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ELECTROCHEMICAL ACTIVITY AND USE OF BIMETALLIC CATALYTIC STRUCTURES WITH DIFFERENT NI CONTENTS

Abstract. This paper presents the results of a study of the effect of nickel particles on the properties of a bimetallic PtNi catalyst for membrane-electrode assemblies in hydrogen fuel cells. A series of samples with controlled Ni particles ranging in size from 6.0 to 15 nm were synthesized using magnetron sputtering with process time ranging from 30 to 120 s. It was found that fluxes obtained with sputtering times of 60–90 s (Ni particle size ranging from 6 to 8 nm) provide a balanced combination of differential characteristics: developed dendritic structure, active electrochemical surface area, catalytic activity and stability during stress testing. It is shown that deviations from the optimal synthesis parameters, either in the direction or by increasing the sputtering time, lead to degradation of either the catalytic activity or the stability of the system.

Keywords: catalyst, electrochemical activity, ion-exchange membrane, hydrogen fuel cell.

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ЭЛЕКТРОХИМИЧЕСКАЯ АКТИВНОСТЬ И ДОЛГОВЕЧНОСТЬ БИМЕТАЛЛИЧЕСКИХ КАТАЛИТИЧЕСКИХ СТРУКТУР С РАЗЛИЧНЫМ КОЛИЧЕСТВОМ NI

Аннотация. В работе представлены результаты исследования влияния частиц никеля на свойства биметаллического катализатора PtNi для мембранно-электродных блоков водородных топливных элементов. Методом магнетронного распыления с варьированием времени процесса от 30 до 120 с синтезирована серия образцов с контролируемым размером частиц Ni от 6,0 до 15 нм. Установлено, что образцы, полученные при времени напыления 60–90 с (размер частиц Ni 6–8 нм), демонстрируют сбалансированное сочетание функциональных характеристик: развитую дендритную структуру, максимальную электрохимически активную поверхность, высокую каталитическую активность и устойчивость в ходе стресс-тестирования. Показано, что отклонение от оптимальных параметров синтеза в сторону как уменьшения, так и увеличения времени напыления приводит к деградации либо каталитической активности, либо стабильности системы.

Ключевые слова: катализатор, электрохимическая активность, ионообменная мембрана, водородный топливный элемент.

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Introduction. Hydrogen energy is a key area of development in the field of alternative and environmentally friendly sustainability [1, 2]. The core of a hydrogen fuel cell is a polymer ion-exchange membrane (IEM), the efficiency of which directly depends on the manufacturer of the catalytic material [3]. Of particular interest in this regard are bimetallic Pt-M (M is a transition metal) nanoparticles, which not only outperform pure platinum in catalytic activity in the respiration recovery mode but also exhibit increased resistance to structural corrosion and degradation [4, 5]. This makes them promising candidates for use in low-temperature fuel cells [6–8].

Previous studies have shown that optimizing the thickness of the catalytic layer is crucial for achieving high performance, productivity and efficiency of membrane-electrode assemblies (MEAs) [9, 10]. However, the effect of the thickness of PtNi catalytic layers formed by a two-stage method, including magnetron sputtering of nickel and subsequent chemical deposition of platinum, has not been sufficiently studied. The thickness of the nickel layer used as an adsorption site for platinum deposition can affect the properties of the entire PtNi catalytic layer [11, 12].

The aim of this study is to optimize the composition and structure of PtNi catalytic structures to improve the efficiency and stability of MEAs intended for use in hydrogen fuel cells.

Materials and methods

A commercial IEM, Nafion 324 (DuPont), was used as the polymer IEM. This membrane belongs to the class of perfluorinated sulfonic acid polymers, the structural basis of which consists of a copolymer of tetrafluoroethylene and perfluorinated vinyl ether with the latter sulfonate compounds ($-SO_3H$) [13]. The presence of these groups is a determining factor, as they are responsible for the proton conductivity of the material – a key property that determines its use as an electrolyte in fuel cells (Fig. 1) [14].

The formation of PtNi catalytic structures occurred in two stages. In the first stage, nickel particles were detected directly on the polymer IEM surface using magnetron sputtering. The residual pressure in the chamber before gas release was 10^{-3} Pa, which resulted in accidental interactions between nickel, oxygen, and nitrogen during the deposition process. Next, Ar working gas was supplied to the chamber at a flow rate of 0.5 l/h. The nickel target was sputtered at a power of 0.150 kW and a current of 1 A, with process times ranging from 30 to 120 s. Scanning electron microscope micrographs are shown in Fig. 2.

The average results of measuring the distribution and size of nickel particles on the surface of the IEM are shown in Fig. 3 and Table 1.

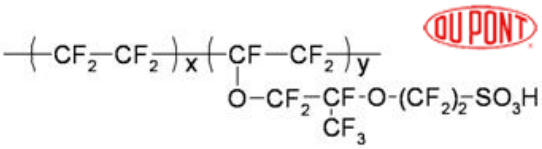

Fig. 1. Nafion perfluorinated polymeric sulfonic acid chain

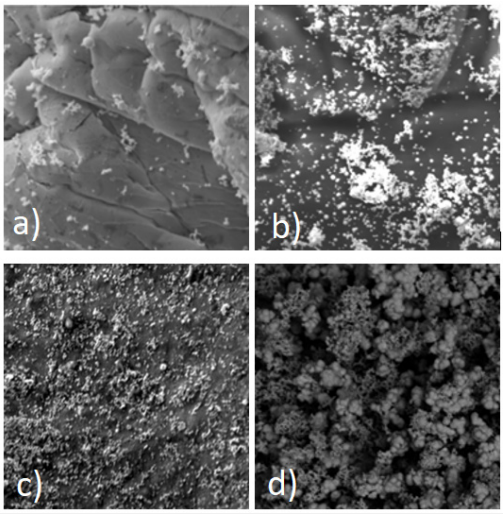

Fig. 2. Catalytic structures with different Ni deposition times: a) 30 s; b) 60 s; c) 90 s; d) 120 s

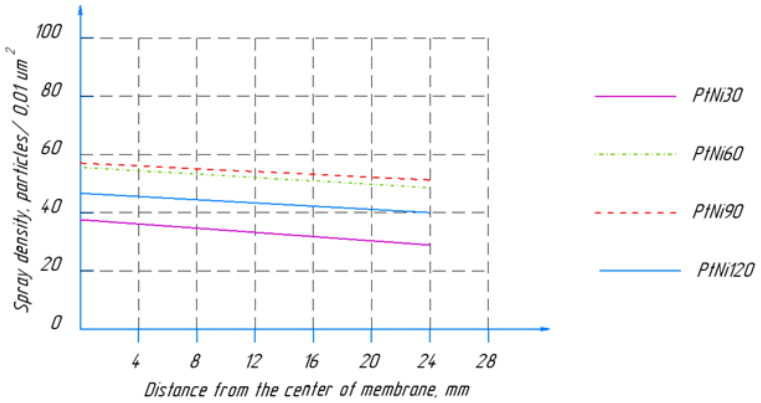

Fig. 3. Sputtering density distribution graph

Table 1

Average particle size of deposited Ni particles by magnetron sputtering

Sample	Average particle size, nm
PtNi30	6.0
PtNi60	9.0
PtNi90	11.8
PtNi120	14.9

In the second stage, a platinum shell was formed by chemical deposition from a solution. The reaction process is shown in the following formulas:

$$H_2PtCl_6 + 2NH_4OH \rightarrow (NH_4)_2 PtCl_6 + 2H_2O; \quad (1)$$

$$N_2H_4 + 4NH_3 \rightarrow 4NH_4^+ + 4e^- + N_2; \quad (2)$$

$$(NH_4)_2 PtCl_6 + 4e^- \rightarrow 2NH_4Cl + Pt + 4Cl^-. \quad (3)$$

The main component of the solution is hexachloroplatinic acid (H_2PtCl_6), whose minimum concentration (0.8 g/L) ensures uniform coating with a long precipitation time (approximately 45 min). Hydrazine (N_2H_4), used in a concentration range of 0.8 to 1.2 g/L, plays a key role in the reduction of platinum from H_2PtCl_6. The addition of concentrated ammonium hydroxide (NH_4OH) in an amount of up to 200 ml/L stabilizes the chemical reaction, ensuring a uniform process. To prepare the IEM for coating, it is pre-soaked in distilled water for 6–12 h at a temperature of 20–22 °C. This step saturates the membrane with water, activating its surface and creating optimal conditions for the formation of a high-quality coating during chemical vapor deposition [15].

Results and discussion

The resulting polymer IEMs were integrated into MEAs to record current-voltage characteristics (Fig. 4), followed by stress testing. Tests were conducted in fuel cell mode in potentiostatic mode in the range from 0.1 to 0.9 V with a sweep rate of 20 mV s^{-1}, with a total number of cycles of 5000. Fig. 5 shows the dynamics of current density changes at a potential of 0.9 V.

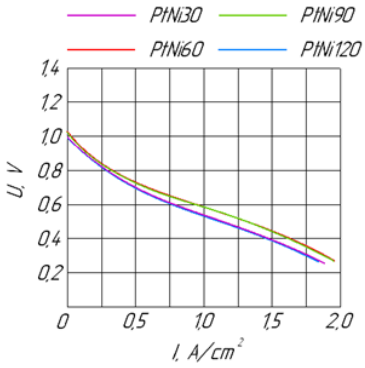

Fig. 4. Comparison of the I–V characteristics of the MEA in a hydrogen fuel cell

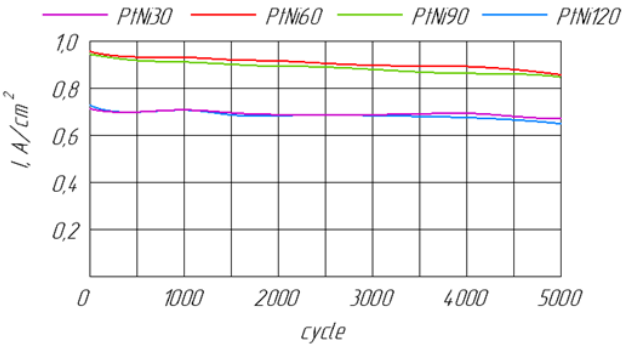

Fig. 5. Graph of current density changes in the MEA over time for polymer IEMs with different Ni amounts

Analysis of the surface morphology and functional characteristics of the MEA reveals that short deposition times result in an insufficient number of nucleation centers [12]. This limits the development of the electrochemically active surface area, which in turn explains the observed decrease in current density despite the high stability of the samples. The superior stability compared to samples obtained with sputtering times of 60 to 90 s is likely due to the increased average distance between particles, which suppresses sintering mechanisms. This assumption is supported by the results of particle size distribution analysis before and after stress testing (Fig. 6).

In contrast, excessively long sputtering times lead to the formation of large agglomerates. Although proton conductivity is retained in such systems, the significant reduction in electrochemically active surface area negatively impacts the shape of the I–V characteristics and the overall membrane performance.

The highest efficiency was demonstrated by structures formed with sputtering times of 60 to 90 s, which corresponded to nickel particles of 6–8 nm in size. These synthesis conditions allowed us to obtain catalysts that combine high electrochemical activity with sufficient corrosion and morphological stability.

Conclusion

The study established a fundamental relationship between the dispersion of the nickel substrate formed by magnetron sputtering and the functional characteristics of the bimetallic PtNi catalyst within the MEA.

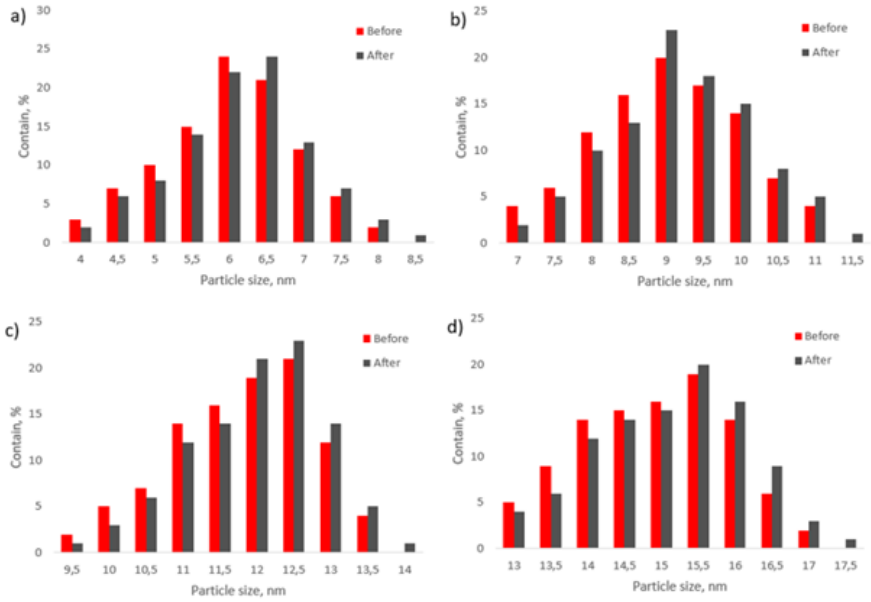

Fig. 6. Nanoparticle size distribution before and after stress testing for polymer IEM with Ni sputtering times of: a) 30 s; b) 60 s; c) 90 s; d) 120 s

The highest efficiency was demonstrated by samples synthesized with sputtering times of 60 to 90 s, which corresponded to nickel particles of 6–8 nm in size. This range achieves an optimal balance between the density of nucleation centers and the prevention of their coalescence, ensuring the formation of a highly developed dendritic structure with maximum electrochemical absorption. Catalysts with these parameters combine high electrochemical activity with sufficient corrosion and morphological stability, confirmed by 5000 stress testing cycles.

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