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Influence of temperature and nitrogen pressure on the test without active gases for high-temperature proton exchange membrane fuel cells

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Abstract: High-Temperature Proton-Exchange Membrane Fuel Cells (HT-PEMFCs) are a candidate for electrical energy supply devices in more and more applications. Most notably in the aeronautic industry. Before any use, an HT-PEMFC is preheated and after that supplied with its active gases. Only at this state, the diagnostics can be performed. A method of testing not requiring a complete start-up would be beneficial for many reasons. This article describes an extended version of the charging and discharging diagnostic method of HT-PEMFCs with no active gases. This extended approach is named "Test Without Active Gases" (TWAG). This paper presents original research on the influence of nitrogen temperature and pressure on the HT-PEMFC response to charging and discharging. A lumped-element model of an HT-PEMFC is also presented. A numerical result of using this model to recreate an experimentally obtained curve is also presented.

Key words: diagnosis, high-temperature PEMFC, state of health, TWAG

1. Introduction

Fuel cells are devices that transform the chemical energy of hydrogen into useful electrical energy. Among them, the most commonly used type is Proton Exchange Membrane (PEM) fuel cells [1, 2]. The main advantages of this kind of fuel cells is their fast startup, flexibility in input fuel, compact design and light weight [1–3]. The main disadvantage is the necessity to use a costly catalyst in the form of platinum.

Due to the rising popularity of electrical energy systems, fuel cells are the subject of extensive studies, many of which concern the modeling of these devices [7–10, 14, 18, 19]. Well-performed modelling work can lead to a good integration of fuel cells as electrical energy sources in distribution network systems [27]. While the behavior of fuel cells during operation has been the subject of many studies [16, 20–23, 25], their properties without active gases are relatively unknown.

Articles on this topic are rare. One can cite a study of the response of a low-temperature PEMFC fuel cell without active gases to charging and discharging with a constant current [10, 11]. This study concluded that the described method shows promise in terms of use as a diagnostic tool for fuel cells' state of health. This approach has not been further developed and applied to different types of fuel cells at varying temperatures and pressures.

There exists, however, another kind of PEMFC, called a high-temperature PEMFC or HT-PEMFC. They use phosphoric acid entrapped in polymer structure as their electrolyte material in place of proton-conducting polymer used for low-temperature PEMFCs. This kind of fuel cell is currently a subject of research as a likely candidate for applications in the aeronautic industry due to its many advantages such as easier cooling, higher tolerance for gas impurities, and no need for membrane humidification which results in simpler water and thermal management systems [1–4]. One of their drawbacks is the required preheating (up to 100°C) to avoid contact between liquid water and phosphoric acid used as electrolytes. Indeed, liquid water pumps this acid and evacuates it to the outside accelerating the aging of the fuel cell. This preheating is carried out before the injection of active gases. Consequently, no diagnosis is made during this start-up as the currently used diagnostic methods, such as electrochemical impedance spectroscopy and polarization-curve acquisition, can only be applied after the cell has been launched.

The goal of the study described in this article was to reapply the methodology previously used in [10, 11] and expand it to high-temperature proton exchange membrane fuel cells filled with nitrogen and neutral gas. The development of a diagnosis method without active gases would be very beneficial for the ease of future applications. In order to normalize the procedure in case of further research, an impact of the method on the state of health of the cell and the influence of two main experimentation parameters (pressure of nitrogen inside the fuel cell and temperature of the fuel cell) were studied.

While fuel-cell models have already been developed and established under active gases, due to their electro-chemical nature, they cannot be used for the analysis of the results described in this article. The available knowledge of fuel cell structure suggests that with no active gases present, it is likely to behave in a manner similar to double-layer capacitors [12]. Due to the nature of the electrodes used in a fuel cell, the internal double-layer capacitors are true supercapacitors. These kinds of capacitors are well studied and their equivalent electric circuit models have been already developed and established [5, 13]. This lays a solid ground for modeling work for fuel cells in a state with no active gases.

The novel aspects of this work include:

– extended methodology of charging and discharging HT-PEMFCs in a nitrogen atmosphere,
– analysis of the influence of nitrogen pressure and cell's temperature on the response of the cell to charging and discharging.

This paper aims to achieve the following objectives:

- developing the methodology called "Test Without Active Gases" (TWAG) based on a previously described "test in the passive state" [10, 11] and preparing the experimental bench setup required to perform it,
- carrying out a series of measurement campaigns on the HT-PEMFC for data acquisition,
- determining the circuit parameters of the equivalent lumped-element model of the HT-PEMFC at no active gases.

2. Literature review

First fuel cells were invented back in the first half of the 19th century [1, 3]. Initially, they have not gained a lot of attention due to the rise of combustion engines, which were much cheaper and easy to apply in the industrial world. Interest in this kind of technology was sparked by the space-race that took place in the 60s and 70s, when alkaline fuel cells were used as electric energy supply for American space rockets [1, 3]. This interest was extinguished a bit in the 80s and 90s but was sparked again at the beginning of the 21st century due to ecological concerns [1–4]. Nowadays, a lot of research is being done with a focus on designing more efficient and easier-to-utilize fuel cell cells. There exists a wide range of publications and books covering the topic of fuel cells and diagnostic methods used to study them [4, 15]. There are, however, significantly few works concerning the behavior and properties of fuel cells in the state with no active gases present [10, 11].

3. Principle of the test without active gases

The studied method was given the name "Test Without Active Gases" (TWAG in short). During this test, a fuel cell filled with a chemically neutral gas (nitrogen) is firstly charged with a constant current until it reaches a chosen voltage or time limit by a device that can act as both a current and voltage source. This part will be called the "charging phase". Next, it is disconnected from the device and set aside for a selected time period. This part will be called the "self-discharge phase". During this time, its voltage is continuously measured. Lastly, the cell is once again connected to the controllable source, which this time discharges it with a set current until there is no more charge remaining in the device. This last part is called the "discharge phase". Figure 1 presents hypothetical curves with two main measured signals. In this figure, T1 and T2 of switching between different phases of TWAG.

These curves are qualitative in nature in order to show the three mentioned transient states. The first one is the voltage of the fuel cell. The second signal is the current absorbed or delivered by the fuel cell. During all the TWAG tests described in this article, an experimental bench utilizing Sefram hardware to measure and record voltage, temperature and pressure were used. A standalone OrigaLys programmable source was used as a current source and to measure and record current. The full scheme of the experimental circuit is shown in Fig. 2.

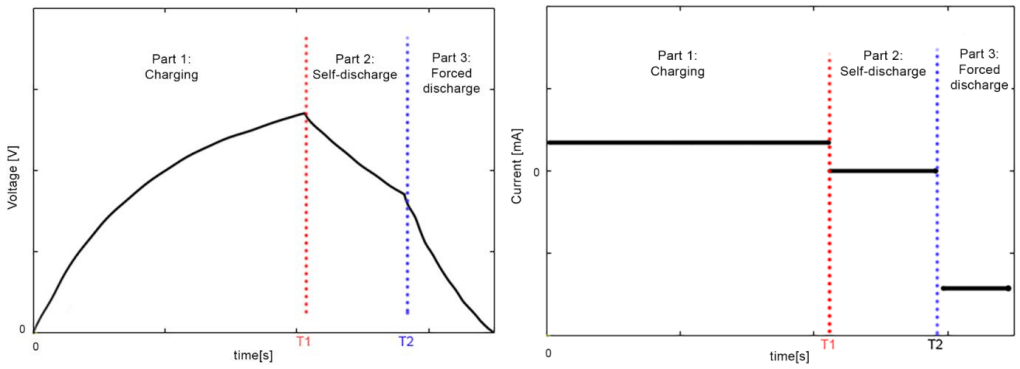

Fig. 1. Typical shape of the voltage waveform of the fuel cell during TWAG (a). Typical shape of current waveform imposed on the fuel cell during TWAG. Times of switch between the phases are marked as T1 and T2 (b)

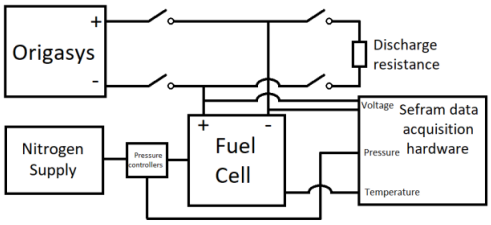

Fig. 2. Schematic of the experimentation circuit used to perform TWAG

4. Experimentation performed

Two experimental campaigns were performed during this study and are described in this paper. The first one had the goal of finding if the TWAG provoke degradation of the Membrane Electrode Assembly (MEA) or any other elements. The second one aimed to determine the influence of the experimental parameters on the test and if standardization of them will be necessary in order to have a consistency of results in any further studies.

4.1. Campaign to study the potential effect of TWAG on the degradation of the MEA

The objective of the first campaign was to study if the TWAG procedure itself has a negative impact on the state of health of fuel cell components. It was decided that the aged MEA would be installed in a single-cell box and used as the study subject. This choice was made for two reasons. Firstly, the used MEA's state of health before the experimentation was already well studied with classical diagnosis methods (V–I curves, impedance spectroscopy). Secondly, if degradation would be significant, no costly parts would be damaged.

The campaign began with the installation of the chosen MEA and the start-up of the fuel cell. After that, an initial diagnosis by the acquisition of the polarization curve was performed. Next,

two experiments were performed. The first experiment consisted of shutting the cell down, one application of TWAG, and a re-start-up. The second experiment consisted of shutting the cell down, ten applications of TWAG, and a re-start-up. Two additional diagnoses by the acquisition of the polarization curve were performed: between the two experiments and after the second experiment. This was followed by shutting off and dismantling the studied fuel cell, which concluded the first campaign.

Figure 3 presents the results of the first campaign. They were used to calculate ΔV, an average difference between cell voltages: before and after the experiment. ΔV for the first experiment was calculated to be 1.4 mV and for the second experiment 1.3 mV. The observed difference could be primarily caused by the processes of shutdown and restart of the cell rather than the TWAG test, as described in [10, 11].

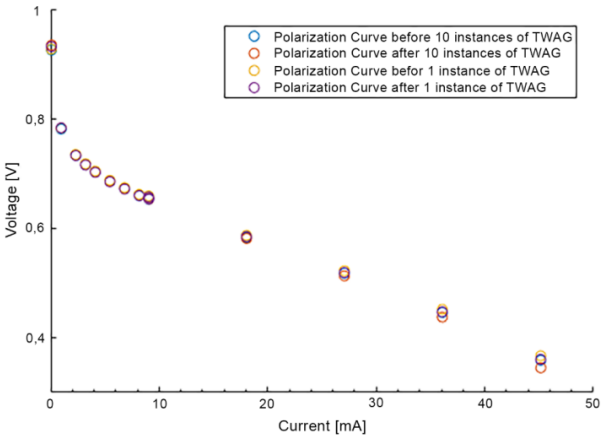

Fig. 3. Results of experimental campaign no. 1

4.2. Campaign to study the influence of nitrogen pressure and temperature on results of TWAG

High-temperature PEMFCs operate in the temperature range of 120°C to 200°C. At these temperatures, some of the components such as gaskets "fuse together" welding the MEAs in one block. This creates difficulty in disassembling a high-temperature PEMFC after initial start-up. Because of this, it would be beneficial to perform a diagnosis of components in a lower temperature. TWAG are viable candidates for this as the test can be performed with no active gases. It was decided, however, that to confirm this viability, it was necessary to study the effect of temperature on curves obtained by this kind of test.

During TWAG, no active gases are present inside the fuel cell. They are replaced by nitrogen (N_2) which is neutral to electrochemical components of the device. After initial tests of TWAG, it was hypothesized that the constant flow of nitrogen may result in impurities in form of hydrogen and oxygen particles being delivered to the electrodes. This would result in the cell exhibiting a voltage of its own and thus unreliability of the observed results. To remove this problem, a procedure was implemented: pressurizing the cell with nitrogen up to a chosen value higher

than atmospheric pressure and then connecting a discharge resistance for a short period of time. It was decided that the chosen value of pressure of N_2 for pressurization is another parameter that could potentially affect TWAG result curves and, therefore, it needed to be investigated.

A campaign focused on the study of the two aforementioned parameters was designed and performed using the same test bench and equipment as the first campaign. Input values of these parameters that were tested are presented in Table 1. A new fresh MEA was used for this campaign. It was installed in a single-cell casing on the day of the launch.

Table 1. Studied values of parameters for the second campaign

No.	**Temperature (°C)**	**N_2 Pressure (kPa)**
1	20	102
2	40	122
3	40	102
4	60	122
5	80	122
6	100	122
7	100	102
8	120	102
9	120	122
10	120	142
11	120	162
12	120	182
13	120	102
14	100	122
15	100	162
16	80	162

Figure 4 presents part of the TWAG results. All of the presented TWAG were performed with an N_2 pressure of 122 kPa with the exception of the curve for 20°C, which was obtained with an N_2 pressure of 102 kPa. The reason was the inability to maintain pressurization at this temperature. An explanation is most probably the fact that the gaskets inside the fuel cell casing were designed for a higher temperature of operation and thus do not keep their sealing properties at 20°C. The current value in the charging state was equal to 1 mA.

An interesting observation was made: temperature has a non-linear impact on the observed response of the studied device submitted to the TWAG test. In the temperature range from 20°C to 60°C the voltage change due to the temperature change is relatively high. Above the temperature 80°C the voltage change is not so sensitive to temperature change. This could be explained

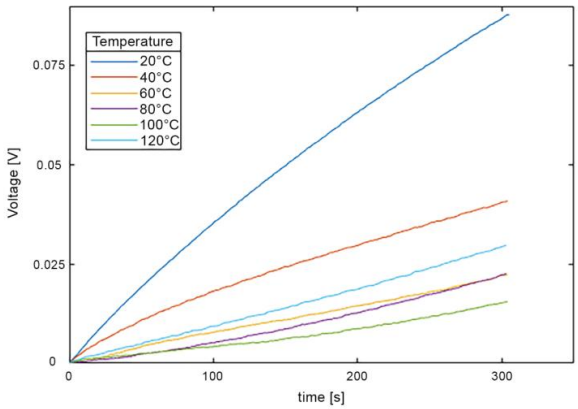

Fig. 4. Charging curves (made out of measured points) for different values of temperature, the N_2 pressure being set to 122 kPa

by a phenomenon linked to a material occurring in this range. The authors especially suspect phosphoric acid, the nature of which changes with temperature. The nature of this occurrence as well as its impact on the applicability of TWAG tests for practical purposes would require additional study. For now, a conclusion was made that in order to ensure relatability of TWAG results to the performance of the fuel cell, further TWAG tests should be executed in temperatures not lower than 60°C.

Figure 5 presents a comparison of self-discharge curves for different values of temperature. It was made in order to determine if the temperature has a similar influence on this part of TWAG as for the charging part. No easily recognizable trends were observed in this case. The curve

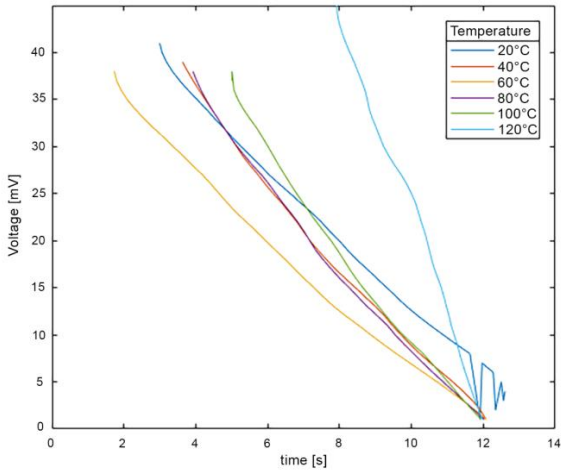

Fig. 5. Self-discharge curves (made out of measured points) for different values of temperature, the N_2 pressure being 122 kPa

that varied most from the rest was the one obtained for a temperature of 120°C. The literature suggested, this part of TWAG best reflects the performance of the MEA [1]. This result suggests this is very likely the case.

Similarly to Fig. 4, Fig. 6 presents curves measured during charging parts of TWAG for 5 different cases during which temperature was at 120°C while pressure varied. In this case, no issue with maintaining the temperature was observed and thus it was assumed as constant for purposes of analysis.

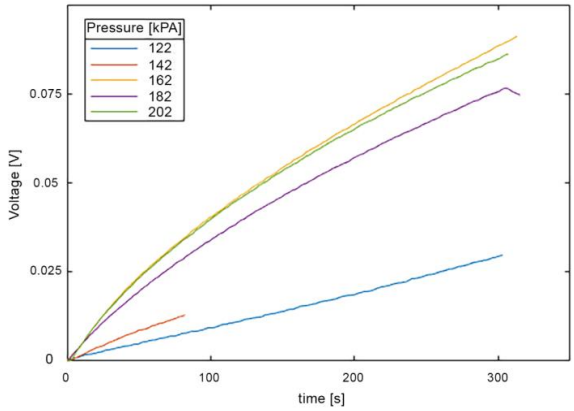

Fig. 6. Charging curves (made out of measured points) for different values of N_2 pressure, the temperature being 120°C

There was however an issue for the curve measured for 142 kPa: due to unexplained reasons, a peak in voltage was observed before the test. This was most probably caused by an error during connecting the discharge resistance. Because of this, the initial voltage was higher than expected and the aimed voltage was achieved quicker. The result is a very short charging slope.

Unlike for the temperature, in this case, it is not possible to indicate a clear dependence of voltage changes due to pressure changes. A clear observation is that the curves for higher pressures (from 162 kPa to 202 kPa) are very similar to each other while the ones for lower pressures of 122 kPa and 142 kPa vary more. The proposed explanation for this observation is that higher values of pressure are better to prevent particles of active gases from the air around the casing from getting inside the fuel cell. Thus, the results of TWAG appear to be more stable with high values of pressure. Thus, it was decided that the value of N_2 pressure in further applications of the TWAG method should be kept higher than 142 kPa.

Like in the case of temperature, self-discharge curves for different values of N_2 pressure were also analyzed, as presented in Fig. 7. Likewise, no clear trend relating to the shape of these curves was found as they all closely resemble each other. This, along with the knowledge that temperature remained constant at a level of 120°C for all presented tests, would be consistent with the previously suggested hypothesis that the fuel cell with no active gases operates in better conditions when the temperature is over 60°C.

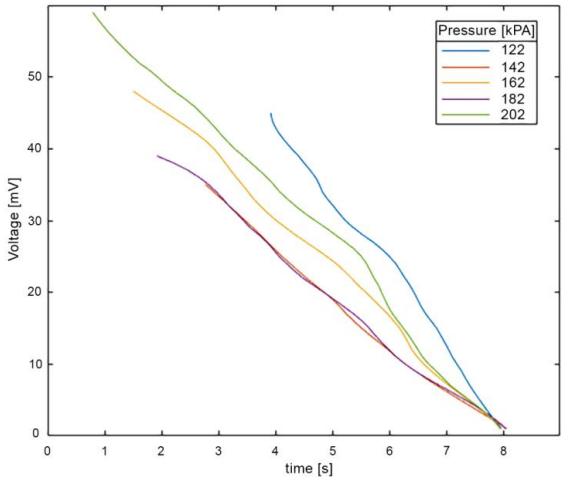

Fig. 7. Self-discharge curves (made out of measured points) for different values of N_2 pressure, the temperature being 120°C

5. Proposed model of HT-PEMFC with no active gases

While fuel-cell models have already been established under active gases, they cannot be applied to the cell with no active gases. It is due to the fact that the elements of this kind of model represent physical phenomena that cannot occur with no active gases present. This represents a challenge of creating another type of model that would enable researchers using TWAG to recreate experimentally obtained curves of fuel cell's response to charging with constant current such as those aforementioned in this article.

The authors of this article have proposed such a model. The basis for it is the model of a double-layer capacitor. It was chosen due to the knowledge of the PEMFC structure, which is very similar to the structure of this kind of component. The authors thus predict that a fuel cell with no active gases should behave similarly to a supercapacitor. The presented model is described by Eqs. (1), (2).

$$G_0 u_0 + G_1 (u_0 - u_1) = i_0(t), \tag{1}$$

$$C_1 \frac{\partial u_1}{\partial t} - G_1 (u_0 - u_1) = 0, \tag{2}$$

where: G_0, G_1 are the conductances, C_1 is the capacity, u_0, u_1 are the node potentials, i_0 is the current, t is the time.

The circuit model equivalent form of the model that has been used for simulations in the Simscape environment, is shown in Fig. 8. Systems of Eqs. (1), (2) represent a variant of the model with only one RC branch.

As shown in Fig. 8, the proposed model contains a resistor called R_{self} which represents the self-discharge phenomena; connected in parallel to this resistor, RC branches represent the capacitive nature of the fuel cell with no active gases. Variants of the model with different

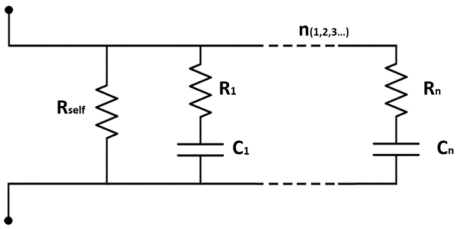

Fig. 8. Equivalent circuit model of a fuel cell with no active gases

numbers of RC branches from one to ten were studied. The studied variants of the model were created in the Simscape environment and used for simulations in order to recreate as closely as possible experimentally-obtained curves. The authors were able to achieve this goal with an already-satisfying accuracy with as low a number of branches as one. The typical curve obtained using the presented model compared to the experimentally-obtained curve is shown in Fig. 9.

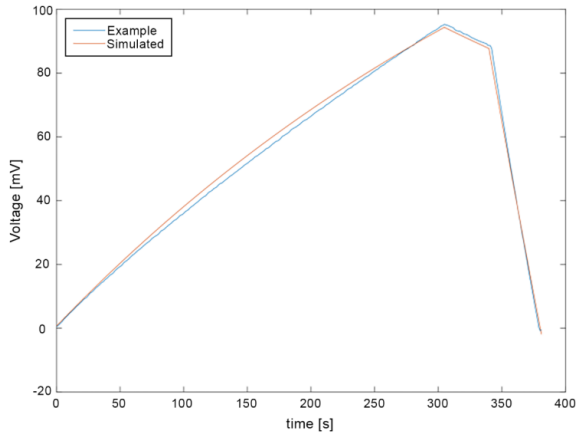

Fig. 9. Comparison of the curve obtained experimentally and the curve obtained through simulation using the presented model with one RC branch

6. Conclusions

In this study, the extended TWAG (Test Without Active Gases) methodology of charging and discharging fuel cells using nitrogen as a reference and neutral gas has been developed. This methodology has been implemented for testing High Temperature PEMFCs in many different sets of input conditions [Table 1]. The advantage of this method is that nitrogen does not cause irreversible chemical reactions in the fuel cell's membrane structure even in its preheating state. As such, the method can be applied before the launch of the fuel cell stack. Any potential defect detected this way can be removed at a stage when a replacement of a single MEA in the stack is possible.

It means that the HT-PEMFCs at no active gases can be modeled by lumped conservative and dissipative elements. The conservative elements are capacitors that represent electric energy stored in the layer structure of the membrane. The dissipative lumped elements represent energy losses due to Joule's law of heating. It is assumed that parameters of the lumped conservative and dissipative elements are sensitive to the physical properties of the layer membrane structure. Therefore, an equivalent circuit model can be used to simulate the steady and transient states of HT-PEMFCs in the absence of active gases for diagnostic purposes.

The TWAG method should be used with a slightly higher pressure inside the fuel cell than the external pressure. This creates a barrier against the penetration of active gases from the external environment and provides the appropriate conditions for test standardization.

On the basis of the conducted research, the following detailed conclusions were obtained:

- The TWAG procedure has no negative impact on the state of health and structure of fuel cell components.
- Nitrogen pressure has no essential influence on current and voltage waveforms at steady and transient states, but should be constant and higher than external pressure.
- The internal temperature of a fuel cell has a significant impact on the voltage value at the cell terminals in the range of values up to about 60°C.
- The value of the internal temperature of the cell must, therefore, be constant and always determined in the cell test using the TWAG method.
- Self-discharge voltage waveforms are not sensitive to nitrogen pressure.
- The equivalent electrical circuit with a single capacitor representing the energy stored in the double-layer membrane fuel cell structure is sufficient to simulate HT-PEMFC charge-discharge states in the absence of active gases with good agreement with the measurement results.
- In order to better match the simulation results with the experiment results, a non-linear model or a model containing a larger number of conservative and dissipative lumped elements should be developed.

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