FIRST INTERNATIONAL CONFERENCE ON ELECTRON MICROSCOPY OF NANOSTRUCTURES

ELMINA 2018

ПРВА МЕЂУНАРОДНА КОНФЕРЕНЦИЈА О ЕЛЕКТРОНСКОЈ МИКРОСКОПИЈИ НАНОСТРУКТУРА

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FIRST INTERNATIONAL CONFERENCE

ELMINA 2018

PROGRAM & BOOK OF ABSTRACTS

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August 27-29, 2018
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Organized by:
Serbian Academy of Sciences and Arts and Faculty of Technology and Metallurgy, University of Belgrade

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FIRST INTERNATIONAL CONFERENCE
ELMINA 2018
Program and Book of Abstracts

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At the beginning we wish you all welcome to Belgrade and ELMINA2018 International Conference organized by the Serbian Academy of Sciences and Arts and the Faculty of Technology and Metallurgy, University of Belgrade. We are delighted to have such a distinguished lineup of plenary speakers who have agreed to accept an invitation from the Serbian Academy of Sciences and Arts to come to the first in a series of electron microscopy conferences: Electron Microscopy of Nanostructures, ELMINA2018. We will consider making it an annual event in Belgrade, due to this year's overwhelming response of invited speakers and young researchers. The scope of ELMINA2018 will be focused on electron microscopy, which provides structural, chemical and electronic information at atomic scale, applied to nanoscience and nanotechnology (physics, chemistry, materials science, earth and life sciences), as well as advances in experimental and theoretical approaches, essential for interpretation of experimental data and research guidance. It will highlight recent progress in instrumentation, imaging and data analysis, large data set handling, as well as time and environment dependent processes. The scientific program contains the following topics:

- Instrumentation and New Methods
- Diffraction and Crystallography
- HRTEM and Electron Holography
- Analytical Microscopy (EDS and EELS)
- Nanoscience and Nanotechnology
- Life Sciences

To put this Conference in proper prospective, we would like to remind you that everything related to nanoscience and nanotechnology started 30 to 40 years ago as a long term objective, and even then it was obvious that transmission electron microscopy (TEM) must play an important role, as it was the only method capable of analyzing objects at the nanometer scale. The reason was very simple - at that time, an electron microscope was the only instrument capable of detecting the location of atoms, making it today possible to control synthesis of objects at the nanoscale with atomic precision. Electron microscopy is also one of the most important drivers of development and innovation in the fields of nanoscience and nanotechnology relevant for many areas of research such as biology, medicine, physics, chemistry, etc. We are very proud that a large number of contributions came from young researchers and students which was one of the most important objectives of ELMINA2018, and which indicates the importance of electron microscopy in various research fields. We are happy to present this book, comprising of the Conference program and abstracts, which will be presented at ELMINA2018 International Conference. We wish you all a wonderful and enjoyable stay in Belgrade.

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Synthesis and Characterization of $Na_{0.4}MnO_2$ as a Positive Electrode Material for an Aqueous Electrolyte Sodium-ion Energy Storage Device

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Due to the increasing use of batteries in everyday life and in industry, there is a need for developing cheaper batteries than the widely used lithium ion batteries. Lower price and higher abundance of sodium compared to lithium mineral resources intensified the development of Na-ion batteries. Aqueous lithium/sodium rechargeable batteries have attracted considerable attention for energy storage because they do not contain flammable organic electrolytes as commercial batteries do, the ionic conductivity of the aqueous electrolyte is about two orders of magnitude higher than in non-aqueous electrolyte and the electrolyte salt and solvent are cheaper. Various materials such as manganese oxides, vanadium oxide and phosphates have been used as electrode materials (cathodic and anodic) in sodium batteries due to high sodium intercalation ability in both, organic and aqueous electrolytes. The most frequently used type of manganese oxides are Li–Mn–O or Na–Mn–O systems due to their tunnel or layered crystal structures which facilitate the lithium/sodium intercalation-deintercalation [1, 2].

In this work, a glycine-nitrate method (GNM) was applied for the synthesis of cathode material $Na_{0.4}MnO_2$. Aqueous solutions of $NaNO_3$ and $Mn(NO_3)_2{\cdot}4H_2O$ were mixed in appropriate volume ratios in order to obtain desired stoichiometry and solid glycine was added in order to obtain a precursor solution with the molar ratio of glycine to nitrate was 1.2:1. The precursor solution was placed in a glass beaker and heated in an oven at 200°C until spontaneous ignition occurred. The ash, resulted from the combustion, was heated at 800 °C for 4 h.

The phase composition and crystal structure were analyzed by XRD on a Philips PW-1050 over a 2θ range 10–70° with a step of 0.05° and a counting time of 5

s. The particle morphology was examined by SEM/FIB (FEI SCIOS Dual Beam microscope) and TEM (FEI Talos F200X microscope) while elemental mapping was performed by EDS analysis. The electrochemical performance was tested by cyclic voltammetry (CV) using Gamry PCI4/750 in a three-electrode cell, consisting of a working electrode, platinum foil as the counter electrode and a saturated calomel electrode (SCE) as the reference electrode in saturated aqueous solution of $NaNO_3$. The working electrode was prepared by mixing 85 wt% $Na_{0.4}MnO_2$ powder, 15 wt% acetylene black and 5 wt% poly(tetrafluoroethylene) (PTFE) binder in N-methyl-2-pyrrolidone and, after homogenization in an ultrasonic bath, the suspension was transferred onto glassy carbon support.

The phase structure of the product annealed at 800 °C was identified as orthorhombic $Na_{0.4}MnO_2$ (JCPDS 27–0749) with tunnel structure which consists of MnO_6 octahedral units shared by corners and/or edges [3]. Also, some additional peaks were observed and identified as Mn_2O_3. TEM and SEM micrographs of obtained $Na_{0.4}MnO_2$ powder material are shown in Fig. 1. Uniform rod-like shaped particles can be observed, with average length and width of 300 nm and 80 nm, respectively. EDS analysis revealed that sample contained Na, Mn, and O in an appropriate ratio.

The cyclic voltammogram were performed in a saturate aqueous solution of $NaNO_3$ at scan rate of 20-400 mVs^{-1} within the voltage range from -1.30 to 1.35 V vs. SCE. The shape of CVs and peak positions were similar for all rates which means that the intercalation/deintercalation process of Na^+ is reversible even at high rates of charging/discharging. The specific capacity (mAh g^{-1}) was calculated as the area under redox peaks of CVs recorded at 20 mVs^{-1} (equivalent to 33 C). The initial discharge capacity of $Na_{0.4}MnO_2$ in $NaNO_3$ solution was 50 $mAhg^{-1}$ while after 15^{th} cycles the values increased by 9%. During cycling, this material demonstrated great efficiency (the ratio of capacity charge and discharge) of ~95%. This indicates that the material synthesized by glycine-nitrate method can be used in aqueous rechargeable sodium batteries [4].

References:

[1] J Liu *et al*, Green Energy & Environment **3** (2018), 20.
[2] F Hu and M Doeff, Journal of Power Sources **129** (2004), 296.
[3] S Liua *et al*, Journal of Power Sources **196** (2011), 10502.
[4] This study was supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia under Grant no III45014 and III45005 and the Serbian Academy of Sciences and Arts through the project F-190 "Electrocatalysis in the contemporary processes of energy conversion".

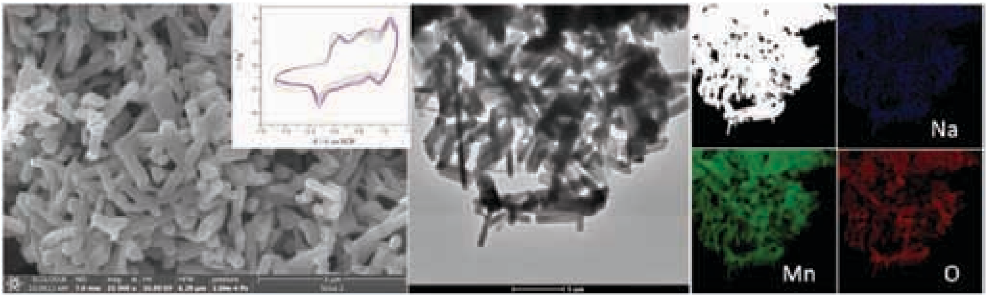

Figure 1. (Left) SEM micrograph of sample with CV in inset; (Middle) TEM micrograph of samples; (Right) HAADF STEM micrograph of samples with appropriate EDS maps of Na, Mn and O distribution.

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