Aleksandar Petričević[1]*, Susanne Holmin*[2]*, John Gustavsson*[2]*,*
Lena Sundberg[2]*, Erik Zimmerman*[2]*, Vladimir D. Jović*[1,]* *

[1]*Institute for Multidisciplinary Research University of Belgrade, Belgrade, Serbia,* [2]*Permascand, Folkets Husvägen 50, SE-841 99 Ljungaverk, Sweden*

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Additional accelerated service life test for testing Ni-Sn coatings under the conditions of industrial hydrogen production

ABSTRACT

The accelerated service life test (ASLT), developed by PERMASCAND and applied in a specially designed flow cell, is presented for the first time in the literature in this work. This test involves the application of six sequences of very high current densities, followed by zero current density, in 5.0 M NaOH at 50 ^{o}C.The test is performed on Ni-Sn coatings electrodeposited onto Ni mesh, which serves as the substrate for catalytic coatings used by PERMASCAND.The results demonstrate that the overpotential (η) for hydrogen evolution reaction (HER) on several Ni-Sn samples is practically unaffected by the test sequences, remaining approximately -180 mV at j = -1000 mA cm^{-2}. The same test was applied to PERMASCAND's commercial cathode, and the results were compared. Additionally, the Ni-Sn samples were tested in a stationary electrolyte H-cell configuration using polarization curves and electrochemical impedance spectroscopy (EIS) measurements. Nyquist plots recorded at four different values of η were characterized by the presence of two semi-circles, indicating the presence of charge transfer reaction and intermediate adsorption reaction. Comparing polarization curves recorded in PERMASCAND's test cell after the ASLT and in the stationary H-cell after the EIS measurements suggests that long-term performance should be assessed using the ASLT developed by the industry.

Key words: *ASLT, Ni-Sn coatings, hydrogen evolution, alkaline solution, Nyquist plots.*

1. INTRODUCTION

Chlor-alkali electrolysis represents one of the most widely used industrial processes for the production of chlorine and caustic soda [1-6]. Industrial electrolysis relies on the use of "dimensionally stable anodes" (DSA) for chlorine production in the anodic compartment, and noble metal coated cathodes for the HER, actually caustic soda production, in the cathodic compartment. Several large industries in Europe produce chlorine and caustic soda through chlor-alkali electrolysis (Arkema, Boregaard, Covestro, Inovyn Kemira, Kemone, Vynova), each of them using different conditions for this process: different anodes and cathodes, different base materials for applying anodic and cathodic catalysts, different cell constructions (mono-polar, bi-polar, zero-gap configuration), different concentration and temperature of NaOH, different flow rates of electrolyte, different diaphragm (membrane) materials, etc. Regarding anode materials, they are all based on Ti plates or meshes, which are activated by applying mixture of Ti, Ru and Ir, mostly through the thermal decomposition of their salts. This process forms a thin layer of TiO_2, RuO_2 and IrO_2 on the Ti surface. The largest producers of DSA's in Europe are Industry De Nora S.p.A. and PERMASCAND. For cathode materials, a variety of materials are used, both as base materials and catalytic layers. For example, Industry De Nora S.p.A employs cathodes with an electrodeposited thin layer of Ni-RuO_2 coating onto Ni mesh 40, while PERMASCAND's cathodes contain catalytic layer of noble metals on much ticker Ni mesh substrate.

The main components of the chlor-alkali cells used in the industrial processes are summarized in Table 1 [7].

The stability of commercial DSA's in industrial chlor-alkali electrolysis is known to be very high, primarily determined by the thickness of the catalytic coating and is practically insensitive to the eventual interruption in electrolysis [1-6].

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*Corresponding author: Dr. Vladimir D. Jović
E-mail: vladajovic@imsi.bg.ac.rs

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Table 1. Characteristics of Various Chlorine/Sodium Hydroxide Electrolysis Cells

Component	Diaphragm Cell	Mercury Cell	Membrane Cell
Cathode	Steel/steel coating with nickel	Mercury flowing over steel	Steel or nickel with a nickel-based coating
Anode	DSA	DSA	DSA
Diaphragm/ Membrane Material	Asbestos and fibrous polytetrafluoroethylene	None	Ion-exchange membrane (fluorinated polymers)

However, cathodes have been observed to lose their catalytic activity during electrolysis, especially during the replacement of old electrodes with new ones in electrolyzer with zero-gap membrane configuration cells. During such replacements, a phenomenon known as polarity inversion of the electrodes occurs, as anodes and cathodes in the rest of cells within the industrial plant are short-circuited. Depending on the type of cells used in the industrial plant, cathode manufacturers can predict the frequency and duration of reverse polarization and develop an appropriate ASLT, simulating certain period of time of their use in industrial plant. However, it is important to note that different industries apply different conditions for chlor-alkali electrolysis, including variations in cell types, cathode materials, and electrolyte concentration and temperature. As a result, their research and development teams create tailored ASLT's for these unique conditions. Unfortunately, the application of these ASLT's is missing in the literature and in almost all papers describing the behavior of certain catalyst for the HER, the "long-term performance" is typically based on electrolysis durations of 50 – 100 hours in a stationary electrolyte setup [8].

The only ASLT for commercial cathodes, developed by Industry De Nora S.p.A., has been partially disclosed in the literature to date [9]. The procedure involves a sequence of galvanostatic polarizations in the HER range, followed by cyclic voltammetry (CV) at 50 mV s^{-1}, with the HER as the negative limit and oxygen evolution as the positive limit. This specific ASLT, with slight modifications, has been applied in our previous works [10-15] on Ni-Sn and Ni-MoO_x coatings, and the results were compared with those of the commercial Ni-RuO_2 (De Nora's) cathode.

In this work, the ASLT developed by PERMASCAND is presented. Since these two companies are the largest producers of cathodes for chlor-alkali electrolysis, presenting their ASLT's is crucial for researchers analyzing catalysts for the HER in alkaline solution. PERMASCAND's test is based on the application of sequences of very high current densities, followed by zero current density. The test is applied on Ni-Sn coatings electrodeposited under various conditions onto Ni mesh substrate used for the application of catalytic coating in PERMASCAND, as well as on the commercial PERMASCAND's cathode (PSC), with the obtained results being compared.

2. EXPERIMENTAL

2.1. Investigations in a stationary H-cell configuration

P.a. chemicals (Sigma - Aldrich, Merck) and extra pure UV water (Smart2PureUV, TKA) were used for solutions preparation. All potentials are referenced vs. the reversible hydrogen electrode (RHE) as η. Ni mesh was pre-cleaned using the procedure described in our previous work on Ni foams [16].

The electrodeposition of the Ni-Sn coating for testing in PERMASCAND was carried out galvanostatically at two current densities using two different solutions: solution A (0.60 M$K_4P_2O_7$ + 0.30 M Glycine + 0.10 M $NiCl_2$ + 0.03 M $SnCl_2$, j = -80 mA cm^{-2}, t = 1250 s) and solution B (0.60 M $K_4P_2O_7$ + 0.30 M Glycine + 0.20 M $NiCl_2$ + 0.06 M $SnCl_2$, j = -100 mA cm^{-2}, t = 1000 s). Two types of Ni mesh substrates were used: sand-blasted and non-blasted. Two larger counter electrodes (Pt-Ti expanded meshes) were positioned in parallel with the working electrode.

Polarization curves and EIS measurements were performed in a H-cell with stationary electrolyte, where the working and counter electrode compartments were separated by a Zirfon^{TM} Perl UTP 500 membrane. A reversible hydrogen electrode (RHE) – HydroFlex (Gaskatel GmbH), connected to the working electrode compartment via bridge and a Luggin capillary, served as the reference electrode. Potentiostat Interface 1010E (Gamry Instruments Inc.) was used for polarization curves and EIS measurements. All measurements were carried out in 5.0 M NaOH at a temperature of 50 ^oC.

Nyquist plots for all investigated electrodes were recorded at four different η values (-60 mV, -75 mV, -90 mV and -105 mV) within the frequency range of 20 kHz to 0.01 Hz, with 20 points per decade and an amplitude of 5 mV RMS. Prior to each measurement, the electrodes were held at the desired η for 100 s to establish a stable current density response. The current densities and solution resistance (R_s) values obtained from EIS measurements were used to calculate the real η_{corr} by correcting the applied η values for jR_s.

2.2. Investigations in a PERMASCAND's test cell

The HER on larger samples was tested in a specially developed PERMASCAND's test flow cell. The working electrodes for the test cell were larger (3.0 x 3.5 cm), while those used for polarization curves and EIS measurements in the H-cell were smaller (1.0 x 1.0 cm). For the testing procedure in PERMASCAND, the following equipment was used: potentiostat/galvanostat Iviumstat.XRi (Ivium) for electrochemical measurements; the electrolyte was heated in a custom-built tank and pumping station; the tank had a volume of 100 liters, and pumipng was achieved by a centrifugal pump. Sodium hydroxide (Analytical reagent grade, Fischer Scientific) was used in the PERMASCAND's test flow cell.

2.3. ASLT procedure developed by PERMASCAND

The ASLT procedure, developed by PERMASCAND and performed in their laboratory, contained 7 steps schematically presented in Figure 1. The procedure begins by recording a polarization curve (corrected for ohmic drop) from -1.0 A cm^{-2} to -0.1 A cm^{-2} and back. The first step, lasting for 1 hour is applied as follows: j = -3.0 A cm^{-2} for 54 s followed by j = 0.0 A cm^{-2} for 6 s (repeated 60 times). Before the second step, another polarization curve was recorded, and this process continues until the 6th step, after which the final 7th polarization curve is recorded.

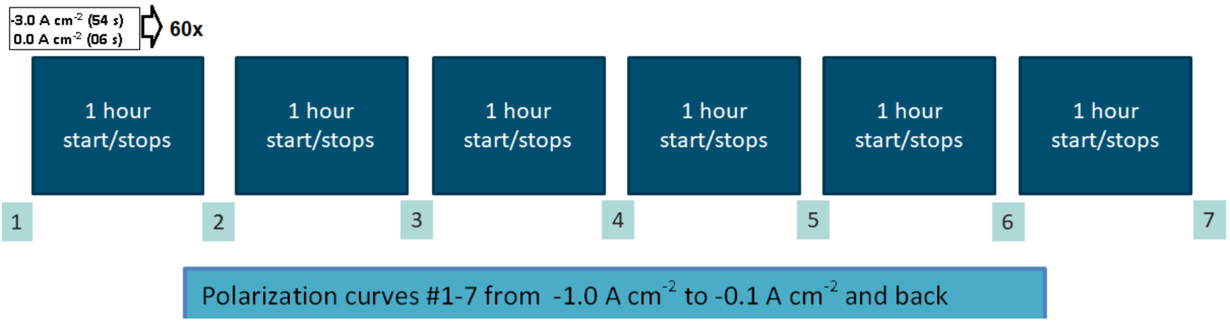

Figure 1. Schematic presentation of the ASLT procedure developed by PERMASCAND

Coating amount for Ni-Sn samples was measured before and after electrolysis with X-ray Fluorescence (NITON XL3t handheld instrument from Thermo Scientific) three times on each sample in electrolyzed spot.

3. RESULTS AND DISCUSSION

3.1. Results of the test procedure developed by PERMASCAND

A total of five Ni-Sn coatings were tested in the PERMASCAND test cell, alongside a Ni blasted mesh and a commercial PERMASCAND cathode (PSC) under the standardized procedure. The coatings were prepared under varying conditions, as detailed below:

Ni-Sn(1) – electrodeposited onto Ni blasted mesh from solution A at j = -80 mA cm^{-2} for 1250 s;

Ni-Sn(2) – electrodeposited onto Ni blasted mesh from solution B at j = -100 mA cm^{-2} for 1000 s;

Ni-Sn(3) – electrodeposited onto Ni mesh from solution A at j = -80 mA cm^{-2} for 1250 s;

Ni-Sn(4) – electrodeposited onto Ni mesh from solution B at j = -100 mA cm^{-2} for 1000 s;

Ni-Sn(5) – electrodeposited onto Ni blasted mesh from solution B at j = -100 mA cm^{-2} for 1000 s.

The corresponding U vs. t dependences for the electrodeposition of these samples are presented in Figure 2. All U vs. t dependences exhibit well-defined voltage plateaus, with differences in voltage values due to different current densities of electrodeposition.

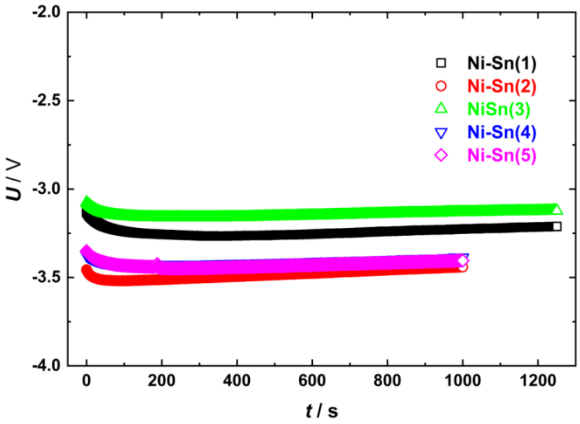

Figure 2. U vs. t dependences for the electrodeposition of all Ni-Sn tested samples

Polarization curves corrected for the ohmic drop (η_{corr}) obtained prior to the test (1) and after each subsequent step (2-7) are presented in Figure 3, as follows: (a) Blasted Ni mesh; (b) Commercial PSC cathode; (c) Ni-Sn(3) sample; (d) Ni-Sn(4) sample; (e) Summary of polarization curves corrected for the ohmic drop (η_{corr}) recorded after all test steps (2-7).

Investigated cathodes exhibited varying behavior during the test. Blasted Ni mesh (Figure 3(a)) showed clear signs of deactivation, with the η_{corr} values in the final polarization curve (7) approximately 130 mV higher than those recorded before the test (1). Commercial PERMASCAND's cathode (PSC) demonstrated activation during the test (Figure 3(b)), particularly during the first step, while Ni-Sn(3) and Ni-Sn(4) coatings (Figure 3(c) and (d) respectively) exhibited only minor sensitivity to the applied stress steps, suggesting relatively stable performance.

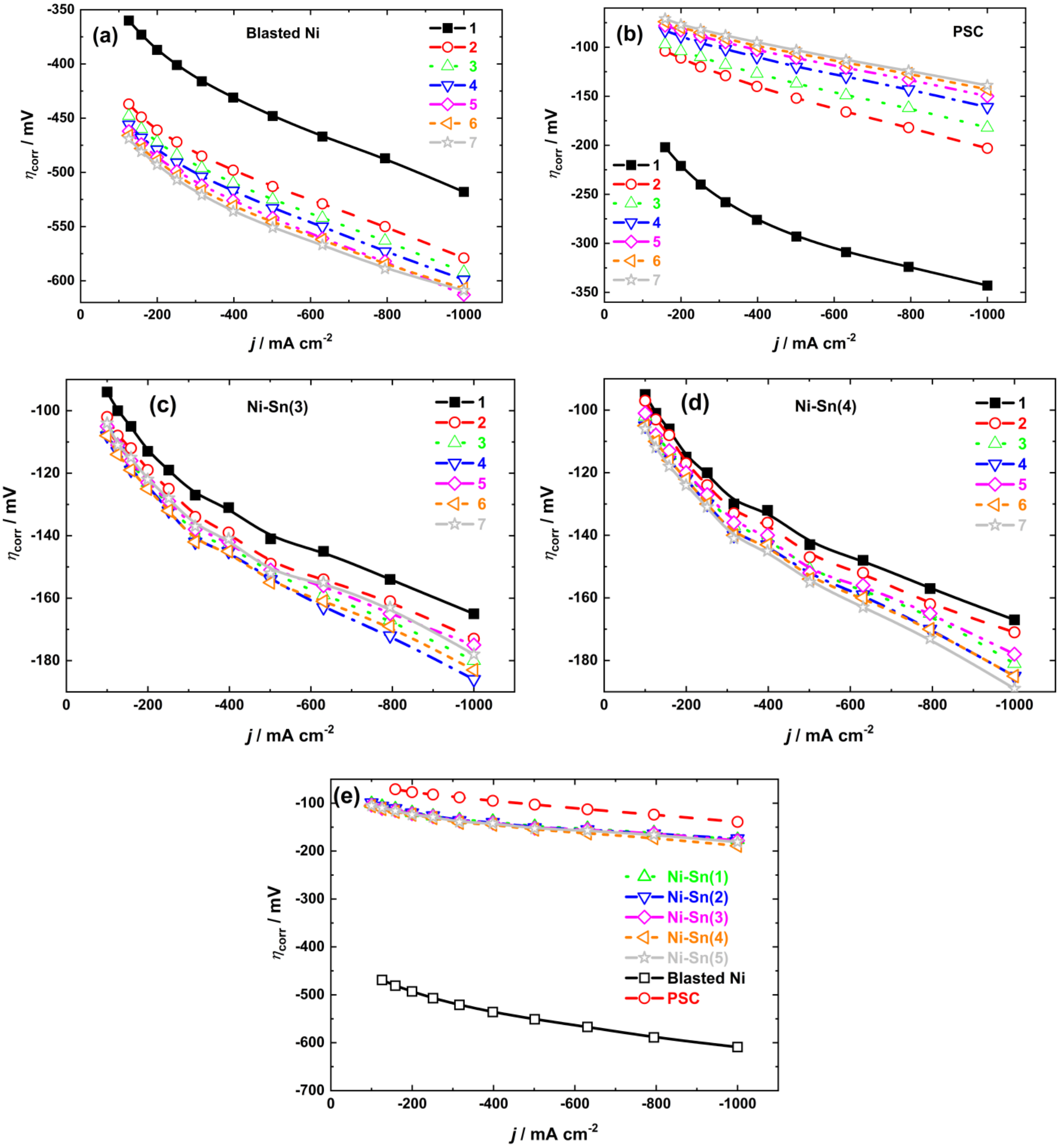

Figure 3. Polarization curves corrected for the ohmic drop (η_{corr}) obtained before the test (1) and after each subsequent step (2-7): (a) For blasted Ni mesh; (b) For commercial PSC cathode; (c) For Ni-Sn(3) coating; (d) For Ni-Sn(4) coating; (e) Comparison of the final polarization curves no. 7 for all investigated samples

As can be seen the lowest η_{corr} values recorded after the test (polarization curve 7) were obtained for the commercial PSC cathode, amounting approximately -140 mV (Figure 3(b)). In contrast, the blasted Ni mesh exhibited significantly higher overpotentials, with η_{corr} values about 500 mV higher. All Ni-Sn coatings demonstrated almost identical polarization characteristics, with a maximum variation in η_{corr} values of only about 10 mV. Among them, the characteristics of Ni-Sn(3) and Ni-Sn(4) are shown in Figures 3(c and d) respectively. An interesting observation was made for the Ni-Sn(3) sample (Figure 3(c)), where η_{corr} values increased during steps 2,3 and 4, followed by decrease through the steps 5 to 7.

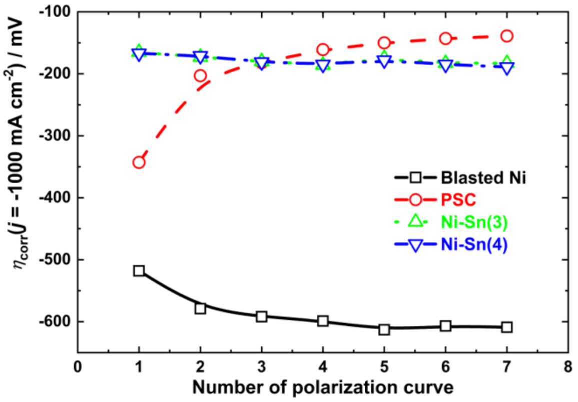

Figure 4. Overpotentials corrected for the ohmic drop (η_{corr}) recorded at $j = -1000\ mA\ cm^{-2}$ as a function of the polarization curve number

This trend is more clearly illustrated in Figure 4, which shows the values of η corrected for the ohmic drop recorded at the highest applied current density ($\eta_{corr}(j = -1000\ mA\ cm^{-2})$) as a function of the polarization curve number.

An interesting observation is that the $\eta_{corr}(j = -1.0\ A\ cm^{-2})$ values for the Ni-Sn coatings remain practically unaffected by the applied test procedure. This suggests that the alternating steps of high and zero current densities do not induce any significant changes in the coating composition. In contrast, the Ni blasted mesh exhibited a noticeable increase in η_{corr} over the course of the test, with values rising by approximately 130 mV from step 1 to step 6 (point 7). The PSC cathode showed a clear activation trend, with the $\eta_{corr}(j = -1.0\ A\ cm^{-2})$ values decreasing sharply during the step 1 from -330 mV to -190 mV and then decreasing slowly to ~ -140 mV at the end of test. Such behavior could be explained by the change of coating composition during the first step producing surface more active towards the HER.

The Sn content in the coatings, measured by X-ray florescence (XRF) at the spot of analysis before and after the test, is presented in Figure 5. As can be seen, the loss of Sn is practically negligible. Based on the findings from our previous study [16], the small amount of Sn that dissolved during the ASLT procedure likely corresponds to the fraction of Sn in the electrodeposit that exist either as free Sn or is incorporated in secondary phases present in the Ni-Sn alloy (Ni_3Sn, Ni_3Sn_4) [17]. This is consistent with the fact that the dominant phase in the coating is Ni_3Sn_2. According to Ref. [16], Ni_3Sn_2 phase is primarily responsible for the high catalytic activity of Ni-Sn coatings towards the HER.

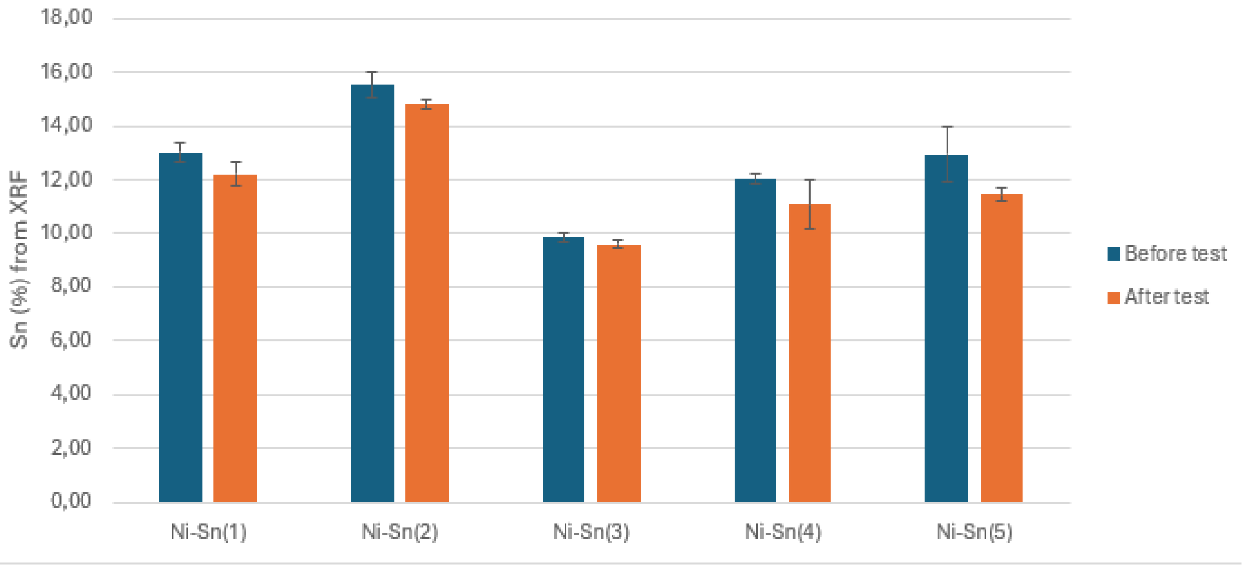

Figure 5. XRF at the spot of analysis before and after the test

3.2. Results obtained in the stationary H-cell configuration

Before and after the EIS measurements, polarization curves were recorded using linear sweep voltammetry (LSV) at a sweep rate of 1 mV s^{-1}, in combination with the current interrupt technique. Figure 6 shows the polarization curves obtained before and after EIS measurements for sample Ni-Sn(3) (a), and sample Ni-Sn(4) (b). Given that the EIS measurements lasted 4 h, a slight change in the polarization curves is to be expected. Interestingly, sample Ni-Sn(3) exhibited slightly lower η_{corr} values after the EIS measurements (a), while sample Ni-Sn(4) showed a slight increase in η_{corr} values after the EIS measurements (b).

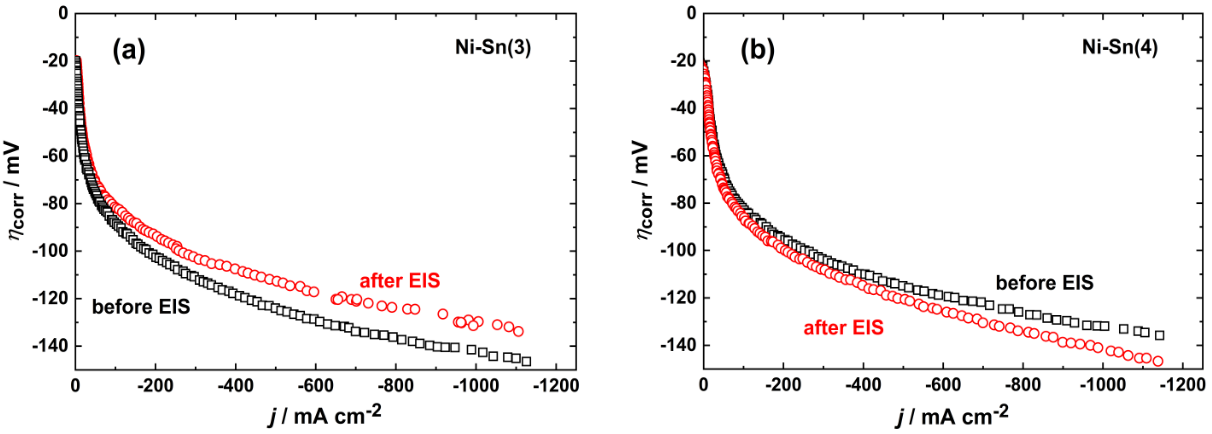

Figure 6. Polarization curves for HER at sample Ni-Sn(3) (a) and sample Ni-Sn(4) recorded before and after the EIS measurements

Nyquist plots recorded at four different values of η are shown in Figure 7. All plots are characterized by the presence of two semi-circles: the first one (at high frequencies) corresponding to the charge transfer reaction, and the second one corresponding to the intermediate adsorption reaction. As expected, an increase in η leads to the smaller semi-circles, reflecting higher values of j and therefore faster kinetics of the HER.

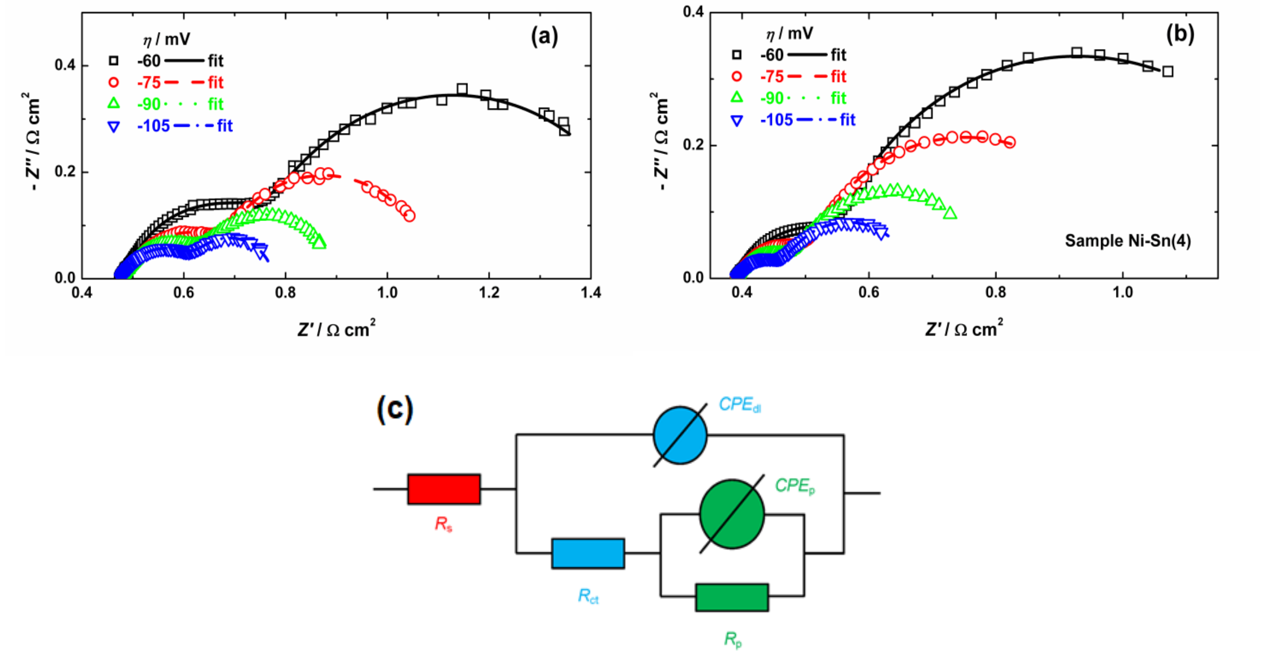

Figure 7. Nyquist plots recorded at different η values (designated in the figure) for Ni-Sn(3) sample (a) and Ni-Sn(4) sample (b). Squares, circles and triangles represent the experimental data points, while the lines correspond to the fitting results obtained using the equivalent circuit shown in Figure 7(c)

R_s – solution resistance; R_{ct} – charge transfer resistance; CPE_{dl} – constant phase element corresponding to the double layer capacitance; R_p – resistance of the electrosorption/ electrodesorption of intermediate; CPE_p – constant phase element corresponding to the adsorption pseudo capacitance.

As reported in Ref. [15], the exchange current densities for the charge transfer reaction ($j_{o,ct}$), intermediate adsorption reaction ($j_{o,ads}$) and overall (total) HER ($j_{o,tot}$) can be determined by analyzing dependences η_{corr} vs. log(R_{ct}^{-1}), η_{corr} vs. log(R_p^{-1}) and η_{corr} vs. log(R_F^{-1}), specifically from the intercepts of those linear dependences at $\eta_{corr.} = 0$. Similarly, the transition time τ_o at the reversible potential for HER can be obtained from the intercept of the log(τ) vs. η_{corr} linear dependence at $\eta_{corr.} = 0$. All respective dependences for samples Ni-Sn(3) and Ni-Sn(4) are presented in Figure 8, with the corresponding values of $j_{o,ct}$, $j_{o,ads}$, $j_{o,tot}$ and τ_o indicated in the figure. It can be observed that the slopes of η_{corr} vs. log(R_{ct}^{-1}), η_{corr} vs. log(R_p^{-1}) and η_{corr} vs. log(R_F^{-1}) dependences are practically identical for both samples, except for that of η_{corr} vs. log(R_p^{-1}) dependence of sample NiSn(4) (Figure 8(b)). Although the values of $j_{o,ct}$ and $j_{o,ads}$ are different, the linear η_{corr} vs. log(R_F^{-1}) dependences for both samples are identical, resulting in the same $j_{o,tot}$ of -1.57 mA cm^{-2}. This indicates that the charge transfer and intermediate adsorption steps are sensitive to the composition (or morphology) of the Ni-Sn coatings, while the overall HER is identical for both samples being in accordance with the polarization curves.

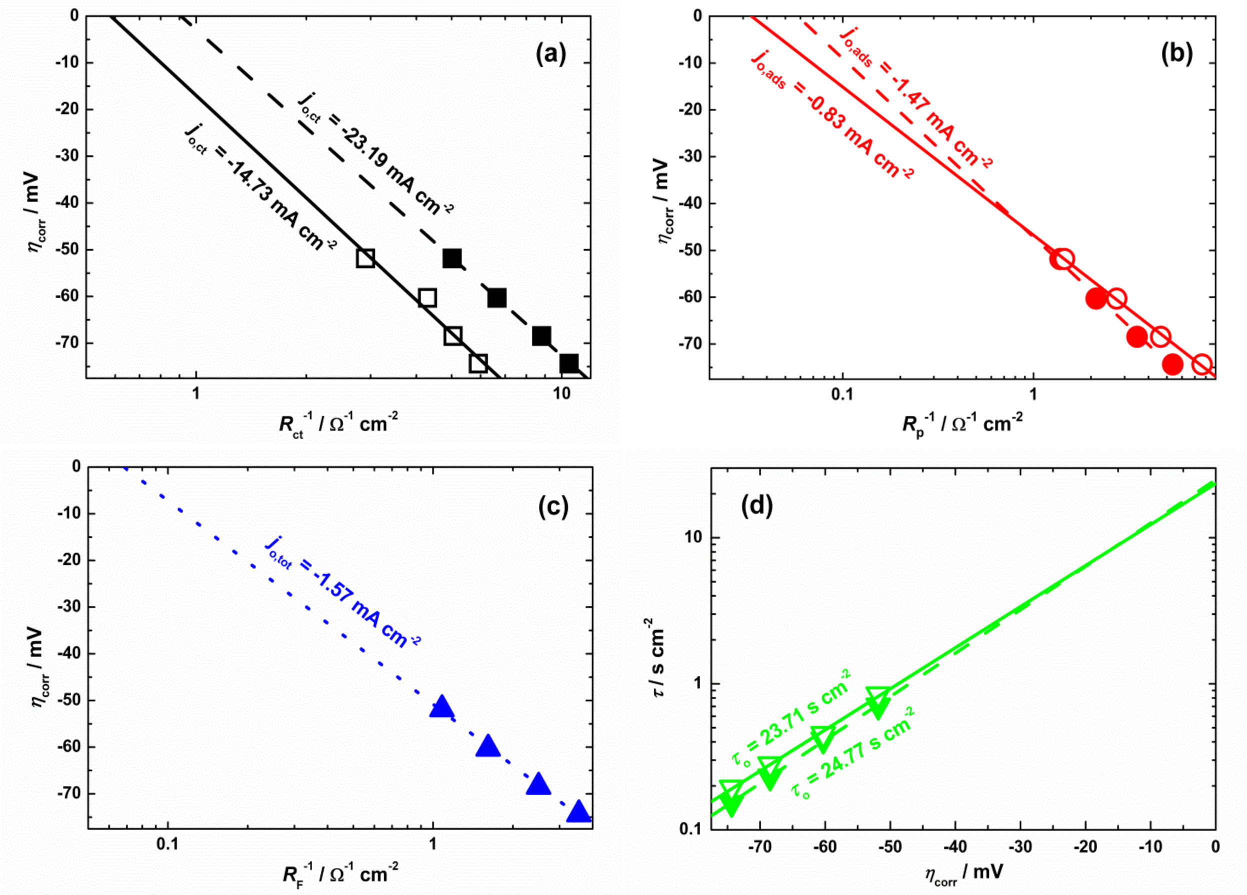

Figure 8. (a) The dependences η_{corr} vs. log(R_{ct}^{-1}), (b) dependences η_{corr} vs. log(R_p^{-1}), (c) dependences η_{corr} vs. log(R_F^{-1}) and (d) dependences log(τ) vs. η_{corr} for samples Ni-Sn(3) (□○△ ▽) and Ni-Sn(4) (■●▲ ▼)

3.3. Comparison of the results obtained in the flow test cell and in the stationary H-cell

Figure 9 compares the polarization curves for samples Ni-Sn(3) and Ni-Sn(4), recorded both in the PERMASCAND's flow cell and in a stationary H-cell. It should be emphasized that the samples tested in the flow cell were analyzed 10 months after electrodeposition, while the samples tested in the H-cell were investigated immediately after the electrodeposition.

Considering presented polarization curves, it can be concluded that the samples tested in the stationary electrolyte exhibit slightly better performance, with lower η_{corr} values across the entire current density range. Of course, comparison of presented polarization curves is not realistic, since those recorded in a stationary electrolyte were not exposed to the 6 steps of PERMASCAND's test procedure. Hence, it should be stressed that electrodes, even if smaller than those used in full-scale industrial plants, must be tested under conditions simulating real industrial process, within a smaller pilot plant.

As previously stated [8], the commonly accepted "long-term test" used in the literature to evaluate the catalytic behavior of electrodes for the HER is useless and this statement is confirmed by the results presented in Figure 9.

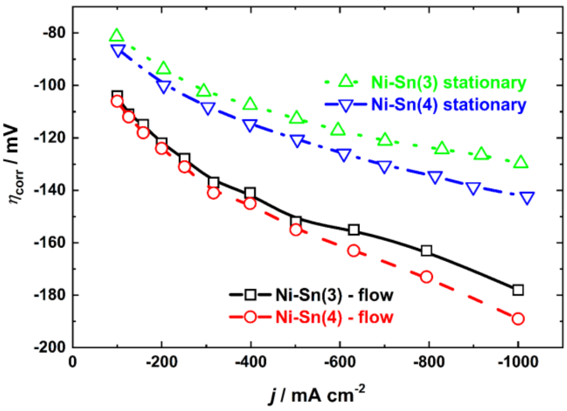

Figure 9. Polarization curves for the HER at Ni-Sn(3) sample and Ni-Sn(4) sample recorded in the PERMASCAND's test flow cell and in stationary H-cell

4. CONCLUSIONS

1. The ASLT procedure developed by PREMASCAND and applied in a specially designed flow cell using 5.0 M NaOH at 50 °C on Ni-Sn coatings electrodeposited onto Ni mesh, used as substrate for applying catalytic coatings in PERMASCAND, has been presented in this work for the first time in the literature.

2. The η_{corr} values for the HER on several Ni-Sn samples were found to be practically insensitive to the test sequences, maintaining values around -180 mV at j = -1000 mA cm^{-2}.

3. The commercial PERMASCAND's cathode exhibited 30-40 mV lower η_{corr} values at the highest current density after the test.

4. Nyquist plots recorded at four different η_{corr} values were characterized by the presence of two semi-circles, indicating the presence of charge transfer reaction and intermediate (H_{ads}) adsorption process.

5. Comparison of the polarization curves recorded in the PERMASCAND's test cell and the stationary H-cell confirmed that long-term electrode performance should be tested using industrial-grade ASLT procedures.

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IZVOD

DODATNI UBRZANI TEST ZA ISPITIVANJE Ni-Sn PREVLAKA U USLOVIMA INDUSTRIJSKE PROIZVODNJE VODONIKA

Prvi put u literaturi prikazan je ubrzani test za ispitivanje Ni-Sn prevlaka u uslovima industrijske proizvodnje vodonika razvijen u kompaniji PERMASCAND. Test je uradjen u specijalno dizajniranoj aparaturi koja simulira uslove rada u hlor-alkalnoj elektrolizi i bazira se na primeni šest sekvenci. Svaka sekvenca traje 1 sar i za to vreme se 60 puta ponavljaju j = - 3000 mA cm^{-2} (54 s) i j = 0 mA cm^{-2}(6 s). Kao elektrolit koristi se 0.5 M NaOH zagrejan na 50 °C. Testirane elektrode su bile prevlake od Ni-Sn legure istaložene na Ni mrežici koja se koristi kao osnova za nanošenje komercijalnih prevlaka u kompaniji PERMASCAND. Pokazano je da je prenapetost korigovana za omski pad napona (η_{corr}) na svim testiranim elektrodama bila praktično nezavisna od broja sekvence ($\eta_{corr} \approx$ -180 mV pri j = -1000mA cm^{-2}). Test je primenjen i na komercijalne PERMASCAND katode i rezultati su uporedjeni, pri čemu su Ni-Sn katode imale znatno bolje karakteristike u prvoj sekvenci, dok su u preostalim sekvencama komercijalne katode posedovale manju prenapetost za 30 - 40 mV. Ni-Sn katode su ispitivane i u stacionarnom elektrolitu (H – ćelija) merenjem polarizacionih krivih i impedansom. Nyquist-ovi dijagrami su bili okarakterisani prisustvom dva polukruga, ukazujući na prisustvo reakcije razmene naelektrisanja i adsorpcije intermedijara (H_{ads}). Polarizacione krive registrovane u PERMASCAND-ovoj test ćeliji nakon završenog testa i u H – ćeliji su uporedjene i potvrdjeno je da se tzv. "dugovremenski test" mora raditi pod uslovima koji su definisani od strane industrijskih razvojnih instituta.

***Ključne reči:** Ubrzani test, Ni-Sn prevlake, izdvajanje vodonika, alkalni rastvori, Nyquist-ovi dijagrami.*

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Aleksandar Petričević https://orcid.org/0000-0001-5954-3569
Erik Zimmerman https://orcid.org/0000-0002-5230-3682
