

MARIJA KORAĆ¹
ŽELJKO KAMBEROVIĆ¹
MILOŠ TASIĆ²
MILORAD GAVRILOVSKI²

¹Faculty of Technology and
Metallurgy, University of Belgrade,
Serbia

²Scientific Research Centre,
Užice, Serbia

SHORT COMMUNICATION

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NANOCOMPOSITE POWDERS FOR NEW CONTACT MATERIALS BASED ON COPPER AND ALUMINA

This paper is a contribution to characterization of Cu-Al₂O₃ powders with nanostructure designed for the production of dispersion strengthened contact materials. New materials with predetermined properties can be successfully synthesized by utilizing the principles of hydrometallurgy and powder metallurgy. The results show a development of a new procedure for the synthesis. The applied characterization methods were differential thermal and thermogravimetric analysis (DTA-TGA), X-ray diffraction (XRD), scanning electron microscopy (SEM), Transmission Electron Microscopy (TEM): Focused Ion Beam (FIB) and Analytical Electron Microscopy (AEM). Nanostructure characteristics, particle size in range 20-50 nm, and uniform distribution of dispersoids in copper matrix were validated.

Key words: electrical contacts; copper; alumina; nanocomposites; characterization.

The intensification in research of nanostructure materials in recent years has occurred primarily due to their attractive potential, *i.e.* mechanical and physical properties significantly improved compared to the conventional grain materials. The synthesis of powders represents the starting and crucial stage in the production of sintered metal materials with required properties. Considering that the starting structure undergoes certain changes during further processing, but remains essentially preserved in the structure of the final product [1], there is an increased necessity for a great number of methods for producing powders.

The production of powders by the thermochemical method is not a new procedure, but in recent years, due to the development of contemporary materials with pre-set properties, there has been an extended interest in this method, especially for the production of nanostructured powders [2-4]. The introduction of fine dispersed particles into the metal matrix have significant reinforcing effects which can be kept at elevated temperatures [2-6]. For such reinforcement, ultra-fine and nano-particles of oxides are suitable, because they, due to their hardness, stability and insolubility in the base metal, represent obstacles

to dislocation motion at elevated temperatures without a significant effect on conductivities, both thermal and electrical [7-9]. The research of dispersion strengthened materials points out the significance of the properties of the starting powders and the importance of the starting structure preserved in the structure of the final product [10].

A very important aspect of dispersion strengthening is an even distribution of oxide particles, their fine dispersion, especially in nanometer scale, and the introduction of as small as possible amount of dispersed particles into the volume of the base metal.

In this research, copper was chosen as a base metal for its high electrical and thermal conductivity, but low mechanical properties. Copper matrix was dispersion-strengthened by alumina through thermochemical route. Alumina particles were chosen due to their exceptional properties, such as high melting point, high hardness, excellent thermal stability and chemical inertness. Also, alumina can increase the temperature of recrystallization and demonstrate the excellent strength at an elevated temperature by pinning grain and sub-grain boundaries of the copper matrix and blocking the movement of dislocations [5,11,12]. The amount of alumina used for dispersion strengthening is usually from 0.5-5.0 wt. % [5], but significant results regarding a particle size could be achieved even with higher amounts like 50 wt. % of Al₂O₃ [13].

Corresponding author: M. Korać, Faculty of Technology and Metallurgy, University of Belgrade, Serbia.

E-mail:

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These materials are widely applied in the field of electronics and electrical engineering as highly conductive materials for the operation at elevated temperatures, as electrodes for spot welding, different contact materials, various switches, thermal and electric conductors, microwave tubes, commutators for starting helicopter engines, relays, catalysts with a high degree of conversion, coatings with low porosity and high adhesion, *etc.* [14].

EXPERIMENTAL

Water soluble nitrates of copper and aluminium, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, were used to synthesize a two-component nanocomposite $\text{Cu-Al}_2\text{O}_3$ powder by the thermochemical procedure through several phases: obtaining 50 wt. % water solution of copper and aluminium nitrates, spray drying at 180 °C for the production of a precursor powder, annealing of the precursor powder in the air atmosphere at 900 °C for 1 h and the reduction of hydrogen atmosphere at the temperature of 400 °C for 1 h. Previous publications by the authors give a detailed description of the procedures [15,16] and process parameters [17,18] for the synthesis of two-component nanostructured composite materials.

The results of determination of fluidness, pouring density and specific area of the obtained nanostructured composites with different amounts of Al_2O_3 dispersed in the copper matrix show that all investigated powders are not fluid and that mean values of pouring density and specific area are the same for different contents of Al_2O_3 (1.04 g/cm³ and 0.75 m²/g, respectively). Also, in previous work of the authors [16] the results of differential thermal and thermogravimetric analysis (DTA-TGA) and scanning electron microscopy can be found, which show the flow of phase transformations during the process of oxidation, as well as the morphology of the obtained powders.

RESULTS AND DISCUSSION

In a prior research [16], the authors used Scanning Electron Microscopy (SEM) for the assessment of the morphology of a produced powder, a particle size and distribution. In recent investigations, the authors used a Focused Ion Beam (FIB) for the analysis.

The FIB is a scientific instrument that resembles a scanning electron microscope. However, while the SEM uses a focused beam of electrons to image the sample in the chamber, a FIB uses a focused beam of gallium ions instead.

Besides finer particles at 10000× magnification, as presented in Figure 1, the agglomerated particles are also noticeable. The agglomerates, size 2-5 μm, are sponge-shaped. Specifically, the agglomeration of finer particles is the result of their large surface, high surface energy, and an effect of the bonding forces between them. Due to the atomic connections at the interface, attracting strains are created, the magnitude of which depends directly on the surface energy of the particles that are in contact. For the production of non-agglomerated powders, which contribute in later stages of the powder processing, surfactants, such as poly (ethylene glycol) [2], and ultrasonic treatment [19,20] could be used.

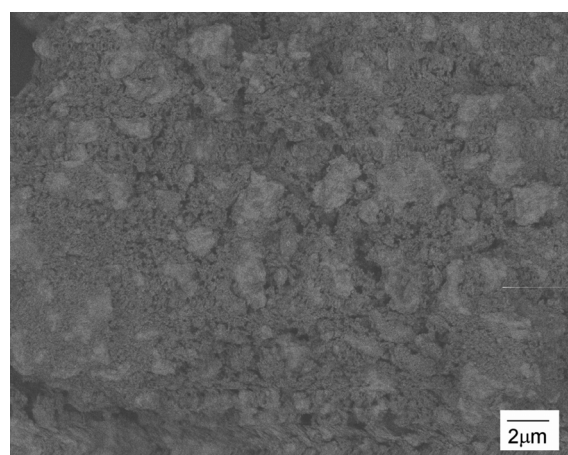


Figure 1. FIB image of Cu-5 wt. % composite powder.

The qualitative X-ray diffraction analysis of the powders was performed using a Siemens D500 PC diffractometer, CuK_α radiation, in the 2θ range 0-100° with a step of 0.02°. X-ray diffraction analysis (XRD) was performed on the powder precursor after spray drying (Figure 2), dried powder after heat treatment (Figure 3) and after the reduction (Figure 4).

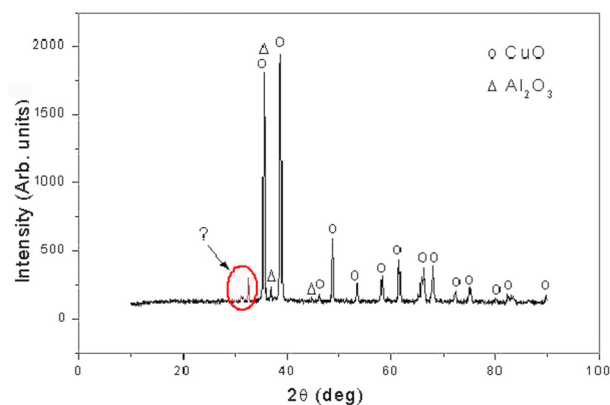


Figure 2. XRD of the Cu-3 wt. % Al_2O_3 powder after thermal treatment.

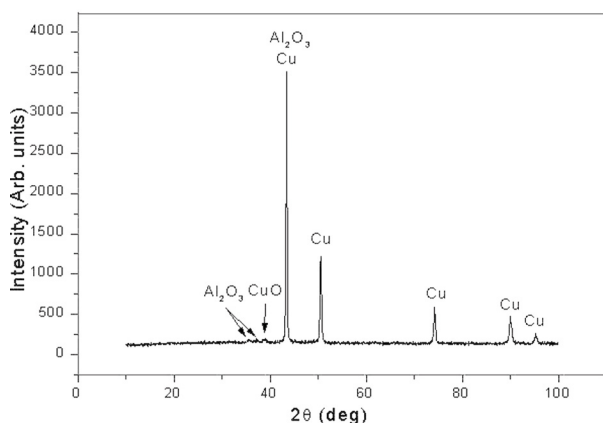


Figure 3. XRD of the Cu-3 wt. % Al_2O_3 powder after reduction.

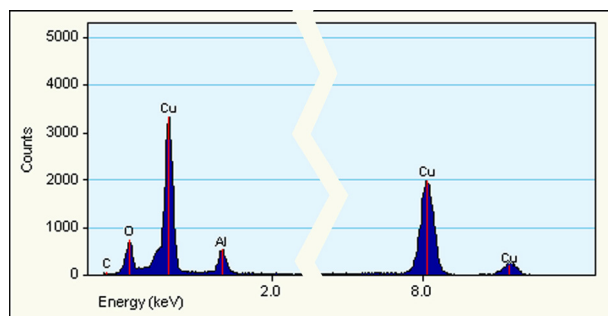
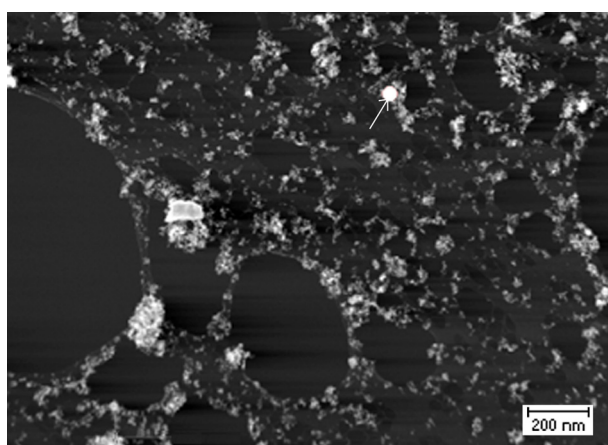


Figure 4. AEM analysis of Cu- Al_2O_3 powder with 5 wt % of Al_2O_3 .

In Figure 2 (the X-ray differential analysis after annealing of dried powder) detected peaks correspond to CuO and Al_2O_3 and the one that could not be identified. According to Lee [14], this peak corresponds to the third phase, $\text{Cu}_x\text{Al}_y\text{O}_z$, which appears in the structure due to eutectic reaction of $(\text{Cu} + \text{Cu}_2\text{O})$ with Al_2O_3 . The formation of this phase is thermodynamically possible on Cu-Al contact surfaces. During eutectic joining of copper and Al_2O_3 , the eutecticum formed by heating up to eutectic temperature expands and reacts with Al_2O_3 creating $\text{Cu}_x\text{Al}_y\text{O}_z$, which is compatible from the both phases on the inter-surface.

XRD of the powder after the reduction show the presence of peaks corresponding to the elementary copper and Al_2O_3 . Also, Figure 3 shows the presence of copper oxide. Retained oxygen after the reduction is a consequence of the formation of third phase in the structure.

The produced powders were analyzed at the National Center for Electron Microscopy, University of California, Berkeley, by Analytical Electron Microscopy (AEM) with corresponding EDX (in marked spot) as presented in Figure 4.

Particles, 20-50 nm in size, are clearly noticeable, as well as the presence of agglomerates >100 nm. Particles are irregularly shaped with the presence of nodular individual particles, with the rough surface morphology. On EDX analysis, the peaks correspond to copper, aluminium and oxygen. Carbon peaks represent the reflection from the grid.

CONCLUSION

The presented characterization of the obtained powders points out the possibility of the synthesis of nanocomposite Cu- Al_2O_3 powders, particle size 20-50 nm, by a thermochemical method. The shape of the particles was irregular with the presence of nodular-shaped particles.

A relatively even distribution of the alumina in the nanocomposite system was achieved during the synthesis of the powder by depositing metals from the solutions of metal salts.

The identification of $\text{Cu}_x\text{Al}_y\text{O}_z$ phase in the structure and studying its influence to the stabilization of the dislocative structure, and thereby to the improvement of mechanical properties and accomplishing of a better combination of mechanical and electrical properties of the sintered systems is an essential aspect of this research and the same will be continued.

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