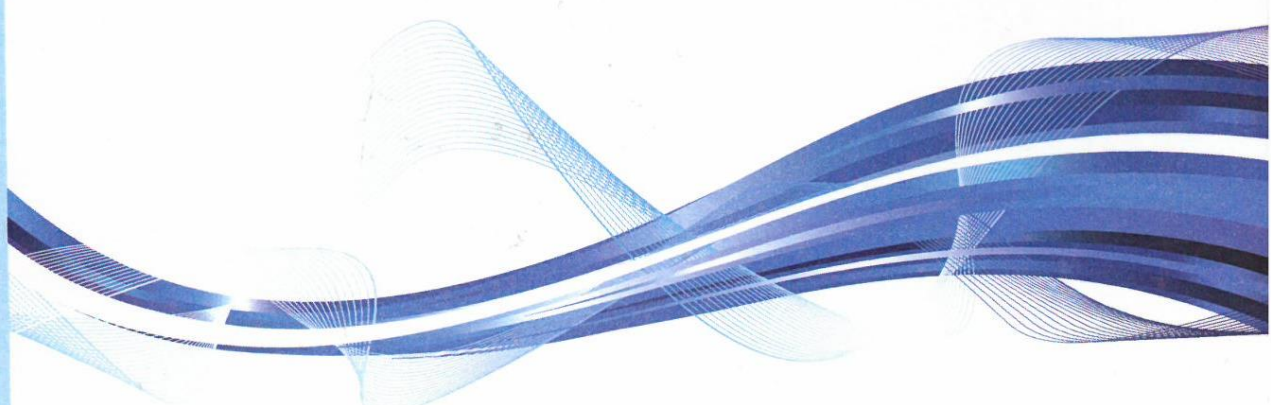


University of Belgrade
Technical Faculty in Bor and
Mining and Metallurgy Institute Bor



**49th International
October Conference
on Mining and Metallurgy**

PROCEEDINGS



Editors:
Nada Štrbac
Ivana Marković
Ljubiša Balanović

Bor Lake, Serbia
October 18-21, 2017

IOG 2017
International October
Conference

**PROCEEDINGS,
49th INTERNATIONAL OCTOBER CONFERENCE
on Mining and Metallurgy**

Editors:

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Technical Editor:

M. Sc. Uroš Stamenković

University of Belgrade, Technical Faculty in Bor

Publisher: University of Belgrade, Technical Faculty in Bor

For the publisher: Dean Prof. dr Nada Štrbac

Circulation: 200 copies

Printed by "Happy trend DOO", Zaječar, 2017

ISBN 978-86-6305-066-2

CIP - Каталогизacija у публикацији - Народна библиотека Србије, Београд

622(082)

669(082)

INTERNATIONAL October Conference on Mining and Metallurgy (49 ; 2017 ; Bor Lake)

Proceedings / 49th International October Conference on Mining and Metallurgy - IOC 2017,

Bor Lake, Serbia, October 18-21, 2017;

[organized by] University of Belgrade, Technical Faculty Bor and Mining and Metallurgy Institute Bor;

editors Nada Štrbac, Ivana Marković, Ljubiša Balanović. - Bor : University of Belgrade, Technical Faculty,

2017 (Zaječar : Happy trend). - XXIII, 664 str. : ilustr. ; 25 cm

Tiraž 200. - Bibliografija uz svaki rad. - Registar.

ISBN 978-86-6305-066-2

a) Рударство - Зборници b) Металургија - Зборници

COBISS.SR-ID 246349324

Bor Lake, Serbia, October 18-21, 2017



Conference is financially supported by
The Ministry of Education, Science and
Technological Development of the
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THE PARTICLE SIZE DISTRIBUTION OF Ag POWDERS OBTAINED BY CHEMICAL AND ELECTROCHEMICAL PROCESSES OF SYNTHESIS

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Abstract

The particle size distribution and the specific surface area (SSA) of silver powders obtained by chemical and electrochemical processes of synthesis have been analyzed. Silver particles produced by a chemical reaction with hydrazine as the reducing agent consisted of agglomerates of approximately spherical grains. The pine-like dendrites were formed by the potentiostatic electrolysis from the ammonium electrolyte at overpotentials inside and outside the plateau of the limiting diffusion current density. The values of average grain size and SSA for the chemically synthesized powder were between those obtained for the powders produced by electrolysis, that is detailed explained and discussed.

Keywords: silver; particle size distribution; specific surface area; chemical reduction; electrolysis

1. INTRODUCTION

Silver has found wide applications thanks to its unique optical, chemical and electrical characteristics [1, 2]. It is used in electronics, optics, as catalysts, gas sensing, chemical and biological sensing, in surface-enhanced Raman scattering (SERS), etc. Due to the strong antiviral, antibacterial and antifungal effects, silver is also used in medicine.

Different methods for the production of metal powders including mechanical comminution, chemical reaction, electrolysis and liquid metal atomization are used in practice [3, 4]. Powders are finely divided solids, smaller than 1000 µm in its maximum dimension. A particle is defined as the smallest unit of a powder. The particles of powder may assume various forms and sizes, whereas the powders, as an association of such particles, exhibit, more or less, the same characteristics as if they were formed under identical conditions and if the manipulation of the deposits after removal from the electrode was the same [3, 4]. The size of particles of many metal powders can vary in a quite wide range from a few nanometers to several hundreds of micrometers. The most important properties of a metal powder are: the specific surface, the apparent density, the flowability and the particle grain size distribution. These properties, called decisive properties, characterize the behavior of a metal powder.

Application of Ag in the powder form in some of the above mentioned areas is closely associated with the shape and size of the particles. In this study, we analyze and compare Ag powders obtained by the chemical and the electrochemical methods of synthesis. For the chemical synthesis, hydrazine as the reducing agent was used. The potentiostatic regime of electrolysis was used for electrochemical production of Ag powders. For that purpose, the ammonium electrolyte was used. From the mentioned properties characterizing the behavior of the powders, the particle size distribution and the specific surface area were analyzed.

2. EXPERIMENTAL

The procedure for the chemical synthesis of Ag powders using hydrazine as the reducing agent was described elsewhere [5].

For the electrochemical synthesis of Ag powders, the potentiostatic regime of electrolysis was used. Silver was electrochemically deposited at overpotentials of 625 and 925 mV from the ammonium electrolyte based on 0.10 M AgNO₃ in 0.50 M (NH₄)₂SO₄ to which was added NH₃ to dissolve present silver sulfate precipitate. The electrolytic production of Ag powders was performed in an open cell, at the room temperature using Pt cylindrical wire as the working electrode. The counter and reference electrode was of pure Ag. Ag powders were removed from the electrode surface every 10 min, rinsed with the distilled water and dried in a tube furnace under a controlled nitrogen atmosphere at 110–120 °C for 16 h.

Morphologies of chemically and electrochemically synthesized Ag particles were characterized using a TESCAN Digital Microscopy scanning electron microscope - model VEGA3, Czech Republic. The particle size distribution curves were obtained using a MALVERN Instruments Ltd, United Kingdom - MASTERSIZER 2000 device. The values of the specific surface area (SSA) were recorded using the Malvern software which controls the apparatus operation and processes the obtained data.

3. RESULTS AND DISCUSSION

The particle size distribution (PSD) curve for the chemically synthesized Ag powder is shown in Fig. 1a. Morphology of Ag particles corresponding to this particle size distribution curve is presented in Fig. 1b. As seen from Fig. 1b, agglomerates consisted of approximately spherical grains were formed by the process of silver reduction with hydrazine. The average size of the particles (Fig. 1a) was about 2.2 μm. The specific surface area (SSA) of this powder has been already determined [5] to be 0.0298 m²g⁻¹.

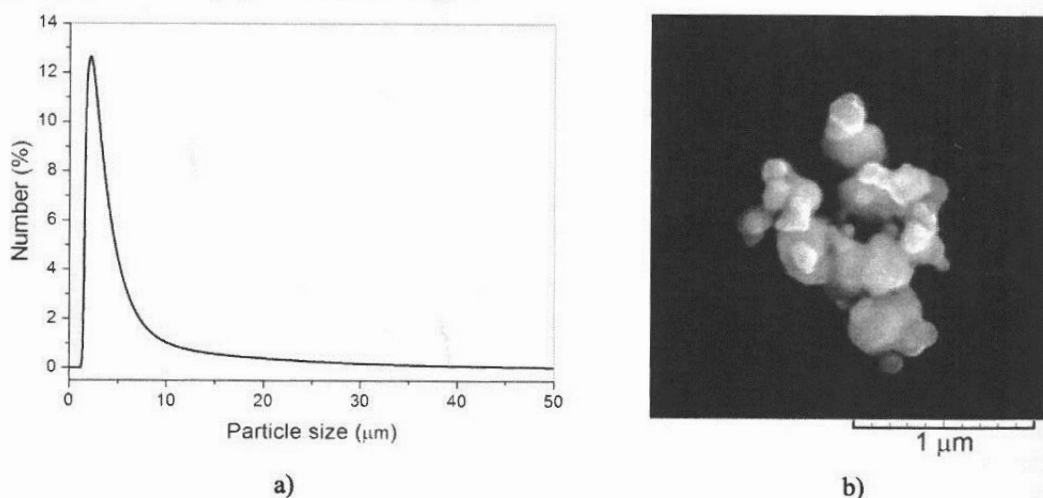


Figure 1 - (a) The particle size distribution curve, and (b) morphology of the chemically synthesized silver powder particles

The particle size distribution curves for the electrochemically produced Ag powders at overpotentials of 625 and 925 mV are shown in Fig. 2a. Increasing overpotential leads to the formation of smaller particles and to narrower particle size distribution curves. Figure 2b shows morphology of the particle obtained at an overpotential of 625 mV, while morphology of the particle obtained at an overpotential of 925 mV is shown in Fig. 2c. For this ammonium electrolyte, an overpotential of 625 mV belonged to the plateau of the limiting diffusion current

density, while an overpotential of 925 mV was for about 225 mV outside this plateau [5]. In the both cases, the 3D (three-dimensional) pine-like dendrites constructed from approximately spherical grains were formed. The average sizes of grains constructing these pine-like dendrites were about: 0.57 μm (625 mV) and 0.25 μm (925 mV). On the other hand, the average sizes of the particles extracted from Fig. 2a were 3.5 μm for the particles produced at 625 mV, and 1.7 μm for the particles produced by the electrolysis at 925 mV. For these Ag powders, the specific surface areas (SSA) were: 0.0208 m^2g^{-1} for the powder produced at 625 mV, and 0.0373 m^2g^{-1} for the powder produced at 925 mV.

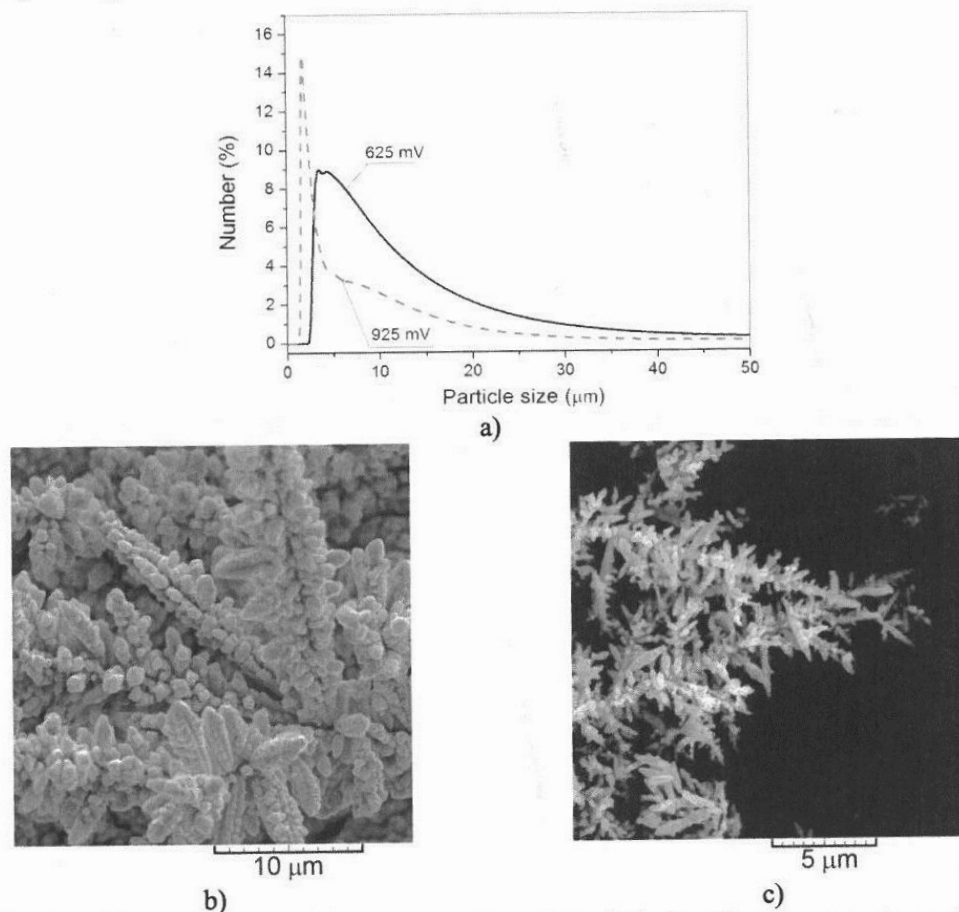


Figure 2 - The particle size distribution curves (a), and morphologies of the silver powder particles obtained by the potentiostatic electrolysis at overpotentials of: (b) 625 mV, and (c) 925 mV

It is clear from the above consideration that the values of PSD and SSA obtained for the chemically synthesized powder were between those obtained for the electrochemically produced powders at overpotentials at 625 and 925 mV. The increase of SSA value and the smaller particle size for the powder produced at 925 mV in relation to those produced at 625 mV can be explained as follows: increasing overpotential leads to the formation of a more disperse deposit characterized by the decreased particle size. This is due to the fact that the increase of overpotential leads to the decrease of the height of protrusion at which dendrites start to grow instantaneously. Hence, increasing overpotential means a larger number of growth sites suitable for growth of dendrites [4]. On the other hand, the velocity of dendrite growth has maximum for some optimal value of the dendrite tip radius. The optimal tip radius decreases with the increasing overpotential. With the dendrite tip radii larger than the optimal value, the difference between maximal and actual velocities of dendrite growth increases with the increasing

overpotential. Hence, smaller particles and narrower particle size distribution curves are expected with the increasing overpotential of powder formation.

4. CONCLUSION

The Ag powder obtained by the chemical process with hydrazine as the reducing agent was compared with the electrochemically produced powders obtained by the potentiostatic electrolysis from the ammonium electrolyte at overpotentials inside (625 mV) and outside (925 mV) plateau of the limiting diffusion current density. The best characteristics, i.e. the highest SSA value and the smallest average grain size showed the powder obtained by the electrolysis at 925 mV.

ACKNOWLEDGEMENTS

This work was supported by the Ministry of Education, Science, and Technological Development of the Republic of Serbia under the research project 172046.

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