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Enikő Tóth Szász

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STUDY OF NH_3BH_3 OXIDATION REACTION ON GOLD ELECTRODES BY *IN SITU* FOURIER TRANSFORM INFRARED SPECTROSCOPY AND DIFFERENTIAL ELECTROCHEMICAL MASS SPECTROMETRY

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Ammonia borane (NH_3BH_3) oxidation reaction (ABOR) was studied on gold electrodes using rotating disk electrode (RDE) and coupled physical techniques: on-line differential electrochemical mass spectrometry (DEMS) and *in situ* Fourier Transform Infrared spectroscopy (FTIR). Non-negligible heterogeneous hydrolysis in the low-potential region was asserted via molecular H_2 detection (Fig. 1). Thus, the number of electron exchanged per BH_3OH^- species is *ca.* 3 at low potential, and only reaches *ca.* 6 above 0.6 V *vs*. RHE. These figures were confirmed by Levich and Koutecki-Levich calculations. The nature of the ABOR intermediates and products was determined using *in situ* FTIR (Fig. 2). While BH_2 species were detected during the ABOR, its adsorption onto the Au electrode proceeds via the O atom, in opposition to what happens during the borohydride oxidation reaction (BOR). Therefore, the mechanism of the ABOR likely differs from that of the BOR. From the whole set of data (RDE, DEMS, FTIR), a relevant reaction pathway was proposed, including competition between the BH_3OH^- heterogeneous hydrolysis and oxidation at low potential, and preponderant oxidation at higher potential. Finally, a simplified kinetic modeling accounting with this reaction pathway was proposed, which nicely fits the stationary (*i vs. E*) ABOR plot.

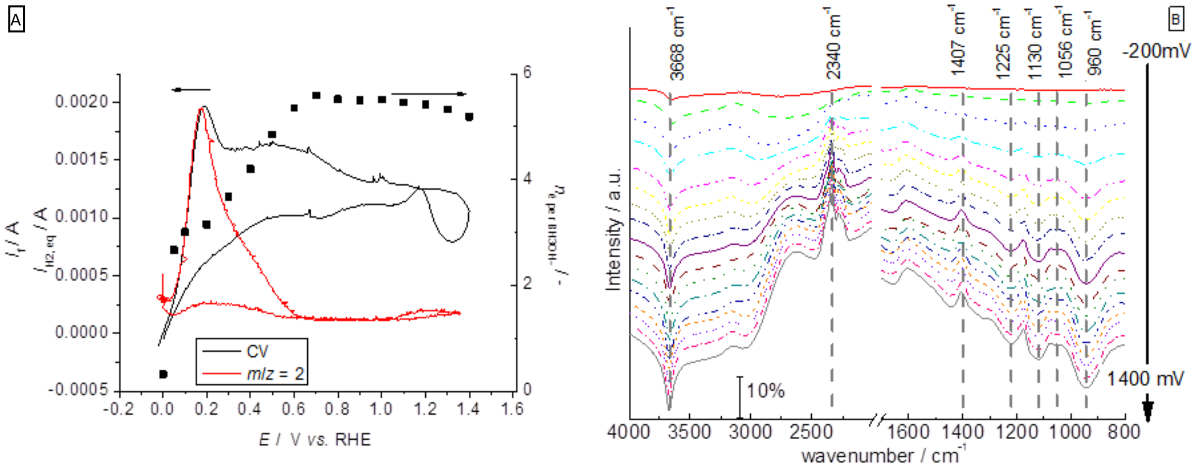

Fig. 1: *(A) ABOR faradaic current (I_f) vs. E, resulting equivalent HER current ($|I_{H2,eq}|$) and corresponding variation of $n_{e^-\ per\ BH3OH^-}$; (B) In situ infrared spectra recorded on a gold electrode in 1 M NaOH + 1 M NH_3BH_3 solution for s-polarized light; R_{ref} = -200 mV vs. RHE.*

Acknowledgements

The authors thank the CAPES/COFECUB for funding (project Ph 598/08).

AB_5-TYPE HYDROGEN STORAGE ALLOY MODIFIED WITH CARBON USED AS ANODIC MATERIALS IN BOROHYDRIDE FUEL CELL

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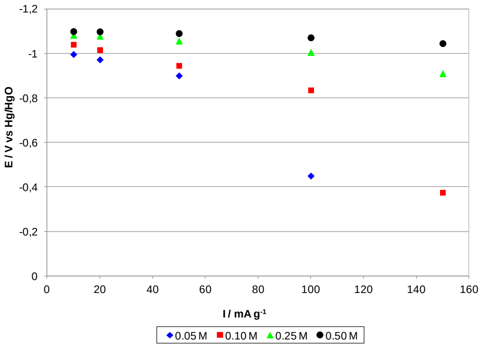
Abstract

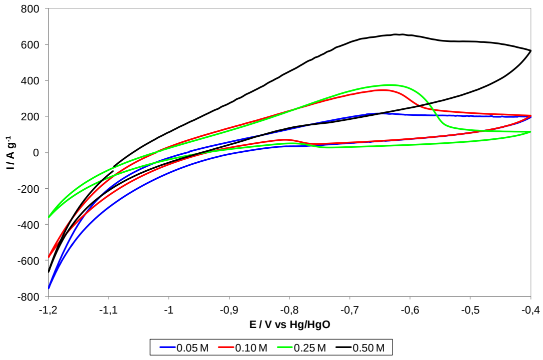
Multicomponent alloys of the AB_5-type are characterized by high electrochemical catalytic activity in the oxidation of hydrogen, therefore, they can be used as an anode materials in borohydride fuel cell. In such a fuel cell, H^- in BH_4^- is oxidized into H^+ directly on the anode.

Anodic oxidation of borohydride can be described by the following equation (1, 2):

$$BH_4^- + 8\,OH^- \rightarrow BO_2^- + 6\,H_2O + 8\,e^- \qquad E^0 = -1.24\ V$$

In this work the electrochemical properties of hydrogen storage alloy: $LaMmNi_{3,55}Al_{0,30}Mn_{0,40}Co_{0,75}$ modified with carbon used as anodic materials in borohydride fuel cell (BFC) are discussed. Composites with a small amount of carbon where obtained by direct pyrolysis on hydrogen storage alloy or carbonization of conducting polymers. This was done for the improvement of the hydrogen electrosorption properties. The samples were characterized by BET surface area measurements and Scanning Electron Microscopy. The electrochemical properties were examined in 6 M KOH solution (vs. Hg|HgO reference electrode) with the addition of different amount of KBH_4. The electrochemical measurements had been carried out using cyclic voltammetry (OCP→-0.4V→-1.2V) and galvanostatic methods.





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STUDY OF THE BH_4^- ELECTROOXIDATION MECHANISM ON Pt CATALYST USING ROTATING RING-DISK ELECTRODE (RRDE)

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Direct borohydride fuel cells (DBFC) display high theoretical potential (1.64 V) and energy density (9.3 Wh g^{-1} at 1.64 V) [1]. However, the anodic oxidation reaction of borohydride (BOR) is complex (8 electrons) and its kinetic is slow [2]. In order to study this mechanism on Pt, several experiments were performed using rotating ring-disk electrode (RRDE) in 1 M NaOH + 0.01 M $NaBH_4$ electrolyte. Cyclic voltammetry was performed on the disk, while the gold ring was held at 0.2 V vs RHE, i.e. a potential low enough to selectively oxidize the BH_3OH^- species produced during the cyclic voltammetry at the disk, but not the BH_4^- of the solution [3]. This system therefore enables the in situ BH_3OH^- detection during the voltammogram of BH_4^- oxidation on Pt.

In this study, the disk of the RRDE is composed of Pt nanoparticles supported on vertically aligned carbon nanotubes (VACNT) [4]. Several electrodes were prepared with different Pt-loading, in order to study the influence of the structure of the active layer towards the BH_4^- electrooxidation on Pt. These results were compared with: (i) differential electrochemical mass spectrometry (DEMS) -measuring the amount of H_2 produced the BH_4^- oxidation- and (ii) in situ infrared spectroscopy measurements [5,6]. From these results, a relevant reaction pathway for BH_4^- electrooxidation on Pt was proposed.

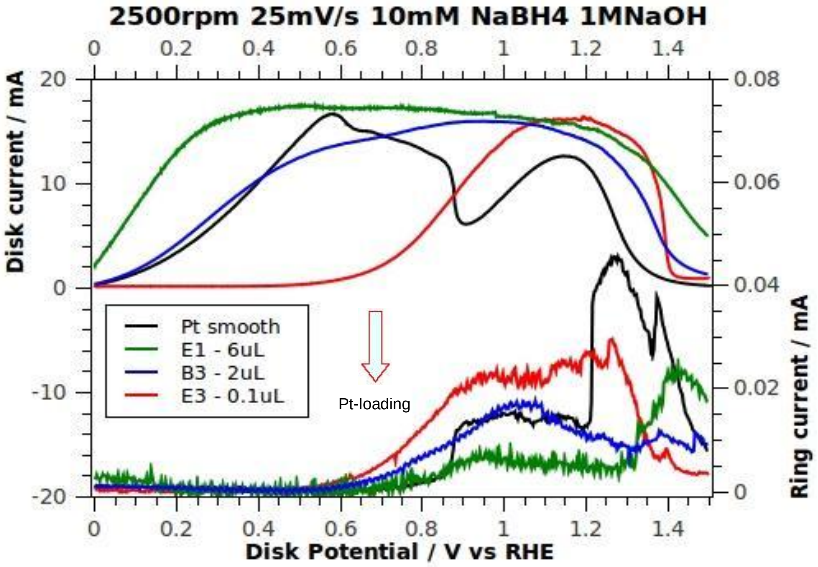

Fig. 1: *RRDE voltammograms of 10 mM $NaBH_4$ in 1 M NaOH at 25 mV s^{-1} and 2500 rpm. The disk was composed of the different Pt on VACNT samples and was swept between 0 and 1.5 V vs. RHE, while the Au-ring was held at +0.2 V vs. RHE.*

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ELECTROCATALYTIC PROPERTIES OF REDUCED GRAPHENE OXIDE IN OXYGEN ELECTRODE

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Abstract

The use of air or oxygen electrode in devices generating electrical energy is very promising since it does not pose environmental problems and makes it possible to save natural resources, such as oil and gas. The current magnitude generated at such gas-diffusion electrode depends on the size of the contact zone of three phases. The electrode itself consists of a catalyst and a support; the interaction between them determines mainly the value of generated current, which depends on the catalyst used. Therefore, two main problems exist: search for efficient catalyst and search for catalytically active and stable support. References (1, 2) showed the advantage of carbon nanotubes as catalyst support. At the present time, in view of the advent of a new carbon nanomaterial such as graphene, a number of papers appeared on its investigation as electrode material for lithium ion batteries (3) and as support for catalysts in fuel cells (4, 5). A structure peculiarity of graphene sheet is that the charge carriers, which have unrestricted freedom to move in the plane, are confined in a narrow space between “walls” bounded by atomic π-orbitals, which are the shortest atomic distance of ~0.3 nm apart, which gives rise to unique electrophysical characteristics and other extraordinary properties of graphene. Therefore, it is of great interest to study the dependence of electrochemical properties on the method for the preparation of reduced graphene as a catalyst support for fuel cell oxygen electrodes. Reduced graphene was prepared by the chemical oxidation of multiwalled carbon nanotubes, followed by the reduction of the resulting product with sodium hypophosphite and sulfite. The product obtained has been investigated by means of a JEM-100 CXII electron microscope. The X-ray phase analysis was carried out by means of a DRON-4 X-ray diffractometer with CuK_α radiation. It has been shown that reduced-graphene sheets are obtained. The electrochemical characteristics of reduced graphene have been investigated in a mockup of oxygen fuel cell with alkaline electrolyte. It has been found that the electrochemical characteristics in the oxygen reduction reaction for graphene obtained by reduction with sodium hypophosphite are much better than those of starting carbon nanotubes. They are superior to the characteristics of oxygen electrodes based on nanocomposites of manganese dioxide deposited on multiwalled carbon nanotubes and remain stable during six-month tests.

Thus it can be concluded that reduced graphene is a promising material for fuel cell oxygen electrodes, and that it has a much lower cost than its commercial samples.

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LOW-COST FUEL CELL MATERIALS MADE FROM NEW SOURCES

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Abstract

Replacing high-cost materials in fuel cells with widely acceptable alternatives is an urgent issue in order to bring this high-tech electric generator into markets in a near decade for stationary as well as transport applications. In this report, some examples of such alternatives are proposed based on new preparation methods, and tested for operation under fuel cell conditions.

The first is to use non-precious catalysts as the cathode material, which is aimed at reducing Pt amounts to less than 0.04 mg cm^{-2} or preferably zero. This is attempted by synthesizing organic metal complexes on carbon powder substrate, and then heat-treating in Ar atmosphere at 400-450°C. Transition metal complexes of N,N'-mono-8-quinolylphenylenediamine (abbreviated as *mqph*, which has three N ligands(1)) were newly synthesized for this purpose, and tested as oxygen reduction reaction (ORR) electro-catalysts in acid media. As transition metals, Mn, Fe, Co, Zn proved to be promising candidates which, especially in combined states, showed good synergistic effect (ladder-step effect) due to electron hopping via shifted redox potentials depending on the metal centre. Later heat-treatment in NH_3 atmosphere was further tested, which proved to be very effective in improving the ORR initiation potential more than 0.1 V. The new catalysts with 20wt% on C revealed ORR initiation potential of 0.9V, compared with 1.0V for 20wt% Pt/C.

The second is carbon paper gas diffusion layer (GDL) made from Japanese paper (Washi), which is carbonized under special treatments to obtain porous materials with good electric conductivity(2). The morphology-retaining carbonization process was developed to prepare porous carbon materials. Washi was treated with dehydrating agents, then carbonized for 1h at 800°C in Ar gas, followed by heat-treatment for 30 min at 1400-2600°C. The porous carbon papers obtained were composed of carbonized micro- and nano-fibrils, and exhibited an electric conductivity 10-50 Scm^{-1} increasing with increasing the heat-treatment temperature. Fuel cell operation tests revealed that good peformance in polarization curves and power density were obtained comparable with a commercial GDL.

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ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY – JUST ONE OF MANY TOOLS TO STUDY BATTERIES AND POWER SOURCES

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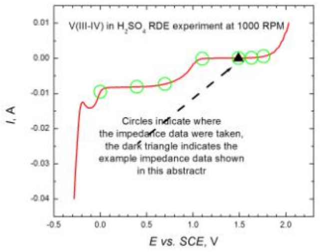
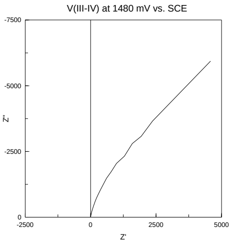
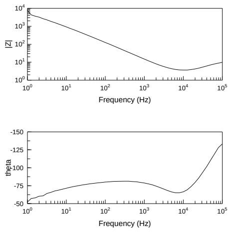
Electrochemical Impedance Spectroscopy (EIS) is one of the many methods available to the researcher in electrochemistry. To make a proper use of the results, one should understand the method and the underlying principles. In this presentation voltammetry on a stationary electrode, as well as rotated disk electrode results are presented together with EIS results. The examples are given on redox of vanadium salts in sulfuric acid, work motivated by the energy storage community in redox flow cells. This presentation summarises highlights, but also pitfalls of impedance measurements and their interpretation, as they are relevant to the practice and theory of electrochemical work.

Redox vanadium electrochemical cell is an interesting concept, which dates back to NASA patents (1) and early work by Skyllas-Kazacos and Grossmith (2, 3). The work has recently received renewed interest and studies were made not only to the chemistry, but also to economical feasibility of this scheme (4). To study the impedance response on the vanadium system was inspired by a recent thesis I had an opportunity to review (5).







The above three figures are an example of a voltammogram taken during an RDE experiment and resulting impedance (the Bode and Nyquist plots) taken at a point shown. The issues discussed in the presentation will focus on the correlation between the voltammetry at a given point and the impedance at the same point, which, in some sense, is a derivative of the voltammetric curve. Although this explanation holds to some extent, the deviations will be discussed in greater detail.

Acknowledgements

Travel support from the ABAF Organizers and their Sponsors is greatly appreciated.

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A NUMERICAL STUDY ON THE EFFECTS OF GAS CHANNEL WETTABILITY IN PEM FUEL CELLS: STRAIGHT CHANNEL

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Abstract

Water management is a technological problem in Proton Exchange Membrane Fuel Cells (PEMFC) that hinders its commercialization because of its impact on the gas flow and power output. The wettability of the gas channel and the gas diffusion layer (GDL) has a great influence on the water management. In this paper, a numerical study has been carried out to examine the effect of the wall and gas diffusion layer wettability on gas channels. The investigation employed a three dimensional numerical simulation using the volume-of-fluid (VOF) method to simulate the air-water flow in a straight micro-channel representing a gas channel in PEMFCs. Nine combinations of various wall/GDL wettability combinations were investigated. The analysis is based on the liquid water behaviour, the amount of water occupying the domain, the amount of water covering the GDL surface, and the total pressure difference. For fixed wall wettability, the pattern of the analysed parameters was changed between uniform cyclic behaviour, random cyclic behaviour and continuous behaviour as the contact angle of the GDL decreased; in addition to an increase in the amount of water covering the GDL and a decrease in the pressure drop. It was found that changing the wall wettability has less significance impact on the analysed parameters when compared to the effect of changing the GDL wettability. Moreover, different wettability combinations were found to give various water behaviours within the gas channel that can be optimised for different fuel cell requirements.

Acknowledgment

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ANALYSIS OF THE PERFORMANCE OF A POLYMER ELECTROLYTE FUEL CELL STACK USING SIMULTANEOUS ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY MEASUREMENTS

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Polymer Electrolyte Fuel Cells (PEFCs) are electrochemical conversion devices that convert the chemical energy released from hydrogen-oxidation and oxygen-reduction processes into usable electrical energy and thermal energy. In order to maximise the impact that the PEFCs can bring worldwide, there is a need to develop a robust and potentially cost-effective way of monitoring and optimising the performance, reliability and operating cost as a function of service life. Electrochemical impedance Spectroscopy (EIS) is an in-situ experimental technique that can unveil the electrochemical and diffusion processes that occur at different rates within the PEFC. EIS has the potential to reveal the internal state and performance of a PEFC without affecting its ability to perform as an energy conversion device. The resulting frequency response from EIS is commonly represented in a complex plane and represents the electrochemical and diffusion mechanisms of the PEFC in the frequency domain. In this study, an analysis of the performance of a commercial PEFC stack using simultaneous EIS measurements has been carried out. The objective of this study is to provide an insight into the change in impedance response of individual cells within a commercial PEFC stack. EIS measurements were carried out through a multichannel frequency response analyser (Z#106 WonaTech) which allowed the measurement of the impedance response of the stack and single cells simultaneously. To validate this study, the mathematical model reported in the authors' previous study [1, 2] based on electrochemical and diffusion theories to simulate the impedance response of a PEFC was considered. The factors that limit the performance of the PEFC stack were evaluated with EIS measurements and the impedance model. The results show that the change in impedance response of individual cells within the stack is attributed to oxygen transport limitations, membrane dehydration and temperature distribution through the stack. This study has demonstrated that EIS is a powerful tool for in-situ diagnosis of a PEFC stack. It is possible to generate a deeper understanding of the internal phenomenological processes by coupling the experimental EIS technique to the fundamental theory.

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THE IMPACTS OF IMAGE RESOLUTION ON PERMEABILITY SIMULATION OF GAS DIFFUSION LAYER USING LATTICE BOLTZMANN METHOD

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The lattice Boltzmann (LB) method has the capability to implement with complex boundaries. It has been increasingly utilized to investigate fluid transport through a polymer electrolyte fuel cell gas diffusion layer (GDL) in the past few years. Recently, in order to accurately reflect the actual GDL as manufactured, the x-ray computed tomography technique has been employed to generate 3D representative structures of GDL samples. The combination of the two advanced techniques has also been successfully applied to study fluid movement through PEFC GDLs in recent publications [1-3]. However, the huge computational demand of the LB method with a high pixel resolution of the x-ray images based 3D geometric model has limited its application to a very small volume of the GDL. In this paper, the influence of resolution variation of the x-ray reconstructed GDL on the resulting absolute gas permeability was examined using LB method. The binary 3D models of the GDL at six different resolutions were acquired by using x-ray imaging technique. Each image was then integrated into a single-phase LB numerical solver to characterize its gas permeability. The results indicate that the resolution of the x-ray reconstructed GDL has a great impact on gas permeability in both principal and off-principal flow directions. Comparing with the original resolution, the coarser pixel sizes can contribute significant changes in the resulting permeability. But conversely, it can reduce computation time to a great extent. It is worth considering an optimum resolution for the representative GDL model which provides a good compromise between computation time and accuracy. It was found that the combined technique has the potential to analyze fluid transport in much larger GDL volumes.

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ENZYMATIC BIOFUEL CELL FOR IMPLANTABLE DEVICES

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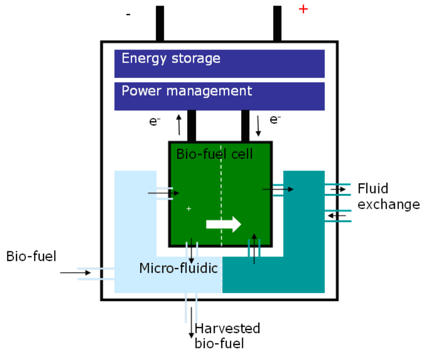
The improvement in quality of modern health-care is closely related to the need for autonomous implantable devices. Biofuel cells (BFC) are a promising approach of energy harvesting to power these devices. In combination with power management and energy storages, BFC may form an essential power source (See fig.1) for future implants [1].

Research efforts for implantable BFC have been focused on the investigation of different biocatalysts and fuels [2]. Among these enzymatic biofuel cells with glucose oxidase (Gox) as biocatalysts and glucose as fuel seem to be most promising due to the high catalytic-specificity, activity, and fuel abundance in body fluids. Therefore, a BFC based on Gox and Laccase (Lac) as biocatalysts for anode and cathode, respectively, is studied and demonstrated experimentally.

Optimization of the BFC performance requires a more deep understanding of the electrochemistry of the energy conversion processes. This can be achieved by developing a model describing the BFC thermodynamics and kinetics. For this purpose the BFC kinetics are performed by different (electro-) chemical methods. Consequently, one systematic modelling system has been set up.

Based on a shuttle reaction mechanism, appropriate using ferrocene (Fc) and 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) as the mediator of anode and cathode separately, a mathematic description has been done, including Nernst-Plank convective diffusion and migration for all (electro-)chemically active species and Navier-Stokes equations describing the movement of solvent. This modelling approach shows good agreement between theoretical description and experimental results (See fig.2).



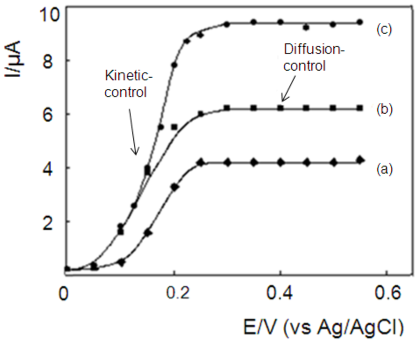


Fig. 1: *(left) The energy storage-power management-BFC concept*
Fig. 2: *(right) Measured (symbols) and simulated (lines) current-potential curves (vs Ag/AgCl) of a BFC system in a buffered electrolyte (PBS pH 7.2) containing 1 mM Fc as mediator, 0.5 g/l Gox and 1 mM Glucose (a); 10 mM Glucose (b); 20 mM Glucose (c)*

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THE IMPACT OF CATHODE GAS DIFFUSION LAYER STRUCTURE ON FUEL CELL PERFORMANCE

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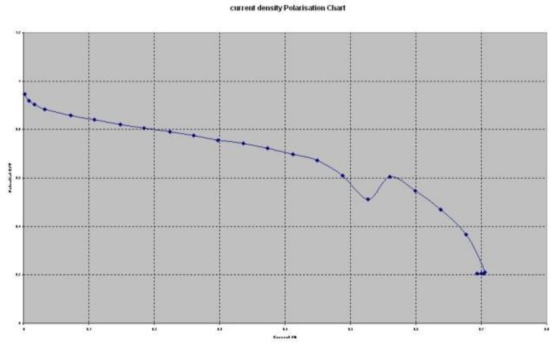
Abstract

Polymer Electrolyte Membrane Fuel Cells (PEMFCs) offer the potential of efficient, sustainable power solutions for the future. A key element in these devices is the Gas Diffusion Layer (GDL). It is critical in transport of reactants across the surface of the fuel cell, humidification of the polymeric membrane, as a support for catalytic materials, and transport of charge around the cell. In this study, three different GDL structures (Paper, Felt and Weave) for cathode GDL materials have been investigated. A Fuel Cell Component Analyser (FCCA) which is capable of testing 4 cells simultaneously has been employed for this study. Each Membrane Electrode Assembly (MEA) was manufactured with identical catalyst and binder loadings. Ex-situe analysis of the MEAs and the GDLs were completed before the FCCA tests. Polarisation curves were recorded as part of the 'conditioning' regime performed. The obtained results showed that significant differences in polarisation response of different GDL structures. These differences reveal changes in resistance loss, mass transport loss, and the flooding effects on Fuel Cells as due to GDL structure.



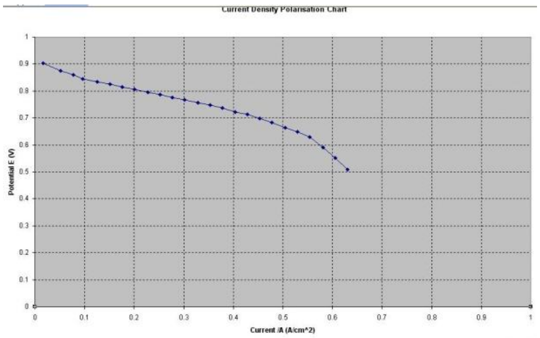


Fig. 1 (left): *Short duration polarisation curve (current density A.cm^{-2}) for woven sample*
Fig. 2(right): *Short duration polarisation curve (current density A.cm^{-2}) for sample Felted*

Acknowledgements:

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DEVELOPMENT OF LITHIUM-ION BATTERY PROTOTYPES FOR ELECTRIC CARS

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Abstract

In the last years, new lithium-ion intercalation materials have been studied at INIFTA (National University of La Plata) as a result of the Argentine government's interest in exploiting large lithium deposits in the north of the country. The project is based on international scientific cooperation with the group led by professors Jiri Vondrak and Marie Sedlarikova, who have been working in the field of new electrochemical energy sources for over two decades. Research in this group focuses on obtaining lithium battery cathodes that should be safe and have a long service life, new anodes based on carbon and silicon materials, and noncombustible organic electrolites. Focus is placed both on the synthesis and characterization of materials suitable for the development of lithium-ion batteries, basically new cathodic and anodic materials, and on the design of a lithium-ion battery prototype. Based on a commercial battery, we are working on the design of a lithium battery powerful enough to drive an electric car.

Currently, the cathodic material, $LiFePO_4/C$, is the most promising active material for high-power lithium-ion batteries and it has been observed that its response is also dependent on the nature of the carbon used as support. This paper presents the preliminary results from the synthesis of $LiFePO_4$ supported on carbon fibers where phosphate and fibers are synthesized together at a single stage. The electrodes built with this material have a good electrochemical response with the typical behavior of $LiFePO_4$.

In addition, the behavior of these materials has been characterized from the viewpoint of the electrochemistry of charge/discharge processes in order to provide knowledge for the global modeling of batteries. A Battery Management System has also been developed to control the operating condition and the state of charge of batteries in service.

STRUCTURAL AND ELECTROCHEMICAL PROPERTIES OF COMPOSITE GEL ELECTROLYTES WITH OXIDE NANOFILLERS

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Abstract

Li-ion batteries with polymer and gel electrolytes are the most promising electrochemical energy storage systems for a variety of applications, including portable electronic devices (laptops, tablets, smartphones, etc.), medical devices, space exploration and military devices. The advantage of Li-ion batteries with non-liquid electrolytes over those with conventional, liquid ones, lies in their enhanced energy density, shape flexibility and, most importantly, improved safety. Safety is the most vital parameter (apart from energy density) for the widespread adoption of electric transportation. Polymer gel electrolytes based on PVdF/HFP microporous membranes/matrices have been proved to be particularly useful due to excellent technological properties and stability (thermal and chemical). There is still room for improvements in this area by the application of ceramic fillers dispersed in the polymeric matrix.

The purpose of this work is to examine different oxide fillers as components of composite polymer gel electrolytes in terms of their impact on certain key factors, like specific conductivity, anodic stability and chemical stability toward lithium. It is demonstrated that chemical functionalization of conventional submicron silica powders leads to a significant improvement of performance. Mesoporous MCM-41 type silica has also been synthesized and tested as potential filler for composite gel electrolytes. The material has been additionally surface functionalized with fluorine-containing functional groups. Both materials have features of ordered mesoporous materials, as evidenced by X-ray diffraction and TEM imaging. The effectiveness of fluorine functionalization has been confirmed by FTIR spectroscopy. Membranes with dispersed mesoporous fillers exhibit markedly enhanced solvent uptake abilities and improved specific conductivities, exceeding 10^{-3} S/cm at room temperature.

Mechanism of beneficial action of dispersed ceramic phases has been concisely discussed. The majority of positive effect can be ascribed to the enhanced capability of absorbing liquid media, which directly improves ionic transport conditions. It is argued, however, that part of the explanation may be connected with special transport conditions existing on the graon boundaries, where space charge layer is formed, providing fast ionic transport pathways for lithium cations.

Acknowledgements

The work has been financially supported by the European Regional Development Fund in the frameworks of the Innovatine Economy Programme 2007-2013, project No POIG.01.01.02-00-015/09.

STUDY OF THE LITHIUM-RICH LAYERED COMPOUNDS $Li[LiMnNiCo]_2$ AS POSITIVE ELECTRODES IN LITHIUM CELLS

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Abstract

High-voltage Li-rich materials for positive electrodes in Li-cells have attracted much attention of the researchers in the field of advanced Li-ion batteries since they are interesting from both scientific and applied viewpoints (1). These materials represent a type of layered $LiMO_2$ compounds, in which a transition 3d-metal M is partially substituted by a structurally compatible $[Li_{1/3}Mn_{2/3}]$ unit. Electrodes based on these integrated materials can operate up to 4.6 – 4.8 V and provide reversible capacities >200 mAh/g.

The main goal of the present work was studying the electrochemistry of $Li[LiMnNiCo]O_2$ electrodes at 30 and 60^0C in relationship with the structural changes of the active material due to the lithium extraction at high anodic potential in the first charge. The $Li[LiMnNiCo]O_2$ materials were synthesized by the self-combustion reaction using Li, Ni, Mn, and Co nitrates, and sucrose. Transmission electron microscopy (TEM) study of these materials demonstrated that they are comprised of nano-domains of both rhombohedral $LiNiO_2$-like and monoclinic Li_2MnO_3 structures, which are integrated and interconnected with one another at the atomic level. It was shown that electrodes comprising the $Li[LiMnNiCo]O_2$ submicron particles demonstrated reasonable cycling behavior at various charge/discharge rates at 30 and 60^0C. They can be cycled successfully at a moderate rate of C/5 at the elevated temperature. At higher rates of charge-discharge at the elevated temperature, these electrodes show reversible cycling performance and capacity ~250 mAh/g in the earlier cycles while capacity fades upon prolonged cycling in coin-type cells.

In this work, using several techniques (X-ray diffraction, convergent beam electron diffraction, high-resolution TEM, and Raman spectroscopy) we revealed possible partial layered-to-spinel structural transition in Li_2MnO_3 and $Li[LiMnNiCo]O_2$ compounds due to electrodes' cycling in lithium cells. The above transition is possible in the first charge process (Li extraction) due to the manganese migration into the interlayer lithium sites that leads to the creation of a spinel-like cation ordering (2).

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ELECTROCHEMICAL AND XPS STUDIES OF PPy/PEG $LiFePO_4$ CATHODE MATERIAL

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Abstract

The polypyrrole-$LiFePO_4$ composites were synthesised by simple chemical oxidative polymerization of pyrrole (Py) monomer directly on the surface of $LiFePO_4$ particles. $LiFePO_4$ is more stable under overcharge or short circuit conditions and it can withstand high temperatures without decomposing [1]. However, an important drawback of this material is its poor conductivity. The decrease of particle size and the nano-layer of carbon coating [2] on $LiFePO_4$ particle surfaces are necessary to achieve a high electrochemical performance. Conducting polymers (e.g., polyacetylene, polythiophene (PTh), polypyrrole (PPy), polyaniline (PANI), etc.) have attracted considerable interest in recent years because of their potential applications as cathode materials in rechargeable lithium batteries. Generally, every functionalization of a surface by polymers allows an accurate tuning of the substrate surface properties. Properties of resulting polypyrrole-$LiFePO_4$ (PPy-$LiFePO_4$) samples (especially conductivity) are strongly affected by the preparation technique, polymer additives and conditions during synthesis. For increasing of PPy-$LiFePO_4$ conductivity we used polyethylene glycol (PEG) as additive during polymerization [3]. The samples were characterized by scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDX), X-ray photoelectron spectrometry (XPS) and charge-discharge tests. PPyPEG hybrid layers decrease particle to particle contact resistance while the impedance measurements confirmed that the coating of PPy-PEG significantly decreases the charge transfer resistance of the electrode material. The initial discharge capacities of this sample at C/5 and 1C are 150 and 128 mAh/g, respectively. The results show that PPy/PEG$LiFePO_4$ composites are more effective than bare $LiFePO_4$ as cathode material.

Aknowledgement

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SYNTHESIS AND ELECTROCHEMICAL LITHIUM INSERTION INTO SKUTTERUDITE-TYPE MATERIALS

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Abstract

In recent years there has been great interest in the study of intermetallic compounds with metals, which can alloy with Li, such as Si, Sn, Al, Sb, with the aim of finding new materials for the anode material of lithium-ion batteries. These materials have shown that the main advantages are higher specific capacity, lower irreversible capacity and improved safety compared to carbon. The main drawback of these compounds is the rapid capacity fade upon repeated cycling resulting from the large volume changes during charge and discharge process. The use of nanosized materials seems to be an effective strategy to alleviate volume change effect. Very recently, intermetallic nano-$CoSb_3$ possessing skutterudite structure has been proposed as potential anode material for Li-ion batteries.

In the present contribution we report wet chemical syntheses (solvothermal and polyol) of nano-sized $CoSb_3$ powders. Impact of synthesis conditions on a number of structural factors (phase composition, particle size distribution, porous structure) is studied and discussed. The materials have been tested towards reversible electrochemical insertion of lithium cations, using galvanostatic cycling and cyclic voltammetry. The electrodes have been prepared by casting the active materials onto the copper current collectors. The electrodes have been tested in Swagelok-type cells with metallic lithium as counter and reference electrode and a conventional lithium-conducting electrolyte. The results have shown that electrochemical performance strongly depends on the exact phase composition, in particular the existence of minority non-skutterudite phases. Lithium cations enter the structural sites of $CoSb_3$ at the potential of about 1 V (vs. Li/Li^+). Cyclic stability of original skutterudite materials is very poor, which results from the severe volume changes upon repeated insertion and deinsertion of lithium. Therefore it is essential to combine the active material with appropriate conducting additive. Impact of the type and ratio of the conducting agent has been also examined and discussed. Our results prove that $CoSb_3$ can reversibly accommodate significant amounts of lithium, exceeding those known for graphite, thus can be considered as promising anode materials for lithium-ion batteries but thorough optimization of electrode composition and discharge conditions are necessary.

Acknowledgements

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SAFETY ENHANCING OF LITHIUM-ION ACCUMULATORS

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Abstract

Thermogravimetric analysis and differential scanning calorimetry were used to study effect of different composition of gel polymer electrolytes (GPEs) on their thermal stability and thus safety of lithium-ion accumulators. Polymer matrixes of GPES were made of different methacrylates (MMA, TSPMA, EOEMA), as a solvent propylena carbonate and sulfolane were used. Moreover, two different lithium salts were tested ($LiClO_4$, $LiBF_4$). Contribution of addtion sulfolane and TSPMA on the thermal stability of the GPEs were observed. Video – recorded comparisons of TGA in the inert argon environment and in common atmosphere were passed.

Acknowledgements

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BROOKITE TIO_2 NANORODS FOR LITHIUM-ION ANODES

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Abstract

This study was aimed to using of new different structural phase of TiO_2 for anodes of Li-ion accumulators. Samples of TiO_2 nanorods with brookite structure were prepared by solvothermal synthesis and calcinated at different temperatures. Structural characterization by XRD and SEM was made. Electrochemical analysis was made by cyclical voltammetry, charge-discharge cycling and electrochemical impedance spectroscopy.

Acknowledgements

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CONNECTING ISSUES OF THE FAST-CHARGERS FOR TRACTION BATTERY TO DISTRIBUTION NETWORK

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Abstract

The power rate of the fast-chargers for traction battery in electric vehicles is increased rapidly in the present day. High-power converters with high frequency pulse transformers are used in this equipment to decrease their size and price. These converters and their input rectifiers have significnat impact on distribution network. This factor must be taken into consideration in the case of mass stationing of electric vehicles.

Distribution net impact can be split into these several basic domains:

- EMC issues, caused by high *du/dt* produced by fast power transistors used in converters - Shielding techniques and input low-pass passive filters can solve these problems successfully.
- Unusual and high local concentration of consumed power - These problems are solved by a complex control of chargers by a global master structure called “Smart grids” technologies.
- Presence of higher harmonics in input current - If a ordinary passive input rectifier with a large DC-link capacitor is used then problems with power factor λ will appear due to the shape of the phase current caused by narrow pulses with high peak value and high RMS value. This is why active filters, generally known as the power factor correctors PFCs, are used for large power applications. However this complicates the power circuitry and decreases the efficiency of the charger. Moreover the price and volume is increasing.

An advantageous alternative solution was proposed. A conception with a passive diode input rectifier and a DC-link with an extremely low capacity is used in this conception. Dimensioning of this DC-link capacitor to ensure mentioned low impact on distribution net is presented.

The final part of the paper demonstrates overall applicability of RN14-140 fast charger pattern via presentation of high harmonics of phase current measurements.

Acknowledgements

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ELECTROLYTES FOR Li-ION BATTERIES OPERATING IN WIDE TEMPERATURE RANGE

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The questions of the lithium-ion power source operation at elevated temperatures are relevant. The safety, safe keeping of the charge, possibility of the high current density discharge depend on the electrolyte conductivity and on processes at the electrode-electrolyte interface.

Electrolytes based on mixtures of high boiling solvents such as tetraethyleneglycol dimethylether (TG), adiponitrile (ADN), polyethyleneglycol dimethylether (200) (PEG-DME) with lithium salts bis(trifluoromethane)sulfonimide ($LiN(CF_3SO_3)_2$) and bis(oxalate)borate (LiBOB) have been studied by thermogravimetric analysis, conductometry, potenhtiodynamic and galvanostatic cycling. It has been shown that thermal stability of electrolytes increases in the next line: PEG-DME - LiBOB < TG - LiBOB < ADN - LiBOB.

Dependence of conductivity on the temperature is illustrated on Fig.1. The maximum conductivity value has been estimated for electrolyte based on PEG-DME-LiBOB (0.7 mol/kg). But other electrolytes under investigation are characterized by high conductivity level which is enough for the Li-ion battery operation.

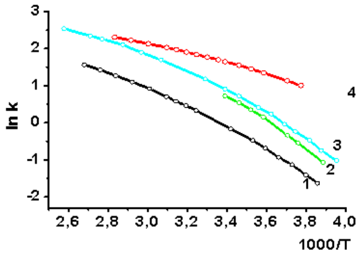

Fig. 1: *Temperature dependence of the electrolyte specific conductivity:*
1 - LiBOB (0.7 mol/kg) – AND, 2 - $LiN(CF_3SO_3)_2$ (1.0 mol/kg) – AND, 3 - LiBOB (0.7 mol/kg) – TG, 4 - LiBOB (0.7 mol/kg) - PEG-DME

Potentials of the electrolyte electrochemical stability have been studied by cycling voltammetry methods using platinum working electrode. Maximum value of the electrochemical stability potential (5.1 V) has been estimated for solutions based on the ADN - LiBOB. The electrochemical stability potential for electrolytes TG - LiBOB (0.7 mol/kg) and PEG-DME - LiBOB (0.7 mol/kg) at ambient temperature equals approximately 4.8 V and 4.6 V correspondingly. The same characteristics have been observed at the temperature 60 °C, but the region of stability is lowered to 4.8 V for the ADN - LiBOB mixture and to 4.5 V for TG - LiBOB and PEG-DME - LiBOB.

Specific capacity of $LiMn_2O_4$ and $LiCoO_2$ cathodes has been studied by galvanostatic cycling in 2016 battery prototype where Li-foil was used as anode. Dependence of specific capacity on the cycle number and on the current density is shown on Fig.2.

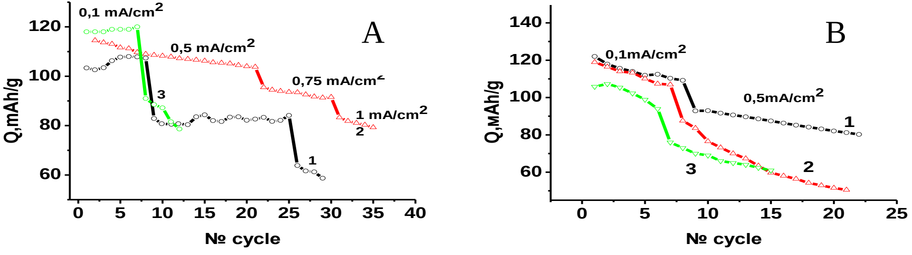

Fig. 2: *Dependence of the $LiMn_2O_4$ electrode specific capacity on the cycle number and on the current density (A – 25 °C, B – 60 °C). Electrolytes: 1 – ADN - LiBOB, 2 – PEG-DME - LiBOB, 3 – TG - LiBOB.*

ELECTROCHEMICAL CHARACTERISTICS OF $Li_xMn_2O_4$ SYNTHESIZED BY MICROWAVE METHOD

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Lithium-manganese spinel ($Li_xMn_2O_4$) is one of the most actively investigated electrode materials for Li-ion power sources. The main advantages of $Li_xMn_2O_4$ in comparison with other electrode materials are a high energy density, environmental friendliness and relatively low cost of production.

Conventional methods for synthesis of $Li_xMn_2O_4$ include quite durable procedure which involves the use of high temperatures resulting in large expenditure of energy. The use of microwave technique enables significant acceleration of the process due to the interaction of microwave radiation with molecules of reactants. The water concentration in the structure of initial materials or in the interior of the reaction medium plays important role because the water provides efficient interaction with the microwave field. Effect of the water content in main components and of their nature upon the structure and specific characteristics of $Li_xMn_2O_4$ obtained by microwave method has been discussed.

Mixtures of $LiCH_3COO·2H_2O$, Li_2CO_3, $[MnCO_3·mMn(OH)_2·nH_2O]$, $Mn(CH_3COO)_2·4H_2O$ were used as initial substances for synthesis. It has been shown that the presence of free water and crystallization water in the initial mixture significantly affects characteristics of $Li_xMn_2O_4$ since the reaction mixture heating is mainly the result of water dipoles rotation in the microwave field. For (10-15) minutes at the microwave oven power 350 W the temperature of the reaction mixture reaches 400 °C. Further synthesis process may be accompanied by a flash and heating of the reaction mixture up to 600-700 °C. The $Li_xMn_2O_4$ lattice parameter has been estimated by X-ray spectroscopy to equal 8.2158 Å to 8.2414 Å depending on the nature of $Li_xMn_2O_4$ samples, and is determined greatly by the Li/Mn ratio in initial mixture. Calculated crystallite size for all samples varies in the range (25.6-27.7) nm. Mn_2O_3 and Mn_3O_4 compounds were found as the main impurities in some $Li_xMn_2O_4$ samples synthesized. Specific capacity of 103 to 122 mA·h/g has been obtained at the discharge rate of 0.2 C. At the discharge rate of 1 C specific capacity drops to (119-95) mA·h/g depending on the nature of $Li_xMn_2O_4$ samples. Average capacity loss during cycling has been estimated as 0.2 % by a cycle.

Thus, application of the microwave synthesis method enables one to reduce significantly the synthesis time and energy consumption while providing sufficiently high electrochemical characteristics of $Li_xMn_2O_4$.

Typical volt-amperes characteristics for different samples of $Li_xMn_2O_4$ are shown on Fig.1.

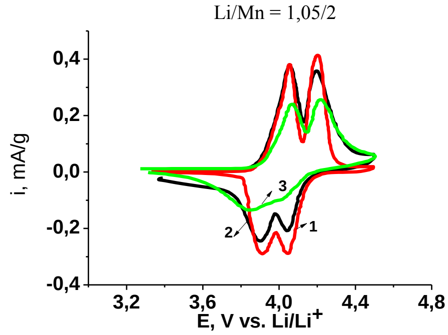

Fig. 1: *Cycling voltamperograms of $Li_xMn_2O_4$ samples. Initial materials:1. LiAc+$MnCO_3$, 2. Li_2CO_3+$MnCO_3$, 3. Li_2CO_3+$MnAc_2$*

ELECTROCHEMICAL DEPOSITION OF TIN AND SILICON STUDIED BY EQCM

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Abstract

Silicon and/or tin were deposited in electrolyte which contained PC or DMC with EC, and tetraethylammonium tetrafluoroborat on copper and nickel substrates. Substrate was cleared by NH_3 and IPA. Experiment were performed in the Atmosbag, which is inflatable polyethylene chamber with built-in gloves that lets man work in a totally isolated and controlled environment. As inert gas, nitrogen was used. Attempts of electrodeposition od tin from $SnCl_4$ were used initially for verification of experimental techniques. Cyclic voltammograms showed differences between deposition with and without $SiCl_4$., there we can see two potential waves, when silicon becomes bivalent and trivalent. EQCM describes increase in weight during deposition. Deposited layers were studied by optic microscopy, electron microscopy and X-ray diffraction. All these electrodeposited layers could be used for negative electrodes in li-ion cells.

EFFECT OF POTASSIUM ON THE STABILITY OF THE STRUCTURE OF $LiCoO_2$

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Abstract

The aim of this study was to improve the properties of cathode material for lithium-ion batteries based on $LiCoO_2$ As a method of manufacturing of the doped base material was chosen the solid phase reaction method, which has been already tested for the production of the basic material. The main goals were reducing the capacity loss during cycling of the material, maintaining the same or achieving higher capacity or increasing the current carrying capacity of the material $LiCoO_2$ by doping the base material with varying amounts of potassium ions or by substitution of lithium by potassium.

Acknowledgments

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ENHANCE CAPACITY AND SAFETY OF LITHIUM-ION ACCUMULATORS

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Abstract

This paper is based on a study of negative materials for Lithium-ion accumulators. In the study of possibilities were considered methods, which would lead to enhance capacity of Lithium-ion accumulators along with improving their safety. In the first part of the study mixtures of negative materials were prepared, these materials should be to reduce or completely remove the irreversible capacity of Lithium-ion accumulators. These mixtures were made by process of lithiation with help of Butyllithium reagent. In this way prepared mixtures already contain lithium ions (cations) which are intercalated to materials. For experimental purposes were chosen commonly uses graphite materials for negative electrodes of Lithium-ion accumulators as natural graphite or expanded graphite. (1, 2, 3, 4)

The second part of study was focused on safety testing of various mixtures (materials). The tests were performed on STA i1500 device, this device offers two different types of analyses. Both analyses works on the principle of temperature changes (temperature is controlled value). The first analysis is Thermogravimetry (TGA), this analytical method assesses the weight change of specimen. The second analysis is named Differential Thermal Analysis (DTA) this method assesses the change of specimen temperature against to the reference specimen.

Further research will deal with another ways that could lead to improve accumulators features.

Acknowledgements

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MODELING AND NUMERICAL SIMULATION OF LITHIUM ION BATTERY

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Lithium-ion batteries are incredibly popular these days. You can find them in laptops, PDAs, cell phones, iPods and so on.

The energy density of lithium-ion is typically twice as high as of the standard nickel-cadmium cell. Most of today's mobile phones run on a single cell. A nickel-based pack would require three 1,2 volt cells connected in series.

Despite its overall advantages, lithium-ion has its drawbacks. It is fragile and requires a protection circuit to maintain safe operation. Built into each pack, the protection circuit limits the peak voltage of each cell during charge and prevents the cell voltage from dropping too low on discharge. In addition, the cell temperature is monitored to prevent temperature extremes. The maximum charge and discharge current on most packs is limited to between 1 C and 2 C.

Acknowledgment

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ASYMMETRIC CAPACITORS BASED ON CONDUCTING POLYMERS AND METAL OXIDE/CARBON COMPOSITES AS ELECTRODES

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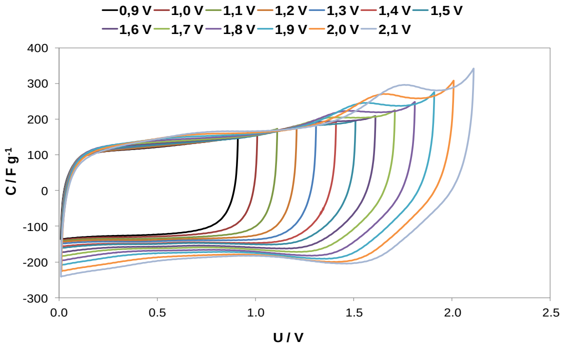
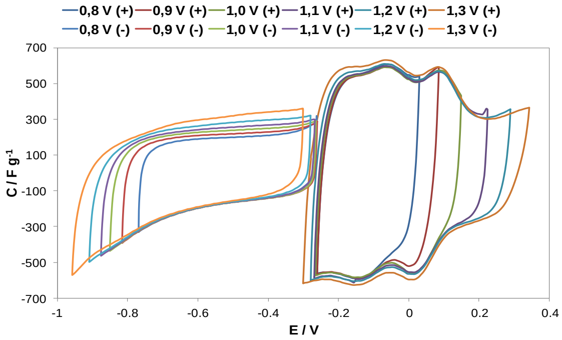
Abstract

The electrochemical properties of asymmetric supercapacitors based on polyaniline (PANI)/activated carbon (AC), polypyrrole (PPy) and poly(3,4-ethylenedioxythophene) (PEDOT) and manganese oxide composite/activated carbon are discussed. The electrically conducting polymers (ECPs) and manganese oxide were prepared using chemical method of synthesis. The active carbon was prepared by carbonization of commercial lignine (Aldrich) and then activated in KOH with a C:KOH ratio of 1:4. The morphology of the materials was observed by SEM. The electrochemical performances of obtained materials in symmetric and asymmetric capacitors were studied in two and three electrode Swagelok® systems using 1 M H_2SO_4 and 1 M Na_2SO_4 as an electrolyte. The electrochemical measurements have been carried out using cyclic voltammetry (CV), galvanostatic charge/discharge and electrochemical impedance spectroscopy. The results showed that PANI and MnO_2/carbon composite provided the higher value of capacity as positive electrodes while the others materials (PPy, PEDOT and AC) should be used as negative electrodes due to their electrochemical properties. The higher value of capacity in asymmetric device was obtained for PANI/AC configuration; additionally, this composite can work in wide potential window, i.e., 1.3 V (CV of PANI/AC in three-electrode system (5 mV/s)). Asymmetric device of MnO_2 composite/AC configuration can work in wide potential window, i.e., 2.1 V (CV in two-electrode system (10 mV/s)). The considerable growth of energy is connected with such configuration in asymmetric capacitors. The results of cyclability showed that such configuration provided high value of capacitance (when compared to the pristine materials) but the drop of capacitance after 5000 cycles with high current regime 2 A/g is also high.





Acknowledgements

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CARBON NANOTUBES AS A SUPPORT OF ELECTRODE ACTIVE MATERIAL FOR ELECTROCHEMICAL CAPACITORS

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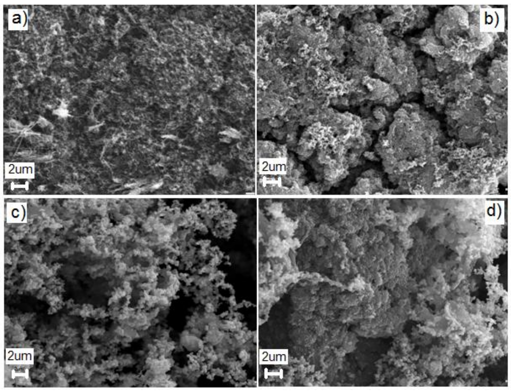
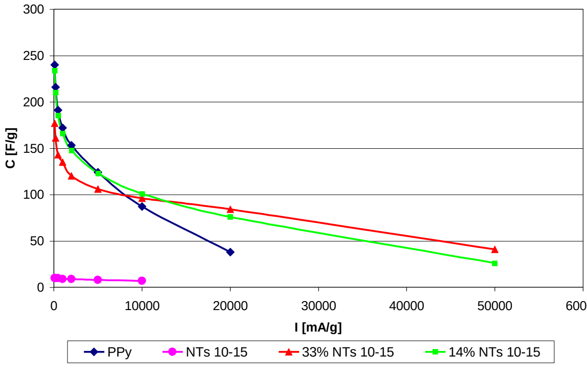
Abstract

Electrochemical capacitors have generated great interest because of their possible use in high power applications such as lasers, propulsion in electric vehicles, etc. There are a number of electrode materials being developed for electrochemical capacitors. In this work the capacitance properties of carbon nanotubes (CNTs) and their composites with conducting polymers (e.g., polypyrrole - PPy) and metal oxides applied as electrode materials for electrochemical capacitors are discussed. The morphology of the composite materials was observed by SEM and TEM. Figure presented below shows SEM micrographs of CNTs (a) and composites with different amount of PPy: 67% as (b), 81% as (c) and 86% as (d). The electrochemical performances of CNTs and composite materials in symmetric capacitors were studied in two and three electrode Swagelok® systems with 1 M H_2SO_4 as an electrolyte. The electrochemical measurements have been carried out by cyclic voltammetry (CV), galvanostatic charge/discharge and electrochemical impedance spectroscopy. It has been shown that carbon nanotubes show moderate values of capacitance. In the form of the entangled network, the material provides good charge propagation and can be tentatively used as a support or additive for different composite electrode materials. The highest values of capacitance were obtained for conducting polymers without additives of carbon nanotubes. Nevertheless, carbon nanotube networks allow to reach high current regime, i.e., even 50 A/g; it was additionally confirmed by cyclability measurements (5000 cycles with current regime of 2 A/g).





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INVESTIGATION OF ELECTROCHEMICAL PROPERTIES OF AB_2/AB_5-TYPE HYDROGEN STORAGE ALLOYS AS ANODIC MATERIALS FOR HIGH ENERGY Ni-MH CELLS

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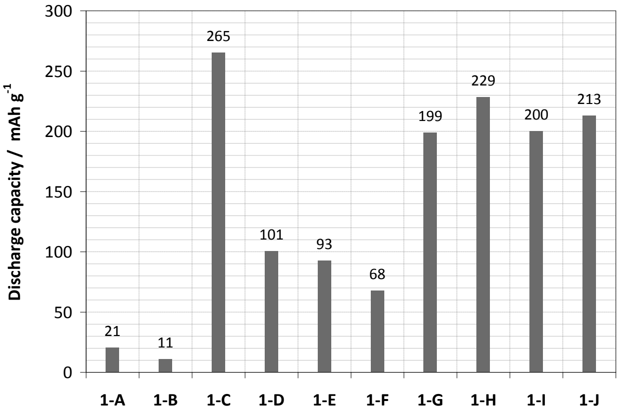
Abstract

Multicomponent alloys used as electrode material in NiMH cells should reversibly absorb hydrogen and be characterized with low self-discharge. The major criteria for application of those alloys as M/MH electrode material for recharge NiMH batteries are as follows: the capacity of the alloy in hydrogen absorption-desorption process, chemical stability in concentrated alkaline solutions, electrode reaction kinetics and fast activation time of the alloy. Those parameters are strongly dependent on the method used to acquire the alloy and its chemical composition.

In the conducted research the electrochemical parameters of 10 multicomponent AB_2 and AB_5 type alloys, obtained by arc melting, were examined. The tests were conducted to determine the specific capacity of hydrogen absorption desorption process, the reversibility of the electrode reactions and the reaction kinetics. The electrochemical measurements have been carried out using galvanostatic charge/discharge method with different current densities and cycling voltammetry.



Chemical formulas	Designation	Type
Hancke phase		
$MmNi_{4,5}Al_{0,5}$	1-A	AB_5
$LmNi_{4,78}Mn_{0,22}$	1-B	AB_5
$MmNi_{3,55}Al_{0,3}Mn_{0,4}Co_{0,75}$	1-C	AB_5
$MmNi_{3,6}Al_{0,4}Mn_{0,3}Co_{0,7}$	1-D	AB_5
$MmNi_{3,35}Al_{0,3}Mn_{0,4}Co_{0,75}$	1-E	$AB_{4,8}$
$MmNi_{3,2}Al_{0,2}Mn_{0,6}Co_{1,0}$	1-F	AB_5
Laves phase		
$ZrCr_{0,8}Ni_{1,2}$	1-G	AB_2
$Zr_{0,9}Ti_{0,1}Mn_{0,6}V_{0,2}Co_{0,1}Ni_{1,1}$	1-H	AB_2
$Zr_{0,9}Ti_{0,1}Mn_{0,6}V_{0,2}Cr_{0,05}Co_{0,05}Ni_{1,2}$	1-I	$AB_{2,1}$
$Zr_{0,5}Ti_{0,5}V_{0,68}Mn_{0,68}Cr_{0,34}Ni_{0,7}$	1-J	$AB_{2,4}$

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INFLUENCE OF THE ELECTROLYTE AGITATION AT THE SYNTHESIS OF THE ELECTROLYTIC NICKEL SULFIDES ON THEIR ELECTROCHEMICAL CHARACTERISTICS IN LITHIUM ACCUMULATOR MODEL

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Electrolytic sulfides of the transition metals (Me=Fe, Ni, Co and bi-Me-compounds) obtained in electrolysis mode without the electrolyte agitation show oneself as the perspective electrode materials for lithium and lithium-ion accumulators (1–5). In the present work it was shown that electrochemical characteristics of the sulfide change with positive effect under the influence of the electrolyte agitation. On the XRD data the sulfides of the metals (Me_xS_y) depose at the cathode with the dominated structure of the nonstoichiometric MeS and Me_3S_4 from aqueous solutions of Me-sulfates in the presence of $S_2O_3^{2-}$ (1). Discharge voltage of the lithium accumulator model on their base increases with sulphur increasing in the Me-sulfide thin layer electrodes. The compound with increased sulphur content (Ni_3S_4) shapes together with (NiS) sulfide at the synthesis of electrolytic Ni sulfides with the electrolyte agitation. It is different from the case of the synthesis without the electrolyte agitation when the main synthesis product is (NiS) sulfide. The maximum activity of the cathode process in lithium accumulator model coresponds to (1.40–1.45) V obliged to electrochemical NiS sulfide transformation. Discharge voltage of (Ni_3S_4) sulfide corresponds to 1.50–1.55 V. Discharge capacity of the Ni sulfide obtained with the electrolyte agitation fall less than one without the agitation at the cycling in the accumulator model with EC (ethylencarbonate, Merck), DMC (dimethylcarbonate, Merck), 1 M $LiClO_4$ (Iodobrom) electrolyte at $i_{dischage}$=0.05 mA/cm^2. The capacity (Q, mAh/g) stabilization of the sulfide compound obtained with the electrolyte agitation carry out in 10-th cycle ($Q_{10\text{-}35}$=490). However the fall of the discharge capacity continues in 20-th cycle (Q_{20}=320) in the case of the sulfide synthesis with same parameters of the electrolysis without the agitation. The hysteresis of the discharge-charge curves that is essential for the case of the Ni sulfide deposition from the electrolyte without the agitation is absents at its deposition from the electrolyte with the agitation. This way the electrolyte agitation at the deposition of the metal sulfides promotes the sulphur increase in deposit structure that increases the energy characteristics of the lithium accumulator.

In contrary of the defined positive agitation effects its negative factor is the sulfide deposition rate fall in the synthesis process. It have been determined that the deposit mass of the sulfide material on stainless steel at the agitation of ($NiSO_4$ – $4.3\div5.0\cdot10^{-2}$, $Na_2S_2O_3$ – $2.5\div3.0\cdot10^{-2}$; pH $3.5\div4.2$) electrolyte falls with the growts of the electrolyte agitation rate. The mass gain is equal 2.00-2.50 mg/cm$^2\cdot$h (without the electrolyte agitation) and 0.20–0.25 mg/cm$^2\cdot$h (with the electrolyte agitation). The formed and adsorbed S^{2-}-ions on the cathode make leave by the convection at the electrolyte agitation changing the phase composition of the deposit and electrochemical characteristics of Ni sulfide interaction with lithium in the accumulator model as the consequence.

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EFFECT OF COBALT ADDITION ON STRUCTURE AND ELECTROCHEMICAL BEHAVIOUR OF NICKEL HYDROXIDE SYNTHESIZED BY CHEMICAL PRECIPITATION METHOD UNDER DIFFERENT CONDITIONS

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Abstract

The paper relates to research work focused on utilization of nickel hydroxide with improved electrochemical properties in rechargeable alkaline batteries where it is used as a positive material.

A treatment of chemical composition, i.e. cobalt amount, and synthesis conditions of nickel hydroxide were carried out for purposes of the product optimizing ensuring excellent electrochemical parameters and their durability in long-term cycling process. Set of several samples with variously modified molar ratios of cobalt to nickel as well as precipitated from mixed nickelous and cobaltous salt solutions in different concentrations of alkaline hydroxide solutions was prepared. All these propositions were based on following conclusions:

Cobalt introduced into lattice of nickel hydroxide improves reversibility of redox reactions on cycled nickel hydroxide electrode (NHE) as well as charge acceptance of the active material via lowered anodic oxidation potential and increased oxygen overvoltage η_{O2} on charged NHE at the same time. Consequently, it means that an improvement of the charge efficiency occurs. Cobalt addition also suppresses presence of “low-density” gamma phase formed during overcharging and thereby increase resistance to undesirable volume changes in cycled active material. We applied cobalt as two species, primarily incorporated through co-precipitation and secondary as surface modification/coating of particles $Ni(OH)_2$.

The pH value has been found to be crucial factor that affected the crystal structure characteristics, such as degree of crystallinity, crystallite size, lattice disorder and crystal growth orientation. The amounts of sulphates and carbonates captured in crystallites also significantly depend on the pH value of the chemical precipitation reaction.

Acknowledgements

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CONCEPT FOR RECOVERY OF VALUABLE COMPONENTS FROM BLACK MASS, SEPARATED FROM Ni-MH BATTERY SCRAP

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Abstract

Method for recovery of Ni/Co and lanthanides (Ln =La+Ce+Pr+Nd) values from black mass, separated from Ni-MH battery scrap, has been presented in abbreviated poster form. According to experimental results crude charge of a very complicated chemical, phase and granulometrical composition should be processed in the sequence of technological blocks of operations.

The first block: preliminary treatment, comprising electrolyte removal and thermal oxidation steps, wherein NaOH/ KOH is being removed, and intermetallic hydrogen storage alloy $LnNi_5$ is being completely oxidized into Ln_2O_3 and $LaNiO_3$, and, by the way, auxiliary organic substances are fully removed, prepares upgraded charge, that is suitable for hydrometallurgical processing.

The second block: collective solubilization and Ln separation steps, comprising collective leaching, wherein Ni/Co and Ln are being effectively leached together in accordance with either hot acid leaching (HAL) or activated hot acid leaching (AcHAL) procedures, which ensures higher rates of metallic nickel digestion, and next - Ln precipitation from leach solution; aforesaid block of steps results in separation of snowy-white sulphatic Ln concentrate ($NaLn(SO_4)_2 \cdot H_2O$) from Ni-Co solution, containing other contaminants (Mn, Fe, Al, Zn, etc.), but almost Ln-free, which could be further processed for Ni and/or Co recovery on oxyhydrolysis – solvent extraction route (experiments in progress).

The third block: Ln recovery product enrichment, comprises desulphatisation step, wherein white sulphatic Ln concentrate (Ln ~ 35%), is converted into yellow hydroxidic Ln concentrate (Ln ~ 65%), and next – calcination step, wherein yellow hydroxidic Ln concentrate (Ln ~ 65%) is being completely transformed into pale-brown oxidic Ln concentrate (Ln ~ 75%).

In performed investigations starting from crude charge, containing ~50% Ni, ~6%Co and ~14%Ln, and following depicted technological route said valuable components have been successfully separately recovered with extraction rates ≥95% for both Ln and Co, and 90-95% for Ni (depending on selected leaching condition).

All three Ln recovery products are marketable, and, moreover both hydroxidic and oxidic concentrates are even more valuable than high-grade bastnäsite (∑REO~ 68%) and monazite ((∑REO~ 45%) concentrates, manufactured from natural mineral sources.

Acknowledgements

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EFFECT OF INHIBITORS ON Zn-DENDRITE FORMATION

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Abstract

The effect of TEPA, Lugalvan P, Pragogal Zn 3400, Alfonal K, Slovasol, Tego Gluksid and Pragolub 1300 additives on the inhibition of Zn-dendrite growth in Zn-Ni batteries was studied. For simulation Zn-Ni batteres, three electrodes systems with Zn as working e., Ni as counter e. and Zn as reference e., with Toray PS0100S as separator and 6M KOH with suspension of ZnO as base for electrolyte were used. For charging and discharging we used Bio-Logic Potentiostat/Galvanostat and current 20 mA for 2 hours. This contribution deals with some experiments with charging and discharging modes. Formation of dendrites was monitored by a microscope, which takes one picture every 6 minutes.

THE EFFECT OF NICKEL CONTENT IN NEGATIVE MASS ON ELECTRODE CAPABILITY IN Ni-MH SYSTEM

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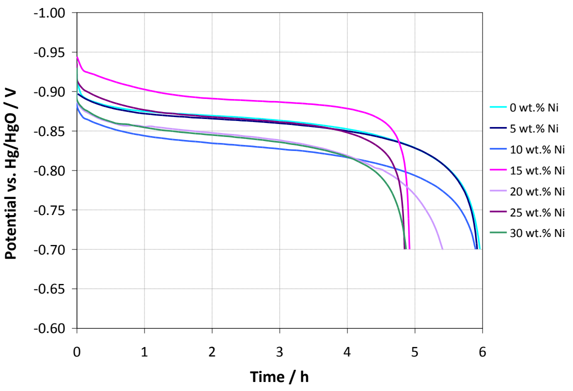
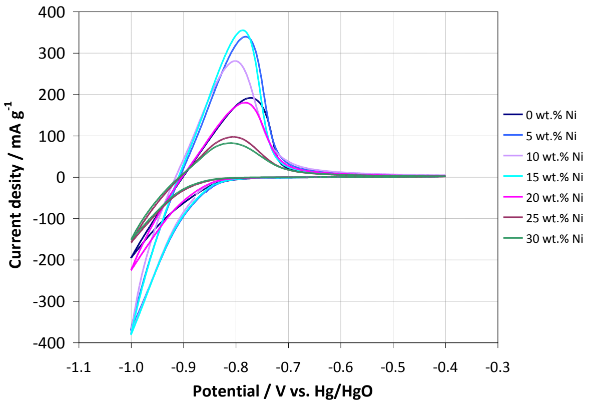
Abstract

The electrochemical properties of alloys reversibly absorbing hydrogen, used as negative electrode material in Ni-MH batteries, are strongly influenced by nickel presence. In the reversibly absorbing hydrogen alloys of AB_5 type, nickel is one of the elements of the B group. The kinetics of electrode reaction, average charge/discharge potential and discharge capacity are depended on the amount of nickel in the alloy.

Nickel in the composition of intermetallic compounds affects the creation of specific crystalline structure while simultaneously decreasing the energy of Me-H bond to a value suitable for the application of the electrode in a Ni-MH-type cell. Moreover, nickel presence in the electrode in metallic form increases the electrical conductivity, catalyzes electrode reactions and the surface hydrogen dissociation.

In the presented study the influence of different amounts of metallic nickel (from 0 to 30 wt.% Ni) on the reaction kinetics, and the MH electrode performance was observed. Research into electrochemical performance of electrodes with those additions was conducted to determine the best composition for the negative active mass of nickel-metal hydride batteries (Ni-MH). The electrochemical properties such as specific capacity, charge efficiency, percent of utilization and cycleability were defined in a half-cell system by cyclic galvanostatic charge/discharge and cyclic voltammetry.





Acknowledgements

The authors acknowledge the financial support from the European Fund of Regional Development within the frameworks of the operating program –"Innovative Economy 2007–2013", under Project No. POIG.01.01.02-00-015/09.

PVA/KOH MEMBRANE FOR ELECTROLYSIS WITH HIGH CONDUCTIVITY

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Abstract

In this paper we are coating crosslinked PVA/KOH membrane with high conductivity to appropriate nonwoven polypropylene fabrics to keep good mechanical properties of membrane. Part of the article will be also confrontation of PVA/KOH membrane with commercial product in conductivity measurements and also in real electrolyser.

Acknowledgements

This work was supported by the grant FEKT-S-11-7 and project CVVOZE CZ.1.05/2.1.00/01.0014.

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CARBON MODIFICATIONS OF AB$_5$-TYPE HYDROGEN STORAGE ALLOY USED AS ANODE MATERIALS IN Ni-MH CELLS

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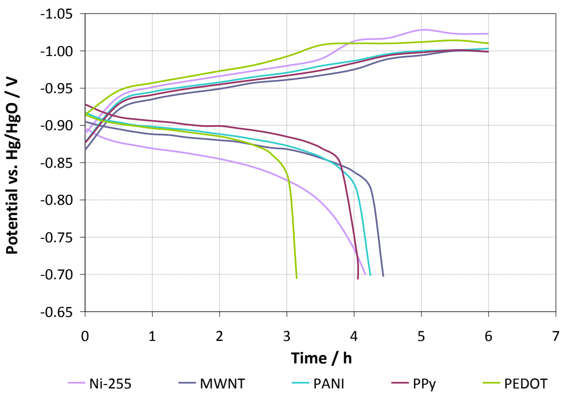
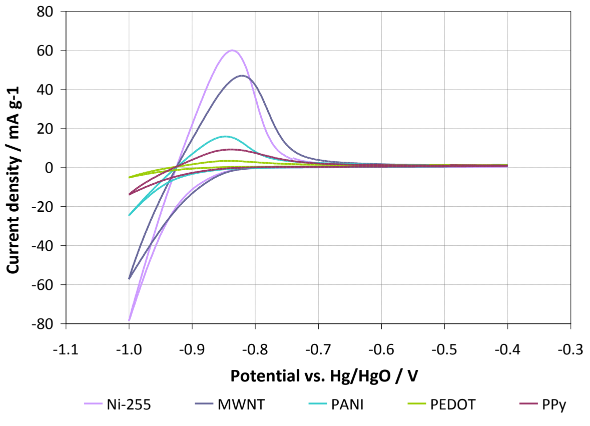
Abstract

The conducted research described the influence of different additions – enhancing the electrical conductivity in electrosorption of hydrogen – on the performance of negative electrode used in Ni-MH cells. Two commercial materials was used as the additions: carbonyl nickel powder 255 type (Inco) and multiwall carbon nanotubes with outer diameter 110-170 nm (Sigma-Aldrich), and also three carbon materials obtained by carbonization of conducting polymers: polyaniline (PANI), poly3,4-ehtylenedioxyhtiophene (PEDOT) and polypyrrole (PPy). The surface morphology of the examined materials was observed using scanning electron microscope SEM EVO® 40 ZEISS. Galvanostatic charge/discharge method with different current densities was used to determine the electrical capacity of tested electrodes. The reaction kinetics was defined with the cyclic voltammetry. The best results were obtained for the electrodes with additions of MWNT > PANI > Ni-255 > PPy > PEDOT.





Acknowledgements

The authors acknowledge the financial support from the European Fund of Regional Development within the frameworks of the operating program –"Innovative Economy 2007–2013", under Project No. POIG.01.01.02-00-015/09.

SURFACTANT ELECTROLYTE ADITIVES EFFECT ON ALPHA NICKEL HYDROXIDE

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Introduction

Nickel Hydroxide is the basic material for positive electrode of alkaline accumulators as Ni-Cd, Ni-MH and Ni-Fe. The three modifications of nickel hydroxide are known, these are alpha, beta and gama.

Nickel Hydroxide can be prepared by mixing of Nickel salt and Hydroxide as KOH or NaOH. Other way of preparing Nickel Hydroxide is electrochemical deposition from Nickel nitrate. Surfactant can slow down recrystalization of Nickel hydroxide.

Experimental, Results and Discussion, Conclusions

Thin layers of Nickel hydroxide were prepared by electrodeposition from Nickel nitrate with various amount of Aluminium nitrate. The concentration of solution was 0,1M $Ni(NO_3)_2$ + $Al(NO_3)_2$ in H_2O. The samples were measured in 6M KOH solution with 2% of Sodium Alkyl Ether Sulfate and Alkyl Dimetyl Benzyl Amonium Chloride (ADBAC) by cyclic voltammetry for 150 cycles. Scan rate 10mV/s, potential window 0-600mV to Hg/HgO reference electrode.

The best capacity was measured on Nickel Hydroxide with 10% Aluminium Aditive measured in KOH electrolyte with ADBAC Tenzide. Higher concentrations of Aluminium aditive causes passivation of Nickel Hydroxide structure and the measured capacity were lower. Nickel Hydoxide electrode with he aditive of cathodic Tenzide in electrolyte has better capacity than pure KOH.

Acknowledgements

The investigations were supported by Ministry of Education of Czech Republic, Project MSM0021630516

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PERFORMANCE OF THERMAL CELLS, BASED ON Mg-$PbSO_4$ ELECTROCHEMICAL SYSTEM, MADE FROM POWDER MATERIAL

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Abstract

One of the types of thermal cells are cells composed of elements in the form of tablets. They are made by a method known as powder technology or pressed technology. This technology is based on the preparation of mixtures of the electrolyte and the cathode in the powder form and then pressing one tablet of specific composition and dimension. Single cell has a cathode, an electrolyte-separator system and anode, which contains a reducing agent in the form of solid metal, bi-metal or metal placed in the matrix. Electrolyte and cathode layers are permanently connected.

This method minimizes waste production and allows constructing batteries more resistant to exposure to vibration that cause leakage of the electrolyte and cathode material, which can lead to short-circuit cells.

The study applied two types of anode (Mg and Mg/ KCl-LiCl 5 wt.%) and the cathode $PbSO_4$ containing percentage additions of graphite and silica. The electrolyte was a mixture of 88 wt.% KCl-LiCl and12 wt.% of SiO_2

The obtained electrode thickness was measured and assessed compactness and underwent a series of the cells and electrical study was performed at temperature ranging from 400 to 500 ^{o}C. Electrical tests were carried out by activating cells electric heaters.

Acknowledgements

The authors acknowledge the financial support from the European Fund of Regional Development within the frameworks of the operating program –"Innovative Economy 2007–2013", under Project No. POIG.01.03.01-00-086/09-01.

SIMULATION AND MATHEMATICAL DESCRIPTION OF CHARGING AND DISCHARGING OF THE LEAD ACID ACCUMULATOR

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In a lead-acid cell the active materials are lead dioxide (PbO_2) in the positive plate, sponge lead (Pb) in the negative plate, and a solution of sulfuric acid (H_2SO_4) in water as the electrolyte.

A chemical reaction takes place between lead plates and sulfuric acid, creating electric current. When the charge is depleted, the battery can be recharged many times.

When the battery is electrically loaded, the sulfate in the acid reacts with the positive and negative plates, forming lead sulfate. Electrons from the reaction flow out from the negative terminal to the circuit and then back into the positive side.

When the battery is charging, the sulfate leaves the lead and becomes sulfuric acid again. The discharge-charge cycle can be repeated thousands of times – unless the battery is discharged too deeply.

Acknowledgment

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ACTIVE MASS AND COLLECTOR CORROSION RESISTIVITY AND IMPEDANCE CHANGES OF PULSE CHARGED LEAD-ACID ACCUMULATOR

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Abstract

This paper describes active mass and collector corrosion resistivity and impedance changes of lead-acid accumulator of electrodes. Data was obtained during experiment with pulse charging cycling of old lead-acid accumulator. Both resistivity changes were measured using four-point direct current method. Impedance changes were observed by electrochemical impedance spectroscopy. Old lead-acid accumulator previously not used for almost half of a year was used in the experiment. Reasons why pulse charging was used follows: increased efficiency of charging, gas generation reduction, corrosion speed reduction, sulfation reversion of old cells (1).

Acknowledgements

This work was supported by the EU CZ.1.05/2.1.00/01.0014 and by the specific graduate research of the Brno University of Technology No. FEKT–S–11–7.

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INFLUENCE OF VARIOUS AMOUNTS OF TITANIUM DIOXIDE IN NEGATIVE PLATE TO IMPEDANCE PROPERTIES OF LEAD-ACID ACCUMULATOR

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Abstract

It has been established that addition of carbon additives to the negative active material of lead-acid batteries increases battery charge acceptance in hybrid electric vehicle mode of operation (1). But the latest findings show that the titanium dioxide has similar effect (2). Hence this paper deals with impedance characteristics of experimental lead-acid accumulators with various amounts of titanium dioxide in active mass of negative electrode. Electrochemical impedance spectroscopy was used for all measurements. Obtained data was subsequently fitted by equivalent circuits. It was investigated that ohmical resistivity lead-acid cell increases with higher amounts of titanium dioxide in active mass of negative electrode.

Acknowledgements

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HEAT CHANGES IN THE LEAD ACID BATTERY CELL

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Abstract

Heat changes in the lead acid battery cell during charging and discharging are affected by the construction of the cell and by method of cycling. They affect the use properties of the electrodes and they can change the capacity and cycle life of the cell. The issue of heat changes in the lead acid cell was studied by several authors (1-4). For correct determination of heat changes it is necessary to calculate the contributions of all types of heat. It is the heat generated or lost due to the electrochemical reactions, heat generated due to the ohmic and polarization losses (Joule heat) and heat lost by contact with the environment.

It were realized practical measurements of temperature changes in the experimental cell equipped with temperature sensors Pt100 (Fig. 1), in different modes of discharging and charging. Theoretical calculations of heat changes in the lead acid cell were confirmed by practical experimental measurements inside the cell. Optimum appears to be modification of charging mode, instead of charging with constant current it is better to use charging with voltage limitation around voltage 2.45 V, reducing both gassing of the cell and rise of the temperature. In constant current charging under unfavourable conditions (elevated ambient temperature and long overcharging) it can be started oxygen and hydrogen cycle, further increasing the cell temperature and it can lead to the collapse of the cell (thermal runaway).

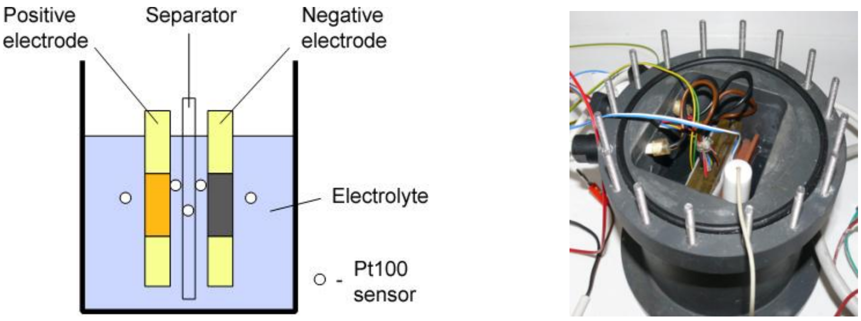

***Fig. 1**: Scheme of the experimental cell with Pt100 sensors*

Acknowledgements

This work was sponsored by the Internal grant No. FEKT–S–11–7 and by the EU project CZ.1.05/2.1.00/01.0014.

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CONDUCTIVE CERAMIC AS AN ADDITIVE OF NEGATIVE ACTIVE MASS OF LEAD ACID BATTERY

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Abstract

When using lead-acid batteries under PSoC regime there are new mechanisms of failures appear known as PCL3. These failures are associated with progressive sulphation of the negative active material and they are caused by insufficient charging of the negative active mass and by intensive oxygen cycle. Possibility to eliminate PCL3 proved to be to use carbon as an additive in the negative active mass [1, 2]. The original concept of the mechanism of carbon function in the negative active mass was based on the high conductivity of carbon, that can to improve conductance of the active mass especially in the last stages of the discharge [1, 3]. In our previous experiments we studied the influence of carbon on the cycle life of the negative active mass in the PSoC cycling [4]. The current experiment was focused on the ceramic material Ti4O7 known as EBONEX, which has similar conductive properties as carbon previously tested. First results indicate, however, that addition of Ti4O7 throughout the tested range of weight percent does not improve the cycle life of the negative active mass during the PSoC cycling.

Acknowledgements

This work was sponsored by the Internal grant No. FEKT–S–11–7 and by the EU project CZ.1.05/2.1.00/01.0014.

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LAPLACE'S PRESSURE INDUCED RHOMBOHEDRAL PHASE IN THE SCANDIA-STABILIZED ZIRCONIA CERAMICS

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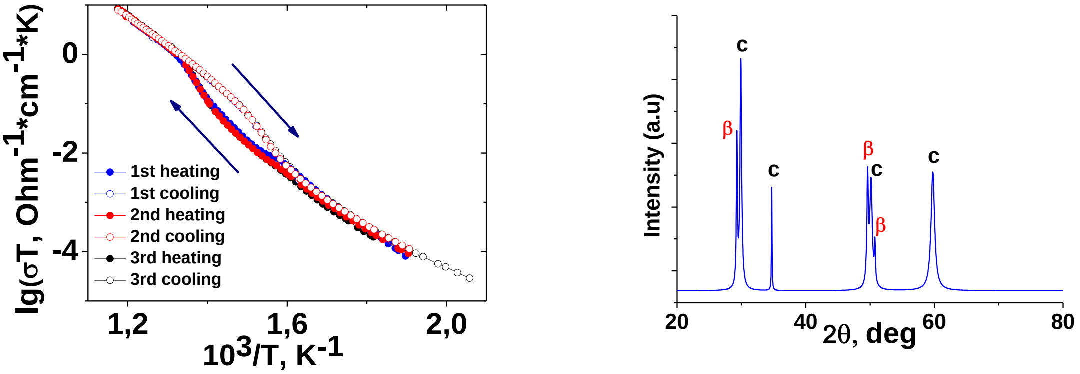
The present study is devoted to the grain-size effect on the conductivity and phase composition of scandia stabilized zirconia ceramics which was produced from DKKK (Japan) powder with composition 89 mol.% ZrO_2 + 10 mol. % Sc_2O_3 + 1 mol. % CeO_2. Variation of grain size in ceramics was obtained using the control of such parameters like temperature, sintering duration and powder pressing conditions.

Decreasing the grain size of ceramics from 16 to 0.6 μm qualitatively changes the form of lg(σ) – 1/T dependence. For ceramics with large grain size (16 μm), curve of ionic conductivity demonstrate the anhysteretic kink at 713 K. For material with the twice smaller grain size (8 μm), this curve obtains the sigmoid form. For fine-grained ceramics (0.6 μm), lg(σ) – 1/T curves demonstrate the hysteretic kink (fig. 1a) caused by the presence of rhombohedral phase. Laplace's pressure grows with decreasing the grain size, and for the grains with a diameter 0.6 μ m it reaches a value 0.33 GPa. This value is enough to transfer the material from the tetragonal phase to rhombohedral phase which has the significantly lower conductivity. This conclusion fully corroborates with our results of X-ray phase analysis (fig. 1b).

All specimens sintered from DKKK powder don't show the previously studied [1] low-temperature kinks on Arrhenius plots of ionic conductivity. However, the ionic conductivity value is reduced to a second for the specimen orthogonal directions within temperature range 640 to 740 K, and anisotropic properties of the specimen are the controlled parameter.



a) b)

***Pic.1:** Arrhenius plots of ionic conductivity (a) and phase composition (b) of zirconia ceramics which was produced from DKKK (Japan) powder with grain size 0.6 μm*

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DYNAMIC VISCOSITY OF SOLVENTS FOR ELECTROLYTES OF LITHIUM-ION ACCUMULATORS

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Abstract

Lithium-ion accumulators are among the power sources suitable (are used), especially for (the) mobile applications, medical devices and electric vehicles. Rheological properties description of the electrolytes is one of the most important non-electrical physical quantities. These properties, in particular the value of dynamic viscosity, are decisive for the selection of suitable electrolyte solvents usable in lithium-ion accumulators.

Solvents mixture of dimethylsulfone and sulfolane with a lithium salt (in particular case $LiClO_4$) appear as potentially suitable electrolyte. The resulting ratio of the components depends on the desired properties to be achieved. The dependence of dynamic viscosity in a wide temperature range is observed to ensure optimal electrical properties. Dynamic viscosity is closely related to electrical conductivity in accordance with Walden's rule.

The vibration method is used to determine the values of dynamic viscosity. Its principle consists in the transfer of current, required for keeping constant frequency and amplitude of the vibrations of the sensor plates immersed in the observed liquid, on the apparent viscosity. Conversion of apparent viscosity on dynamic is conditioned by knowledge of the sample density. In the case of the realized experiment, viscometer A&D SV10 and analytical balance Ohaus Explorer EX223 with extension density determination kit, using Archimedes' law, were used. The temperature stabilization of the measured sample is realized by using the bath thermostat Medingen KT40. The realized laboratory workplace allows continuous monitoring of viscosity and density in the temperature range by using remote access over the Internet.

The aim of this experiment is to find the optimal composition of the electrolyte from the perspective of achieving the lowest dynamic viscosity and to determine its temperature dependence. The value of activation energy is calculated from the measured temperature dependence of dynamic viscosity of electrolyte.

Acknowledgements

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COLD ISOSTATIC PRESSING EFFECT ON MICROSTRUCTURE AND ELECTRICAL CHARACTERISTICS OF ScCeSZ CERAMICS

Akimov, G. Ya., Komysa, Yu. A.

The effect of cold isostatic pressing (CIP) on the phase composition, sintering behavior, and conductivity of 10Sc1CeSZ ceramics has been investigated. Specimens were isostatically compacted from commercial DKKK (Japan) powder at pressures up to 1000 MPa and sintered at 1100 °C or 1500 °C. The densities obtained were between 5.71 and 5.89 g/cm^3. Sintering at 1500 °C yielded pure cubic phase in all specimens while sintering at 1100°C resulted in significant share of rhombohedral (β) phase at all CIP pressures except for 500 MPa. In good accordance with the phase composition, conductivity values for all specimens sintered at 1500 °C were quite close reaching 0.043 S/cm at 700 °C while for specimens compacted at 200 MPa or 1000 MPa and sintered at 1100°C they were much less reaching only about 0.020 S/cm at 700 °C. The specimen compacted at 600 MPa and sintered at 1100 °C revealed highest conductivity about 0.047 S/cm at 700 °C. Taking into account small average grain size 0.98 μm and high sintered density 5.84 g/cm^3, this material can be considered as the best choice for IT-SOFC.

USE OF DIMETHYL SULFONE IN APROTIC ELECTROLYTES

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Abstract

The aim of this work is to determine the influence of dimethyl sulfone on electrical properties of electrolytes. Dimethyl sulfone is a adjacent product by many chemical manufactory. The key thought of this article is to find a possibility to use of this adjacent product. In this work is dimethyl sulfone used as additive into commonly applied solvents that are used for aprotic electrolytes. There will be established properties in low flammable solvent such as sulfolane too. The article will discussed specific conductivity of this solvents mixtures.

Acknowledgements

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MEMBRANES FOR ALKALINE ACCUMULATORS

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Abstract

In this paper are given global information about our recent research in the field of membranes for alkaline accumulators. Various types of experimentally prepared membranes were tested. We were focused on ionic conductivity and dendritic resistance measurements and on stability in highly alkaline liquid electrolytes. Different materials for electrode coating were also tested as a ionic conductivity membranes and as a holders for active electrode material.

Acknowledgements

This work was supported by the grant FEKT-S-11-7 and project CVVOZE CZ.1.05/2.1.00/01.0014.

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EFFECT OF FUNCTIONALYSED SILSESQUIOXANES ON LITHIUM CATION TRANSPORT IN PEO BASED POLYMER ELECTROLYTES

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Abstract

The demand for the most efficient energy storage and conversion device results in the need for the development of cheap, reliable, durable and most of all environmentally-friendly batteries. The most promising and widely studied are lithium cells that offer highest energy densities and cell voltages among all battery systems known (1). However environment and safety issues prohibit production of large Li-ion batteries in the present technology because of the liquid electrolyte inside. In order to bypass these issues the suggestion by Armand et al. of using solid polymer electrolytes has been widely explored over last three decades (2).

One of the cheapest, most environmentally-friendly and most promising materials are polymer-salt complexes based on lithium salts and poly(ethylene oxide). The studies on these complexes showed low room temperature conductivities. Works on this materials and their modifications have been mostly devoted to enhancing the ion transport properties (3).

Currently, in our laboratory improvement and increasing conductivity of composite dry polymer electrolytes based on PEO is being researched. It is done by adding to PEO lithium salt and functionalysed silsesquioxanes with different morphological structure.

One of the most intriguing groupe of nanocomposite is silsesquioxane. A silsesquioxane is a compound with the empirical chemical formula $RSiO_{3/2}$ where Si is the element silicon, O is oxygen and R is either hydrogen or an alkyl, alkene, aryl, arylene group which can design composite properties on a molecular level. Silsesquioxanes can have a cage like structure,(see Figure 1), which is most commonly cubes.

The present work shows the preparation route as well as basic structural and electrochemical characteristics of the resulting composite dry polymer electrolytes with different weight ratio of functionalysed silsesquioxanes to poly(ethylene oxide).

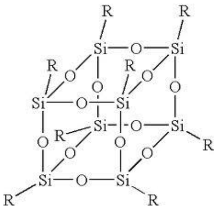

Fig. 1: *Silsesquioxanes cage structure*

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MECHANISMS OF IONIC CONDUCTION IN PVA BASED NANOCOMPOSITES

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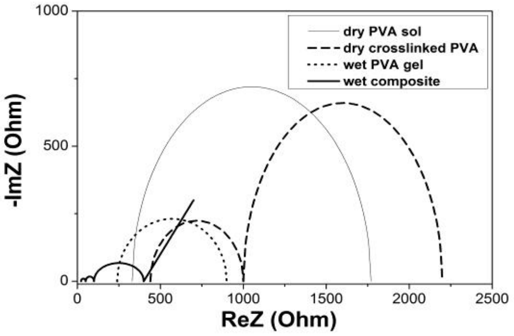
End performance of fuel cells with polymer membranes as a base of advanced energy storage systems is determined by selectivity of the electrolyte in respect to fuel crossover. Polyvinyl alcohol (PVA) based composites are most selective up-to-date polymeric electrolytes with proton conductivity. Best PVA-based compositions in wet condition have conductivity nearly as high as Nafion but are much less methanol permeable. Raising conductivity of polymeric membranes by the order would allow reaching DMFC efficiency and power density at the SOFC level but at room temperatures (1). The aim of this work was to study mechanisms of ionic conduction in cross-linked PVA/$Al(OH)_3$ membranes in all material processing stages using high resolution electrochemical impedance spectroscopy (HR-EIS) with a high capacitance cell (2).



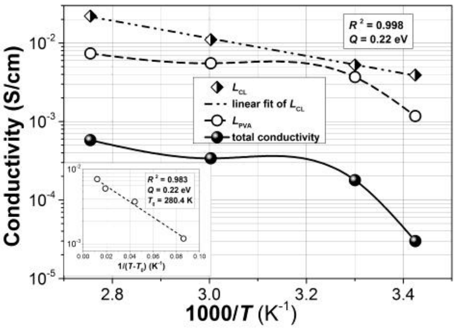


Fig. 1: *(left) Fitted impedance spectra of PVA based membranes.*
Fig. 2: *(right) Arrhenius and VTF (insert) plots of total conductivity and lines L_{PVA} and L_{CL}.*

Dry PVA sol or wet PVA gel show one EIS line (L_{PVA}), conductivity of wet electrolyte being several times lower (Fig. 1). Cross-linked PVA reveals the second line (L_{CL}) with nearly the same total conductivity. Filler yields no resoluble extra lines but reduces total width by order or more if optimize filler content and working temperature. Cross-linked PVA electrolytes are hence essentially multicomponent unlike Nafion. Protons in such material have to sequentially overcome both linker-associated interlayers and PVA blocks very poorly permeable by methanol.

Mechanisms of conduction in these two constituents are understandable from Fig. 2. Temperature dependence of interlayer conductivity (L_{CL}) obeys the ordinary thermal activation with activation energy 0.22 eV that corresponds to Grotthuss hopping in bound water (3). PVA block conductivity (L_{PVA}) obeys the Vogel-Tammann-Fulcher (VTF) model with zero-free-volume temperature 280.4 K describing viscous motion of chain segments (4).

It can be concluded that the best selectivity should be attainable in PVA based composite electrolytes optimized for not only filler kind and content but also matrix block structure and cross-linking agent.

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PVA MEMBRANES FOR THE PEM FUEL CELLS

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Abstract

In this paper are given global information about recent low temperature polymer electrolyte membrane (PEM) fuel cells. Furthermore, experimental preparation and conductivity measurements of polyvinyl alcohol (PVA) polymer electrolyte membranes are described. Fundamental characteristics of fuel cells containing PVA membranes were measured (e. g. volt-ampere and power characteristics).

Acknowledgements

This work was supported by the grant FEKT-S-11-7 and project CVVOZE CZ.1.05/2.1.00/01.0014.

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CRITICAL CRACK LENGTH FOR THE INITIATION OF UNSTABLE PROPAGATION IN RELATION TO FRACTURE TOUGHNESS AND THE EVALUATION OF STRESS AND DEFORMATION CHARACTERISTICS OBTAINED FROM TENSILE TESTING

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Abstract

The paper is concerned with the relation between temperature and critical crack length for the initiation of a brittle crack unstable propagation at the yield strength level stress of high-strength low-alloy (HSLA) steel. For the purpose of calculation, results of dynamic fracture toughness test were applied; the experiment was supplemented with measuring stress and deformation characteristics in a determined test temperature range. For comprehensive assessment of test temperature effects, a fractographic analysis of fracture surfaces was conducted. The results show that the risk of brittle failure occurrence may be expected over the entire range of test temperatures -80 °C to 100 °C; at yield strength level stress.

STUDY OF BIOCOMPATIBLE ZrN AND ZrN/DLC COATING DEPOSITED ON MEDICAL TOOLS

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Abstract

The paper presents results of study of mechanical and chemical properties of ZrN and ZrN/DLC coatings. These coatings were deposited on martensitic stainless steel surgical instruments by using PVD arc deposition. After treatment, SEM microscopy, laser confocal microscopy, microhardness and indentation test were used to characterize surface morphology, thickness, hardness and adhesion.

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APPLICATION OF WIND TURBINE TO TEACHING

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Abstract

The aim of this article is to introduce a possibility of use a wind turbine in education process. The students will be able to measure the efficiency of wind energy to electricity transformation. To reach this goal there will be a laboratory workplace assembled. The workplace will contain a 100 W wind turbine, lead accumulator, measuring instruments and multiplexor. The measurement will be carried out be using a software interface special created to this purpose.

Acknowledgements

This work was supported by FRVŠ 2227/2012/G1 Measuring workplace to investigate the wind energy characteristics and its application to teaching and specific research FEKT-S-11-7 Materials and technologies for electronics.

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STRUCTURAL ANALYSIS OF $CoSb_3$ BASED POWDERS OBTAINED BY DIFFERENT SYNTHETIC ROUTES

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Abstract

Lithium-ion batteries are state-of-the-art power sources for portable electronic devices. due to their high working voltage and energy density. Carbon-based materials are currently used as anode materials due to the flat charge and discharge plateau and excellent cycling stability[1]. Great interest has been turned to metal or alloy anodes due to their extremely larger capacity compared to those of carbon-based materials. Among these alloys, which can be potential anode materials for secondary Li-ion batteries, the Sb-based intermetallic compounds received much interest in recent years. Alc´antara et al. [2] first reported $CoSb_3$ as the anode materials for Li-ion batteries [1]. The intermetallic compounds exhibit slightly smaller Li-storage capacity than the pure metals, but often show an improved cycling stability. Generally, the intermetallic compound consists of an active species and an inactive species. It is believed that the active species can electrochemically react with Li when polarized to the sufficiently low potential and provides the anode capacity, while the inactive species offers an inert matrix to support the active species and alleviates the volume effect associated with the active species during the charge and discharge cycling [3].

In our present work, $CoSb_3$ powder was synthesized by solvothermal method and by sintering and mechanical milling. The study of the influence of synthesis on the structure and properties of $CoSb_3$ was performed using the following techniques: XRD (Fig.1), SEM, TG and textural parameters was determined using nitrogen adsorption isotherms.

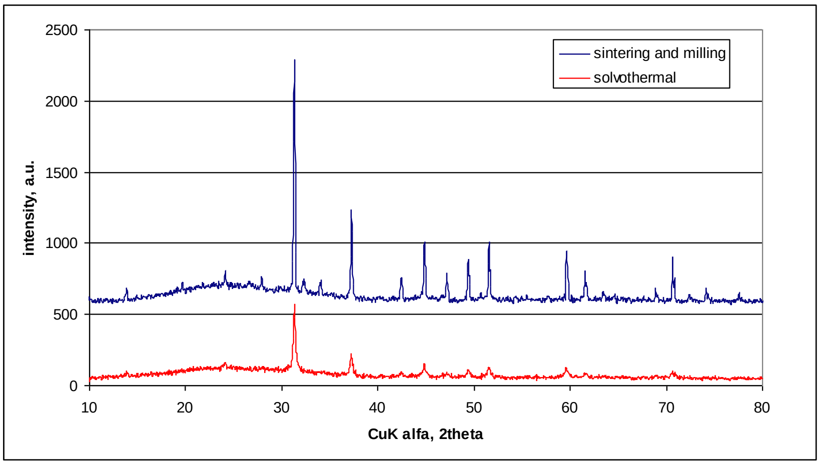

Fig. 1: *XRD patterns of powders $CoSb_3$ obtained by two different routes.*

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THE HYDROGEN BUS AS A MATHEMATICAL MODEL IN MATLAB© – COMPARISON WITH ITS ACTUAL OPERATION

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Abstract

The paper firstly outlines control strategies for all three cooperating energy sources of TriHyBus (Primary source: 50 kW PEM fuel cell, secondary energy sources: 22 Li-Ion traction batteries, 4 supercapacitors) and conditions for their cooperation. For optimization of routine operation of the bus a mathematical model in MATLAB© - Simulink© has been developed at Department of Power engineering, Faculty of Electrical engineering, Czech Technical University in Prague for needs of UJV Řež. The bus is supposed to drive on a fixed route in a city (public transport), so the data about speed and altitude could be acquired in advance, and the optimal acceleration will be computed with MATLAB© according the specific algorithm described in the paper. The solution of power flow consists of control strategies for fuel cell, charging of batteries and supercapacitor, and conditions for both limiting situations: lack and surplus of energy on the main bus. Possible extensions of the model to achieve more precise data and better results are discussed. In the second part of the paper the MATLAB© model is tested and verified by data obtained from the actual operation of the bus and the conclusions about differences between the model and the actual operation are made.

HIGH-POWER CHARGER FOR SUPER-EL ELECTRIC PASSENGER CAR

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Abstract

Lack of the High-power fast-chargers is one of most important factor limiting the expansion of electric vehicles. Fast-chargers enable to charge the traction accumulators during short time of periods (ones of hours or tens of minutes). Nowadays „on board“ fast-chargers are required instead of heavy and large stationary solutions. This is why new requirements on high power/mass and power/volume ratio are demanded in this area. Also an acceptable low price of these fast-chargers is important.

This paper introduces a function pattern of that fast-charger, which is intended for SUPER-EL electric passenger car. The mentioned properties are achieved using a non-traditional simple construction with a DC/DC converter operation at a very high switching frequency. Progressive silicon-carbide semiconductors are used in this converter. Charger provides output parameters 480 V/100 A. The introduced functional pattern RN48-480 is constructed as an auxiliary portable device (not „on board“), however its dimensions are very small and the driver of the electric car can carry it steadily in the vehicle. Then the charger can be plugged-in to the common grid 3x400 V anywhere to charge the traction accumulator.

Acknowledgements

This work has been prepared under the support provided by research projects FEKT-S-11-14, FR-TI1/061 of Ministry of Industry and Trade of the Czech Republic and Center for Research and Utilization of Renewable Energy no. CZ.1.05/2.1.00/01.0014.

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PARASITIC EFFECTS IN POWER CIRCUITS OF HIGH POWER BATTERY CHARGERS

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Introduction

This article deals with a new battery charger concept with nominal power of 16 kW. Minimum possible size, weight and the lowest price were required while keeping the simplicity and reliability of the equipment. For this reason it was necessary to use an innovative circuitry and design and an extremely high switching frequency of 100 kHz.

Experimental, Results and Discussion, Conclusions

Due to the usage of high switching frequency and the nominal power of 16 kW, it was necessary to suppress or eliminate parasitic effects in the power circuits of the charger converter. These parasitic effects did not exist or were negligible at lower switching frequencies. It was necessary to solve following problem tasks in the development of the device:

- Design of the input rectifier (EMI problems, power factor LAMBA increasing).
- Influence of the leakage inductance of the high frequency transformer on the equivalent internal resistance (in the output of the converters.
- Reverse recovery effects in the secondary rectifier diodes.
- Forward recovery effects in the demagnetizing diodes (primary side of the converter).

New semiconductors (transistors Cool-MOS and diodes based on SiC) seem to be the right choice together with an appropriate topology of the power circuit. The whole device (fast-charger) was built and test measurements were provided. The functionality required at the nominal power was verified.

Acknowledgements

This work was solved in the frame of the faculty project FEKT-S-11-14 Utilization of new technologies in the power electronics, further it has been supported by the European Commission under the ENIAC CA-E3Car-2008-120001 E3CAR - Nanoelectronics for an Energy Efficient Electrical Car project.

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USAGE OF THE FUEL CELL-POWERED ELECTRIC DRIVE IN AVIATION

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Abstract

Weight and consumed space of the fuel cell systems are one of the most important factors limiting its utilization in electric airplanes. Possibilities of its implementation in electric airplane are frequently discussed issues. Power – mass relationship depending on installed power of the fuel cell shows, that the power/mass ratio is unsatisfactory, when powering electric motor directly from fuel cell. Configuration with high-power small-capacity battery and relatively small fuel cell is more profitable. Some calculations were done for functional pattern of the small airplane VUT 051 RAY, its take-off weight is about 600kg and its max power is 55kW. There are some practical measurements on the fuel cell system by Ballard presented in this paper, which can documents disputableness of its usage in airplane. Hydrogen management (hydrogen tank, cooling system, armatures etc.) still represents outstanding issue due to high volumes and weight of the whole fuel cell system. Current progress in the field of the Li-Ion accumulators, generally decreases importance of hydrogen-drive systems and turns attention to simply accumulator powered airplane (1).

Acknowledgements

This work has been prepared under the support provided by research projects FEKT-S-11-14, FR-TI1/061 of Ministry of Industry and Trade of the Czech Republic and Center for Research and Utilization of Renewable Energy no. CZ.1.05/2.1.00/01.0014.

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RECYCLING OF PHOTOVOLTAIC CELLS WITH THE POSSIBILITY OF RE-USE SOME COMPONENTS

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Abstract

Development and installation of photovoltaic (PV) is reaching a large number around the world. Taking into account that the lifetime of photovoltaic panels is about 25 years, the question occurs, how we deal with destruction of many such panels in the future. Currently, the old panels are disposed in two basic ways - environmental disposal and recycling. Each option has its pros and cons. We must take into account both the costs of disposal but also on its environmental impact. As for recycling, we can minimize the amount of residual waste from recycled PV modules, we focused on recycling methods with the possibility of using some components that remain after recycling. Recycling must be taken into account all the materials which the PV modules are manufactured, including materials that are used to encapsulate and protect the PV cells. Major recyclable materials for PV panels, which need to be addressed as aluminum, glass, plastic components and to a small extent by heavy metals are. Whether or not worth re-use some components in the manufacture of solar panels, we investigated on the basis of measurements of individual properties and parameters of such a newly manufactured article properties of commercially manufactured articles.

Acknowledgements

This work was supported by the Ministry of Education (project FEKT-S-11-7) and Ing. Radim Bařinka from Solartec company.

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THE WAYS HOW TO MEASURE THE CHARACTERISTICS OF THE SOLAR CELL

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Introduction

Several ways or methods how to measure the properties of the solar cells or cuts exits. In this paper 3 methods are discussed. One of them is focused on infrared thermography which is nondestructive and really effective. We can find the cracks, the hot-spots and the short circuits very fast without turn-off the system.

Experimental, Results and Discussion, Conclusions

Thermographic measurement is able to record the temperature changes much faster than the contact measurement. The best performance is obtained with FLIR i7 thermocamera. This type is the highest model of the basic line. The complete measuring system contains DC power supply, multimeters, solar analyser, solar exposure meter and thermocamera as well. The V-A characteristics of the solar cells are measured and in parallel the termograms are measured and then they are evaluated by the professional FLIR Reporter software.

If parts of the solar panel are hotter than others, the warm areas will show up clearly in the thermal image. Depending on the shape and location, these hot spots and areas can indicate several different faults. The temperature rise of a cell or of part of a cell indicates a defective cell or shadowing.

The thermographic inspection of photovoltaic systems allows the fast localization of potential defects at the cell and module level as well as the detection of possible electrical interconnection problems.

Acknowledgements

This work was supported by the FRVŠ project 2333/2012 and by the specific graduate research of the Brno University of Technology No. FEKT–S–11–7.

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LONG TIME MONITORING OF HEAT DISTRIBUTION IN PHOTOVOLTAIC MODULE BY THE THERMOVISION

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This paper deals with long time monitoring of photovoltaic module in situ by the thermovision camera. The focus of measurement was to monitor and compare the heat distribution and the defects distribution in the module through the work in real condition. The measured was taken every week in same time and the condition of measuring was noted. The output results were recalculated to the relative value to be possible to make correlation of degradation rate and heat distribution.

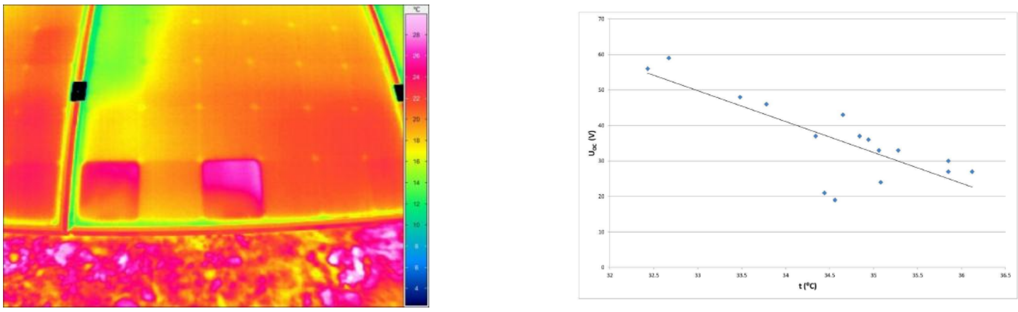

***Fig 1:** a) (left) The tested area of PV module. b) (right)Dependence of temperature and open circuit voltage.*

Ones per week in same time was taken thermovision pictures of photovoltaic module connected to the stable load and also the ambient temperature, light intensity and notes of weather condition were taken. The efficiency of solar module ones per month was measured and the correlation of heat distribution was made.

$$degradation = \frac{P_{mSTC1} - P_{mSTC2}}{P_{mSTC1}} = \frac{228 - 226{,}8}{228} = 0.5\%$$

Conclusions

The long time monitoring of the chosen solar module heat and defect distribution was made. The correlation with the calculated efficiency and also with the defect distribution by the electroluminescence was made and shows the possibility of thermovision monitoring for foreseeing of the destruction of photovoltaic module.

Acknowledgement

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V-I CHARACTERISTIC MEASUREMENT OF SOLAR CELL

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Introduction

This paper deals with creating a workplace for automatic measurement of VA characteristics of photovoltaic cells in the LabVIEW. Challenge is to create a functional program, which automatically measures the VA characteristics not only in the lighted cells, but also in the shaded cell (diode mode). The program calculates the power and important parameters, such as short-circuit current, open circuit voltage, maximum power, current and voltage at maximum power article. The program allows you to export the measured values to a text file. In the last chapter of this work is made control measurements of three samples of monocrystal photovoltaic cell by the measuring workplace. The measurement is compared with the reference device Solar Cell Tester

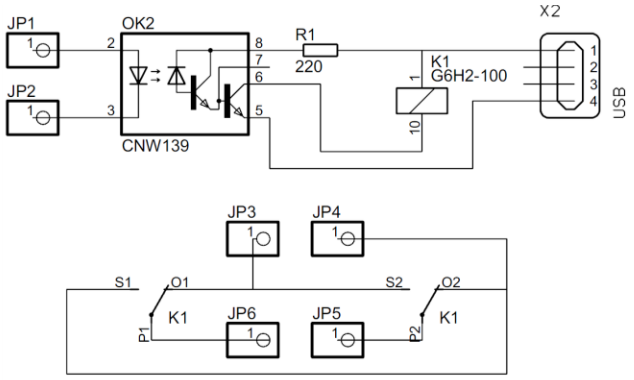

Fig. 1: *The schema of the involvement of the polarity switch*

Conclusions:

The software is designed for the measurement of the VA characteristics of PV cell lit by source of light-powered from AC and DC sources. Therefore, the measurement takes a relatively long period (5 – 100 second depending on the set the number of steps). In the event that the source of illumination is running on battery or DC source, it is possible to shorten the period of averaging the measured values and thus greatly reduce the total time measurement.

Acknowledgements

The authors gratefully acknowledge the financial support by research center CVVOZE CZ.1.05/2.1.00/01.0014 and the project FEKT-S-11-7.

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DESIGN AND OPTIMALIZATION OF CONCENTRATOR SOLAR PANELS COOLING SYSTEM USING COMPUTER SIMULATION

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Photovoltaic concentrators are systems that use lenses or mirrors to concentrate sunlight on photovoltaic cell. This allows us to use a smaller size of the photovoltaic cell to achieve the desired output corresponding to the large-screen panels. In these solar systems more powerful solar cells can be used which would be too expensive in relation to the generated output without using concentrators.

Unfortunately, concentrating of sunlight brings disadvantages associated with higher heating. Light radiation is converted into electrical energy only partially, the remaining (greater) part is converted into thermal energy. If this heat is not removed by additional cooling components, the efficiency of energy conversion is decreased and the excess temperature may occur in the total destruction of systems with high energy concentration.

Acknowledgment

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Sn-Co NANOWIRES: HIGH-PERFORMING ELECTRODES AS ANODES FOR LITHIUM-ION BATTERIES

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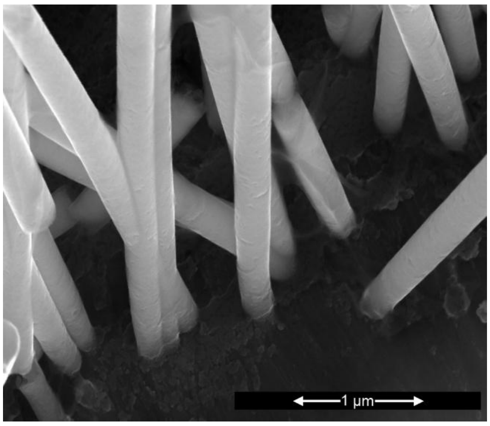
Sn-based amorphous alloys were electrochemically deposited inside pores of a nanostructured polycarbonate membrane. A patented method allows to grow nanowires (NWs) directly and firmly onto a copper current collector. Figure 1 shows a detailed SEM image of the contact zone, after template removal.

Tin is the active material and it reacts with lithium through several steps, up to $Li_{4.4}Sn$. Lithium alloying causes volume expansion due to specific density changes, inducing mechanical stresses, which can be better absorbed by amorphous Sn-M alloys (where M is a transitional metal) instead of pure Sn.

In the present work, it has been investigated how to produce amorphous Sn-Co nanostructured alloys in a wide range of compositions. Changing electrolytic bath composition, it was possible to control alloy composition and crystalline structure~~s~~, attaining the best compromise between higher specific capacity (due to tin content) and higher life cycle (also due to the amorphous structure of the sample). Moreover, the particular shape of nanowires addresses strain mainly lengthwise, avoiding tin agglomeration.



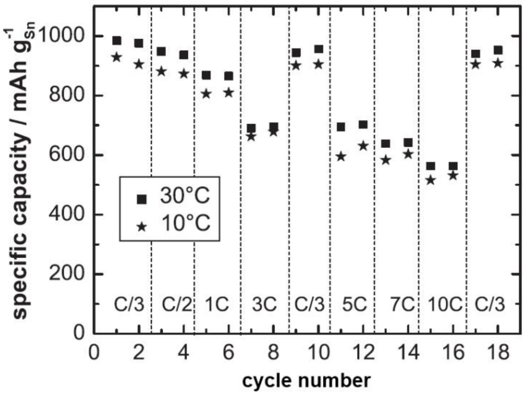


Fig. 1: *(left) SEM image of Sn-Co NWs onto copper current collector (dark grey film).*
Fig. 2: *(right) Discharge specific capacity of Sn-Co NWs vs. cycle number at 10 and 30 °C, at different C-rates.*

The nanostructured Sn-Co alloys were characterized as anodes for lithium-ion batteries. In comparison to carbonaceous anodes, enhanced performances were observed in terms of higher C-rate, and capacity at two different temperatures (see figure 2). Performances of these anodes were also investigated in a battery configuration with $LiFePO_4$ as cathode, with or without addition of vinylene carbonate to improve coulombic efficiency.

Acknowledgments

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INFORMATION ABOUT THE ELECTROCHEMICAL SOCIETY

Introduction

ABAF 2012 is co-sponsored by the Electrochemical Society, Inc (ECS). ECS was founded in 1902 as an international nonprofit, educational organization concerned with a broad range of phenomena relating to electrochemical and solid-state science and technology.

The Electrochemical Society has over 9,000 member scientists and engineers in over 75 countries worldwide, as well as roughly 100 corporations and laboratories, which hold corporate membership. ECS objectives are:

- to advance the theory and practice of electrochemistry, solid-state science, and allied subjects;
- to encourage research and dissemination of knowledge in these fields;
- and to assure the availability of adequate training and education of fundamental and applied scientists and engineers in these fields.

First founded as the American Electrochemical Society in 1902, the name of the Society was changed to The Electrochemical Society already in 1930 to more accurately reflect its international activities and membership. Presently, more than 50 % of members reside outside USA. ECS has grown and continued to respond to the changing technical needs and interests of its members; and in 2000, adopted the official acronym of ECS. The tag-line name of ECS is "The society for solid-state and electrochemical science and technology," encompassing its long tradition to advance these fields. ECS continues to disseminate information through individual membership, corporate membership, student services, technical journals, a quarterly news magazine, books, technical meetings, and awards; and to provide networking contacts through ECS thirteen Divisions. Geographically, members can also associate with Sections. For example, most members in Europe are members of the European Section.

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The Society actively recruits students and gives them the opportunity to get organized in student chapters. One of the active chapters is also in Brno, the site of this year's ABAF meeting. Student membership can be obtained at significant discount.

ECS maintains digital library of its collection of publication. Access to the library, with certain number of downloads, is part of the membership benefits package.
Full information can be obtained at www.electrochem.org.

(contributed by Petr Vanýsek, pvanysek@niu.edu)

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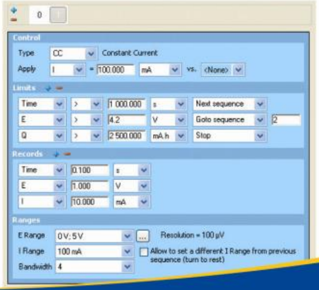

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Control

Type CC Constant Current

Apply I = 100.000 mA vs. <None>

Limits

Time > 1 000.000 s Next sequence

E > 4.2 V Goto sequence 2

Q > 2 500.000 mA.h Stop

Records

Time 0.100 s

E 1.000 V

I 10.000 mA

Ranges

E Range 0 V; 5 V ... Resolution = 100 µV

I Range 100 mA ☐ Allow to set a different I Range from previous sequence (turn to rest)

Bandwidth 4

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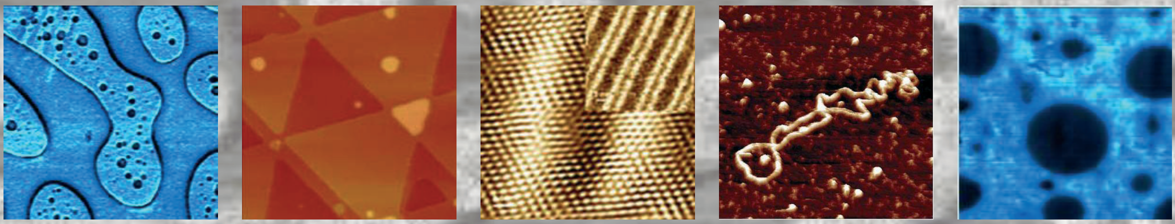

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