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Cold start of proton exchange membrane fuel cell using build-in catalytic heater

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Abstract. The paper report on the cold start of fuel cell with proton exchange membrane (PEMFC) at $-40\text{ }^{\circ}\text{C}$ using a catalytic heating unit integrated directly into the PEMFC bipolar plates. This technical solution increases the heat transfer efficiency up to 60% due to direct contact of the membrane-electrode assembly with the heating unit, and ensure a successful cold start of the fuel cell from $-40\text{ }^{\circ}\text{C}$ to an operating temperature of $35\text{ }^{\circ}\text{C}$ within 6 minutes at air flow rate of 150 mL/min. The hydrogen flow rate is $45\text{ cm}^3/\text{s}$, which corresponds to a hydrogen concentration in the air flow of *ca.* 1.8 vol.%, which is below the autoignition point and ensures the safety of the proposed method. Uniform distribution of heat over the bipolar plates surface prevents dehydration and thermal degradation of the membrane electrode assembly components and improve the PEMFC performance after cold start.

1. Introduction

The main advantages of fuel cells with proton exchange membrane (PEMFC) are high efficiency and zero emission, as well as fast load response, long service life, and a high level of fire and explosion safety. Regarding the PEMFC applications in cold climate conditions (*i.e.* at subzero temperature) there is a problem of water freezing in the volume of the device elements. Ice formation makes it difficult to successfully restart the system and causes permanent component damage and degradation of system performance [1–3]. To eliminate these negative effects, several cold start strategies and corresponding technical solutions are proposed. These scenarios include moisture removal by gas purging of water-containing elements (for example, using dry nitrogen) [3–16], vacuum drying, and the use of antifreezes [11] (for example, an aqueous solution of methanol), heat exchangers, bifunctional microporous layers, the use of materials that prevent freezing or do not degrade when frozen, running the system at constant current or maximum power, etc. These methods ensure the device self-starts at $-5 \dots -10\text{ }^{\circ}\text{C}$. The use of methanol as an antifreeze (40% methanol-water solution) provides successfully PEMFC self-start at $-15\text{ }^{\circ}\text{C}$. In the case of deeper freezing of devices at $-30 \dots -50\text{ }^{\circ}\text{C}$ the assisted heating is needed to provide fast start-up [17–37]. The main solutions are internal and external heating, namely hot air-blowing, electrical heating methods, and the use of catalytic hydrogen-oxygen recombiner. Compared



with other assisted heating methods, the catalytic recombination reaction has the advantages of system design, implementation and high heating efficiency [25].

In [6, 36], an external heating method based on the idea of a catalytic recombiners to generate a large amount of heat in a short period of time was described. A separate device was used for a catalytic recombination reaction under pressure, which produced a heated gas that exchanged heat with the components of the PEMFC. The highly efficient catalytic recombiner based on the catalytic oxidation of hydrogen for supplying heat to a hydrogen storage system was proposed in [38]. The authors of the patent [39] have developed a catalytic hydrogen-oxygen recombiner for PEMFC start-up at subzero temperatures with possibility to regulate the final temperature. The main advantage of such systems is the high speed of heating. On the other hand, in the case of external heating, the heat transfer coefficient is still not high due to the use of gases as coolants to heat the device. This method needs for additional sources of hydrogen and increases the total cost and size of the installation. Internal heating is provided by adding hydrogen to the air flow (cathode) and/or adding oxygen to the hydrogen flow (anode) to generate heat in the catalytic layers due to recombination reaction on the Pt active sites [40, 41]. Modeling studies demonstrate [42] that this internal catalytic reaction affects the temperature distribution and improves cold start performance. The main disadvantages include high concentrations of gas additives in flows up to 20 vol.% and the possibility of local overheating, resulting in its dehydration and thermal degradation of the membrane electrode assembly (MEA) components.

In this paper we studied a cold start method assisted by using a catalytic heating unit mounted in direct contact with the MEAs of PEMFC stack. The design of heating unit consists of three reactors for hydrogen-oxygen recombination reaction (recombiners) directly built into bipolar plates. This solution increases the PEMFC heating efficiency due to the direct contact of the MEA (*i.e.* gas diffusion layers [43]) with heated bipolar plates. The parameters of the heating unit were investigated. Successful assisted cold start process of the PEMFC at $-40\text{ }^{\circ}\text{C}$ has been experimentally demonstrated.

2. Materials and methods

2.1. Catalytic recombiner

The catalytic hydrogen recombiner is a steel tube (Fig. 1a) with an inner diameter of 3 mm and a length of 60 mm, loaded with a catalyst. Spherical γ -alumina granules fraction size 0.6–1.0 mm with 1 wt % deposited platinum were used as a catalyst. The particle size of platinum does not exceed 5 nm. Three catalytic recombiners are built into the bipolar plates (BP) of the PEMFC, as shown in Fig. 1b, and together form a catalytic heating unit. Placing recombiners in bipolar plates is the same in the cost, and do not exceed the total costs of producing the PEMFC stack and a separate catalytic heating unit both in machining and materials.

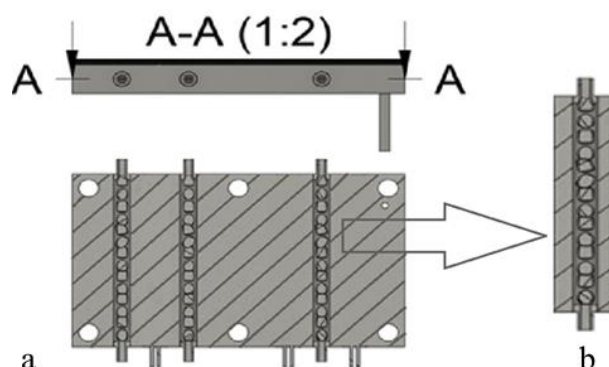


Figure 1. Catalytic heating unit (a) and catalytic recombiner (b) embedded directly into the BP.

The construction material of the presented BP is corrosion-resistant steel. The use of small diameter recombiners (inner diameter 3 mm) provided to avoid a significant increase in the BP thickness. The

recombiner have come into direct contact with a conductive material for effective heat transfer, most often metal. However, the thickness of the metal plate can be reduced if the recombiners are not located in the thickness of the plate but are welded on the outside. Porous plates can be also used, including those made using 3D printing.

2.2. Catalytic recombiner testing

The scheme of the experimental setup is shown in Fig. 2. The catalytic recombiner was placed inside the flow channel of the experimental chamber with a diameter of 25 mm, through which a hydrogen-air mixture was driven. The temperature on the recombiner surface was measured with a Uti165A thermal imager through a $2 \times 65 \text{ mm}^2$ observation window as shown in Fig. 2. Thermal maps were obtained using a thermal imager at certain points in time, which were stored on an SD card, and then processed using the program included in the software of the Uti165A. Additionally, the temperature at the center of the catalytic recombiner was measured using a chromel-copel thermocouple immersed in the catalyst bed. Low subzero temperatures were also recorded with a thermocouple due to the limitations of the thermal imager. The thermocouple signal, after amplification on a precision operational amplifier OP177, was digitized and transferred to the processing program, which showed a graph of temperature changes in real time.

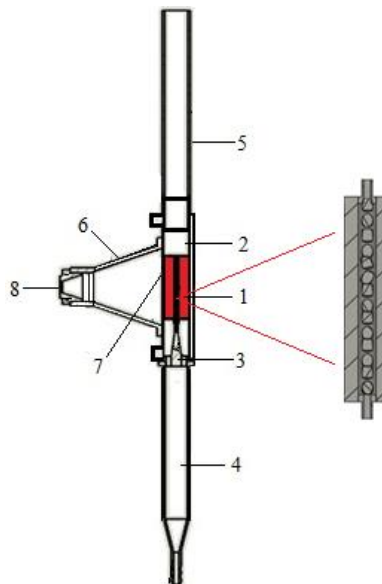


Figure 2. Scheme of an experimental setup for testing a catalytic recombiner: 1 - catalytic recombiner, 2 - flow chamber, 3 - holder for the recombiner, 4 - gas mixing chamber, 5 - removable outlet pipe, 6 - sighting tube for a thermal imager, 7 - observation window, 8 - sealed adapter.

2.3. Calculation of the catalytic recombiner parameters

The release of heat on the catalyst granules in the heating unit leads to an increase in air temperature in accordance with the equation [43]:

$$\frac{E_h(n-n_0)}{N_A} vS = \frac{n}{N_A} vSC_{pa}(T - T_0) \quad (1)$$

where N_A is the Avogadro constant with an exact value of $6.02214076 \times 10^{23} \text{ mol}^{-1}$ (unit of reciprocal moles), E_h is the enthalpy of combustion of 1 mole of hydrogen (240 kJ/mol); $n-n_0$ is the difference in hydrogen concentrations before and after the recombiner; n is the gas concentration at the recombiner outlet; $C_p \cong 7/2 R$ is the heat capacity of the mixture at constant pressure, equal to approximately the heat capacity of air, since the percentage of hydrogen is insignificant, v is the air flow velocity, S is the

cross-sectional area of the cylinder with the catalyst. If we translate the concentration of hydrogen into volume percentages C , then from (1) the equation for the increment of the flow temperature is as follows:

$$T = T_0 + \frac{E_h}{c_p 100} (C - C_0) = T_0 + T_a \Delta C \quad (2)$$

here $T_a=82$ K/vol.% is the temperature characterizing the increase in the temperature of the gas mixture during combustion on the catalyst of 1 volume percent of hydrogen. This assumes no heat loss.

2.4. Calculation of the catalytic heating unit parameters

The calculation of heat transfer processes in the channels of the BP through which the heated air flows is presented. It is assumed that the air at the entrance to the channels contains hydrogen and is heated due to its recombination on the platinum catalyst at the initial section of the entrance to the channels.

On the walls of the channel, a heat exchange process occurs, as a result of which the air is cooled, and the BP warms up. The distribution of air temperatures T_g and BP temperature along the channels T_m is described as follows [25,43]:

$$\begin{cases} \frac{\partial T_m}{\partial t} = \frac{\beta_{gm}}{C_m} \int_0^L (T_g(x) - T_m) dx \\ \frac{\partial T_g}{\partial x} = -\frac{\beta_{gm}}{c_{pa} Q_a} (T_g(x) - T_m) \end{cases} \quad (3)$$

where β_{gm} is the heat transfer coefficient between the walls of the BP channel and the heated air; L is the length of BP channels; C_{pa} is the heat capacity of air at constant pressure; C_m is the heat capacity of the BP; Q_a is the air flow.

It is assumed that the BP is well thermally insulated from the external environment and has a constant temperature.

Solution of equations (3):

$$\begin{cases} T_g(x) = T_m + T_{gmax} \exp\left(-\frac{\beta_{gm}}{c_{pa} Q_a} x\right) \\ \frac{\partial T_m}{\partial t} = T_{gmax} \frac{c_{pa} Q_a}{C_m} \left(1 - \exp\left(-\frac{\beta_{gm}}{c_{pa} Q_a} L\right)\right) \end{cases} \quad (4)$$

where T_{gmax} is the maximum temperature of hydrogen - air mixture $T_{gmax} = T_m + \frac{x_H \xi_H \varepsilon_H}{c_{pa}}$; x_H is the hydrogen concentration in the inlet air flow (relative units); ξ_H is the degree of hydrogen combustion (relative units); ε_H is the amount of heat evolved when 1 cm³ of hydrogen burned ($\varepsilon_H \approx 11$ J·cm⁻³).

The second part of the solution of equations system (4) is an expression that characterizes the rate of BP heating. As expected, the maximum BP heating rate is formally observed at an infinite flow rate, if the degree of hydrogen combustion is not considered:

$$\frac{\partial T_m}{\partial t} = T_{gmax} \frac{\beta_{gm} L}{C_m} \quad (5)$$

An important value is also the value of the BP heating efficiency η_{rec} , which is equal to the fraction of the heat of hydrogen recombination, which is spent on heating the BP. This value is calculated as follows:

$$\eta_{rec} = 1 - \exp\left(-\frac{\beta_{gm}}{c_{pa} Q_a} L\right) \quad (6)$$

When analyzing the effect of air flow on the heating efficiency and rate, it should be noted that in transient flow mode in the channel (from laminar to turbulent), the heat transfer coefficient β_{gm} depends on the air flow through the Nusselt number Nu as follows:

$$\begin{cases} \beta_{gm} = Nu \frac{\lambda}{d} \\ Nu = 0.023 Re^{0.8} Pr^{0.4} \end{cases} \quad (7)$$

where Re is the Reynolds number; Pr is the Prandtl number; α is a constant independent of air flow.

Thus, analyze the described equations it can be finally conclude that with an increase in air flow increases the heating rate of the BP and decreases the heating efficiency BP (6). With an increase in the hydrogen concentration in the air, the heating rate decreases, which requires an increase in air flow (3) and causes an increase in pressure losses. Based on equations (4)-(6) the requirements for high efficiency of BP heating (low air mixture flow rate) lead to an increase in the BP heating time. To reduce the heating time of the BP while maintaining high efficiency the hydrogen concentration should be maximized and the diameter of the recombiner reduced or increased their length L (5). However, an increase in the hydrogen concentration above 4% vol. unsafe due to the risk of hydrogen igniting when mixed with air. A decrease in the internal diameter of the recombiner leads to a nonlinear increase in gas-dynamic resistance and energy consumption for compression of the hydrogen-air mixture. The same, although to a lesser extent, is correct as the length of the recombiner channels increases.

2.5. Assembling of PEMFC stack

In the work, a demonstration setup of a mobile power supply based on PEMFC short stack (3 cells) with a rated power of 50 W was used. For the manufacture of module components and assembly of cells and stack, the techniques presented in detail in [7, 44] were used. The working area of a rectangular electrode ($5.0 \times 8.5 \text{ cm}^2$) was about 42.5 cm^2 . Main components of MEAs included:

- Membrane - Nafion™ 212
- Anode - carbon cloth ELAT LT 1400, coated with a catalytic layer based on Pt/C¹⁰ (40 wt. % of Pt on Vulcan XC-72 with 10 wt. % of PTFE DISP 30 Fluoropolymer Dispersion), catalyst loading was 0.5 mg/cm^2 , ionomer content in the catalytic layer was about 15 wt.% (dispersion D1021 Nafion™ Dispersion - Water based 1100 EW at 10 wt%)
- Cathode - carbon cloth ELAT LT 1400, with a catalytic layer based on Pt/C (40 wt. % of Pt on Vulcan XC-72), catalyst loading was 0.5 mg/cm^2 , ionomer content in the catalytic layer was about 15 wt.%.

MEAs are placed between BP with a build-in catalytic heating unit. An open-cathode scheme was chosen as the design of the PEMFC stack. Fig. 3 shows the general view of the module and the assembly steps of the MEA cathode inside the PEMFC stack (Fig. 3 (1,2,3)). BPs tightened with six studs.

The purpose of the BP is to maintain compressive stresses in the PEMFC to ensure contact between the electrodes and the membrane, as well as to pressurize the adhesive layers of the frames. In addition, the BP serves to fasten the hydrogen supply fittings. Finally, BPs act as a catalytic hydrogen recombiner for the cold start procedure of the PEMFC stack.

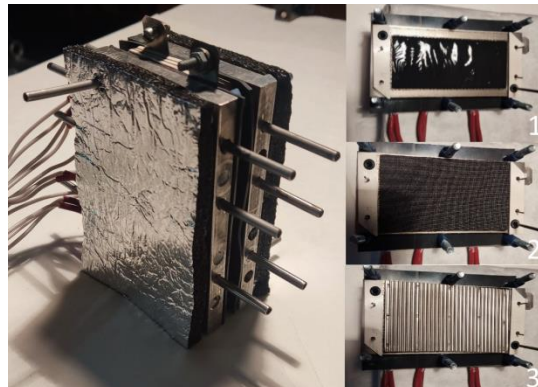


Figure 3. Image of the PEMFC stack in a heat-insulating covering with a built-in catalytic unit and the process of PEMFC cathode assembling (1,2,3).

2.6. Testing a module with an embedded catalytic heating unit in conditions of subzero temperatures

The PEMFC stack was frozen in a Pozis (Paracels) freezer. The PEMFC stack was placed in a freezer and cooled down to $-40\text{ }^{\circ}\text{C}$. After freezing the module was connected to the laboratory test setup.

The stack was heated by using the heat from the combustion of hydrogen in the air flow in the catalytic unit to the given temperature in the range from 20 to $80\text{ }^{\circ}\text{C}$ (the temperature in the freezer was maintained at $-40\text{ }^{\circ}\text{C}$). Monitoring of the characteristics, namely the temperature and current density of the stack at a cell voltage of 0.5 V, was carried out every 3 minutes until the temperature reached $0\text{ }^{\circ}\text{C}$, then every 15 minutes.

Fig. 4 shows a diagram of the experimental setup. PEMFC heating is carried out at a constant temperature with the help of a catalytic heating unit embedded in the BP. To do this, hydrogen from the H_2 tank and air from the compressor are fed into the gas mixing container, and then the gas mixture (a mixture of hydrogen with a concentration of 0.2-3.0 vol.% with air at a constant flow rate of 150 mL/min) is fed into the catalytic recombiners, which ensures heating of the BP of the PEMFC stack. An ATN-8060 electronic load (Atacom, Russia) is connected to the electric contacts of the PEMFC. The temperature in the MEA volume is recorded using a thermocouple.

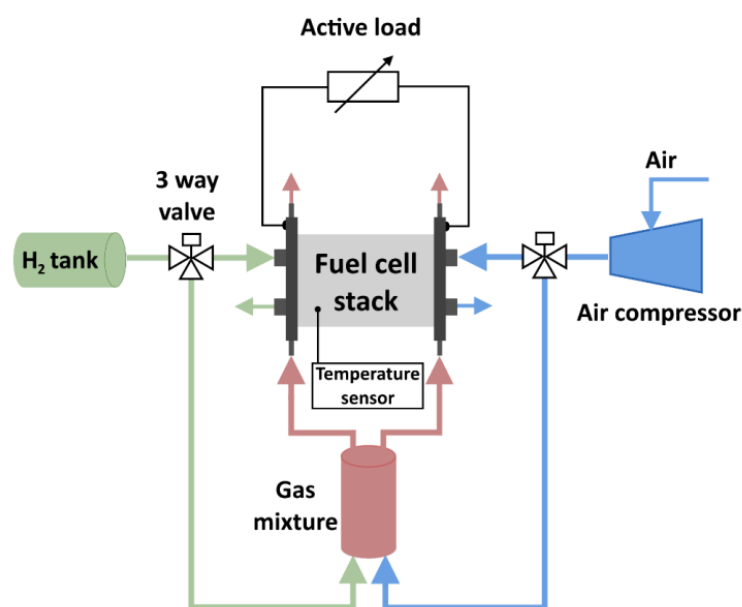


Figure 4. Diagram of a laboratory test system.

The i -V curves were recorded using an ATN-8060 electronic load in the potentiodynamic mode in the voltage range from 0.9 to 0.1 V (sweep rate 4 mV/s). The working gases were supplied without humidification at atmospheric pressure and ambient temperature ($-40\text{ }^{\circ}\text{C}$). The flow rates of working gases were: 160 mL/min for hydrogen, and 500 mL/min for air.

The electrochemical impedance spectroscopy (EIS) data was recorded using an electrochemical workstation CorrTest CS350 (CorrTest, China) with an impedance module at a signal amplitude of 10 mV in the frequency range from 50 kHz to 0.1 Hz. The Nyquist plot was recorded at a voltage of 0.5 V. All measurements were carried out with an identical assembly and the same preparation procedure and compares the obtained data with each other.

3. Results and discussion

3.1. Heating module specifications

Fig. 5 and 6 shows the results of optimization the operation of the catalytic hydrogen recombiner. Analysis of thermal images (Fig. 5a) provided to obtain the distribution of temperature increase along

the height of the recombiner depending on the hydrogen concentration in the input flow (Fig. 5b), as well as the average temperature of the recombiner.

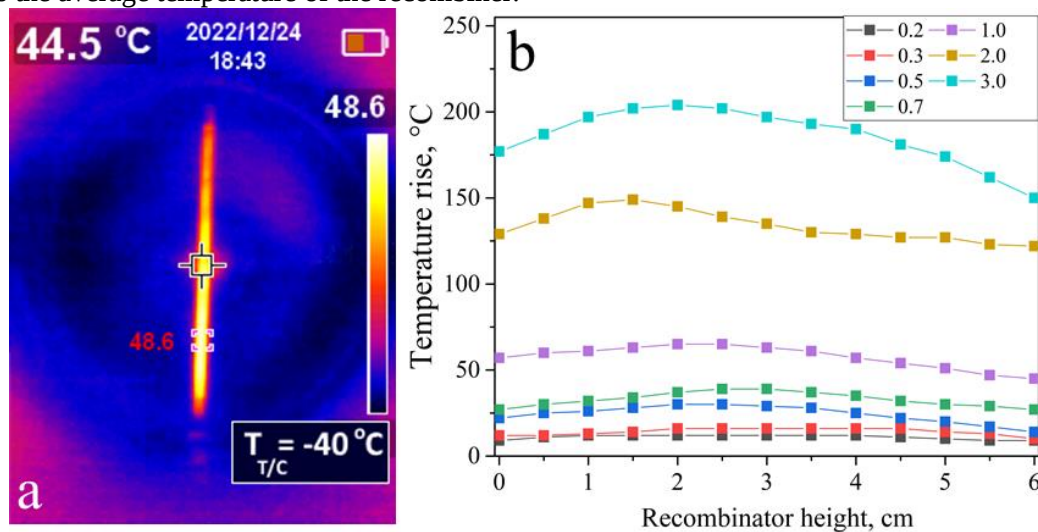


Figure 5. (a) Thermal image of the catalytic recombiner at 5 minutes after the start of the reaction when entering the stationary oxidation process (hydrogen concentration in the air flow is 1.5 vol.%). Lower temperature limit was measured by a thermocouple. (b) Distribution of temperature rise along the height of the recombiner depending on the hydrogen concentration in the input flow.

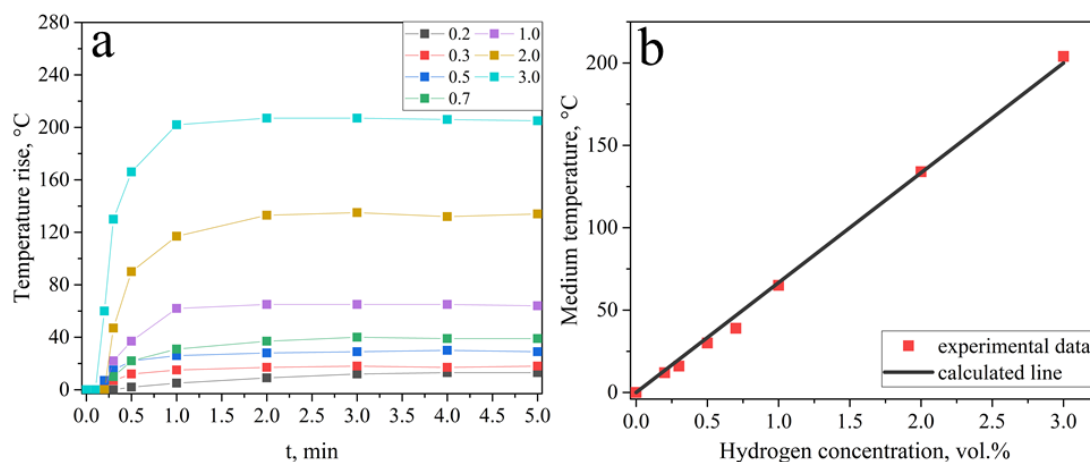


Figure 6. (a) The dependence of the heating time of the recombiner to a given temperature. (b) The dependence of the increase in the temperature of the combustion chamber on the concentration of hydrogen in the input flow, obtained experimentally and calculated according to the presented equations (1) - (2).

The heating time of the BP depends on the hydrogen concentration in the air flow and decreases with increasing concentration from 4 minutes at a concentration of 0.2 vol.% to 1.5 minutes at a concentration of 3.0 vol.%. The temperature characterizing the increase in heat of the gas mixture during combustion of 1 volume percent of hydrogen on a catalyst in a closed system is about 82 °C. In our experiment, the characteristic temperature according to linear interpolation is about 68 °C and increases with an increase in the hydrogen concentration in the flow. The heat utilization efficiency is about 85%, which is a consequence of heat loss through the chamber walls and through radiation. The efficiency of a single recombiner can thus reach 90%. However, the use of a built-in heating unit (consisting of three recombiners) will lead to larger heat losses. The use of the calculation model of the BP heating provided to estimate the efficiency of heating at 60%.

Using the obtained dependences, the conditions for heating the PEMFC assisted by the built-in catalytic heating unit from a temperature of $-40\text{ }^{\circ}\text{C}$ to a cell operating temperature of $35\text{ }^{\circ}\text{C}$ were selected.

3.2. Cold start of PEMFC

Hydrogen and air flows were supplied at $-40\text{ }^{\circ}\text{C}$ (air from the cold environment) and a relative humidity of no more than 20%, which was affected the efficiency of the PEMFC stack (Fig. 7a). The maximum power received was 0.12 W/cm^2 or 15 W from the PEMFC stack vs 50 W rated power. Activation losses (Fig. 7b) are observed both in the anode and pronounced in the cathode spaces, which is caused by the low temperature and humidity of the input gases, as well as the use of an open cathode for air supply.

Fig. 7 shows the i - V curves and the Nyquist plot of the PEMFC before freezing.

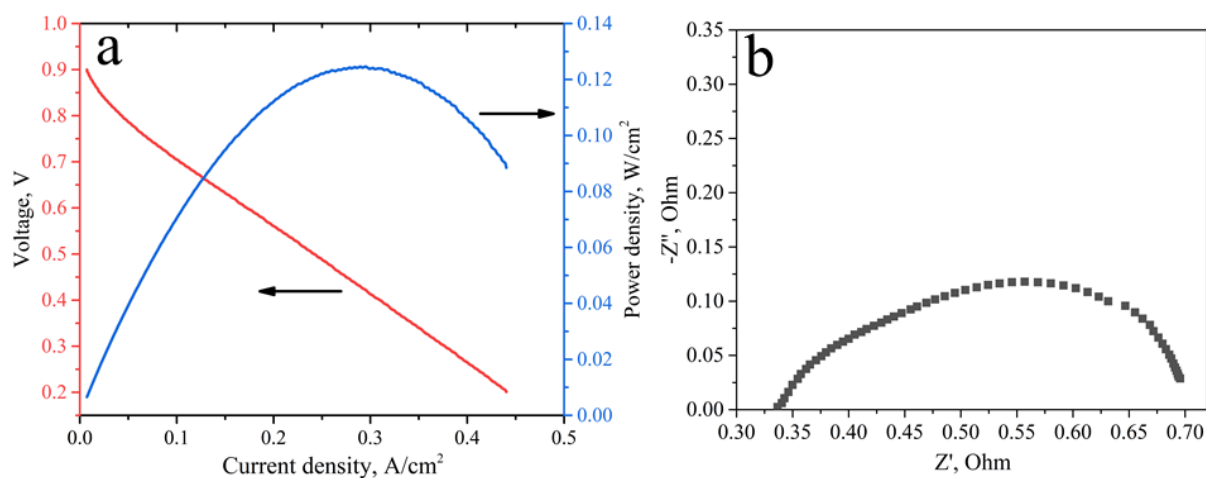


Figure 7. i - V curves (a) and the Nyquist plot (b) of the PEMFC before freezing.

Fig. 8 shows the characteristics of the cold start of the PEMFC when the BP is heated to 20, 40 and $60\text{ }^{\circ}\text{C}$, namely, the dependences of the current density and temperature of the PEMFC on time, and i - V curves before freezing and after cold start. The stack is started up in the maximum power mode at a voltage of 0.5 V, which provided the optimal mode for reaching the operating characteristics [25, 45]. The module was heated by a built-in heating unit, a hydrogen-air mixture was used with a hydrogen concentration in the flow of 1.2, 1.8 and 2.2 vol.%, respectively, at a BP temperature of 20, 40 and $60\text{ }^{\circ}\text{C}$.

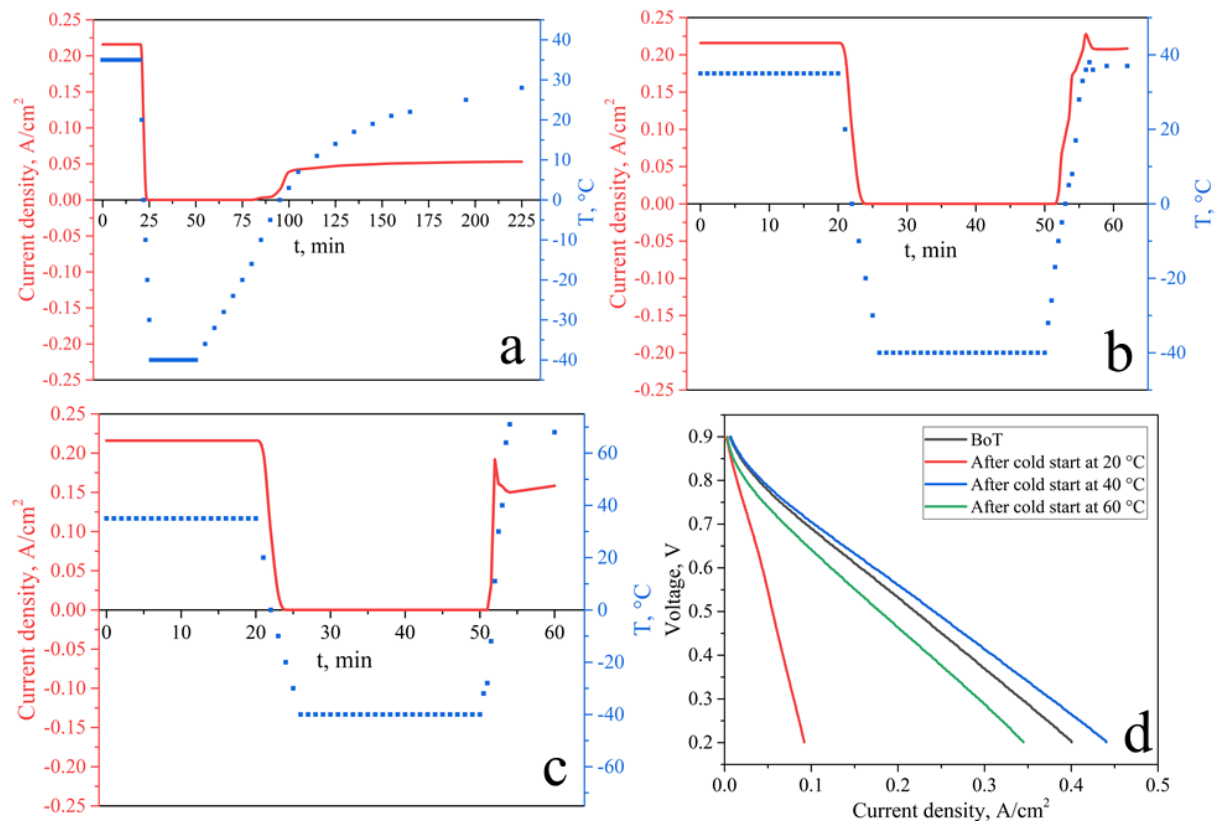


Figure 8. Dependence of the current density and temperature of the PEMFC stack on time during a cold start (a, b, c) and the *i*-*V* curves after a cold start (d) when the end plates are heated to 20 °C (a), 40 °C (b) and 60 °C (c).

As can be seen from Fig. 8a, heating the BP to 20 °C is not sufficient for a successful cold start, since further self-heating of the PEMFC is not sufficient to reach operating temperatures. Excessive startup time leads to irreversible degradation processes of the MEA components. Based on the results of the EIS, after the start of the PEMFC under conditions of subzero temperatures, the resistance increased significantly, which is associated with the intensive degradation of the MEA, the drying of the membrane, the destruction of the catalytic layer and the disruption of the three-phase interface ionomer-catalyst-support. The PEMFC was defective and did not work, thus cold start failed. This effect is caused by the duration of the heating process due to the low temperature of the heating block. The total warm-up time was more than 2 hours.

Fig. 8b shows the dependence of the current density and temperature of the PEMFC stack on the time after freezing and heating to the operating temperature when the BP are heated to 40 °C. The total warm-up time was 6 min. Uniform temperature distribution over the area of the BP ensures startup safety without local overheating and thermal destruction of the MEA components. As a result, the cold start was carried out successfully, the initial values of the output power of the PEMFC were reached, and the water balance of the membrane and catalytic layers was maintained. In this case, the hydrogen flow was 45 cm³/s. Hydrogen concentration in the air flow was about 1.8 vol.%.

Fig. 8c shows the current density and temperature of the PEMFC as a function of time during a cold start when the BP are heated to 60 °C. The total startup time was 1.5 minutes. A significant drop in current density is noticeable, since a rapid increase in temperature in the PEMFC stack causes significant dehydration of the ionomer, which leads to an increase in resistance of the electrocatalytic layer and of the membrane. The output power of the PEMFC decreased, which indicates the shortcomings of this cold start scenario of the stack. Uncontrolled overheating of the cell is also observed due to self-heating

of the stack because of an electrochemical reaction, which will also lead to further degradation of the MEA components.

When comparing the i - V curves after a cold start at different heating temperatures, it can be concluded that the scenario is most effective when using close values of the BP temperature and the operating temperature of the PEMFC. In comparison with other options, heating at 40 °C to the operating temperature is carried out rather quickly and at the same time smoothly, provided to control the heating process and prevent dehydration and thermal destruction of the MEAs components. If the operating temperature of the stack is significantly exceeded, the membrane dries up, which leads to a deterioration in the PEMFC performance.

4. Conclusions

The paper proposes a cold start scenario for the PEMFC stack assisted with a heating unit built-in to the bipolar plates. The heating unit is a hydrogen recombiner tube filled with a catalyst. Using of the built-in unit has increased the efficiency of heat transfer up to 60% and reduce the hydrogen concentration in the input flow to 1.8 vol.% in an air flow of 150 mL/min. An increase in air flow leads to an increase in the heating rate and loss of heat transfer efficiency. The hydrogen flow rate is 45 cm³/s. Assisted cold start processes with build-in catalytic heating unit in proton exchange membrane fuel cell stack has carried out at the start temperature not higher than −40 °C and the parameters for the PEMFC normal operation have determined. Cold start assisted with a heating unit heated to 40 °C was provided the best cold start conditions, including a warm-up time of about 6 minutes and the stability of the PEMFC performance after freezing and subsequent start-up. Fast and smooth heating of the build-in unit and the stack allows controlling the start-up process and ensures the safety of the MEAs components from local overheating and subsequent thermal dehydration and degradation. The proposed method reduces the consumption of hydrogen for heating.

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Nomenclature

N_A	Avogadro constant with an exact value of $6.02214076 \times 10^{23} \text{ mol}^{-1}$ (unit of reciprocal moles)
E_h	enthalpy of combustion of 1 mole of hydrogen (240 kJ/mol)
n_0	gas concentration at the recombiner inlet (mol)
n	gas concentration at the recombiner outlet (mol)
C_{pa}	heat capacity of the mixture at constant pressure (7/2 R)
C	gas concentration at the recombiner outlet (vol.%)
C_0	gas concentration at the recombiner inlet (vol.%)
v	air flow velocity (cm ³ s ^{−1})
S	cross-sectional area of the cylinder with the catalyst (cm ²)
T_0	temperature for the recombiner inlet (K)
T	temperature for the recombiner outlet (K)
T_a	temperature characterizing the increase in the temperature (K/vol.%)
ΔC	difference in hydrogen concentrations (vol.%)
T_g	distribution of air temperatures
T_m	BP temperature along the channels
β_{gm}	heat transfer coefficient
L	length of BP channels (cm)

C_m	heat capacity of the BP
Q_a	air flow (cm^3s^{-1})
T_{gmax}	maximum temperature of hydrogen (K)
x_H	hydrogen concentration in the inlet air flow (relative units)
ζ_H	degree of hydrogen combustion (relative units)
ε_H	amount of heat evolved when 1 cm^3 of hydrogen burned ($\text{J}\cdot\text{cm}^{-3}$)
η_{rec}	heating efficiency (relative units)
Re	Reynolds number
Pr	Prandtl number
α	constant independent of air flow

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