepared by electrochemical techniques (electro-deposition), a cell consisting of two electrodes made of copper plate (anode) and a steel plate (cathode). Current densities 1, 5, 10 A/dcm^2 were used; all electro deposition experiments were carried out at ambient temperature. The dissolution of copper at the anode and the reduction of copper ions at the cathode were collected as a powder at the steel plate. Scanning electron microscopy and x-ray diffraction analysis were performed. Results showed that a particle size was a function of current density applied, as current density decreased, smaller particle size obtained. Removal of electro deposited copper over substrate was easier at lower current densities used, due to less adhesion of copper to substrate.

Keywords: nanomaterial, nano copper, electrolysis, electrode deposition.

Introduction

Nanotechnology has become the most fashionable science since the end of the last century.
Since then, a lot of effort has been made to achieve numbers of multifunctional materials with simple synthetic procedure. At the same time, investigations of easy processing for subsequent applications have become more and more popular as well. Chemical and electrochemical preparation methods are one of the possible and powerful options for the fabrication of a new class of nanomaterials. Many popular methods used for nanostructures fabrication need to involve very expensive devices like: UHV chambers equipped with MBE or sputtering, etc. It is useful to develop methods which are less expensive and lead to similar quality final products. From the application point of view, methods which allow to perform mass production in a relatively easy way were sought. Such a method is electrochemistry, which allows us to deposit a large variety of materials in many different forms from various solutions. Chemical methods also provide an opportunity to obtain nanomaterials in big quantities. Recently, hybrid nanomaterials and nanocomposites have been studied extensively because of new opportunities of the fabrication of a novel class of materials which use nanostructures as building blocks. Such subsequent hierarchical ordering of constituent nanoelements often enhance their needed magnetic, electrical, optical, structural and mechanical properties and extinct unneeded one [1]. In many cases, the tubular or elongated structures have a significant advantage over the round ones due to a possible selective interaction with the environment with or without linkage chemistry. Electrochemical deposition is a very attractive method because the process is simple and effective. There is a huge variety of possibly reduced ions, and it has no limitation as far as sample shape and size are concerned. Deposition of the layer can be done both in constant, also called direct (DC), and accelerating current (AC) modes depending on the needs, possibilities or applications and wires characteristics. In the first case, constant current is applied to electrodes whereas in the second one the potential of working electrode is controlled. Electrochemical deposition of nanowires is a technique which combines either bottom-up or top-down approaches. This is due to the fact that the wires have grown atom by atom manner and it can be obtained in the matrixes which were subjected to anodization
process during which the nanopores are obtained in bulk material.
Before the templates can be used for the deposition of, e.g., nanowires or nanotubes, it
should obey few requirements:
- it should be chemically inactive in a particular process;
- deposited material should wet the pores wall;
- deposition process should not be chaotic and start from the bottom or from the wall of

ISSN: 2277-9655
Scientific Journal Impact Factor: 3.449
(ISRA), Impact Factor: 1.852

the pore and go upwards or to the center, respectively.

Nano materials constitute an emerging sub-discipline of the chemical and materials sciences that deals with the development of methods for synthesizing Nanoscopic particles of a desired material and with scientific investigations of the nanomaterial obtained (2-7). Nano materials have numerous possible commercial and technological applications including uses in analytical chemistry (8-11), electronic, optical and mechanical devices (2,3,12), drug- delivery (13), and bio encapsulation (14). In addition, this field poses an important fundamental philosophical question - how do the properties (e.g., electronic, optical, magnetic) of a nanoscopic particle of a material differ from the analogous properties for a macroscopic sample of the same material?

Nano-copper is the basic raw material indispensable of good conductivity, high strength of the copper nanoparticles have received much attention and exhibited excellent applications for their good friction reduction and wear resistance properties [15]. Many influencing factors have been considered and the tribological behaviors of Cu nanoparticles as additive have been investigated, such as the concentration of nanoparticles in oil, sliding speed, applied load, contact form of friction pairs and lubricating oils [16]. However, few researchers have studied the high temperature tribological behaviors of Cu nanoparticles. It is well-known that the actual service temperature of lubricating oil is very high (90−100 ℃ or even higher than 150 ℃). Therefore, it is very important to study the influence of temperature on the lubricating properties of Cu nanoparticles as additive.

Furthermore, although considerable efforts have been made to understand how nanoparticles work as additives in oil to reduce friction and wear, their mechanism is still not very clear and the uncertainties exist between the following two viewpoints: ball bearing mechanism [17], and film forming mechanism [18]. For the ball bearing mechanism, chemical/mechanical reactions do not occur, and the researchers consider it still as an assumption without direct experimental evidence due to the deficiency of in situ characterization method. Recent studies [19] indicate that local overheating in friction may initiate deposition of copper and forming of a thin protective film on the worn surface, hence, the friction and wear have been reduced. However, few researchers have described the mechanical properties of the protective film [20]. It is clear that the mechanical properties of materials strongly influence the tribological behaviors, especially for coatings and thin solid films on a metal substrate. Hence, measuring the micro mechanical properties (Nano hardness and elastic modulus) of the protective film that is formed by Cu nanoparticles would be helpful to understanding the tribological mechanism of nanoparticle additives and providing reference data for their practical application as lubricating materials.

In this work, the aim is to investigate the effect of current density, on electro deposition of Nano copper powder and then on particle size.

Experimental procedure

Copper electrical leads 6 mm in diameter were used as working anodic electrodes. Steel plates having 100 cm2 total surface areas were used as cathodes over which electro deposition of copper to take place. Prior to the plating process, steel sheets were mechanically polished with 0.5 µm Buhler suspension, and then washed and ultrasonically cleaned. Saturated copper sulfate solutions were prepared and used as the electrolyte, no addition to acids or fluxes were made in order to make the adhesion on steel sheets weak and ease its removal.

Current densities 1, 5, 10 A/ dcm^2 were used; all electro deposition experiments were carried out at ambient temperature. Each test was run for 30 minutes. The collected copper powder on the cathodes was collected dried by hot air stream for particle size determination. The experimental setup is shown in Figure 1.

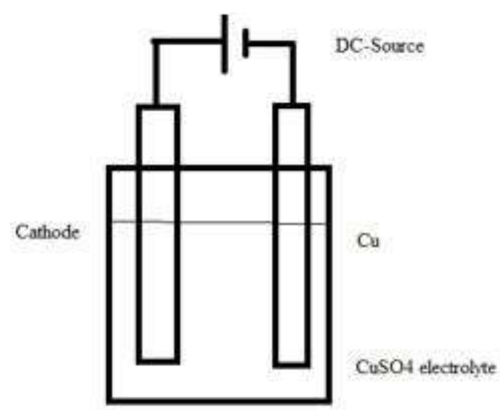

Figure 1: The experimental setup

Results and discussion

Two kinds of deposition processes can be considered with controlled potential or current.
Current controlling demands two electrode systems with voltmeter and ampere-meter devices. Controlling of the potential adjusted to a stable value is performed in three electrode setups, where, besides working and reference, a counter electrode is needed.

ISSN: 2277-9655
Scientific Journal Impact Factor: 3.449
(ISRA), Impact Factor: 1.852

This electrode should be chosen in such a way that it has a significant surface so that the current obtained in the electrolysis process is not changing its potential. This electrode is applied to the working electrode's constant current, which stabilizes constant potential in the reference electrode. A potential value of the electrode is controlled by potentiostat. Accelerating current mode demands a two electrode device, where working and reference electrodes can be made from the same material. Constant current deposition can be carried out without adjusting other current parameters before the deposition process. In case of deposition with the controlled potential, its value should be calculated from the equations described below to assure large enough process efficiency. It is also possible to run voltoamperometric or chronoamperometric curves and on its ground a characteristic value of the deposition process is established. Such specific potential has to be calculated for every type of electrolyte and electrodes. Electrodeposition is a method by which ions from the solution are deposited at the surface of the cathode (working electrode). This process can run parallel to electrolysis, and deposited material can form a continuous layer, wires or tubes when prestructured matrixes are used. The amount of the deposited material, thickness of the layer or wires length depends on the deposition time matrix structure. Electrolysis is a number of processes taking place in electrolyte and at electrode interfaces while electrical current is applied from external sources. In electrolysis the change from electrical energy to chemical potential takes place. The most important reactions proceed at the interfaces electrode metal/solution. When the electrode is dissolved and in oxide form is present in the solution, this electrode we call anode. The opposite electrode – cathode, is one where ions are reduced and metallic surface is formed. During the electrolysis process, large gradients of ions concentrations are observed. The reactions taking place at the electrodes
are described in the following form [12]:

$$C(-): Ox_A + z_A e \rightarrow \mathrm{Red}_A$$
$$A(+): \mathrm{Red}_B \rightarrow Ox_B + z_B e$$

The removal of electro deposited copper over steel sheets was easier as current density decreased, as current density increased the adhesion of copper on steel sheet cathodes increased, this is in agreement with previous studies findings (21).
As current density increases, the hitting force on the cathode would be higher due to higher potential. Removal of electrodeposited copper over cathodes using 10 A/dcm2 is shown in Figure2, and, Figure3. Figure3 shows the x-ray analysis of this powder, it is noticed that the powder contained Fe, C, and S other than copper. Mechanical removing process of copper caused scrubbing some of steel sheet.

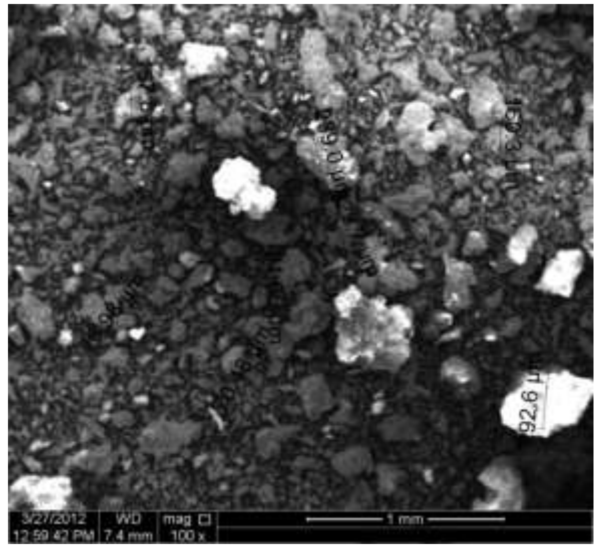

Figure 2: The micrograph of collected copper powder over when using 10 A/ dcm^2, current density

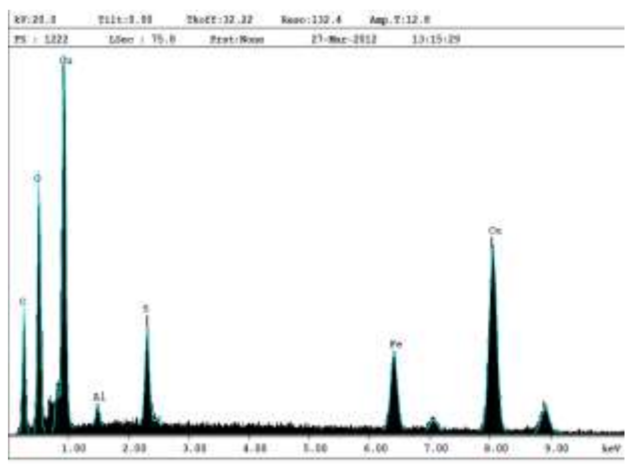

Figure3: X-Ray analysis of copper collected with 10 A/dcm2, current density.

Particle size of copper powder was in the range 20 µm- 300 µm. Micrographs of copper collected when using 5 A/ dcm^2 are shown in Figures 4,5, the x-ray analysis is shown in Figure 6. As is shown in Figures 4 and 5, particle size is smaller than those electrodeposited at 10 A/adm2, particle size was in the range 0.6-4 µm. Lower current density used allowed slower and smaller copper electro deposition on substrate. X-Ray analysis showed some Fe existence in the powder but less quantity when compared to powder collected over the substrate

ISSN: 2277-9655
Scientific Journal Impact Factor: 3.449
(ISRA), Impact Factor: 1.852

when using 10A/ dcm^2 current density; this was due to easier removal of copper powder over substrate.

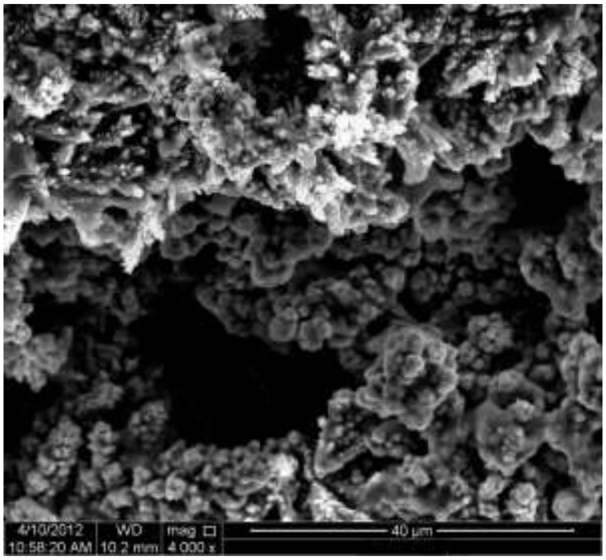

Figure4: SEM micrograph of copper powder electro deposited at 5 A/ dcm^2.

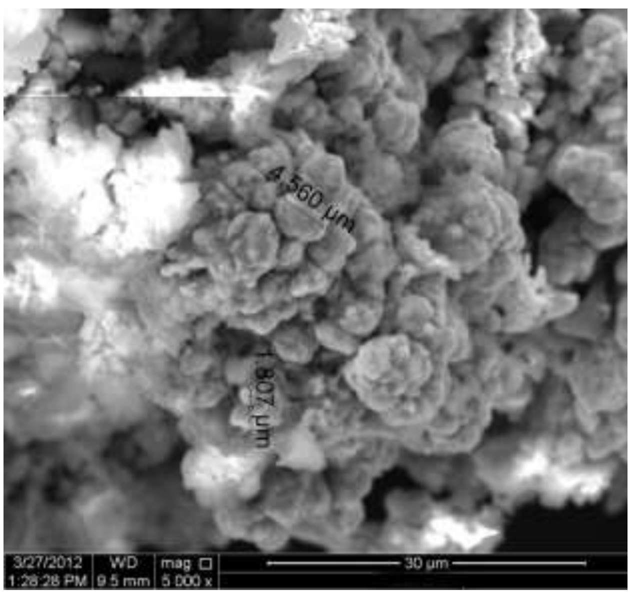

Figure5: Another SEM micrograph of copper powder electro deposited at 5 A/ dcm^2.

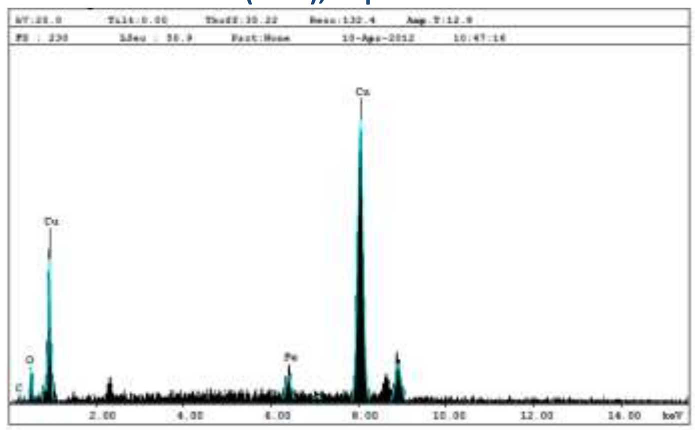

Figure 6: X-Ray analysis of copper powder electro deposited at 5 A/ dcm^2.

Best results were obtained with 1A/dm2 current density; this is shown in SEM micrograph Figure 7, and X-Ray analysis, Figure 8. Particle size down to 50 nm were obtained, this could be due to slower electro deposition of copper powder over substrate. X-Ray analysis (Figure 8) of this powder showed no noticeable contamination of powder with Fe.

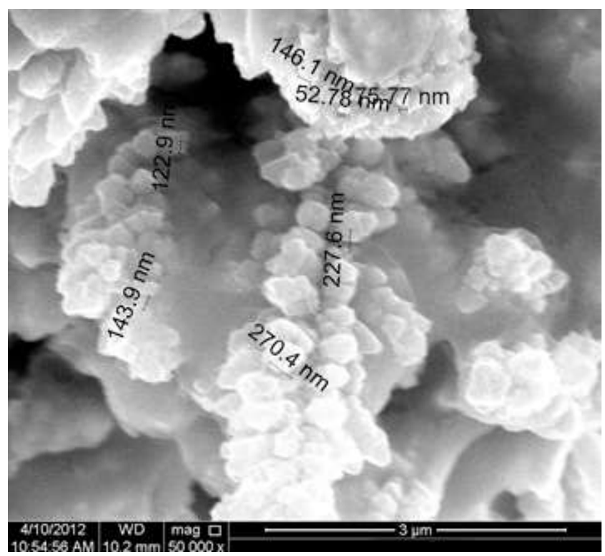

Figure 7: SEM micrograph of copper powder electro deposited at 1 A/ dcm^2.

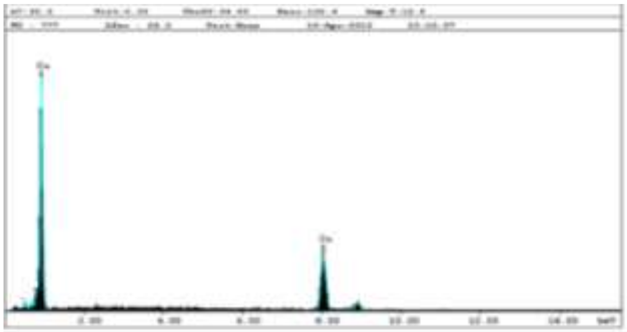

Figure 8: X-Ray analysis of copper powder electro deposited at 1 A/ dcm^2.

Conclusions

1- It is possible to get copper powder by electro deposition technique
2- Particle size is a function of current density
3- Particle size is directly proportional to current density used in electro deposition
4- Adhesion to substrate is also directly proportional to current density.

References

1. *Romero P. G., Sanchez C., Functional Hybrid Materials, (wiley, Weinheim, 2003) p 86*
2. *Bate R. T., Sei. A., 258: 96 (1988).*
3. *Science (special issue on nanomaterials), 25.4: 1300 (1991).*
4. *Martin C. R., Science. 266: 1961 (1994).*
5. *Martin C. R., Chem. Mater.. 8:1739 (1996).*
6. *Martin C. R., Ace. Chem. Res.. 28: 61 (1995).*
7. *Hulteen J. C., and Martin C. R., 1. Mater. Chem.. in press.*
8. *Medcalf E. A., Newman D. J., Gorman E. G., and Price C. P., Clin. Chem., 36:446(1990).*
9. *Simo J. M., Joven J., Cliville X., and Sans T., Clin. Chem., 40: 625 (1994).*
10. *Bangs L B., Clin 1., Immunoassav. 13: 127 (1990).*
11. *Rangs R, Pure Appl. Chem.. 68: 1873 (1996).*
12. *Ozin A., Adv. Mater.. 4: 612 (1992).*
13. *Gref R., Minamitake Y., Peracchia M. T., Trubetskoy V., Torchilin V., and Langer R., Science. 263: 1600 (1994).*
14. *Parthasarathy R., and Martin C. R., .1. Appl. Polv. Sei.. 62: 875 (1996).*
15. *YU Li-yan, HAO Chun-cheng, SUI Li-na, CUI Zuo-lin. Study on the improving frication and wear properties of lubrication oil with nanoparticales [J]. Journal of material science Engineering, 2004, 22(6): 901-905. (in Chinese).*
16. *Kharlmov V, Zolotukhina L V, Frishberg IV, Kishkoparov N V . The influence of Cu-sn alloy fine powder on mass transfer subjected to sliding friction [J]. Friction and Wear, 1999, 20: 333-338.*
17. *YU He-long, XU Bin-shi, XU Yi, WANG Xiao-Li, LIU Qian. Design of wear-out-failure in-situ repair parts by environment-friendly nanocopper additive [J]. Journal of Central South University of Technology, 2005, 12(S2): 215-220.*
18. *LIU Qina, XU Yi, SHI Pei-jing, YU He-long, XU BIN-shi. Analysis of self-repair films on friction surface lubricated with nano-Cu additive[J]. Journal of Central South University of Technology, 2005, 12(S2): 166-169.*
19. *Prentice G.A., Journal of Applied Electrochemistry, 28(1998).*
20. *Wu Y., Xiang J., Yang C., Lu W., Liber M.C., Nature 430 (2004) 61*
21. *Kim B. H., Jung J.H., Hong S.H, Joo J., Epstein A.L, Mizoguchi K., Kim J.W., Choi H.J., Macromolecules 35 (2002) 1419; W.U. Huynh, J.J.Dittmer, A.P. Alivisatos, Science, 295 (2002) 2425*