Current Density Driven Morphological Transitions in Copper Powder Electrodeposition within Nitrate Electrolytes

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ABSTRACT

Powdered copper is one of the key raw materials in advanced manufacturing processes and energy-related systems, where particle morphology and surface texture directly influence compaction behavior, sintering, and the final performance of the component or electrode. In this study, the effect of current density on the morphology of electrochemically deposited copper in a nitrate-based electrolyte was systematically investigated. Copper deposition was carried out galvanostatically from an aqueous copper nitrate solution containing polyvinylpyrrolidone (PVP) as an additive, under controlled hydrodynamic and thermal conditions. In all experiments, the electrolyte temperature, copper nitrate concentration, PVP concentration, sulfuric acid content, and deposition time were kept constant, and only the current density was varied. After each test, the deposits were collected, washed, dried, and then examined by scanning electron microscopy (SEM) at two different magnification levels.

Keywords: *Copper powder morphology, Copper nitrate electrolyte, Current density, Electrochemical deposition, Polyvinylpyrrolidone (PVP)*

1-Intrduction

Copper (Cu) is a technologically vital non-ferrous metal whose face-centered cubic (FCC) structure is associated with excellent electrical and thermal conductivity. These properties have made copper indispensable to modern industry, in particular for the electrical and electronics sector, which accounts for a major fraction of its global consumption[1]. Also, due to its suitable thermal conductivity and acceptable corrosion resistance, it is widely used in heat exchangers, heat-transfer parts, and industrial equipment [2]. In parallel, the metal's high recyclability and its pivotal role in emerging clean-energy technologies—such as electric vehicles and large-scale power transmission networks—have further underlined its strategic importance in recent decades[3]. Besides conventional bulk uses, copper in powder form has become an essential feedstock for a variety of advanced processes. It is extensively employed in powder metallurgy, the fabrication of porous and functional parts, conductive inks and pastes, certain additive manufacturing routes, and various energy-related systems. In all of these applications, the particle size distribution and morphology of the powder are key design parameters: they control compressibility, accessible surface area, sintering kinetics, and ultimately govern the performance and reliability of the final component or electrode[4].

2-Importance of Morphological Characteristics of Powder

Conventional routes for producing metal powders, such as mechanical milling and various atomization techniques, offer only limited flexibility when precise control over particle shape, narrow size distribution, and low contamination levels is required[5]. As an alternative, electrochemical deposition has attracted considerable interest as a versatile method for preparing copper powders with a broad range of morphologies—from relatively compact grains to highly dendritic structures—because the process is governed

by adjustable parameters including current density, electrolyte composition, and operating conditions [6]. A proper understanding of the electrochemical behavior of copper ions in solution, as well as the associated reduction and oxidation reactions, is therefore essential for analyzing and tailoring the deposition process. In copper deposition systems, metallic copper is formed at the cathode through the reduction of Cu ions, while the anodic reaction involves either dissolution of copper (for a soluble anode) or oxygen-evolving reactions such as water oxidation when an inert anode is used[7].

In general, metal powders are not merely intermediate products but functional starting materials whose processing behavior—such as flowability, apparent density, and size distribution—has a direct impact on the properties and reliability of the final part. Nikolić and Popov highlighted that specific surface area, apparent density, and particle size distribution are among the key descriptors that govern powder performance, and that these quantities are strongly dictated by the conditions under which the powder is generated[8]. Accordingly, for any copper-powder production route, accurate control and quantitative evaluation of particle size and morphology are both scientifically important and practically necessary. In electrochemically produced copper powders, a variety of particle shapes and surface textures can be obtained, and factors such as current density, temperature, and Cu-ion concentration are known to exert a pronounced influence on particle size, morphology, and even structural characteristics[9].

Several investigations have specifically addressed the role of current density in electrolytic copper powder production. It has been shown that variations in current density markedly alter the morphology and properties of the resulting copper powder[6]. Under galvanostatic conditions, dendritic structures are frequently observed, and their occurrence can be rationalized in terms of the underlying electrochemical conditions and mass-transport regime[9]. In another study, the effect of current density on the morphology and structure of copper powder/deposit was investigated, showing that changing the current can even affect structural features [10].

In this paper, the influence of current density on the morphology of electrochemically deposited copper powder in a nitrate electrolyte was systematically examined by using SEM observations.

3. Research Method

Electrolyte used in this study was prepared based on copper nitrate salt, and double-distilled water was employed as the solvent. Prior to each run, the solutions were thoroughly homogenized in order to minimize potential variations arising from solution non-uniformity. In these experiments, different current densities were employed as one of the key operational parameters affecting growth and morphology, and the effect of current density on the morphology of electrochemically deposited copper in a nitrate medium was examined and presented using SEM images.

To ensure repeatability, the order of reagent addition was kept constant, and the mixing conditions—including stirring time, stirring speed, prevention of vortex formation, and standardization of hydrodynamic conditions—were controlled and maintained. In addition, ***the electrolyte volume and the electrode immersion depth were kept constant*** *so that variations in mass transfer arising from these factors would not influence the results. Sufficient time was allowed for temperature stabilization prior to the onset of electrodeposition, and before applying the current, the solution was visually inspected for clarity and uniformity. Electrodeposition was initiated by applying a constant current, and the duration of each run was set according to the experimental design.*

After the predetermined time had elapsed, the current was switched off and the electrodes were carefully removed in order to minimize mechanical damage to the formed structures. To reduce potential carry-over effects, the cell and associated equipment were washed as required, and the subsequent run was performed following the same controlled sequence. After each run, the resulting deposit and powder were collected. To minimize the influence of residual electrolyte on morphology observation, a stepwise washing procedure was employed: initially with water to remove soluble salts, followed by ethanol to facilitate drying and reduce particle agglomeration. Finally, the samples were dried under controlled conditions, and ***SEM*** *images were acquired.*

4. Results and Discussion

To specifically evaluate the influence of current density on the morphology of copper electrodeposits, all operating parameters were kept constant, and only the level of current density in the nitrate electrolyte was systematically varied. The fixed deposition conditions are summarized below:

- *Electrolyte temperature:* ***25 °C***

- *Copper nitrate trihydrate concentration:* ***36.2 mg/L***
- *polyvinylpyrrolidone (PVP):* ***300 ppm***
- *Deposition time:* ***60 min***
- *Sulfuric acid concentration:* ***11.5 ml***

SEM micrographs were acquired at two comparable length scales (100 µm for an overview and 10 µm for higher-magnification morphology) to enable a direct visual comparison between the two cases.

In Figure 1, the surface morphology of the copper deposit obtained at a current density of 1 A·cm^{-2} is shown at two different magnifications. In the low-magnification image (scale bar 100 µm), the deposit exhibits a relatively compact and dense structure with a strongly oriented, striated surface. The entire surface is covered by almost parallel ridges and grooves, while only a limited number of small nodular agglomerates can be observed on top of this background. The high-magnification micrograph (scale bar 10 µm) confirms that the deposit is composed of elongated ridges with micrometer-scale height differences and irregular nodular protrusions with typical sizes of about 1–3 µm. At this current density, no coarse dendritic arms or open, highly porous regions are observed, indicating that the growth at 1 A·cm^{-2} remains moderately stable and the resulting copper layer is still relatively coherent and compact.

Figure 2 presents the surface morphology of the copper deposit obtained at a higher current density of 3 A·cm^{-2}, again at the same two magnification levels. In the low-magnification SEM image (scale bar 100 µm), bulky nodular/cauliflower-like clusters protruding from the surface are clearly visible, which demonstrate strong growth localization and a loss of planarity and uniformity of the deposit. The high-magnification image (scale bar 10 µm) shows that these clusters are built from numerous platelet- or flake-like crystallites arranged in a disordered, radially stacked manner, leading to a significantly more porous and heterogeneous texture compared with the deposit obtained at 1 A·cm^{-2}.

A comparison of Figures 1 and 2 indicates that increasing the current density from 1 to 3 A·cm^{-2} drives the system from a relatively stable growth regime, in which a dense layer with an anisotropic ally striated texture is formed, to an unstable regime dominated by mass-transport limitations and tip-enhanced growth. Under these conditions, rapid depletion of Cu^{+2} ions near the cathode and the resulting concentration gradients promote the formation of three-dimensional nodular and cauliflower-like features, accompanied by an increase in surface roughness and porosity.

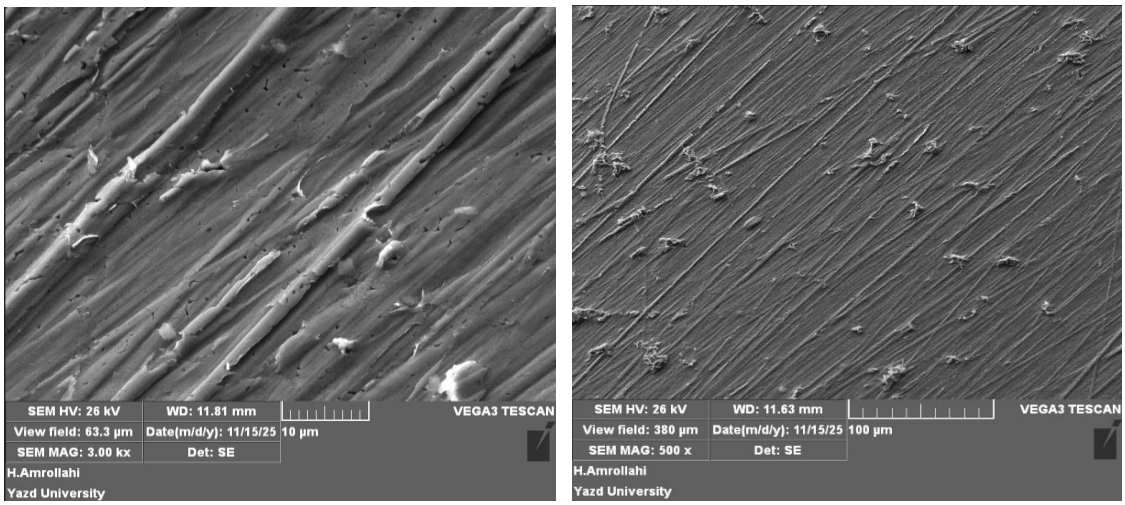

Figure 1. SEM images of electrochemically deposited copper in a nitrate electrolyte at current density 1A. Cm^{-2}, at two magnifications: (a) 100 µm and (b) 10 µm

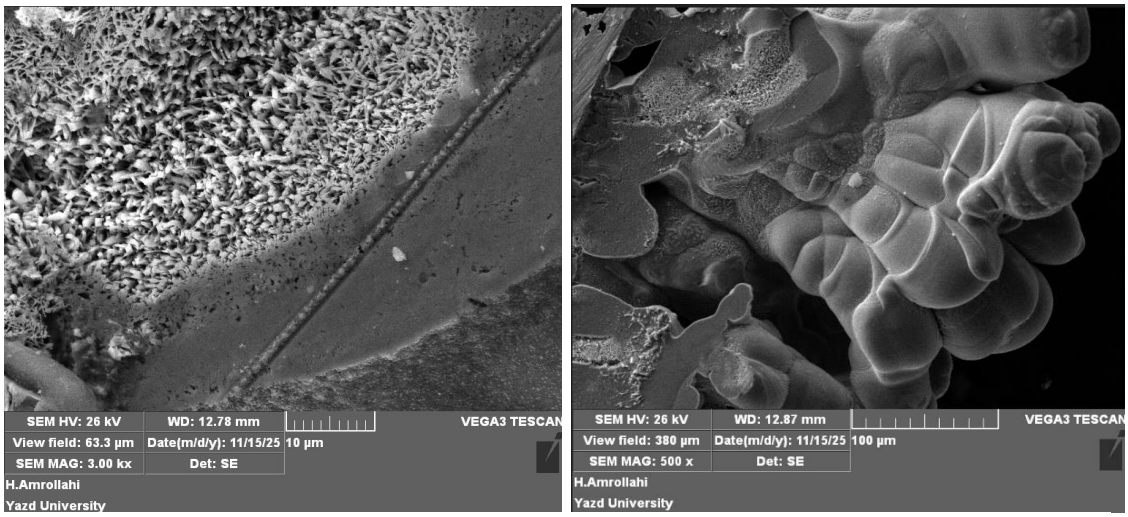

Figure 2. SEM images of electrochemically deposited copper in a nitrate electrolyte at current density 3A. Cm^{-2}, at two magnifications: (a) 100 µm and (b) 10 µm.

5. Conclusion

Increasing the current density from 1 to 3 $A{\cdot}cm^{-2}$ shifts the copper deposition from a stable, compact striated morphology to an unstable regime dominated by nodular/cauliflower-like growth with higher roughness and porosity. This confirms that current density is the primary parameter governing the microstructural evolution of electrodeposited copper powder.

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