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Ni-FOAM MODIFIED BY ‘’DIP AND DRYING’’ METHOD - EFFECT OF TRANSITION METAL SALTS ON THE ELECTROCHEMICAL PROPERTIES

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Abstract: *The paper presents the application of facile one-step “Dip and Drying –DDM” method for deposition of different transition metals (Fe, Co and Ni) on nickel foam (NF) substrates. The obtained modified electrodes are tested as bi-functional electrocatalysts for overall water splitting reaction. The influence of the used metal salts (chloride, sulphate or nitrite) on catalytic efficiency of the modified NF electrodes toward the hydrogen (HER) and oxygen (OER) evolution reactions in 1M KOH is also investigated. The performed electrochemical experiments - cyclic voltammetry, (CV) and low sweep voltammetry, (LSV) - indicate good catalytic performance, originated from the combined effect of electrochemically active constituents, 3D interconnected porosity of the substrate, and its high electrical conductivity, which in turn, resulted in large active surface area available for partial electrode reactions. The electrodes obtained from sulphate baths accelerated both HER and OER, over-performing the rest of the investigated samples and could be considered as potential candidates for application in industrial alkaline electrolysis.*

Keywords: *transition metals, nickel foam, alkaline electrolysis, electrocatalysts*
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1. Introduction

Electrochemical water splitting is commonly recognized as a promising strategy for clean and efficient storage of renewable energy to meet the growing scale-up energy demand of society. [1] Currently, the state-of-art catalysts for hydrogen (HER) and oxygen (OER) evolution reactions proceeding on cathode and anode during electrocatalytic water splitting are platinum group metals (Pt, Ir, and Ru). They are the most efficient catalysts; however, their availability, stability, and cost are serious obstacles for large-scale commercialization [2].

In the recent years, enormous efforts have been devoted to development of efficient and economical OER and HER catalysts based on earth-abundant materials, such as transition metals oxides/hydroxides [3-6], sulfides [7-10], phosphides [11-14], carbides [15,16], selenides [17,18], borates [19], phosphates [20], and alloys [21-25]. The objective is to overcome the limitations of the water splitting process, e. g. the significant overpotentials and slow kinetics and thus, to accelerate the partial electrode reactions. Some of transition metal-based catalysts could be also employed as electrocatalysts with bi-functional activity (in respect both of HER and OER) to thus simplify the design of water splitting system and concurrently reduce the product cost [26].

To find industrial application, the catalysts currently in use should have higher current densities ($\geq$500 mA.cm^{-2}) at low over-potentials ($\leq$ 300 mV) and long-term stability at intensive gas evolution. [27-29].

The point is about recent developments in ‘’self-deposited’’ nanostructured materials on a commercially available 3D Ni foam substrate by “Dip and Drying method’’ (DDM). The NF is stable catalytic support with high internal electrical conductivity (1.43 × 107 S m^{-1} at 20 °C). Its structure allows the catalyst growth in 3D manner and easy escape of the formed gas bubbles during the partial electrode reactions without damaging the electrode surface. The goal of the research presented is the formation

of deposits in the volume of nickel foam with optimized structure, high activity, and low cost and their investigation as potential electrode candidates for both partial reactions of water electrolysis in alkaline media.

1. Experimental

2.1. Chemicals

The nickel foam is obtained from Good Fellow Cambridge LTD and specified as follows: thickness 1.6 mm, density 0.45 g.cm3; 95% pores per centimeter 20; purity 99.5%; sample size 150x150 mm. Prior to catalyst deposition, nickel foam is cut into 1 cm^{-2} pieces and is washed several times with acetone to remove surface and pore contaminations (NF-AC-denoted samples). Then, the cleaned NF-AC pieces are treated with 10% HCl (acid washed NF-AW denoted samples) for 30 minutes in sonificator to increase the active surface area.

The following salts are used as metal precursors: $CoSO_4.7H_2O$ (MERK), $Co(NO_3)_2.6H_2O$ (Valerus), $CoCl_2.6H_2O$ (Reachim), $NisO_4.7H_2O$ (Valerus, BG), $NiCl_2.6H_2O$ (Valerus), $Ni(NO_3)_2.6H_2O$ (Valerus), $FeSO_4.7H_2O$ (Valerus), $FeCl_3.6H_2O$ (Valerus), $FeCl_2.4H_2O$ (MERK) and $FeN_3O_9.9H_2O$ (Alfa Aesar). All aqueous baths used are prepared using distillated water (DI).

2.2. Synthesis of electrocatalysts via DDM on NF

‘’Dip and Drying Method”(DDM) is a novel method for rapid deposition of metal oxide nanoparticles on metal substrates patented recently [30]. The formation of highly active catalyst comprising nanoclusters of γ-FeOOH covalently linked to γ-NiOOH support on NF and obtained from $FeCl_3$ bath via DDM has been reported previously [31]. A schematic diagram of the method is presented in Fig.1.

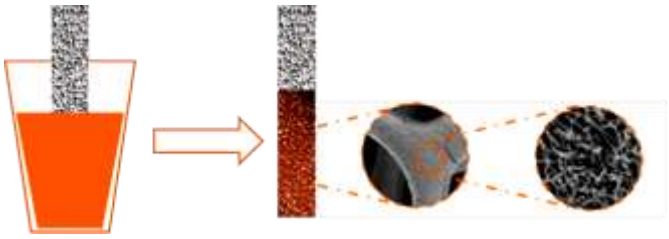

Fig. 1. Schematic diagram of the DDM for preparation of modified nickel foam catalysts

The main advantages of the method are low synthesis temperature, high reproducibility and efficiency, low cost of the used materials.

During the DDM, NF-AW samples are treated in 0.01 M solutions of Co, Fe, and Ni metal salts (nitrates, sulphates and chlorides) for 30 minutes at room temperature. Prepared electrodes are dried in air overnight and then investigated.

2.3. Test procedure

Physical characterization

The surface structure and morphology of the samples are studied by X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The diffraction data are collected using X-ray diffractometer Philips ADP15 with Cu-Kα (λ=1.54178 Å) radiation at a constant rate of $0.2^0.s^{-1}$ on angle range of $2\Theta=10\text{-}90^0$. The diffractograms are given in SI file.

Electrochemical tests

Electrochemical measurements are conducted using Galvanostat/Potentiostat POS 2 (Bank Electronik, Germany) in a three-electrode electrochemical cell with modified NF electrode as working electrode, a reversible hydrogen electrode (RHE) as reference electrode and Pt-wire as counter electrode. The working area of tested electrodes is 1 cm^2. The electrolyte (1 M KOH aqueous solution) is argon saturated during all electrochemical measurements.

The cyclic voltammograms (CV) are recorded in the potential range between

hydrogen and oxygen evolution with scan rate of 100 $mV.s^{-1}$. The OER and HER polarization curves are obtained by linear sweep voltammetry (LSV) with a scan rate of 1 $mV.s^{-1}$. The electrochemical measurements are carried out at room temperature.

3. Results and discussion

The crystalline and phase structures of the deposited films are characterized by XRD analysis. Due to the similarity in the nature of the metals, no difference in the diffractograms of the electrodes obtained by DDM is observed. The presence of other metals or metal oxides is not registered as well, probably due to the low thickness of the obtained deposits which necessitates the use of other methods capable to determine the exact composition of the modified NF electrodes.

Initial information about the electrocatalytic performance of the prepared electrodes is gathered applying cyclic voltammetry and linear sweep voltammetry.

The obtained CV and LSV curves of the bare NF electrodes treated only in acetone (NF-AC) and then acid washed (NF-AW) are presented in Fig. 2.

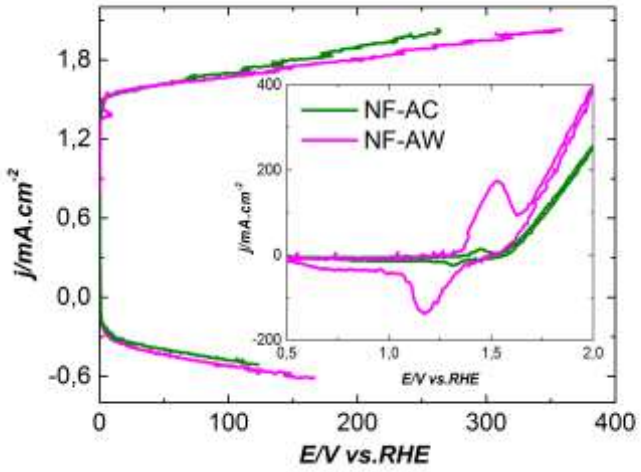

Fig. 2. LSV and CV (inset) of the bare activated NF-AC and NF-AW electrodes in 1M KOH

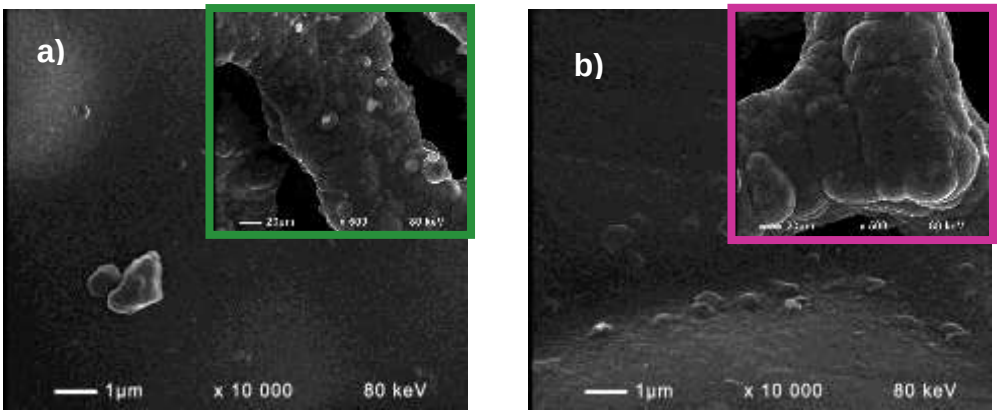

Fig. 3. SEM images of the NF-AC (a) and NF-AW (b) activated electrodes

As can be seen, the treatment in acid (NF-AW) has a strong influence on the nickel foam electrochemical performance. The redox peaks are well depicted –an isolated $Ni^{2+/3+}$ oxidation peak on the CV with a midpoint potential at 1.52 V vs. RHE is observed

followed by OER onset at potential of ~1.63V and a reduction peak at potential ~1.17 V during the reverse potential scanning. Both partial electrode reactions are visibly accelerated.

The observed effects are attributed to increase of the metal roughness during the acid treatment (as seen in the SEM images shown in Fig. 3), resulting in turn, in higher active surface area available for the proceeding electrochemical reactions.

Three series of electrodes are prepared on as-activated NF-AW by DDM varying the type of the used metal salts (chlorides, sulphates, and nitrates). Their CV and LSV curves are compared in Fig. 4-6.

The electrochemical behavior of electrocatalysts deposited from aqueous solutions of nickel salts is compared in Fig. 4. From the CV curves (inset in Fig. 4) a shift of the oxidation peaks to more positive potentials is observed following the order E_{ox} (NF-AW) ≈ E_{ox} ($NiCl_2$) < E_{ox} ($NiNO_3$) < $E_{ox}NisO_4$). The position of the first reduction peak also depends on the type of the nickel salt. The peak potential shifts in negative direction in order as follows: E_{red}(NF-AW) > E_{red}($NiCl_2$) > E_{red}($NiNO_3$) > E_{red}($NisO_4$).

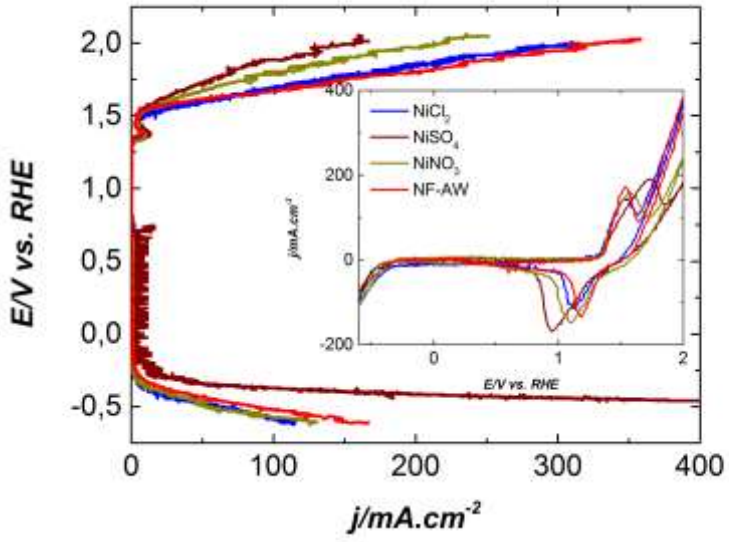

Fig. 4. Anodic and cathodic LSV polarisation curves and CVs (inset) of NF modified with Ni^{2+} from different salts

The registered displacements of the peaks suggest that both electrode reactions (oxygenation of metal surface and reduction of oxygen coverage) are not facilitated on the modified m NF-AC electrodes and proceed faster on the bare NF-AW electrode. The LSV results confirm this suggestion in regard to OER. It is seen that the shift of E_{ox} on CV curve to more positive values corresponds to decrease in OER efficiency in order as follows: NF-AW ≈ $NiCl_2$ > $NiNO_3$ > $NisO_4$. However, for HER the effect of the type of nickel salt is different. The LSVs show a decrease of HER efficiency in case of electrocatalysts deposited from chloride and nitrate baths, while on NF-AW electrodes modified in $NisO_4$ bath the rate of reaction is improved significantly compared to bare NF-AW.

The CV and LSV curves of the electrocatalysts deposited from Fe^{2+}- and Co^{2+}-baths are presented in Fig. 5 and Fig. 6 respectively. The observed effects of the type of metal salt on OER and HER are not much different. On the CV curves similar shifts of both E_{ox} and E_{red} peak potentials are observed. The chemical nature of the anions in the Fe^{2+}-baths does not affect significantly the activity of the electrodes toward both OER and HER.

In contrast, form NF-AW treated in cobalt containing baths more distinct effects are observed. The OER proceeds with higher overvoltage on samples modified in nitrate

solutions. Initially (at low overpotentials), the rate of oxygen evolution on the rest of the samples follows the order: $CoSO_4$ < $CoNO_3$ < $CoCl_2$ < NF-AW, while at higher overvoltages all three electrodes behave identically. At the same time, the efficiency of HER is superior on m NF-AW treated in $CoSO_4$ bath and follows the order $CoCl_2$ < $CoNO_3$ < NF-AW < $CoSO_4$.

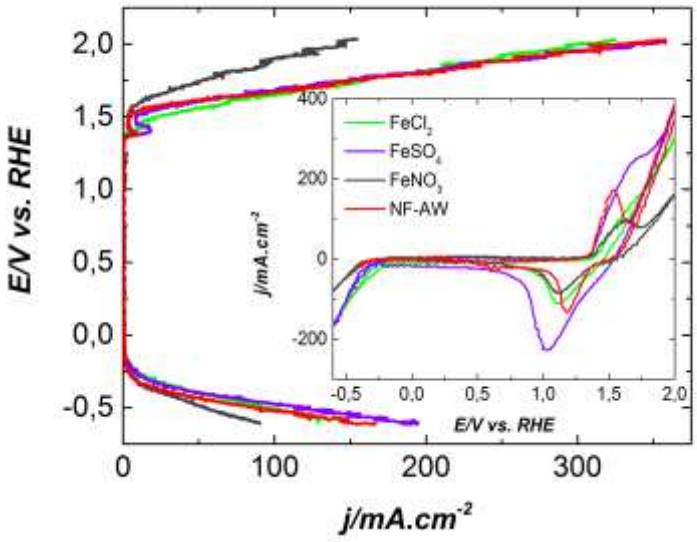

Fig. 5. Anodic and cathodic polarisation curves of NF modified with Fe^{2+} salts, cycling voltammograms (inset) in 1M KOH

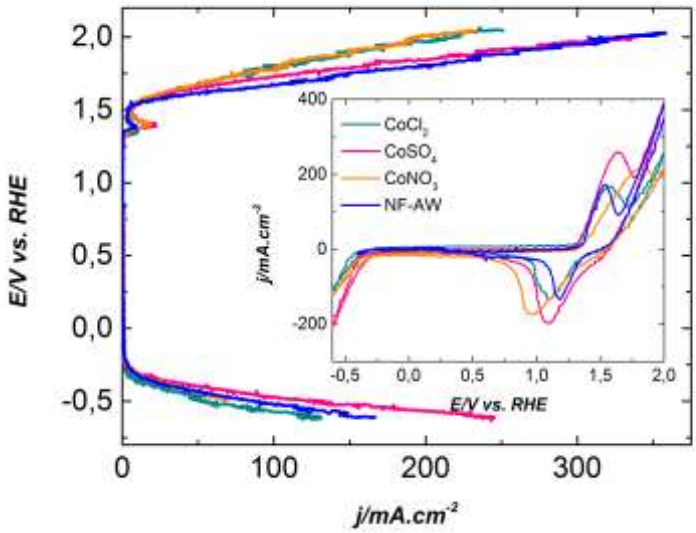

Fig. 6. Anodic and cathodic polarisation curves of NF modified with Co^{2+} salts, cycling voltammograms (inset) in 1M KOH

To distinguish better the effect of the bath (particularly anions type) used to modify the NF surface on electrode performance, in Fig. 7 are compared to the reaction rates (in $mA.cm^{-2}$) for HER (Fig. 7a) and OER (Fig. 7b) at constant potentials of -0.4 V and 1.6 V, respectively.

For all three metals, the chemical nature of the salt in which the substrate is treated, has an effect on electrochemical performance of the electrode. The performance of the samples treated in chloride and nitrate baths is improved in regards to HER, while OER is visibly deteriorated. The most distinct effect is observed for the electrodes deposited form sulphate baths. For these samples, the OER starts a bit later than on NF-AW; however, the rate of the reaction at higher overpotentials does

not change. In contrast, HER is visibly facilitated.

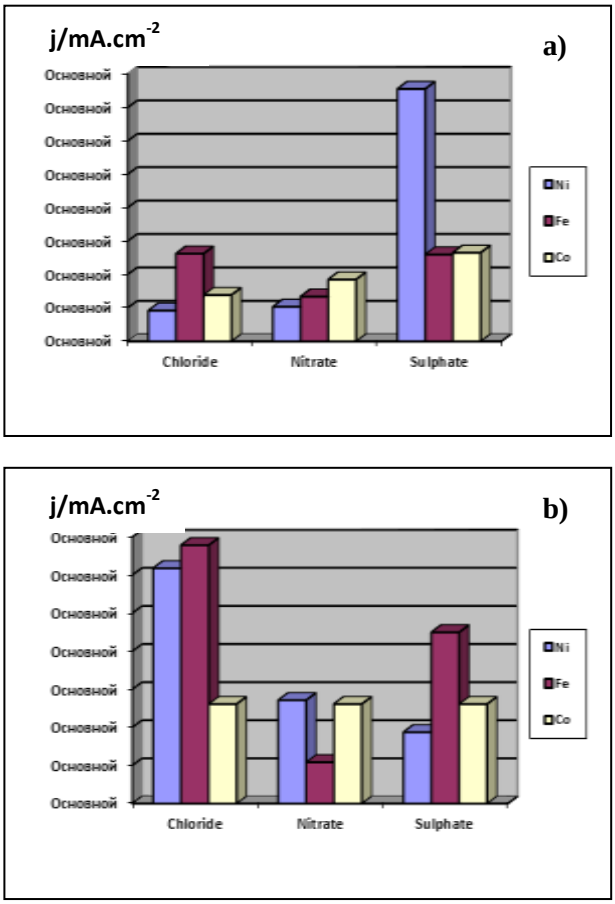

Fig. 7. Comparison of the efficiency of HER and OER on .modified nickel foam (mNF-AW) electrodes in 1M KOH

Conclusions

The research investigates the electrochemical activity of NF electrodes modified by treatment in aqueous solutions of various transition metal (Ni, Fe, Co) salts (chlorides, nitrates, sulphates It found that the direct deposition of nanostructured catalysts via DDM improves the performance of the resulting electrocatalysts providing a higher electrochemically active surface and synergism between the deposit and the conductive substrate. The results reveal that the type of the anions plays an important role in OER and HER performance of electrocatalysts. It confirmed that the straightforward in-situ growth of metal-metal oxide nanostructures (M = Ni, Fe, Co) on the surface of the redox-etched NF-AW foam is as an efficient, easy-fabrication and feasible approach for synthesis of sustainable electrocatalysts for overall water splitting.

Acknowledgments

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"DALDIRMA VƏ QURUTMA" ÜSULU İLƏ KEÇİD METALLARIN DUZLARI İLƏ MODİFİKASİYA OLUNMUŞ NİKEL-KÖPÜK ELEKTRODLARIN ELEKTROKİMYƏVİ XASSƏLƏRİ

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Məqalədə nikel köpük NF (Ni-FOAM) elektrodları üzərinə müxtəlif keçid metallarının (Fe, Co və Ni) çökdürülməsi üçün sadə bir mərhələli "daldırma və qurutma" metodunun (Dip and Drying – DDM) tətbiqindən bəhs edilir. Alınan modifikasiya olunmuş elektrodlar suyun parçalanma reaksiyası prosesində bifunksional elektrokatalizator kimi sınaqdan keçirilmişlər. Həmçinin modifikasiya üçün istifadə olunan metal duzların (xlorid, sulfat və ya nitrit) 1M KOH məhlulunda, hidrogen və oksigenin ayrılma reaksiyalarında elektrodların katalitik effektivliyinə təsiri də araşdırılmışdır. Aparılan elektrokimyəvi tədqiqatlar - tsiklik voltametriya (CV) və kiçik potensialın

dəyişmə sürətinə malik voltametriya (LSV) – göstərir ki, elektrokimyəvi aktiv komponentlərin, elektrodun üçölçülü məsaməliyin, böyük aktiv səthin yaranmasına gətirən yüksək elektrik keçiriciliyinin birgə təsiri nəticəsində yaxşı katalitik xarakteristikalar nümayiş etdirən elektrodlar alınır. Sulfat vannalarından alınan elektrodlar digər tədqiq edilən nümunələrlə müqayisədə həm hidrogenin, həm də oksigenin ayrılma reaksiyalarını sürətləndirdiyi üçün, sənayedə qələvi elektrolizində tətbiq oluna biləcək potensial namizəd sayıla bilər.
Açar sözlər: nikel köpük, keçid metallar, qələvi elektroliz, “daldırma və qurutma” metodu

ЭЛЕКТРОХИМИЧЕСКИЕ СВОЙСТВА ПЕНОНИКЕЛЕВОГО ЭЛЕКТРОДА, МОДИФИЦИРОВАННОГО СОЛЯМИ ПЕРЕХОДНЫХ ЭЛЕМЕНТОВ МЕТОДОМ «ПОГРУЖЕНИЯ И СУШКИ»

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В статье представлено применение простого одностадийного метода «погружение и сушка» (*‘’Dip and Drying’’ method* –DDM» для осаждения различных переходных металлов (Fe, Co и Ni) на подложки из пеноникеля NF (Ni-FOAM). Полученные модифицированные электроды испытаны как бифункциональные электрокатализаторы для реакции расщепления воды. Также исследовано влияние используемых солей металлов (хлорида, сульфата или нитрита) на каталитическую эффективность модифицированных NF электродов по отношению к реакциям выделения водорода и кислорода в 1M KOH. Проведенные электрохимические исследования - циклическая вольтамперометрия (CV) и вольтамперометрия с низкой разверткой (LSV) - свидетельствуют о хороших каталитических характеристиках, обусловленных комбинированным действием электрохимически активных компонентов, трехмерной пористости подложки и ее высокой электропроводности, приводящей к большой активной поверхности, доступной для частных электродных реакций. Электроды, полученные из сульфатных ванн, ускоряли реакцию выделения водорода и кислорода, превосходя остальные исследованные образцы, и могут рассматриваться как потенциальные кандидаты для применения в промышленном щелочном электролизе.
Ключевые слова: пеноникель, переходные металлы, метод погружения и сушки, щелочной электролиз