

POLITECNICO DI TORINO

Master's Degree in Energy and Nuclear Engineering



Master's Degree Thesis

Setup of an experimental test-bench for remote testing of
solid oxide cells. Test-bench commissioning and
test-protocols definition for the characterization of single
cells for fuel cells applications

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Abstract

The present work aims at providing a user guide for the correct utilization of an experimental test-bench, designed for remote testing and characterization of single solid oxide cells. The test bench development has been financed as part of *TeacHy*, an EU H2020 project with the goal of supplying graduate education in form of a MSc course on fuel cells and hydrogen technologies. A specific web-based interface, *TeacHy*, has been designed for the remote control of the test rig, allowing students from every part of the world to be involved in remote experimental sessions.

The provided user's guide investigates instruments and physical connections as well as *TeacHy* operation. A standard test is analysed and explained, to provide a guide for future experiments.

Finally, a gas flow control system has been implemented from hardware to software and presented in this work. The system is a simplified version of the one implemented in the remote test-bench, with a lower number of controlled flows and a local user interface. Despite the lower grade of complexity, the implemented system is based on the same type of hardware and software of the bench, allowing to better understand the development and management of the actual test rig.

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Chapter 1

Introduction

1.1 TeacHy Project

TeacHy is an EU H2020 project that has the goal of supplying graduate education in form of a MSc course on fuel cells and hydrogen technology, founded in response to the increasing need for trained staff in this sector, due to its progressive establishment in the market. Among different institutions, *Politecnico di Torino* also collaborates in this European project. Several modules are included in the MSc compulsory courses: the "Remote Laboratory" one focuses on testing of Solid Oxide Fuel Cells (SOFCs). The remote testing is made possible by a test bench of *Politecnico di Torino*, located in the CO₂ Circle Lab (CCL) at the Environment Park S.p.A.. Students will be able to remotely control a set of variables and participate in SOFC's test experience.

The present work aims at introducing the test rig, providing a hardware and software user's guide. The test bench will be described in its physical connections and components. *Teachy* online software, which allows test-rig remote control and developed specifically for this project, will also be outlined in all of its functionalities.

1.2 Solid Oxide Fuel Cells

1.2.1 General Aspects

A fuel cell is a system able to directly convert chemical energy into electrical energy. It receives as an input a fuel (such as hydrogen) and an oxidant (such as oxygen), which are involved in an electrochemical reaction, producing electricity as long as heat and exhausts.

Fuel cells include mainly three components: an anode, a cathode and an electrolyte. The anode is the electrode receiving (continuously) the fuel stream and driving

an oxidation reaction. The cathode is the electrode receiving (continuously) the oxidant stream and driving a reduction reaction. The electrolyte, in between anode and cathode, allows ions migration (from cathode to anode or viceversa, according to fuel cell's typology). Electricity can be produced if the cell is connected to an external circuit that allows the flow of electrons from anode to cathode. Among the different currently available fuel cell's technologies, the present work will focus on solid oxide fuel cells (SOFC). SOFCs are high temperature fuel cells; in figure 1.1 their working principle is illustrated, with hydrogen as fuel media.

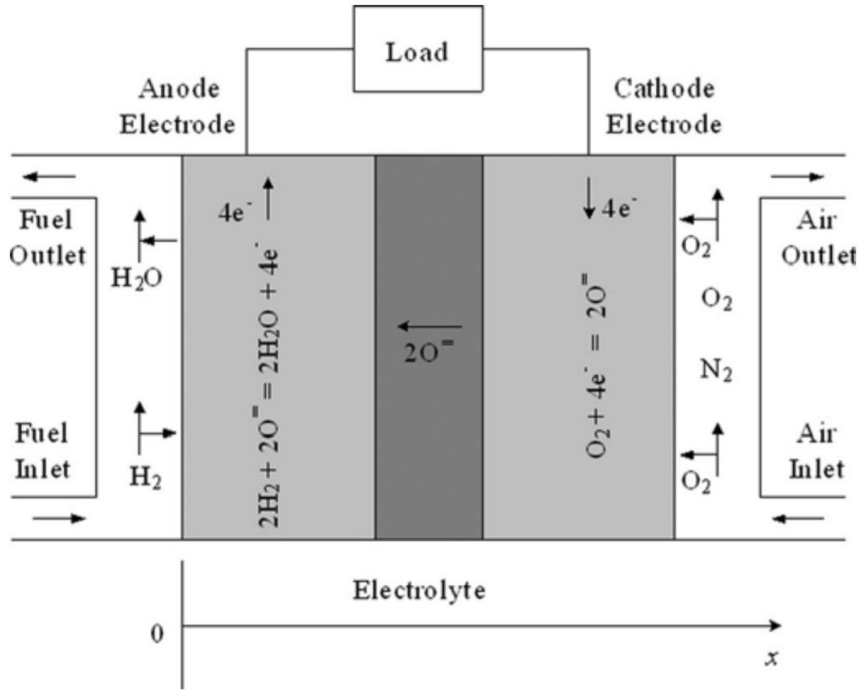
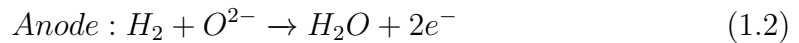
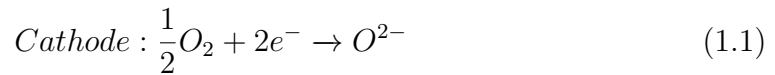


Figure 1.1: Solid oxide fuel cell scheme [1]

In the cathode compartment, oxygen molecules react with the electrons coming from the anode through the external electrical circuit, producing O^{2-} ions. These negative ions are conducted through the electrolyte towards the anode, where they react with the supplied hydrogen molecules, producing water vapour and electrons, which are conducted through the external circuit.

Therefore, the electrochemical half reactions are:



The total electrochemical reaction is:



1.2.2 Nernst potential and Polarization curve

It is demonstrated that the open circuit voltage of a fuel cell (OCV), known as Nernst potential or as Equilibrium voltage, is given by the following equation:

$$E = -\frac{\Delta g_{react}(T_{cell}, p_0)}{z_F \cdot F} + \frac{R \cdot T_{cell}}{z_F \cdot F} \cdot \ln \frac{\sum^R \left(\frac{p_i}{p_0}\right)^{\gamma_i}}{\sum^P \left(\frac{p_i}{p_0}\right)^{\gamma_i}} \quad (1.4)$$

Where: T_{cell} is the cell operating temperature; p_0 is the reference pressure; Δg is the Gibbs free energy variation of the electrochemical reaction (negative in case of fuel cell); R is the ideal gas constant; F is the Faraday constant; z_F is the charge number of the fuel chemical specie, corresponding to the number of electrons involved in the oxidation reaction of the fuel specie (2 in case of hydrogen); p_i are the partial pressures of reactants and products.

Nernst potential E represents the voltage gradient across the cell in open circuit condition, so in case of equilibrium, when no current is flowing through the external circuit. As noticeable from the Nernst equation, E is directly proportional to reactant's partial pressures (while it is inversely proportional to product's partial pressure). Therefore, feeding air at the cathode compartment instead of pure oxygen will result in a lower open circuit voltage.

When closing the external circuit of the fuel cell, the system exits from the equilibrium condition. As a result of irreversible losses, a voltage decrement is introduced with respect to the open circuit voltage and the system can be characterized by a current-voltage curve, denominated *Polarization curve* (figure 1.2).

The first region of the curve, at low current densities, is dominated by activation losses. These losses are related to the kinetic of the charge transfer processes occurring at the anode and cathode during oxidation and reduction reactions. The high operational temperature of SOFC plays an important role, favouring the kinetic of charge transfer, making the activation losses almost negligible.

The second region is dominated by ohmic losses and has a linear trend. These losses are related to the migration of charged species (electrons and ions) and can be expressed through the Ohm Law as [2]:

$$\Delta V_{Ohmic} = i \cdot (\rho_{electrolyte} \cdot L_{electrolyte} + \rho_{anode} \cdot L_{anode} + \rho_{cathode} \cdot L_{cathode} + R_{contact}) \quad (1.5)$$

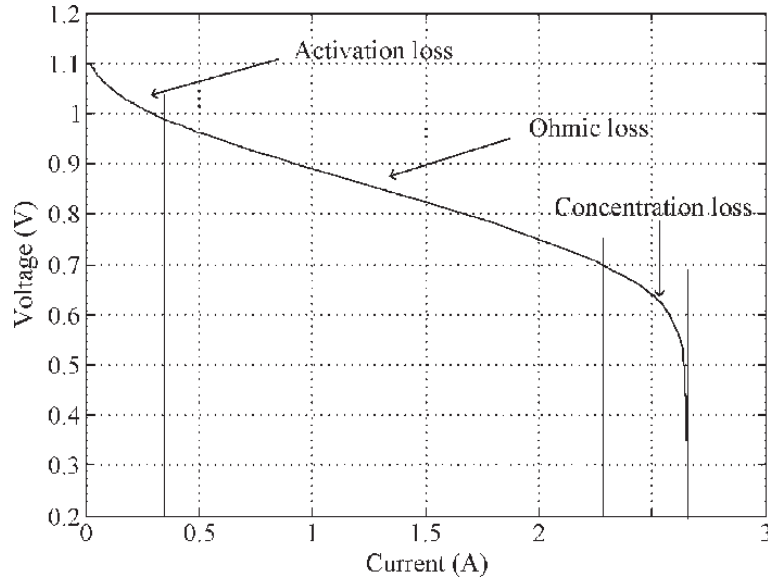


Figure 1.2: Polarization curve: voltage trend with increasing current [3]

Being ρ the resistivity [Ωm] of the specific conductor media, and being L its thickness. Electrode's resistivity is negligible compared to the electrolyte's one, therefore ohmic losses derived from ions migration through the electrolyte represent the prevailing term.

The third region, at high current densities, is dominated by concentration/diffusion losses. These losses are derived by the difference in reactants concentration between the bulk region and the active point of reaction. In fact, at high current densities, a mass transport problem arises: the time employed by reactants to reach the catalyst site is no longer sufficient to keep up with the increasing demand. This can lead to a starvation of molecules available for reaction. Starvation can be very dangerous and should be avoided, since the catalyst itself can begin to oxidise in order to provide electrons. The high temperature of SOFC promote molecular diffusion in the electrodes, limiting the diffusion losses.

1.2.3 State of the art materials

State of the art materials for anode, cathode and electrolyte are chosen basing on the specific requirements and constraints that each component should fulfill. In fact, material science plays a fundamental role in solid oxide fuel cells, providing the basis for future improvements.

- The electrolyte should assure an high ionic conductivity at fuel cell's operating temperature, necessary for the migration of O^{2-} ions from cathode to anode.

At the same time, it must have a high electronic resistivity and it must be impermeable to gas diffusion, therefore very dense. Being in contact with both anodic and cathodic compartments, chemical stability with respect to oxidizing and reductive atmospheres is very important, as long as thermomechanical stability. At the present, the material dominating the market is yttria stabilized zirconia (YSZ): zirconia (ZrO_2) doped with 8%mol. of yttria (Y_2O_3). Doping, on one hand, enhances the hopping mechanism of O^{2-} ions through the lattice: the substitution of Y (trivalent) with Zr (tetravalent) introduces vacant positions (holes), exploited by O^{2-} ions for migrating from cathode to anode [4]. On the other hand, doping is necessary for stabilizing the cubic crystal structure: otherwise, zirconia would change its structure by increasing the temperature [4].

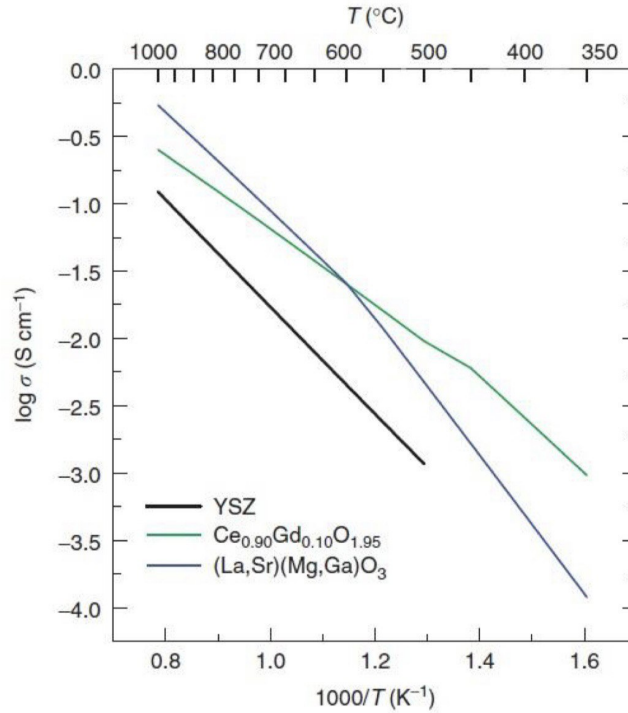


Figure 1.3: Conductivity trend of different electrolyte materials with respect to temperature [5] [2]

As represented in figure 1.3 [2], other materials with higher ionic conductivity with respect to YSZ have been considered, such as gadolinium doped CeO_2 (CGO) or $(La, Sr)(Mg, Ga)O_3$. Anyhow, YSZ is the one assuring the best long-term stability.

- The anode material must ensure high temperature stability (both chemical and mechanical), good catalytic activity and high electronic and ionic conductivity. A suitable porosity for hydrogen diffusion and water migration is also important: the oxidation reaction occurs at the triple phase boundary (TPB), which represents the meeting point of electronic, ionic and gaseous phases [2]. Moreover, a good thermomechanical compatibility with the electrolyte layer must be guaranteed through a negligible mismatch between the coefficient of thermal expansions of the two materials. Otherwise, residual mechanical stresses could lead to crack formation, and consequent propagation, at the anode-electrolyte interface. State of the art material for the anode electrode consists of Nickel/YSZ cermet. YSZ assures an adequate porosity (around 30 %) to the electrode while providing an adequate ionic conductivity. Nickel particles, which provide the catalytic activity for the fuel oxidation reaction, are spread in the ceramic network [2].

Cermet anodes are anyhow subjected to long term deterioration, mainly resulting from nickel particles agglomerations [6]. In response, multilayered anodes are being developed, characterized by a graded porosity [7]: beside presenting a higher long term stability, they also assure adequate fuel oxidation and thermomechanical compatibility. Generally, a traditional cermet layer is employed, to guarantee a sufficient porosity for molecular diffusion as long as catalytic activity. A second thinner and denser layer is paired, facing the electrolyte interface, assuring high electrochemical activity and minimizing the mismatch in the coefficients of thermal expansion.

- The cathode should exhibit similar properties to the anode. A good electronic conductivity is necessary for the transport of electrons from the external circuit to the triple phase boundary [2]. Suitable oxide ions conductivity should be guaranteed. Temperature stability, sufficient porosity and a good catalytic activity for the reduction of oxygen molecules are also important. Same considerations with respect to the anode should be done regarding the coefficient of thermal expansion.

State of the art for cathode electrodes involve perovskite materials (ABO_3 structure), which are mixed ionic and electronic conductors (MIEC). The most common one is $La_{0.8}Sr_{0.2}MnO_3$ (LSM), where Sr doping is employed to increase electronic and ionic conductivity. However, the trend for solid oxide cells is going towards a reduction of the operating temperature, where LSM presents limited ionic conductivity and catalytic activity [2]. Therefore, $La_{1-x}Sr_xCoO_{3-\lambda}$ are also taken into account: they provide a very good oxide ions conductivity as well as a high electronic conductivity. The main drawback is the production of zirconates when YSZ electrolytes are employed, leading to a reduction of conductivity at the cathode/electrolyte interface [2]. Other

possible solutions involve $La_{1-x}Sr_xFeO_{3-\lambda}$ (LSF).

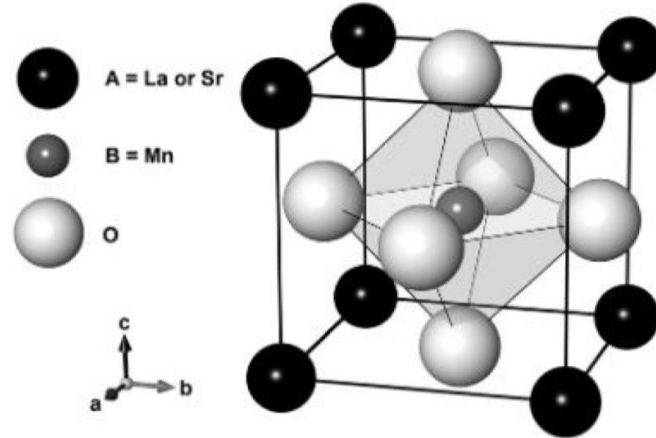


Figure 1.4: Perovskite cubic structure $La_{0.8}Sr_{0.2}MnO_3$ of cathode electrode [2]

1.2.4 Advantages and disadvantages

Solid oxide fuel cells, as a consequence of their high operating temperature, experience both advantages and disadvantages with respect to low temperature cell technologies.

As previously mentioned, the kinetic of charge transfer benefits a lot of the high temperature: this results in a less expensive catalyst (Nickel) compared to low temperature fuel cells. Moreover, nickel catalyst is more tolerant with respect to molecules containing carbon, which allows an higher fuel flexibility. Therefore, differently from low temperature cells, SOFC can work with a large variety of fuels, such as H_2 , CH_4 , NH_3 , biogas, syngas.

Another advantage is the availability of high quality heat, which can be exploited for many applications.

SOFC are also characterised by an higher electrical efficiency and they can work in reverse mode, as solid oxide electrolyzers: in fact, same materials are employed for SOECs and SOFCs.

On the other hand, there are also some drawbacks. More expensive materials, high temperatures resistant, are needed; an higher degradation rate (and a shorter life expectancy) is present as a consequence of the high temperature. Finally, long start-up and shut-down times are required, making SOFC technologies not suitable for load-following applications (eg. transports).

1.3 Solid oxide cell tests and characterization

1.3.1 Test configuration

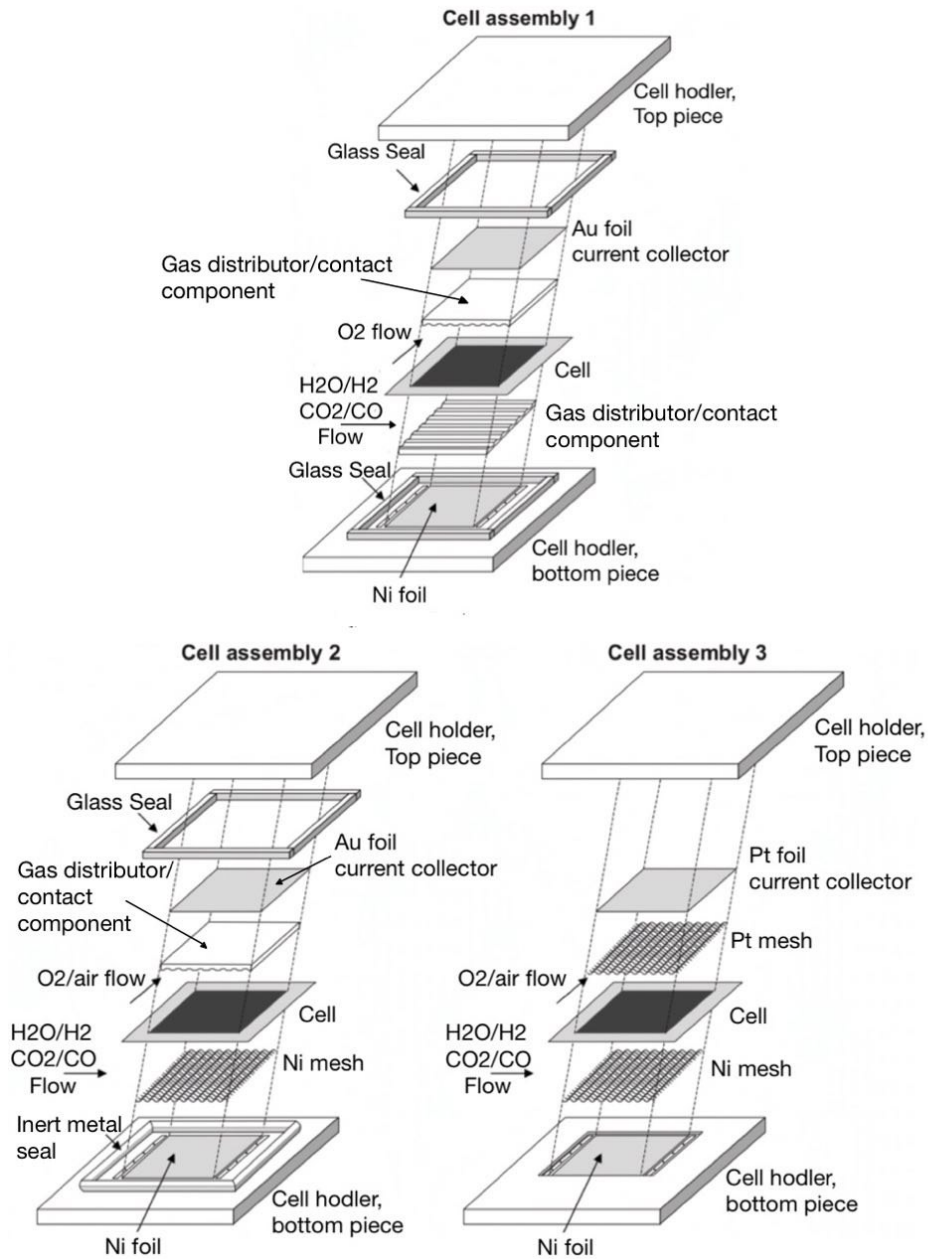


Figure 1.5: Three possible assemblies for planar single SOFC testing [8]

Laboratory's test bench is devoted to test and characterization of planar solid oxide cells. Cell's testing is of fundamental relevance, both commercially and technologically. It allows to obtain information on cell's performance, sources of losses, on long term response and detrimental phenomena. A proper testing facility is necessary to carry out the required experiments. A common set-up configuration can be found in literature [8] [9], as shown in figure 1.5, and will be discussed in this chapter, although small changes can be introduced.

The SOFC is usually placed in between two distributor plates: to allow current collection the fuel plate is in contact with a Nickel foil while the air plate is in contact with a platinum/gold foil. Gas sealing are employed to ensure gas tightness and prevent gas mixing. Everything is sandwiched in between two blocks of alumina, which is an insulating material, equipped with proper inlet/outlet channels for gas supply. The fuel mixture can be previously humidified through a water bubbler or through an evaporator. The test house is enclosed in a furnace, allowing to reach and maintain cell's operating temperature.

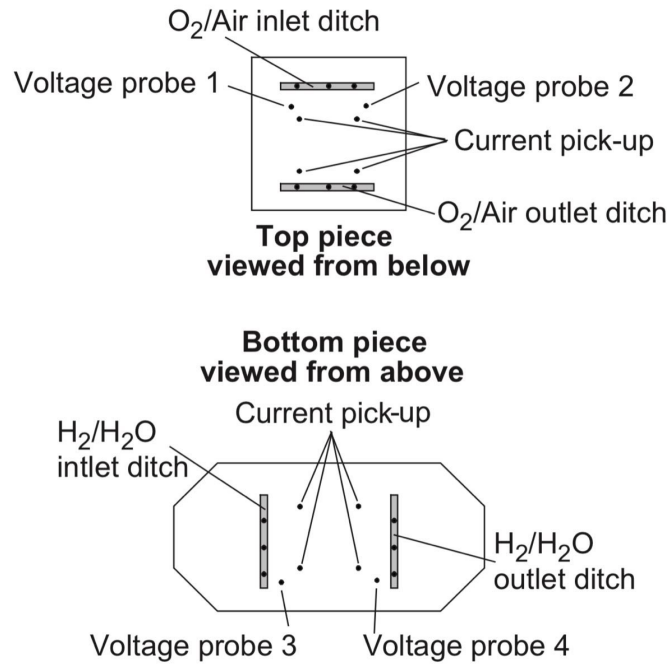


Figure 1.6: Configuration for voltage and current probe for planar SOFC testing [8]

The correct monitoring of cell's voltage and current is essential for SOFC's tests and characterization. As shown in figure 1.6, it can be useful to place four voltage

probes: in this way, both cell's voltage and in-plane voltages can be detected. Cell's voltage is given by the voltage difference between the two electrodes of the cell: monitored by probes 1-3 at the inlet and by probes 2-4 at the outlet. In-plane voltage is instead given by the voltage difference inside a single electrode: monitored by probes 1-2 for the air electrode and by probes 3-4 for the fuel electrode. The in-plane voltage is small compared to the cell's voltage, but it is useful to detect an uneven current distribution in the electrodes.

In figure 1.6 also current pick-up points positioning is illustrated. In correspondence to the current pick-up points, the meshes are welded to current probes, which are connected to an external electronic load. These probes, made of platinum, ensure high current conduction at elevated temperature, without being affected by oxidation or deterioration.

Cell's temperature monitoring is also necessary. Thermocouples are placed in a similar way to the voltage probes, thanks to proper holes drilled in the alumina blocks. Therefore, they are not in direct contact with the electrodes: ideally, temperature should be measured exactly on the site of the electrochemical reaction. For this reason, a small temperature error arises, which should be taken into account when accurate results are needed.

An alternative way to compute the actual cell's temperature can be found in literature [8]. An illustrative example is reported in figure 1.7: impedance spectra (which will be analysed in the following section) are recorded with an interval of 0,1 [A/cm^2], allowing to derive the ohmic resistance. Knowing materials conductivity and ohmic resistance, the temperature difference between the thermocouple site and the electrode active site is computed. The difference between measured and computed temperature is derived for both electrolysis and fuel cell mode.

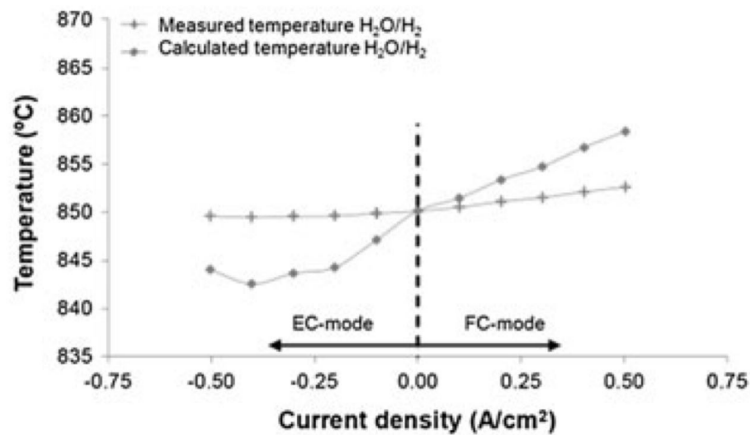


Figure 1.7: Difference between measured temperature and temperature computed by considering the ohmic resistance obtained via impedance spectroscopy [8]

1.3.2 Standard tests and characterization of SOCs

When the set-up configuration is ready, and before testing the cell, it is necessary to carry out a reduction of the fuel electrode of the SOC (NiO/YSZ): from nickel oxide to metallic nickel [2]. Even though many procedures are available in literature, a common path consists in feeding the fuel electrode with a small amount of H_2 (4-10 %) diluted in air. The cathode is instead fed with pure air. Gradually, the H_2 percentage at the anode should be increased, until pure H_2 mixture is obtained. At the same time, open circuit voltage of the cell should be monitored: when reduction is completed, OCV should be close to the theoretical Nernst potential. It should be underlined that the Nernst potential will never be reached, due to imperfect sealing and to small currents crossing the electrolyte (which is not an ideal ionic conductor).

At this point, the actual test and characterization procedures can be implemented.

- **Polarization test** is commonly used to assess cell's performance at different operating conditions. The aim of the test is to obtain the current-voltage characteristic of the cell. Starting from Open Circuit Voltage condition (corresponding to 0 [A]), the current set point must be gradually increased with small and regular steps, until voltage threshold limit is reached (e.g around 700 [mV]). Beneath the voltage threshold, deep polarization may cause irreversible deterioration of the cell [10]. Each step should last for at least 60 seconds, by operating the electronic load devoted to the cell in galvanostatic mode (e.g constant current). At each current level, the average voltage value is measured for the polarization curve computation.

For each step, also cell's power can be computed, by multiplying current and voltage, and current-power curve can be built. By dividing current and power for the active surface of the cell, current density and power density can respectively be obtained.

Other important quantities that can be derived from the polarization test are *Fuel Utilization factor FU*, *Oxidant Utilization factor OU* and *Area Specific Resistance ASR* [10]. When hydrogen is used as fuel media, these quantities can be computed as follow:

$$FU = \frac{Q_{H_2,react}}{Q_{H_2,in}} = \frac{I \cdot 22.414 \cdot 60 \cdot 1000}{2F \cdot Q_{H_2,in}} \quad (1.6)$$

$$OU = \frac{Q_{O_2,react}}{Q_{O_2,in}} = \frac{I \cdot 22.414 \cdot 60 \cdot 1000}{2F \cdot 0,21 \cdot Q_{air,in}} \quad (1.7)$$

$$ASR = \frac{OCV - V}{i} \quad (1.8)$$

where: Q_{react} are the reacted volumetric flow rates expressed in [Nml/min] and computed though the Faraday Law; Q_{in} are the actual volumetric flow

rates sent at the anode/cathode compartments; I is the cell's current; F is the Faraday constant (96,485 [C/mol]); OCV is cell's open circuit voltage; V is the cell measured voltage at the current density i .

FU and OU express respectively the fraction of spent fuel and reacted oxygen with respect to the total inlet supplies. ASR , expressed in [Ωcm^2], is a performance indicator for SOCs: it provides the slope of the polarization curve normalized on the active surface of the cell.

It can be meaningful to carry out a parametric analysis, by computing the polarization curve after changing one parameter at a time (and keeping constant the rest), to assess and compare cell's performances under different operating conditions [10]. The main parameters on which one can act are: hydrogen content at the fuel electrode (keeping constant the total flow rate), air flow rate at the air electrode, operating temperature (according to cell's operative range) and humidification rate.

- **Electrochemical Impedance Spectroscopy** (EIS) allows to break down the overall loss of the cell into its different contributions, also enabling to understand which component is firstly subjected to deterioration and under which conditions. Therefore, EIS is a powerful tool in investigating losses and degradation processes in solid oxide cells, which is the first step towards performance and durability improvements [11]. It is usually coupled with the polarization test for the characterization of SOCs.

EIS technique relies on the principle for which different loss mechanisms take place at different frequencies [2]. When applying an AC current to the SOC:

$$i(t) = I \cdot \sin(\omega t) \quad (1.9)$$

an AC voltage response can be detected:

$$v(t) = V \cdot \sin(\omega t + \phi) \quad (1.10)$$

Where: ϕ is the phase shift between AC voltage and current, ω is the frequency, V is the voltage's amplitude and I is the current's amplitude. It is possible to compute the AC impedance as:

$$Z(\omega) = \frac{u(t)}{i(t)} \quad (1.11)$$

It is a complex number, which comprises a real part (a resistance) and an imaginary part (a reactance). By plotting the imaginary part versus the real part, the Nyquist plot can be obtained, which is the main result of the EIS

technique. As visible from figure 1.8, the impedance spectra reported in the Nyquist plot is made up of different arcs, each one with a peak at a different frequency (usually indicated with an arrow). In the figure, it is also possible to distinguish the two contributions in which the total resistance of the SOC can be split: the pure resistive ohmic resistance (with no imaginary part) and the polarization resistance [4].

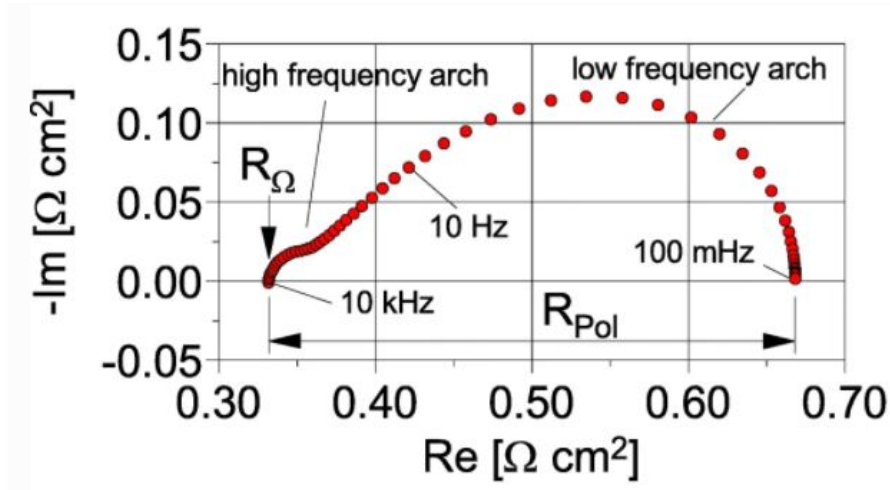


Figure 1.8: Example of a Nyquist plot, highlighting ohmic and polarization resistance [12]

A Bode plot should also be included, reporting the frequencies (in logarithmic scale) on the abscissa and both the impedance's modulus and phase shift on the ordinate [11].

It is common use to adopt an equivalent circuit (ECM) to model the impedance spectrum of the Nyquist plot. The circuit's electrical components (e.g resistances, capacitors, inductors) are used to represent complex loss mechanisms. In the left part of figure 1.9, a simple equivalent circuit is represented, for illustrative purpose [2]. The circuit is made up of a resistance R_e connected in series with two RC elements. Resistance R_e primarily accounts for the electrolyte losses. Each one of the two RC elements represents an arc on the Nyquist plot (represented in the right part of the figure), and their sum gives the polarization resistance of the cell. The arcs of the Nyquist plot correspond to activation phenomena, reactions at the anode/cathode, gas conversion and gas diffusion. Therefore, each RC element accounts for one of these phenomena.

More complex and detailed equivalent circuits are reported in literature. In place of the capacitor, a constant phase element (CPE) is usually connected

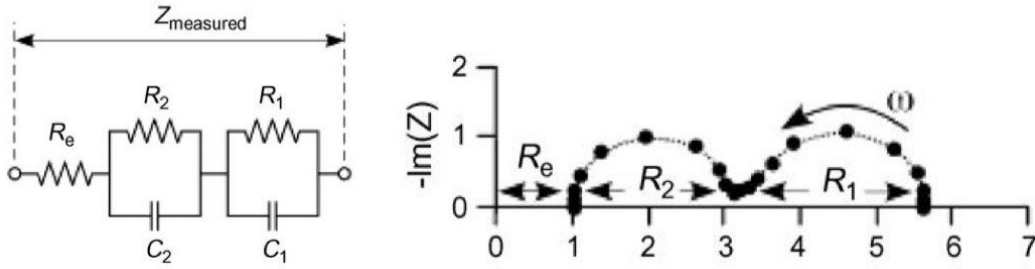


Figure 1.9: Example of a simplified equivalent circuit (left) referred to a Nyquist plot (right) [2]

in parallel with the resistance, allowing to represent inhomogeneous systems. In figure 1.10 the ECM presented by Barford et al. [2] is shown.

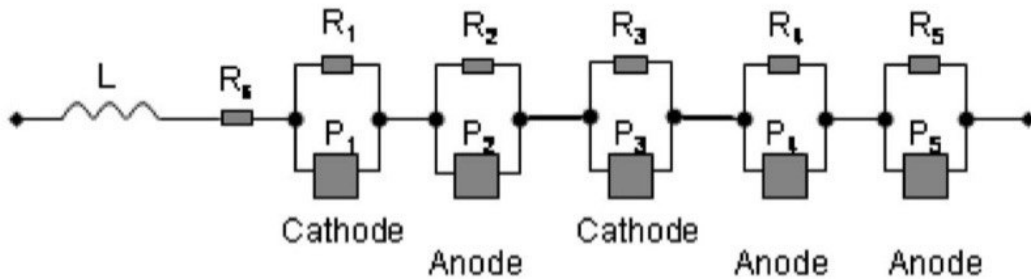


Figure 1.10: Equivalent circuit proposed by Barford et al. [2]

Five constant phase elements (P_i) are present, each one connected in parallel with a resistance (R_i); an inductor (L) and a resistance (R_s) connected in series are also present.

Elements RP_1 and RP_3 are related to cathode's loss mechanisms: the first one represents the losses derived by the transfer of O^{2-} ions at the electrolyte/cathode interface; the second one deals with the oxygen dissociative adsorption and transport to triple phase boundary [2]. Elements RP_2 , RP_4 and RP_5 are related to anode's loss mechanisms: respectively, the first one is associated with losses due to electrochemical polarization, the second one with gas diffusion and the third one with gas conversion [2]. Inductor L represents the inductance of the measurements leads, while resistance R_s is associated to the electrolyte's ohmic resistance.

The highest frequency arc corresponds to the first RP element and the lowest

one to the last one.

When performing an EIS, two main aspects should furtherly be considered [8]:

- Measurements should be performed in a frequency window ranging between 0.1 Hz and 100 kHz, with a sufficient number of recorded points (e.g about 10-15 points per decade). Moreover, electronic noise derived from electronic devices of the test bench should be minimized by correctly setting the frequency.
- Different operative conditions should be analysed in order to derive useful information about loss mechanisms. When changing a parameter, the others should be kept constant.

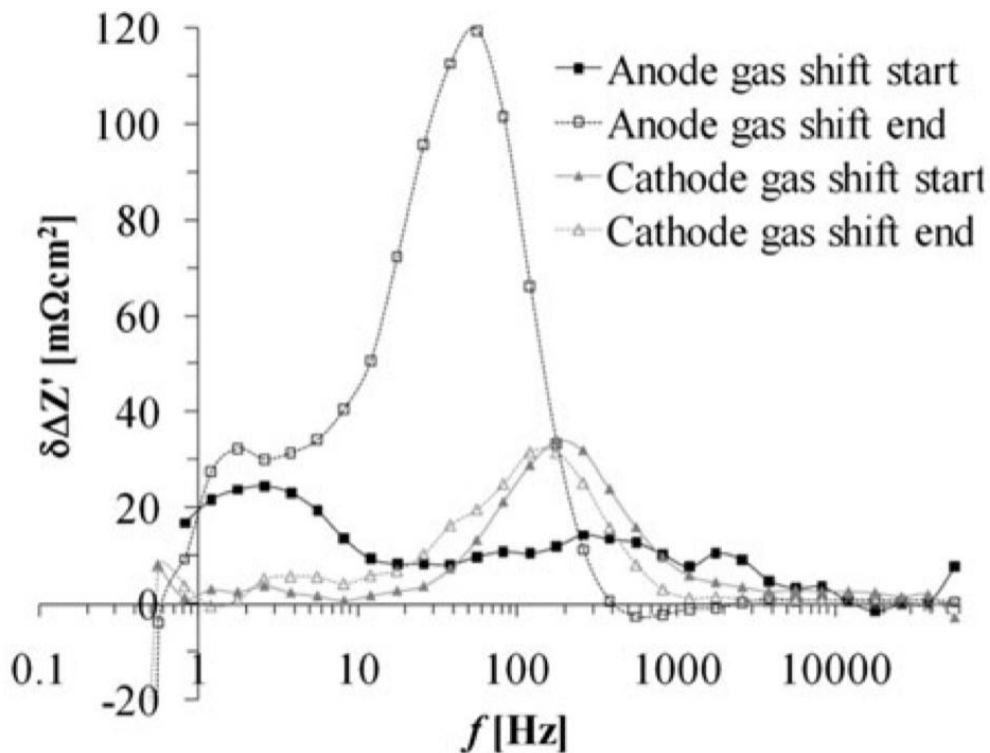


Figure 1.11: ADIS: gas composition changed at one electrode at a time (at OCV at 750 [°C]) before and after a long term SOFC test [8]

Changing the operational temperature while keeping constant the supply-gas compositions enables to distinguish thermally activated processes (e.g electrochemical processes taking place in the electrodes) from non-thermally activated ones (e.g gas diffusion and other physical phenomena). On the other hand, changing the supply-gas composition while keeping

constant the temperature is useful to highlight how much each loss is affected by the inlet composition. Moreover, changing the gas-composition of one electrode at a time underlines which contributions are related to which electrode. In figure 1.11, an example of "Analysis of differences in impedance spectra" (ADIS) is reported for illustrative purpose. In particular, gas-composition is changed at the cathode before and after a long-term test; same procedure is repeated for the anode. Gas shift impedance spectra are recorded at 750 [°C] at open circuit voltage, before and after the long-term test. For the cathode, the two recorded impedance spectra are quite close. For the anode, a remarkable difference is visible among the first-recorded impedance spectra and the post-test one. This allows to deduct that, differently from the cathode, the anode has strongly suffered the long-term test.

In figure 1.12 the total resistance break down into its different contributions is represented, as a result of an EIS analysis.

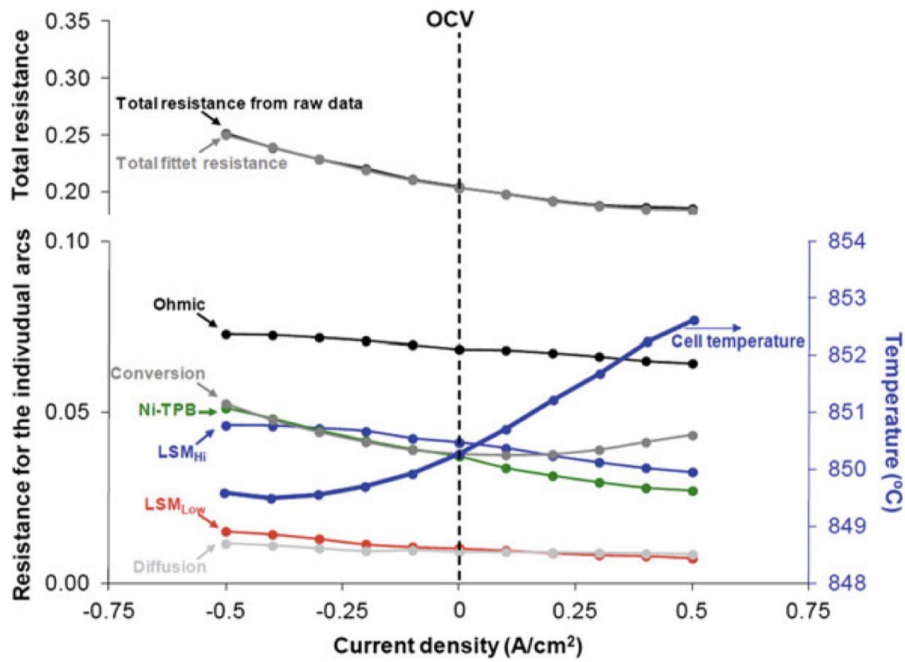


Figure 1.12: Total resistance break down into its different contributions as a function of current density; results of an EIS analysis conducted at 850 [°C] for an anode supported cell Ni/YSZ|YSZ|LSM/YSZ, with a fuel mixture of 50% H_2 – 50% H_2O [8]

EIS analysis for full SOC does not always allow to split the total loss into

all its contributions, due to impedance arc overlapping or poor quality of the spectra. Anyhow, it is always possible to extract information on the *total* polarization resistance and on the ohmic resistance.

- **Galvanostatic and Potentiostatic tests** are durability tests, useful to check cell's stability, durability and degradation over time.

In the galvanostatic test, a steady state DC current set point is imposed for a certain amount of time: both short-term (e.g around 200 consecutive hours) and long-term tests (e.g around 3000 consecutive hours) are possible. A constant voltage level during the whole time of the experiment proves cell's performance stability; otherwise, a voltage drop would be present in SOFC operation mode, highlighting a degradation rate. Higher the slope of the curve, higher the degradation rate. Before and after the galvanostatic test, a polarization test should be performed: in absence of performance degradation, the two polarization curves should overlap.

In the potentiostatic test, a steady state DC voltage set point is imposed, and similar conclusion to the galvanostatic test can be drawn by observing the current trend.

It can be meaningful to compute the Area specific resistance over time, allowing to compare the two test methodologies [13]. When a degradation rate is present, the ASR value will increase over time.

- **Pressurization test.** A pressurization test can be performed by introducing a variation to the previously described test configuration.

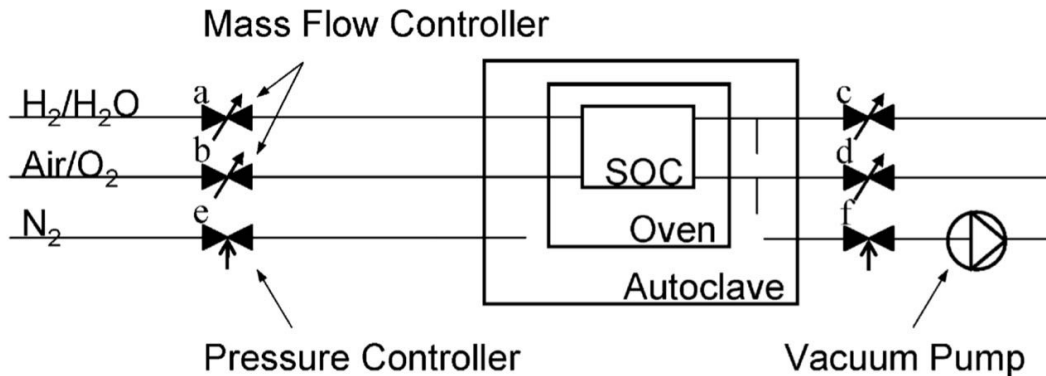


Figure 1.13: Schematic configuration and main components for the pressurization test of single planar SOFC [8] [14].

As reported in literature [14] and visible in figure 1.13, the oven containing the test house is placed inside an autoclave filled with flowing N_2 . Two pressure

controllers (e and f) are used to control the pressure inside the autoclave. Mass flow controllers a and b are used to control the supply gas flow rates towards SOC's electrodes. Mass flow controllers c and d assure that the autoclave and electrodes have the same gas pressure (SOC's exit lines are opened to the autoclave environment). With this test configuration it is possible to test cell's performances with gas pressure between 0.4 and 10 bar (absolute) at 750 [°C]. For tests with less than 1 [bar], a vacuum pump, connected to the autoclave exit line, is used in place of mass flow controllers c and d, allowing the gases of SOC's electrode to mix in the autoclave.

The cell can be tested at different pressures, by performing polarization curves and EIS analysis: OCV, ASR, power density values can be derived.

In figure 1.14 an example of polarization curves under different pressure conditions are reported [14].

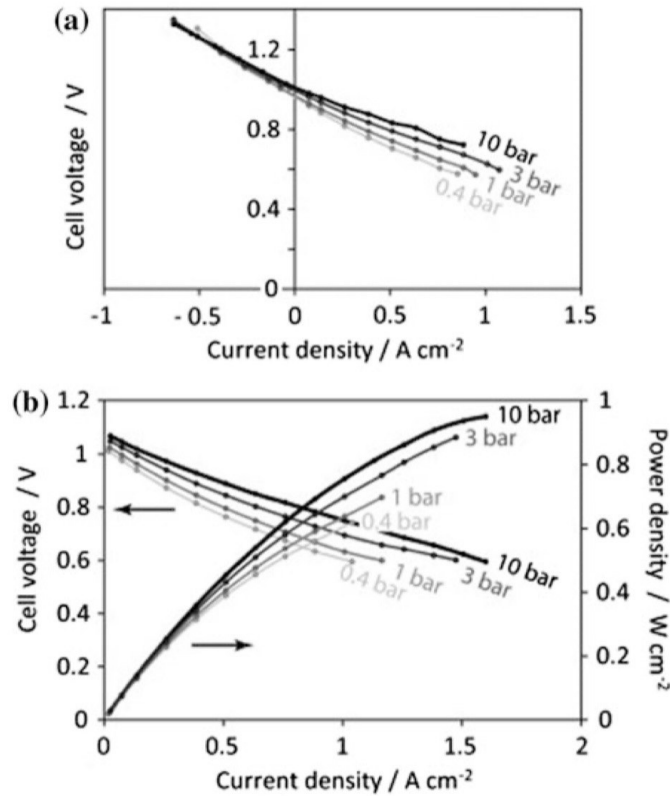


Figure 1.14: Results of a pressurization test, reporting polarization (a) and power density (b) curves as a function of pressure [8] [14]

- **Impurities test.** Cell's degradation may occur as a consequence of the presence of impurities in the anode/cathode compartments. Impurities may have different sources, not always easy to detect: among them, the feeding gases themselves, which are hardly totally pure. Impurities segregation may occur at the electrode's triple phase boundary, causing its blockage and, consequently, an increment of cell's polarization resistance [15].

In literature [8] [15] it is possible to find examples of tests aimed at investigating the impact of supply gases containing impurities. Cell's durability is firstly tested with the as-received gas. Afterwards, the test is repeated under the same operating conditions but with cleaned gas, analysing the differences.

In figure 1.15 an example is reported. A galvanostatic durability test (of about 600 consecutive hours) has been performed in electrolysis operation mode: the red line represents the voltage trend when SOEC's fuel electrode is fed with the as-received gas mixture (CO_2 -CO). Blu line represents the voltage trend with clean gas supply. As it is clearly visible, the red line shows a voltage increment, and so a degradation rate, which is not present in the blu line, although same test conditions were applied. In fact, the as-received gas, analysed through gas chromatography, showed a 20 ppb sulfur concentration: although small, this sulfur content is sufficient to induce a performance degradation, as highlighted from the presented graph.

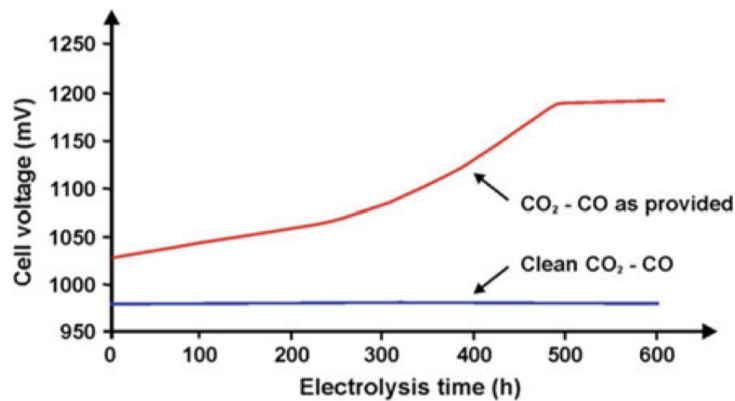


Figure 1.15: Durability test (galvanostatic) of SOEC fed with gas containing 20 ppb of sulphur (red line) and with cleaned gas (blue line)

- **Other characterization techniques.** The above mentioned tests and characterizations can be combined with ex situ tests. The previously described experiment configuration (and the test bench of *Politecnico di Torino*) does not allow to perform these tests, which can give important complementary

information. For this reason, these ex-situ tests will not be explored. Anyhow, among them, Scanning electron microscopy (SEM) is worth-mentioning: it allows to analyse solid oxide cells on a nano-scale, useful to examine degradation and aging effects [8]. As reported in literature [8], SEM can be combined with focused ion beam, allowing to reconstruct 3D electrode structures: triple phase boundary lengths, tortuosity and other detailed information can be extrapolated.

Chapter 2

Components and Connections of the Test Bench

2.1 Schematic diagram and description

The Solid Oxide Cell characterization requires a proper testing environment: the test rig under analysis is intended for this purpose. In this second section, a technical description of the test bench is given, in order to provide a user's guide about different components, their physical connections and, eventually, operation and maintenance.

Referring to the schematic diagram of figure 2.1, it is possible to identify the main subsystems of the test rig:

- **Gas distribution lines**, to allow the flow of CO , CH_4 , H_2 , CO_2 , N_2 and air from the laboratory gas manifolds towards the fuel cell compartments;
- **Mass Flow Controllers**, to allow the control and monitoring of the flow rates, in order to get the desired mixtures;
- **Controlled Evaporator Mixer**, which can be used to humidify the gas flow before entering the cell;
- **Water distribution line**, used eventually for humidification;
- **Oven**, which involves also the housing of the fuel cell, used to reach and maintain the desired operating temperature while hosting the cell;

- **Electronic Auxiliaries**, one dedicated to the oven and one dedicated to the cell;
- **Data Acquisition and Control System and Operator interfaces**, used to control and monitor the test bench's instruments.

All the above mentioned subsystems and components will be described more in detail in the following sections. Moreover, a piping and instrumentation diagram of the system is provided in the Appendix A.

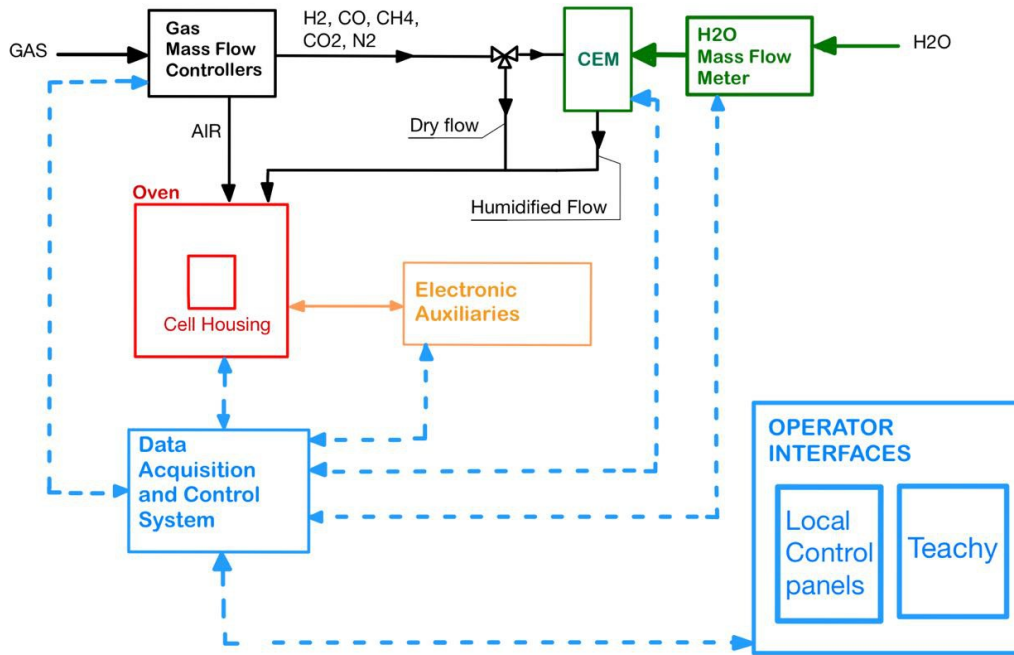


Figure 2.1: Test bench main subsystems and connections [4]. Continuous lines represent physical flow exchange, dashed lines represent data exchange

2.2 Mass Flow Controllers and Gas Distribution Lines

2.2.1 Description

Mass flow controllers (MFCs) and gas distribution lines are exploited to send the desired gas mixtures to the cell's compartments. *Bronkhorst EL-FLOW* mass flow controllers [16], designed especially for laboratory purposes, are employed in the

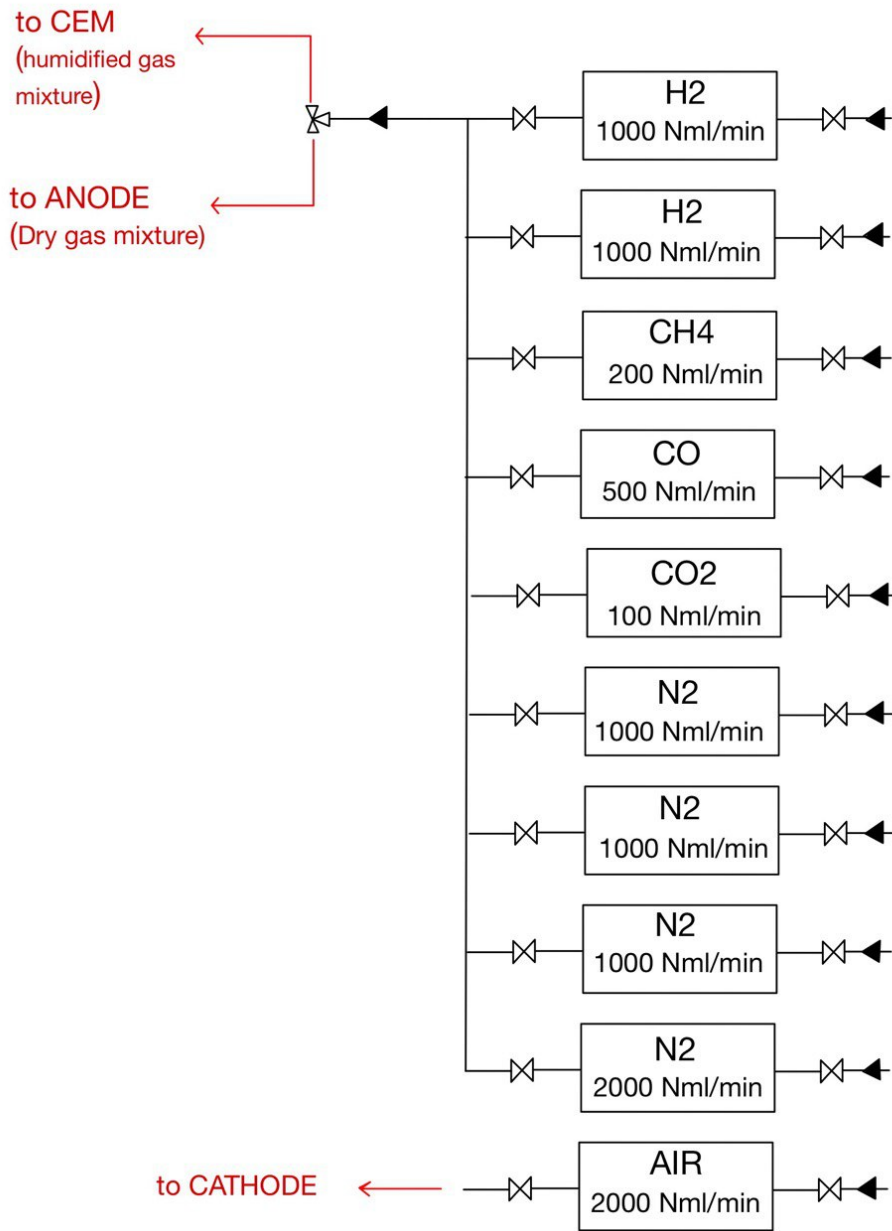


Figure 2.2: Mass Flow Controllers connections

test bench. In particular, as visible from the schematic diagram of figure 2.2, there are ten gas flow meters:

- **MFCs for Fuel gas mixture.** Nine are devoted to control H_2 , CH_4 , CO ,

CO_2 , N_2 flows. Downstream, they are connected to the same distribution line, allowing to create the gas mixture to be sent at the anode compartment, in SOFC operation mode. Before entering the cell, a three-way valve is present on this line, allowing to decide weather to humidify or not the fuel gas mixture.

- **MFCs for Air flow.** The last one controls the air flow sent to the cathode in SOFC operation mode. Downstream, it has its own distribution line, heading directly to the cell.

Upstream, the gas flow controllers are connected to laboratory's gas manifolds, through proper stainless steel pipes (6 mm). A pressure reducer is present between the laboratory's distribution line and the test-bench lines. In fact, the gas coming from the laboratory has a pressure of 30 [bar], much higher than the MFCs operating pressure. Manually-controlled valves are positioned both upstream and downstream of each gas MFCs.

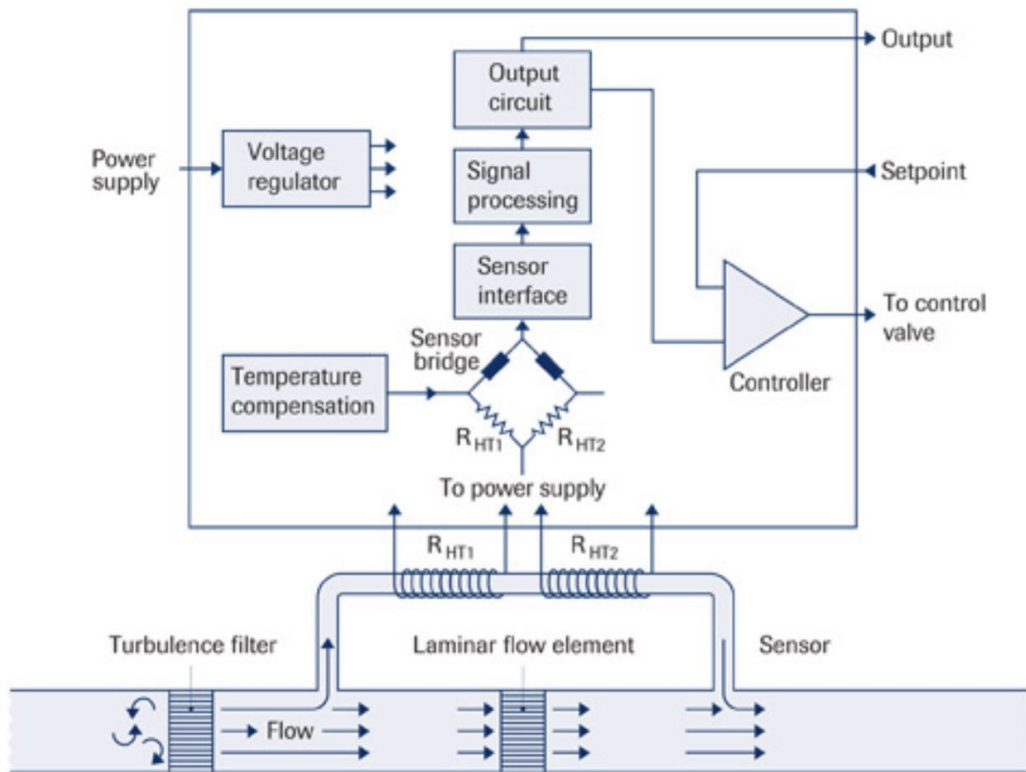


Figure 2.3: Mass flow controllers working principle [17]

The measuring principle of Bronkhorst mass flow controllers relies on an a small bypass tube, equipped with two heating elements and two temperature recorders

[17]. Only a fraction of the gas stream flows through this tube and it is heated up through the two heaters, as shown in figure 2.3. The temperature difference between the two heating elements is measured: it is proportional to the total flow rate; higher the flow rate higher the temperature difference. Therefore, the total flow rate is directly derived and its value is sent to the microprocessor of the MFC, where it is compared to the set point value imposed by the operator. According to the difference between the measured value and the set point value, the position of the electro-magnetic control valve is adjusted in few milliseconds: the desired flow rate is obtained.

2.2.2 Specifications and safety requirements

Each mass flow controller is designed to operate with a specific gas and in a certain range of flow rates. The internal sealing materials are compatible with the specific gas: before using different gas media it is important to check the compatibility with the internal seals. For this purpose, the operator should refer to the table in figure 2.4, from Bronkhorst instruction manual [18]. The sealing material integrated in each MFC can be checked by using the specific serial number.

Gas	Sealing material		
	Viton®	EPDM	Kalrez®/FFKM
Air	✓	✓	✓
H2 (Hydrogen)	✓	✓	✓
CH4 (Methane)	✓	✗	✓
CO (Carbon monoxide)	✓	✓	✓
CO2 (Carbon dioxide)	max 10 bar(g), 50°C	✓	max 10 bar(g), 50°C (ED compound ¹ max 30 bar(g))
N2 (Nitrogen)	✓	✓	✓

ED: Explosive Decompression resistant

Figure 2.4: Gas compatibility with different sealing materials [18]

Moreover, the functional properties reported in the label of each mass flow controller must match with their specific application. Table 2.1 reports the main specifications for each mass flow meter.

No particular maintenance is recommended if the mass flow controllers are operated properly, avoiding thermal/pressure shocks and vibrations and using clean media. More accurate results can be obtained after a warming time of 30 minutes.

Proper checks have been performed in order to detect possible gas leakages along the lines, valves and connections: each line was closed and pressurised, verifying

Table 2.1: Mass Flow Controllers specifications

Name	Media	Range [Nml/min]	Upstream p [bar]	Downstream p [bar]
MFC1	H_2	0-1000	3	0
MFC2	H_2	0-1000	2.1	1
MFC3	N_2	0-1000	2.1	1
MFC4	N_2	0-2000	2.1	1
MFC5	AIR	0-2000	3	0
MFC6	N_2	0-1000	2.1	1
MFC7	N_2	0-1000	3	0
MFC8	CH_4	0-200	3	0
MFC9	CO_2	0-100	2.1	1
MFC10	CO	0-500	2.1	1

that the pressure did not decrease over time. It is advisable to do a periodic check since leakages must be avoided while dealing with potentially harmful gas (such as H_2 , CH_4 , CO): Swagelok joints assure a good performance for this purpose. Moreover, a gas detection and alarm system is installed in the laboratory: in case of dangerous leakage, an acoustic and visual alarm is produced along with electricity supply interruption.

The test bench has been found to respect the regulation in terms of security. Consequentially to an ATEX analysis [19], potentially dangerous zones have been detected in proximity to upstream and downstream valves of the MFCs dealing with H_2 , CH_4 and CO. It has been prescribed to avoid the presence of any electrical component (MFC included) inside the dangerous zones. In figure 2.5 it is possible to verify the observance of this prescription.

The dangerous zones have been positioned and represented in accordance to the ATEX analysis: in particular, their shape and magnitude depend on the gas media crossing the valve and on its relative pressure. Concerning H_2 , the dimensions and volumes of upstream and downstream dangerous zones, prescribed by ATEX, are respectively represented in figures 2.6a and 2.6b.

Concerning CH_4 , the dimensions and volumes of upstream and downstream dangerous zones, prescribed by ATEX, are respectively represented in figures 2.7a and 2.7b.

Finally, concerning CO, the upstream dangerous zone has a spherical shape with a radius of 0,057 [m], while the downstream dangerous zone has a negligible extension. All upstream dangerous zones have an higher volume compared to the downstream zones due to the higher relative pressure of the gas: around 2,5 [bar] upstream

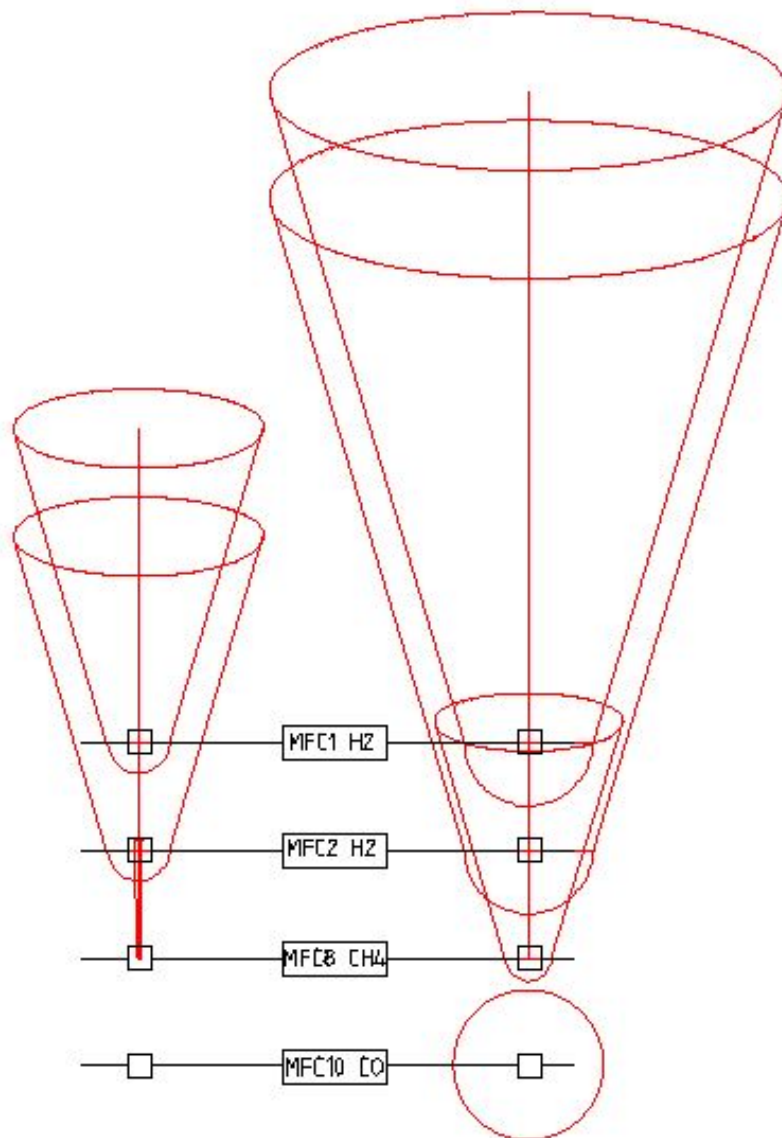
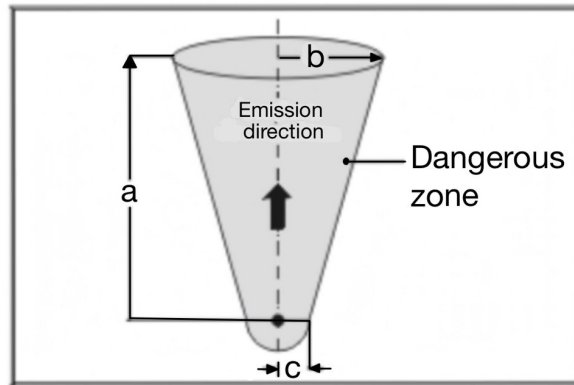


Figure 2.5: ATEX dangerous zones

versus 0,02 [bar] downstream.

As noticeable from figure 2.5, the mass flow controllers do not come in contact with any dangerous zone.

For more detailed information, ATEX document can be consulted on request, see Appendix B.



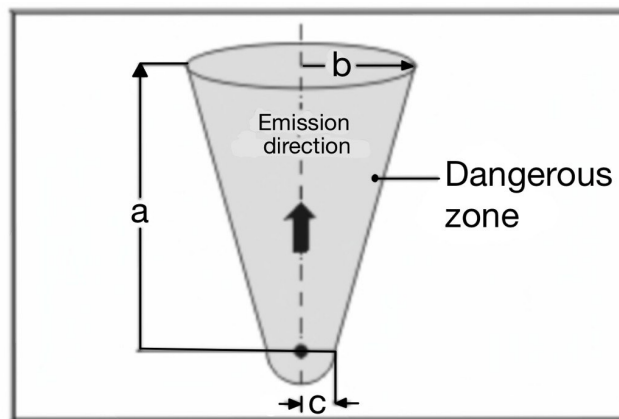
a [m]: 0,494

b [m]: 0,197

c [m]: 0,049

Volume dangerous zone [m³]: 0,02670727

(a) *Upstream dangerous zone: a zone 2 is determined, with a conical shape facing up and with dimensions specified in figure*



a [m]: 0,238

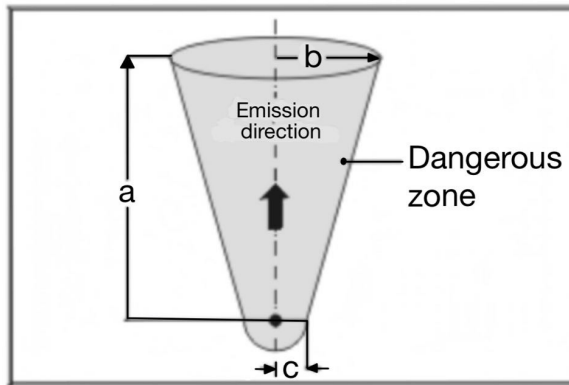
b [m]: 0,095

c [m]: 0,024

Volume dangerous zone [m³]: 0,002982168

(b) *Downstream dangerous zone: a zone 2 is determined, with a conical shape facing up and with dimensions specified in figure*

Figure 2.6: Hydrogen dangerous zones, prescribed by ATEX analysis



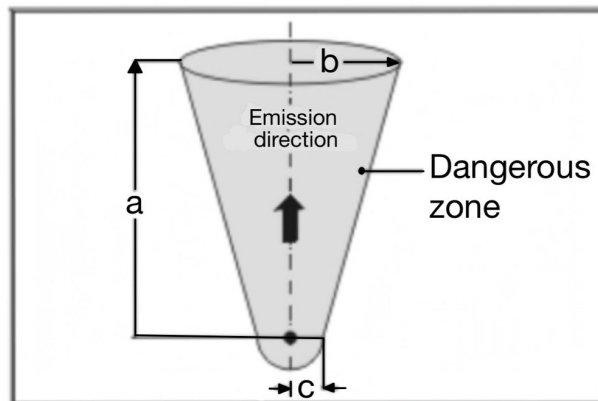
a [m]: 0,179

b [m]: 0,072

c [m]: 0,018

Volume dangerous zone [m³]: 0,001283005

(a) *Upstream dangerous zone: a zone 2 is determined, with a conical shape facing up and with dimensions specified in figure*



a [m]: 0,094

b [m]: 0,037

c [m]: 0,009

Volume dangerous zone [m³]: 0,0001820498

(b) *Downstream dangerous zone: a zone 2 is determined, with a conical shape facing up and with dimensions specified in figure*

Figure 2.7: Methane dangerous zones, prescribed by ATEX analysis

2.2.3 Mass Flow Controller's Calibration

In order to assess the percentage errors of each mass flow controller, *Definer 220* flow meter was employed. The instrument is a volumetric flow meter, which implements a positive displacement gas flow measurement technology (DryCal) [20]. It uses a frictionless piston in a measuring tube. During the measurement, the gas is diverted to the flow measuring tube, entering beneath the piston, which moves with the flow stream. The flow is determined by measuring the time the piston takes to move along the tube of known length (optical measure).

Definer 220 was connected downstream, in series with the tested MFC: a flow rate was set by the operator and the values measured by the mass flow controller itself and by *Definer 220* flow meter were transcribed. The procedure was repeated for three different flow rates. Finally, the percentage errors were computed, in order to assess the instrument's accuracy (both in control and measure). In Appendix C, the results are reported for each Mass Flow Controller, with the exception of MFC9, which is currently not available.

Although some mass flow controllers were not as much accurate as others, the percentage errors have found to be in an acceptable range for this Test Bench purposes, especially when moving towards high mass flow rates.

When more than one mass flow controller is available for a single gas (as in the case of H_2 and N_2), it is advisable to use the most accurate one. Among the four mass flow controllers dealing with N_2 , MFC4 is the less precise one. For this reason, it has been selected to be used only in case of emergency, to send a mass flow rate of N_2 to the fuel cell (procedure that does not require too high accuracy).

More detailed information about gas mass flow meters can be found in Bronkhorst manual [18] and brochure [16].

2.3 Controlled Evaporator Mixer

2.3.1 Description and Connections

The Controlled Evaporator Mixer (CEM) is made up of three main components: a control valve, a mixing chamber and a heater. In the test bench under analysis, the CEM is integrated with a liquid flow meter and with the gas line, by means of a three-way valve. CEM allows water atomization and injection into the dry gas flow: the latter, acting as a carrier, transports the liquid water into the Temperature Controlled Heater, where water is completely vaporized. The produced humidified gas exits from the bottom of the evaporator. A temperature sensor is implemented to monitor the internal temperature of the heater, while a safety switch prevents overheating.

The liquid flow meter ([21]) has both measuring and control functions: it communicates with CEM's mixing valve. In this way, it's possible to control the water flow rate to be mixed with the gas flow. The employed water, coming from the laboratory manifold, is purified before entering the liquid flow meter at an absolute pressure of around 3 [bar].

The carrier gas, coming from the gas flow controllers, passes through a manually controlled three-way valve. When humidification is necessary, the dry gas is sent to the CEM's mixing chamber; otherwise, it is directly sent to the evaporator's outlet. CEM's schematic is represented in figure 2.8.

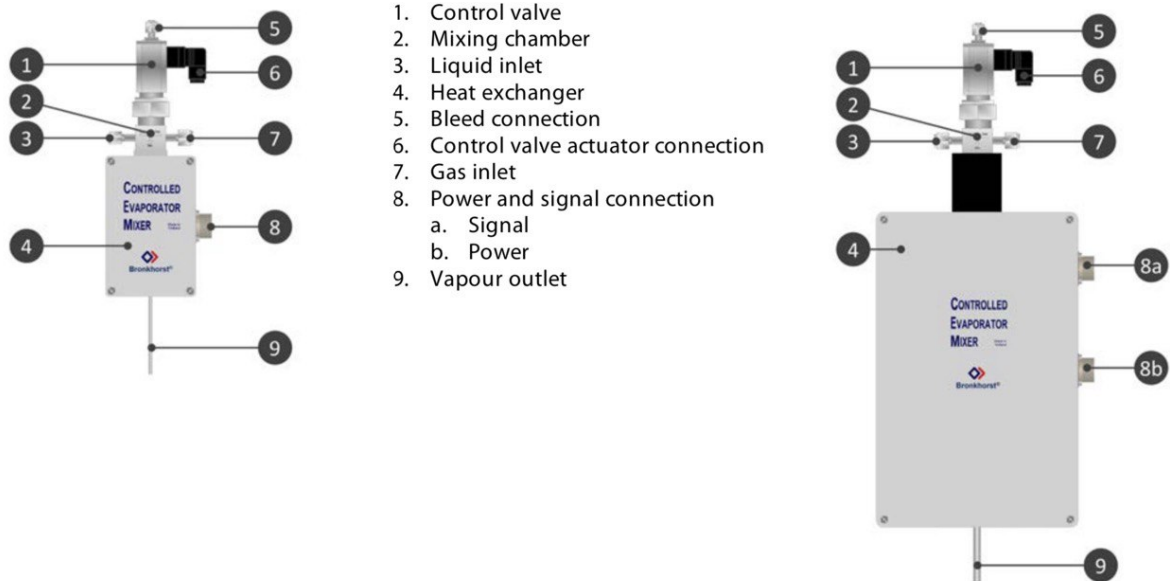


Figure 2.8: CEM's schematic [22]

2.3.2 Assembling procedure and main connections

According to CEM's Bronkhorst Manual [22], the basic assembly and connections are reported in this section, with reference to the specific components of the test bench.

The CEM has been mounted vertically, with the mixing valve in the top part and the vapour outlet pointing down. It's recommended to install it on a rigid and stable structure, far from mechanical vibrations and/or heat sources, by using proper mounting holes. At this point, it's possible to connect the CEM with the other components of the test bench:

- The outlet of the liquid flow meter has been connected with the liquid inlet of the CEM. The inlet of the liquid flow meter is instead connected to the water line.
- The gas inlet of the CEM has been connected with the outlet of the three-way valve, which is connected to the gas line coming from the gas flow controllers. In this way, when dry gas is needed, the three-way valve can be manually adjusted to by-pass the CEM system, sending the gas directly to the evaporator outlet.
- The evaporator outlet is connected to the dry gas line by means of a three-way junction and is then directly connected to the cell's feeding line. This portion of the gas line must be equipped with an external resistance, necessary to avoid condensation.
- By means of an actuator cable, the liquid flow meter has been connected to the mixing valve of the CEM.

2.3.3 Normal Operation

In this section, according to Bronkhorst manual, the usual procedure of CEM's operation is introduced. In fact, a proper operation of this device is recommended in order to avoid possible malfunctions. Therefore, any time a humidification of the fuel mixture is required, the following steps must be followed:

- At first, the required setpoint for the heater's temperature should be obtained through *FLUIDAT® on the Net* [23]. Thanks to this software, using the liquid and gas flow rates as input variables, it's possible to compute the Temperature needed to evaporate the liquid and to prevent vapour condensation at the outlet of the heater.
- Put the setpoints of the liquid flow meter and of all gas flow controllers to 0%
- Enter the previously computed Temperature setpoint for the heater.
- Pressurize the liquid and gas lines to the required values. The liquid pressure should never overcome the gas pressure, to prevent liquid from flowing towards the gas flow controllers.
- Bleed the mixing valve if necessary. See next section for bleeding procedure.
- Insert the required setpoints for the gas flows.
- Wait until both gas flows and heater's temperature have reached the set value.

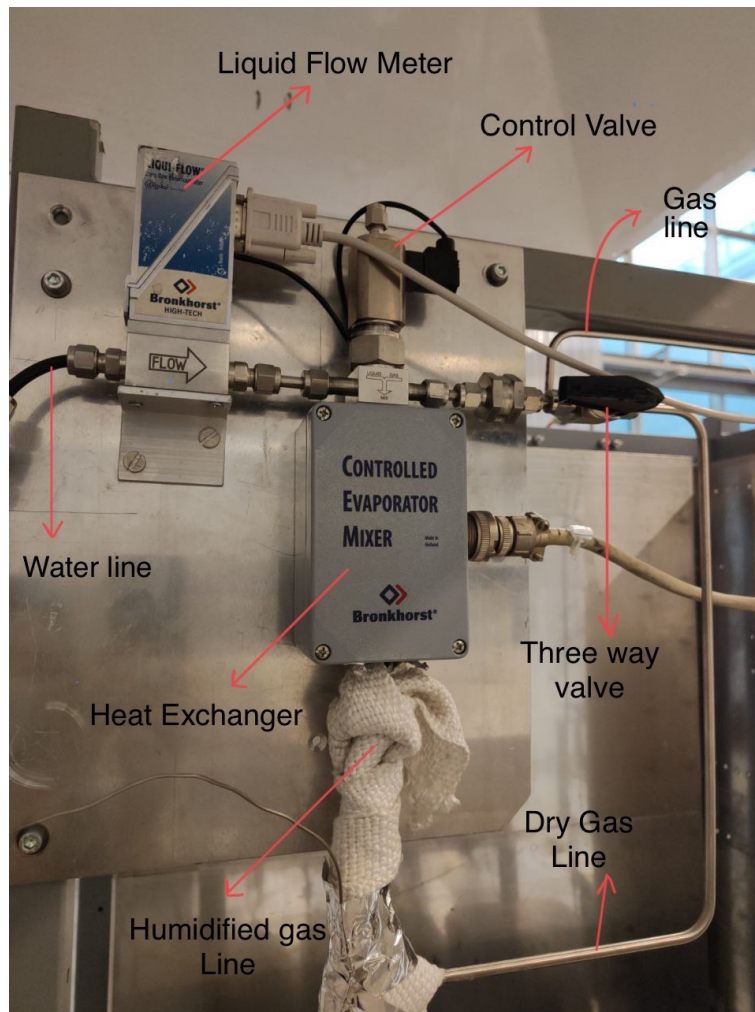


Figure 2.9: CEM installation in the laboratory

- Gradually increase the liquid flow setpoint up to the desired value.

To stop vapour production, a proper procedure should be followed as well:

- Change the liquid flow setpoint up to 0%.
- Change the CEM temperature setpoint to ambient temperature.
- Change the carrier gas flow setpoint up to 0.

2.3.4 Maintenance

A bleeding procedure is recommended from time to time in order to keep free of gas both the dead space and the liquid piping of the mixing valve. In particular, bleeding procedure is recommended:

- When first using CEM.
- After purging.
- Periodically.

The bleeding procedure is the following:

- Close the mixing valve by putting water setpoint to 0%.
- Pressurize the liquid inlet.
- Slowly open the bleed connection on top of the mixing valve until liquid starts to flow out.
- Close the bleed connection as soon as all gas has escaped through the mixing valve.

A purging procedure is helpful in order to prevent clogging of CEM's components and it's advisable:

- Before changing fluid.
- When using corrosive, reactive or explosive media.
- Before and after shutdown periods of more than one week.
- Every 3 months.

For the purging procedure, an inert and dry gas is advisable: for this purpose, N_2 is used in this test bench.

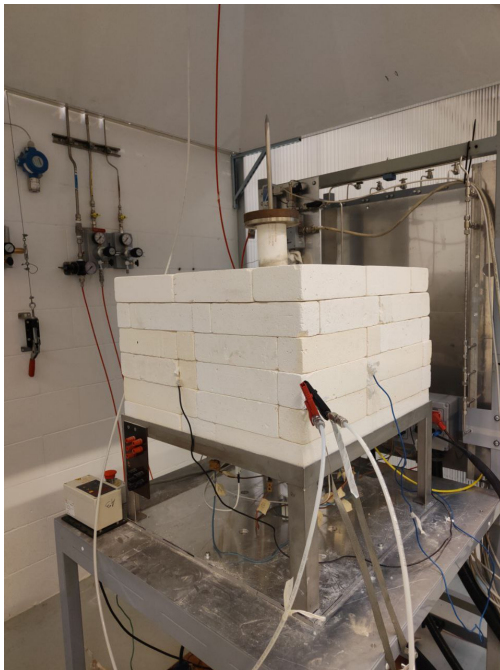
- At first, a N_2 supply line should be connected to the inlet of the liquid flow meter and to the gas inlet of CEM.
- Pressurization of N_2 supply line should be performed.
- Set the N_2 flow rate up to 100% and purge for at least 30 minutes.
- Change the N_2 flow to 50 %, open the bleed connection on the top of the mixing valve and purge for 10-15 minutes.

- Close the bleed connection.
- Set the liquid flow rate up to 100 % and purge for at least 30 minutes.
- Put the N_2 flow setpoint to 0% and de-pressurise the N_2 supply line.
- Put the liquid flow setpoint to 0 %.

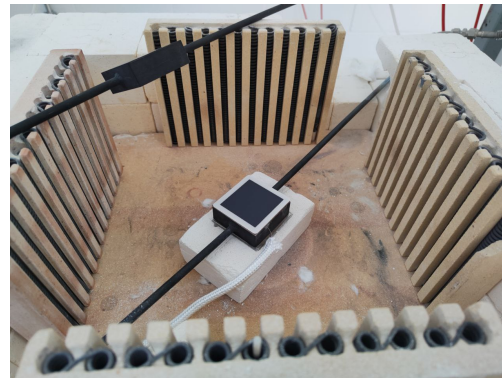
More detailed information about CEM can be found in Bronkhorst manual [22] and brochure [24].

2.4 Oven

2.4.1 General Description



(a) Oven in assembled configuration



(b) Inside view of the oven, with the four electrical resistances and cell's housing

Figure 2.10: Oven: inner and outer view

The oven of the test bench is used in order to reach the fuel cell's operating temperature, and to maintain it constant during the whole time of the experiment, by compensating for heat losses towards the external environment. It is mainly composed by its walls, made of refractory bricks, that can be removed and re-arranged, and by four electrical resistances, placed at the four sides of the oven.

Thermocouples, with ceramic coating, are hosted inside each resistance, in order to keep track of the temperature during experiments. Inside the oven, temperature is not equally distributed and the temperature gradient must be taken into account: a fifth thermocouple can be eventually placed to monitor the temperature in the central zone of the oven, where the cell is placed. In fact, the temperature detected by the four thermocouples placed inside the resistances is higher than the central temperature. The ceramic basement of the oven has proper holes to allow the crossing of thermocouples and electrical connections. The oven's configuration is represented in figure 2.10b and figure 2.10a.

2.4.2 Fuel cell's housing

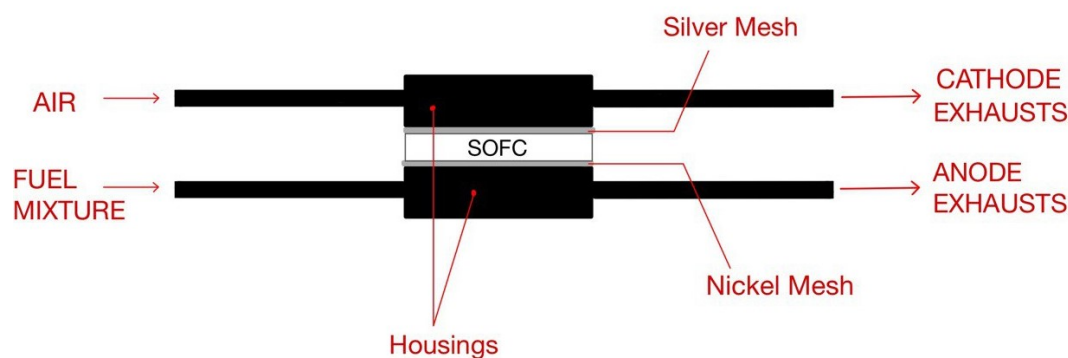
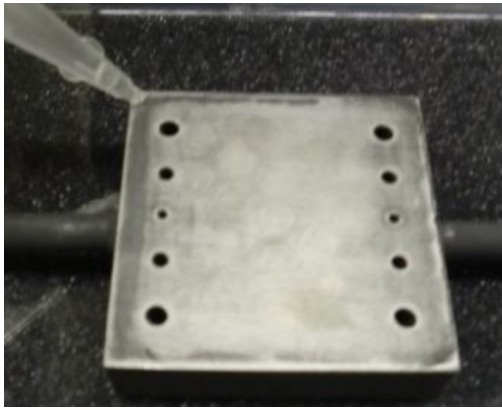


Figure 2.11: Schematic of the housing of the cell

The oven hosts the fuel cell [25] thanks to a proper housing configuration. The cell is placed between two steel housings, with square cross section, that allow gas distribution through the cell thanks to small inlet and outlet holes, as it is possible to notice in figure 2.12a. The lower housing is the one responsible for the distribution of the fuel-mixture and is therefore in contact with the anodic compartment, in SOFC configuration. The upper housing is instead responsible for the air flow distribution and it is in contact with the cathodic compartment. Each one of the two housings is welded with an inlet and an outlet steel pipe. The inlet pipe allows the connection with the feeding gas lines (air and fuel mixture) while the outlet pipe collects the exhausts gas. The latter are eventually sent to a condenser and then conveyed to the hood. Moreover, the two outlet pipes (delivering the exhaust gas) are also employed as current carriers: at the oven outlet, they are connected to the electrical load through two wires, stapled to the pipes, represented in figure 2.12b.

Between the housings of the cell and the anode/cathode compartments a nickel and

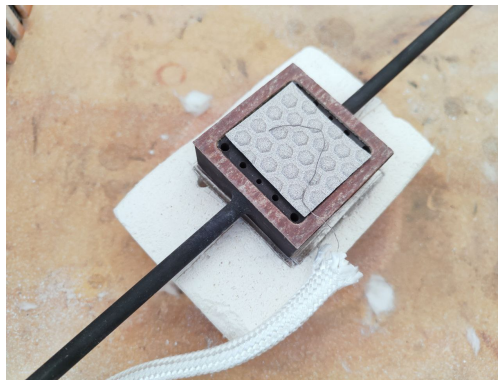
a silver mesh are respectively positioned, to better distribute the current; between the grids and the cell, two thin platinum wires are placed. They cross the oven's walls and are connected to proper terminals that allow to collect the voltage signal and to transfer it to the Data Acquisition System. Moreover, sealing frames (mica) must be employed to seal the housing, assuring gas tightness. In figure 2.12c the lower housing, equipped with the metallic grid, the platinum wire and the frame sealing, is represented.



(a) Cell housing with visible inlet and outlet holes for gas distribution



(b) Wires stapled to the outlet pipes



(c) Lower housing equipped with sealing frame, metallic grid and platinum wire.

Figure 2.12: Oven: cell's housing configuration

The cell's assembly will be thoroughly described in chapter 4. In the top-central part, the oven is closed by an alumina cylinder, equipped with proper weights: this configuration allows to put the fuel cell in compression, enhancing adherence among its components and increasing its performances.

2.4.3 Temperature control

The four electrical resistances inside the oven are fed with a DC current by the power supply. The DC current is regulated according to the set-point temperature and to the temperature ramp imposed by the operator: usually, to not induce too high thermal shocks on the cell and on other components, a ramp of 30°C/h is set, up to the desired temperature. A control unit (Eurotherm) is used for the regulation of the oven power. On one hand, it receives as input signals the operator's commands (temperature ramp and set point, set through Teachy software or manually) and the temperature recorded by one of the four thermocouples. On the other hand, it sends an output signal to the power supply unit, about the power to send to the resistances in order to get the desired temperature in the desired amount of time.

2.5 Electronic Auxiliaries

The test bench under analysis involves two electronic auxiliaries: the one responsible for providing power to oven's resistances and the electronic load devoted to the cell.

- The power supply devoted to the oven (*Delta Electronika B.V SM300 Series*, [26]) sends a DC current to the electric resistances. As explained in the previous section, the power to be sent is computed by the Eurotherm unit, according to the temperature set point and ramp imposed by the operator. The four resistances inside the oven are connected in parallel with respect to the power supply.
- The electronic load devoted to the cell (*Kikusui PLZ-4W Series*, [27]) is coupled with a compensation power supply, used to amplify the signal of the cell's voltage, which is very low (the open circuit voltage is of the order of 1-2 V). The internal circuit, in SOFC operation mode, is represented in figure 2.13: it guarantees high performances and velocity in current control and load simulation, by employing transistors, which act as variable resistor. The operator can set, through Teachy software (or manually through the LCD), the desired current. The electronic load, receiving the signal, will be able to ask for that specific current to the SOFC. In response, the SOFC will deliver the required current to the electronic load, and the voltage at the cell's

terminals will change consequently. The LCD of the electronic load always displays the cell's voltage, power and current, measured at the load input terminals.

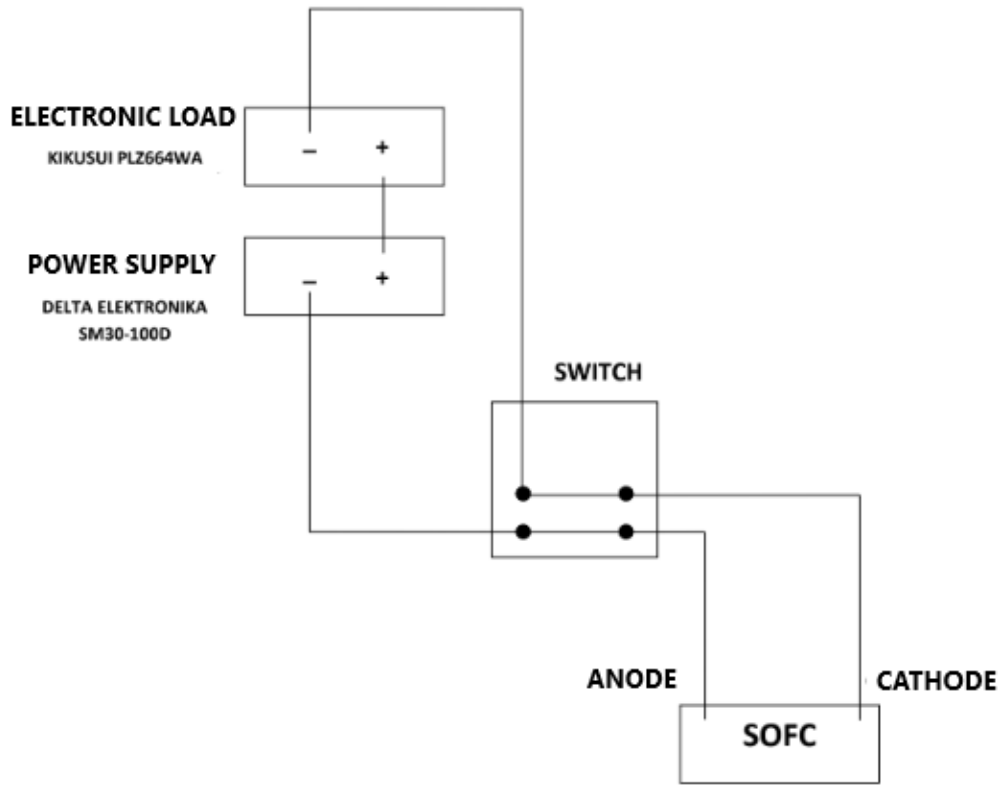


Figure 2.13: Internal circuit of the electronic load devoted to the cell

More specific information about the cell electronic load and power supply/compensation load are reported in their respective manuals [27],[26].

2.6 Data acquisition and control system

The test bench can be almost entirely operated in remote: tests and characterizations of the fuel cell can be performed through Teachy software, even if a physical supervision in the laboratory is always required for starting the experiment and switching the manual valves and switches. Through the software the operator can monitor and control the several over-mentioned devices: this is made possible by a proper data acquisition and control system. Of course, along with the remote option, also a local control is possible, thanks to manually controlled terminals.

- **Remote Control.** The remotization of the test bench has been performed by exploiting National instruments hardware and *Teachy* software, as shown in figure 2.14.

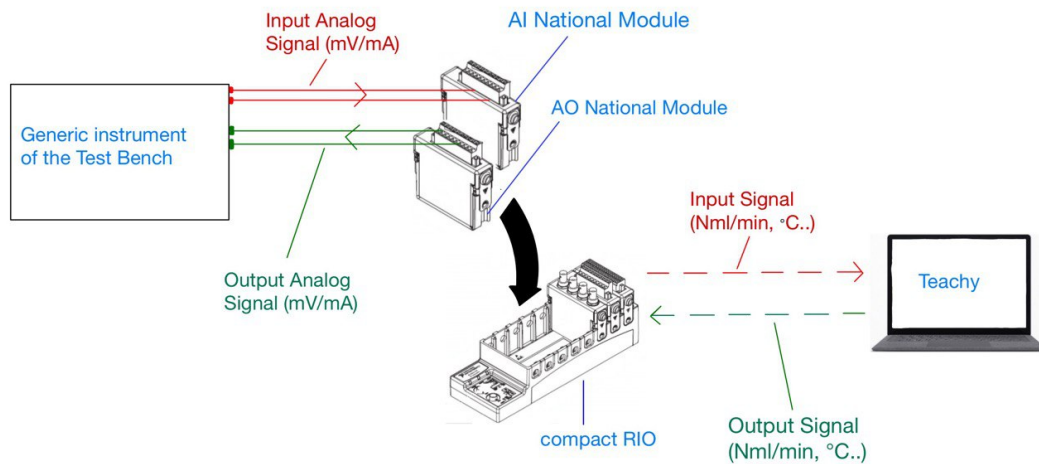


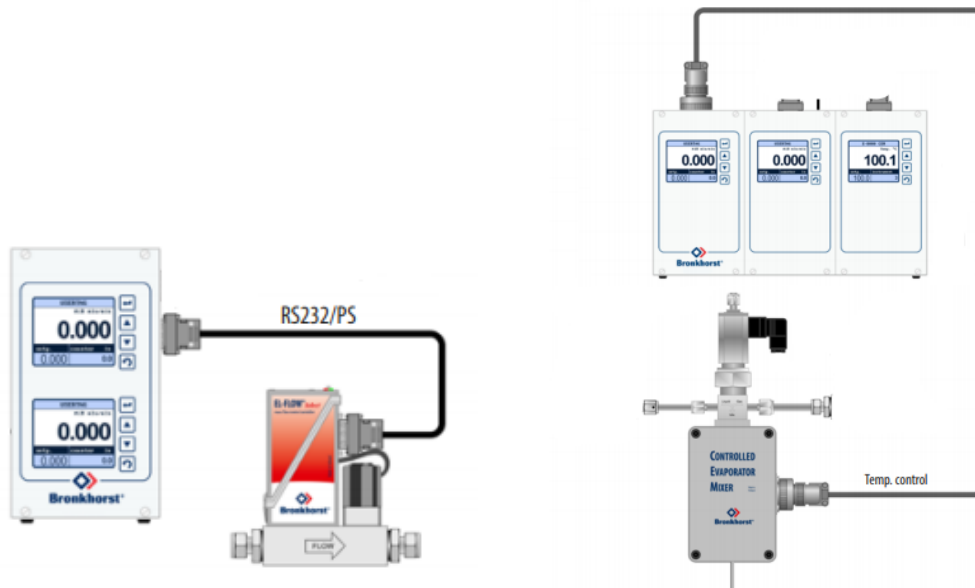
Figure 2.14: Connections for remote control. Continuous lines represent physical connections, dashed lines represent VLAN connection. [28]

In particular, thanks to proper cables, the terminals of each instrument of the test bench have been connected to the National I/O modules, installed on compact RIO units [29]. Compact RIO has been programmed using *LabVIEW* software, in order to properly conditioning the incoming/outgoing signals. In fact, the I/O signals exchanged between each instrument of the test bench (mass flow controllers, thermocouples, Eurotherm unit, electronic load) and

the National modules are analog signals in [mA] or [mV].

The CRIO unit communicates (via LAN) with *Teachy* software, that represents the user interface, in which the operator can read the incoming conditioned signals or send commands. *Teachy* software and the database hosting the recorded data are installed on *Politecnico di Torino*'s virtual machines. *Teachy* software is accessible via browser from any PC with internet connection. The variables that can be remotely controlled are: gas flow rates, water flow rate, temperatures inside the oven, temperature of CEM, fuel cell's current. The fuel's cell voltage can only be read.

- **Local Control.** *Bronkhorst* digital readout/control system is employed ([30],[31]), allowing local control and monitoring of the test bench's flow rates and CEM's temperature. It is connected to each mass flow controller by means of 9-pin D-sub cables, allowing power-up and RS232 communication. A proper cable allows also the electrical connection with CEM's internal resistance and thermocouple, for temperature readout and control. A simplified connection scheme of these two devices is represented in figure 2.15.



(a) *Single MFC simplified connection-scheme* (b) *CEM's simplified connection scheme*

Figure 2.15: Connections for the local readout/control unit

Through the unit's displays the operator can read/control gas/water flow rates

and CEM's temperature.

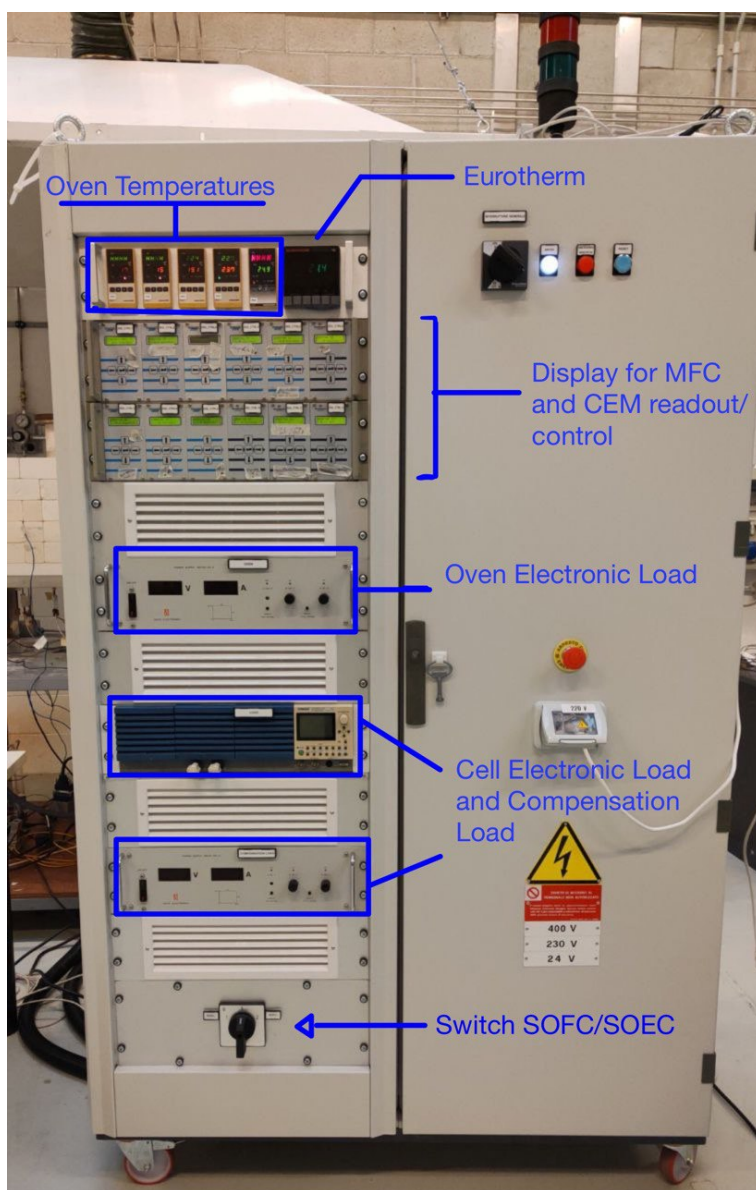


Figure 2.16: Cabinet with local readout/control units

The Eurotherm unit allows to manually impose set point temperature and ramp; also the electronic auxiliaries (oven's power supply, cell's electronic and compensation loads) can be locally controlled. Moreover, a local switch allows to change from SOFC to SOEC mode, which would not be possible through remote operation. It is also possible to put the test bench in emergency mode,

switch it off or reset it. The cabinet containing all locally-controlled commands is represented in figure 2.16.

Chapter 3

Teachy Software

3.1 Introduction to user's guide

Teachy project has the aim of developing a remotely-controlled test bench for the characterization of SOEC/SOFC cells. The control system of the test rig is based on a local hardware installed on the bench for managing the input/output signals from the instruments, a control software and a database installed on a server, and a web-based interface, available from any PC with internet connection, that allows the remote testing of a SOEC/SOFC. The present chapter is intended to lead the operator through the correct utilisation of *Teachy* software ([32], [33]) in all of its functionalities. In order to properly manage the test bench, an adequate understanding of its connections and components, presented in Chapter 2, is recommended.

Teachy environment is accessible from the following web address:

<https://teachy-www.polito.it:8085/Polito>

Through which the operator is brought to the login page. Login requirements differ according to the operator's account:

- **Root account:** it must be directly created through the Database. Its main privileges are to create *Administrator*'s accounts, manage and visualize them.
- **Administrator account:** besides having access to all control and management functions of *Teachy*, it can also create and manage the *User*'s accounts.
- **User account:** it has access to a restricted number of actions and can login only in a specific range of time, chosen by the *Administrator*.

Through the chapter, all possible actions and functionalities of *Administrator* and *User* accounts will be thoroughly outlined, underlying their roles and limitations in carrying out experimental sessions for test and characterisations.

3.2 Administrator account

The *Administrator* account can enter the system at any time, without any restrictions. Once logged in, the overall menu is the one reported in figure 3.1.



Figure 3.1: Menu Administrator's account

The different sections will be analysed one by one.

3.2.1 Home

This section is mainly used to alert the user if profile-activation email have not been received. When there are no warnings, the inscription "No alert found" is present, as noticeable from figure 3.1.

3.2.2 Users Management

This section can be used by the Administrator to create new User's accounts and manage them. It is divided into two subsections:

- **Create User:** the administrator must fill the boxes reported in figure 3.2 with user's information. In particular, the requested data are: user's email, registry number (which corresponds to the Institution official No), University, first and last name. The registered email will be the one with which the user will have access to *Teachy* software. By pressing "Create", an email will be sent to the specified email address in order to complete the registration.

Create a new Account

Email _____

Registry number _____

University _____

First Name _____

Last Name _____

Create

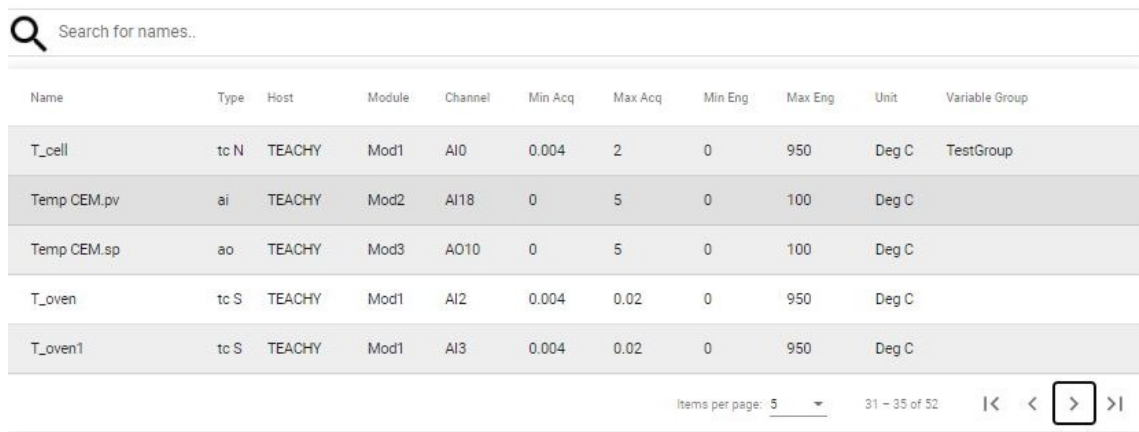
Figure 3.2: Create User

- **User List:** all the created users are visible in this section and can eventually be removed by the Administrator by pressing "Delete". Along with the user's email, also its state is present: "active" if he has completed the registration, otherwise "passive".

3.2.3 Variable Management

This section allows the management of the different variables handled by *Teachy* software. It is divided into two subsections:

- **Variable List:** in this subsection, a table with all variables is present. As visible in figure 3.3, the table reports: variable name, variable type (analog input/analog output), module (number of the National module which receives the signal), channel (number of the channel which receives the signal), minimum and maximum values (both of the signal and of the respective physical quantity), unit and variable group.



Name	Type	Host	Module	Channel	Min Acq	Max Acq	Min Eng	Max Eng	Unit	Variable Group
T_cell	tc N	TEACHY	Mod1	AI0	0.004	2	0	950	Deg C	TestGroup
Temp CEM.pv	ai	TEACHY	Mod2	AI18	0	5	0	100	Deg C	
Temp CEM.sp	ao	TEACHY	Mod3	AO10	0	5	0	100	Deg C	
T_oven	tc S	TEACHY	Mod1	AI2	0.004	0.02	0	950	Deg C	
T_oven1	tc S	TEACHY	Mod1	AI3	0.004	0.02	0	950	Deg C	

Figure 3.3: Variable List

The name of each variable present in the table is followed by the indication ".pv" or ".sp", which are important to understand how we can manage them. A variable ".pv" is associated to an analog input signal (ai type) and can only be read by the operator. On the other hand, a variable ".sp" is associated to an analog output signal (ao type) and is therefore controllable by the operator. Most variables appear both as ".pv" and as ".sp": in this case the operator can both send a command and read the value measured by the specific instrument. In the table, some spare variables are also present, not associated to any

specific instrument: they can be modified and used in case a new instrument is inserted in the test bench.

When the maintenance mode is enabled, the operator has the possibility to delete or modify the variables present in the table: at the end of each line a "Delete" and an "Update" push-buttons will appear. After introducing the desired adjustments, the operator must press on "Send to cRIO" (present beneath the table of figure 3.3 in maintenance mode) to send the modifications to the CompactRIO.

- **Create Variable:** this subsection appears only when the maintenance mode is enabled and allows to create a new variable. The operator should complete the form reported in figure 3.4: the boxes must be filled with the same information appearing in the *Variable List* table. Before proceeding, the operator must be aware of the connections among test bench's instruments and National modules, of the signal's ranges and types. For this reasons, this subsection should generally not be used without the support of technicians.

Create a new Variable

Variable Name	
Type ▼	Host
Module	Channel
Min Acq	Max Acq
Min Eng	Max Eng
Unit	Variable Group

Create

Figure 3.4: Create New Variable

3.2.4 Experiment Profiles

Differently from the *Administrator*, *Users* can have access only to a strict number of variables: this is required since the wrong setting of some variables could lead to cell's failure.

In this section, the *Administrator* can select the set of variables to which *Users* can have access to, called Experiment Profile. There are two main subsections:

- **Create Experiment profile:** allows the creation of a new Experiment Profile. As represented in figure 3.5, the *Administrator* should select the variables belonging to the experiment profile by ticking on them. Then, he should insert, under "Configuration name", the name for the new Experiment profile. Finally, he should create it by click on "Create".

Create a new Experiment Profile

Configuration Name _____

Choose variables:

Search for names..

<input type="checkbox"/>	Name	Type	Host	Module	Channel	Min Acq	Max Acq	Min Eng	Max Eng	Unit	Variable Group
<input type="checkbox"/>	MFC10 CO 500.pv	ai	TEACHY	Mod2	AI1	0	5	0	500	Nml/min	
<input type="checkbox"/>	MFC10 CO 500.sp	ao	TEACHY	Mod3	AO1	0	5	0	500	Nml/min	
<input type="checkbox"/>	MFC1 H2 1000.pv	ai	TEACHY	Mod2	AI6	0	5	0	1000	Nml/min	

Items per page: 3 10 - 12 of 52 << < > >>

Create

Figure 3.5: Create Experiment Profile

- **Experiment Profile list:** here, all the created experiment profiles are displayed and can eventually be updated or deleted. Both the name of the experiment profile and the selected variables are visible.

Search for names..

Configuration Name	Variables	
Polarizzazione	Vcell.pv, current.sp, current.pv, MFC1 H2 1000.pv, MFC5 AIR 2000.pv, MFC7 N2 1000.pv, T_oven.pv	Delete Update

Items per page: 5 1 - 1 of 1 << < > >>

Figure 3.6: Experiment Profile List

3.2.5 Group Management

The next step in the creation of an experimental session, is to select and manage the group of *Users* that will be involved in the experiment and the duration of the test. This is possible through three subsections:

- **Create Group:** allows the *Administrator* to create a new Group (figure 3.7), by choosing the group name, the Experiment configuration (among the Experiment profiles previously created), the start and the ending time. The system does not allow the overlapping in time of different groups. Moreover, the Group's start time should be greater than the actual time. Pop-up warning messages show up when these two constraints are not respected, as shown in figure 3.8.

Create a new Group

Group Name

Experiment Configuration

Start time

dd/mm/yyyy hh:mm

End time

dd/mm/yyyy hh:mm

Create

Figure 3.7: Create Group

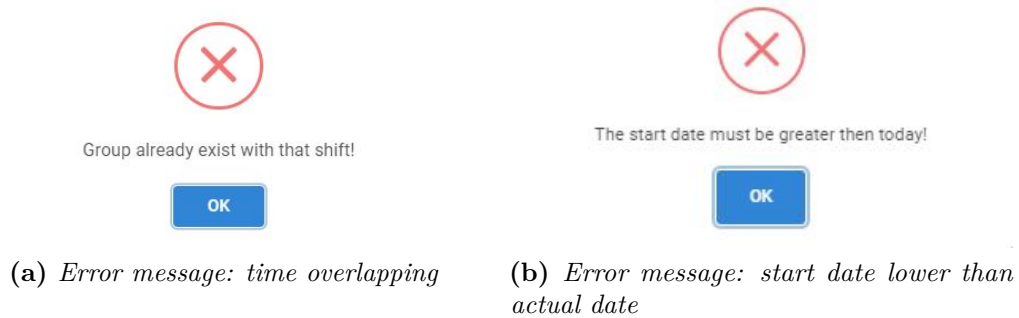


Figure 3.8: Errors messages in group creation

- **Assign User:** after creating a Group, the *Administrator* has to choose the *Users* to allocate and in which role. In fact, two main categories are present: the "Head User" and the "Read only User". The first one has the opportunity to both modify and visualize the assigned variables: just one user can assume this role. The second one can only visualize the experiment's development: more than one user can assume this role.
In this subsection, a table with all the created groups will appear: by clicking on the line corresponding to the desired group, a pop-up window will open. Here, it will be possible to select which user to assign as "Head User" or as "Read only User".
- **Group List:** a table, resuming all the created group's characteristic, is reported and the *Administrator* can modify or delete them.

3.2.6 Script

This section allows to upload and run a script, which can be useful to execute a set of commands in series. The *Administrator* can create the desired script through a dedicated software, *Teachy-Script-Editor* (section 3.4). The script file must be uploaded in *.json* format.

By pressing the button "Browse", the *Administrator* can choose the desired script file from the PC. Through the button "Upload", the script can be uploaded: if the procedure ends well, a pop-up window will appear confirming the correct loading, otherwise warning about the eventual error.

When a file is uploaded but not executed, the inscription "<script-name> currently uploaded" will appear: the *Administrator* can run it by pressing on "Run <script-name>".

Once run the script, the inscription "<script-name> currently running" will show

up, along with the button "Kill <script-name>", to be pushed when the script must be interrupted.



Figure 3.9: Script section

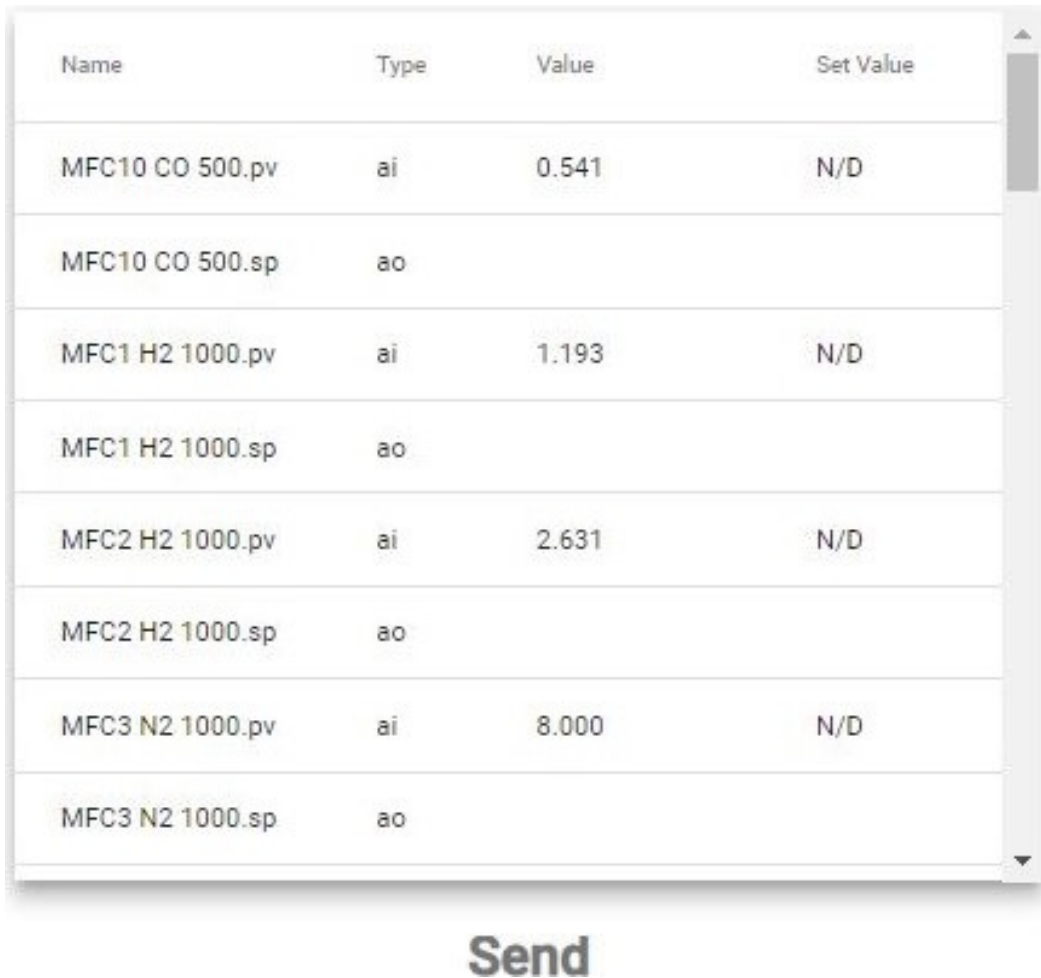
When the uploaded script contains errors, it will be automatically shut down by the system.

3.2.7 Experiment section

The Experiment section is fundamental while carrying out an experiment. In fact, it allows the real time management of variables and the monitoring of alarms and system's status.

- A table, positioned in the left part of the screen, allows the variables management (figure 3.10). Four columns are present, containing variable name, variable type, current value and set value. "Type" column specifies whether the signal is an analog input or an analog output. As mentioned in section 3.2.3, ".pv" variables are analog input variables, so they can only be read by the operator. On the other hand, ".sp" variables are analog output variables and the operator can impose their value. "Value" column specifies the real time value of the variables, allowing a constant monitoring of the system. The "Set Value" column corresponds to the value set by the operator for the specific variable. In case of ".pv" variables, the inscription "N/D" appears. In

order to enter a set point for a certain variable, the *Administrator* should click on the respective row: a pop-up window will open, where the value can be written. If the inserted value exceeds the limit, a warning message will inform the operator, as shown in figure 3.11. To send the set points to cRIO unit, the *Administrator* should click on the button "Send". More than one variable can be sent at a time. If one or more variable's values have been set but not sent, a warning message "Variables not sent" will appear.



The image shows a table with four columns: Name, Type, Value, and Set Value. The table contains eight rows of data. Below the table is a large button labeled 'Send'.

Name	Type	Value	Set Value
MFC10 CO 500.pv	ai	0.541	N/D
MFC10 CO 500.sp	ao		
MFC1 H2 1000.pv	ai	1.193	N/D
MFC1 H2 1000.sp	ao		
MFC2 H2 1000.pv	ai	2.631	N/D
MFC2 H2 1000.sp	ao		
MFC3 N2 1000.pv	ai	8.000	N/D
MFC3 N2 1000.sp	ao		

Send

Figure 3.10: Experiment section: variables management

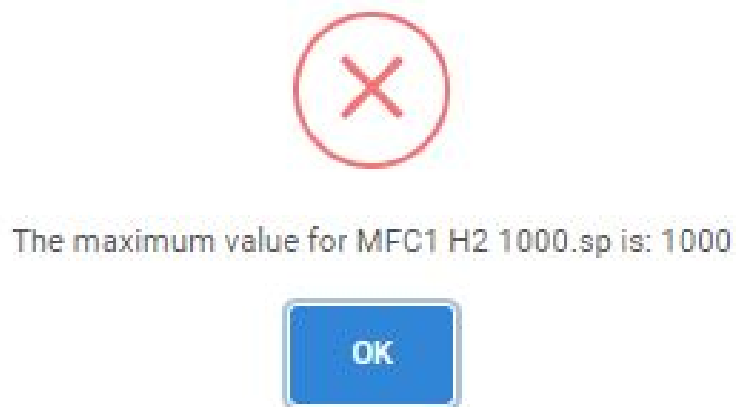


Figure 3.11: Error message: out of range

- A table, positioned in the right-bottom part of the screen, reports all systems alarms. Under "Timestamp" the time and data of the error are reported. Under "Content" the error code is present.

When an error occurs while the *Administrator* is logged in, a pop-up window with the alarm information will appear and the alarm will be added to the list. During the alarm condition, variables can not be changed. Moreover, after the problem has been fixed, a button to send the error "Acknowledge" to cRIO will show up.

Old alarms can be removed from the directory with the button "Delay".

An error in loading cRIO alarms may occur from time to time: in this cases, a pop up error message will appear (figure 3.13).

Alarm List

Timestamp	Content
-----------	---------

Figure 3.12: Experiment section: Alarms



Figure 3.13: Error message: error in loading cRIO alarms

- A box positioned in the top right part of the window allows to monitor the system's status. In particular, it reports information about the status of the normal log and of the fast log (enabled/disabled), the Script conditions (idle/uploaded/running) and the connection of *Teachy* to the cRIO unit (Connected/Disconnected).



Figure 3.14: Experiment section: system's status

3.2.8 Logs Configuration

Teachy allows to store data when required. Data coming from the test bench are not normally saved on the Database, to not occupy too much space. Therefore, to enable data logging, *Logs Configuration* section is created.

The *Administrator* can choose among normal log (DB LOG) and fast log (HSDB LOG - High Speed Database Log). The first one allows to store data coming from every instrument of the test bench every 60s. The second one allows to store data more frequently: since this option involves a large number of information, it can be enabled only for a restricted group of variables. The two logs can be activated simultaneously. For example, it could be useful during experimental sessions to have a fast log of cell's voltage and current and a normal log of other variables.

As represented in figure 3.15, the ending date is required for setting the DB LOG; both the ending date and the variable's Group Name are instead required for the HSDB LOG. Each variable can be associated to a group during the maintenance mode (section 3.2.9). After entering the required parameters, the *Administrator* should press on "Send" to enable the desired log. When a log is enabled, an inscription reporting its ending time appears below the "Send" button. The *Administrator* can choose to end a log at any time by pressing on "Stop".

When a Script is launched, the DBLOG starts automatically and the current section is not needed: if the *Administrator* tries to launch a log while a script is running an error message will show up (figure 3.16).

Set DBLOG

Send Stop

Set HSDB

Send Stop

Figure 3.15: Logs Configuration: DBLOG and HSDBLOG



Figure 3.16: Error message: log during script running

3.2.9 Command

In this section four main actions are possible:

- **Safe:** when pressing on "Safe", the *Administrator* sets the system in safe operation and all variables set points are automatically put to 0, with the exception of N_2 flow of MFC4, which is set to 1000 [Nml/min].
- **Maintenance Mode:** by enabling the maintenance mode, the operator can modify/add/delete variables and send the new configuration to cRIO unit. In fact, only when the maintenance mode is active the subsection "Create Variable" appears and the subsection "Variable List" is editable. To warn the operator, a red line appears in the top part of the window, along with a triangular warning indicator (figure 3.18). The Experiment Section 3.2.7 is not available and the user can not log in. The operator can disable the maintenance mode at any time.
- **cRIO Configuration:** useful to ask to cRIO the actual variable's configuration.
- **Error Acknowledge:** this action is possible only in case of alarm and it is necessary to send the acknowledge of solved errors to cRIO.

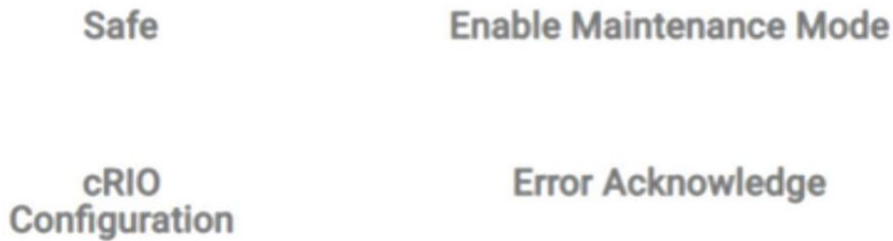


Figure 3.17: Commands

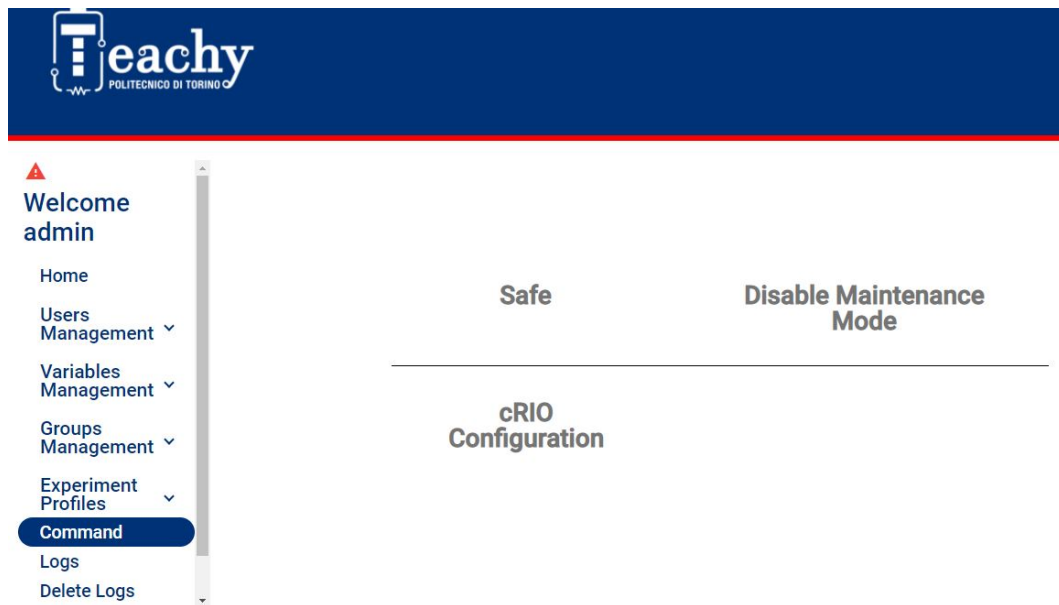


Figure 3.18: Maintenance Mode

3.2.10 Logs

In this section the operator can have access to the stock of data present in the Database and export them.

As represented in figure 3.19, the *Administrator* should enter the desired variables to be plotted: the variables can be selected from the drop-down menu and a new one can be added by pressing "Add Variable". To remove a variable the button "Remove Variable" is present. Moreover, the *Administrator* should choose the temporal range inside which plotting the variables: from the timetable the start and end times can be selected.

Finally, by pressing "Send", a new window is opened (figure 3.20). Here, a plot

reports the values of the chosen variables with respect to time. When more than one variable is present, different colours are adopted and a caption positioned below the plot helps distinguish them. By pressing "Reload logs", the variables are updated to the current time. The button "Change variables" can be used to change the previously inserted parameters. By clicking on the right-top menu the operator can print the chart, download it as an image or export the data in .xlsx or .csv formats.

Choose the variable to show:

Add Variable **Remove Variable**

Variable

Start time

End time

Send

Figure 3.19: Logs section

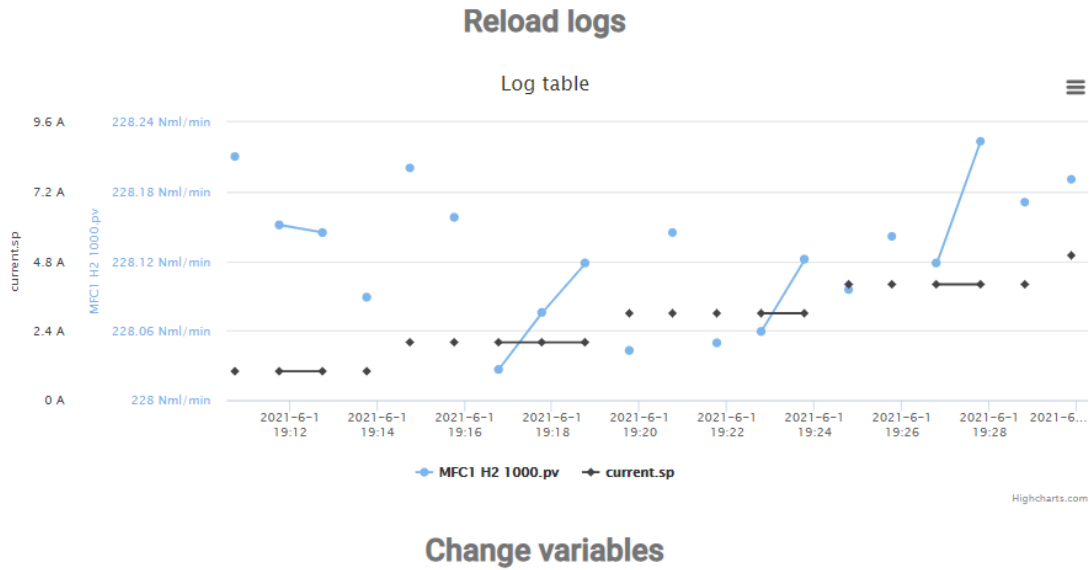


Figure 3.20: Plot Example

3.2.11 Delete Logs

This section allows the *Administrator* to delete data present in the database. In particular, start and end times should be inserted: by pressing "Send" all logged data of that time range will be deleted from the database.

Choose the cancellation time:

Start time

End time

Send

Figure 3.21: Delete Logs section

3.2.12 Log out

By pressing the "log out" section, the *Administrator* will be logged out of the system.

3.3 User account

User's access to *Teachy* environment is totally managed by the *Administrator*. As explained in section 3.2.5, the *User* must be associated to a Group to be able to log in. If the group is not present or not active at the moment of the log in, the *User* will be warned with a message, reported in figure 3.22.

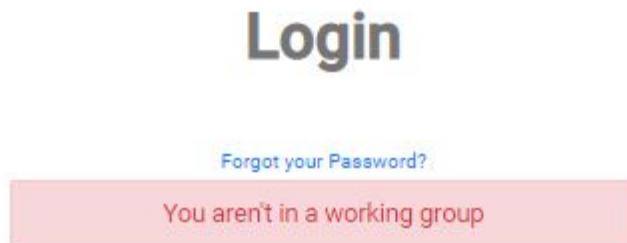


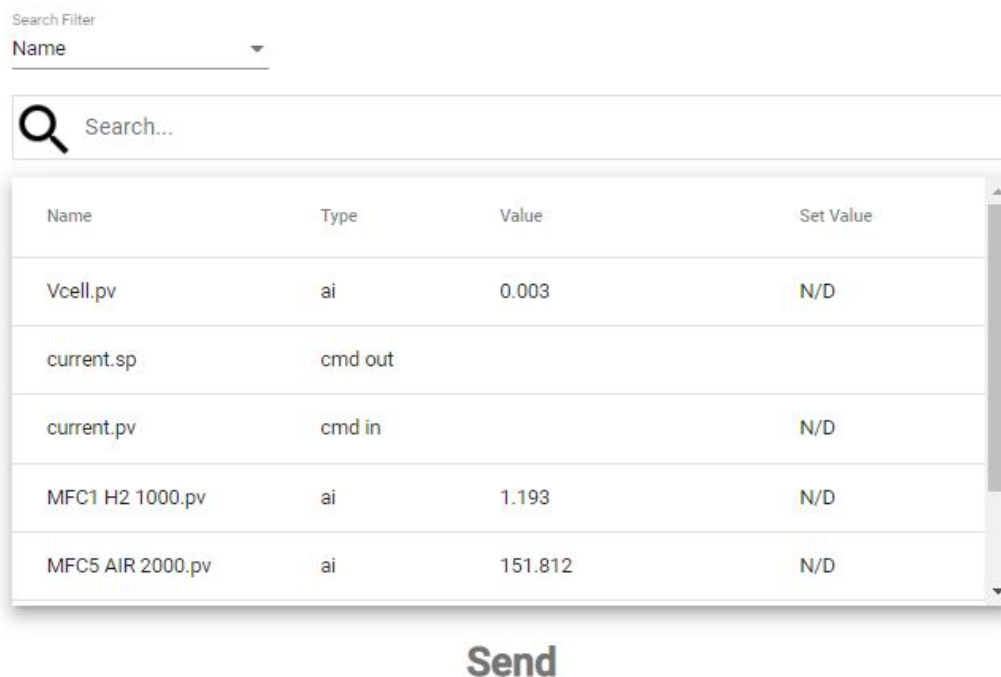
Figure 3.22: Login warning: no working group



Figure 3.23: User General menu

If the login ends successfully, the *User* can have access to its working environment (figure 3.23). With respect to the *Administrator*, the number of actions is limited. In fact, the *User* is thought to be a student, with a limited experience.

3.3.1 Experiment section



Name	Type	Value	Set Value
Vcell.pv	ai	0.003	N/D
current.sp	cmd out		
current.pv	cmd in		N/D
MFC1 H2 1000.pv	ai	1.193	N/D
MFC5 AIR 2000.pv	ai	151.812	N/D

Send

Figure 3.24: User experiment section

In this section the user can monitor and/or manage the variables during the experiment sections. A table is present, reporting variables name, type (ai/ao), current value and set point. The available variables are the ones chosen by the *Administrator* during the creation of the "Experiment Profile".

As discussed in section 3.2.5 two categories of users can be assigned: the "Head User" and the "Read only Users". Only the first one is able to send commands to the test bench: by clicking on the row corresponding to a specific variable, a pop-up window will open, allowing to insert the desired set point. By pressing on "Send" the set points will be sent to cRIO unit. If a variable is set but not sent, a message will warn the Head user.

The "Read only Users" will be notified of the introduced changes and will be able to follow the experiment but can not introduce any command.

In case of Alarm status or maintenance mode, no commands can be introduced and a warning message will inform the users.

3.3.2 Logs

Similarly to the *Administrator*, *Users* can have access to a portion of data stored in the Database. In this case, not all information are accessible: the *User* can visualize only the variables selected by the *Administrator* in the range of time of duration of the belonging Group. Therefore, as reported in figure 3.25, the *User* can select the desired variables from the drop-down menu. By pressing on "Send", a new page will open, containing a plot equivalent to the one presented in section 3.2.10 for the *Administrator*.

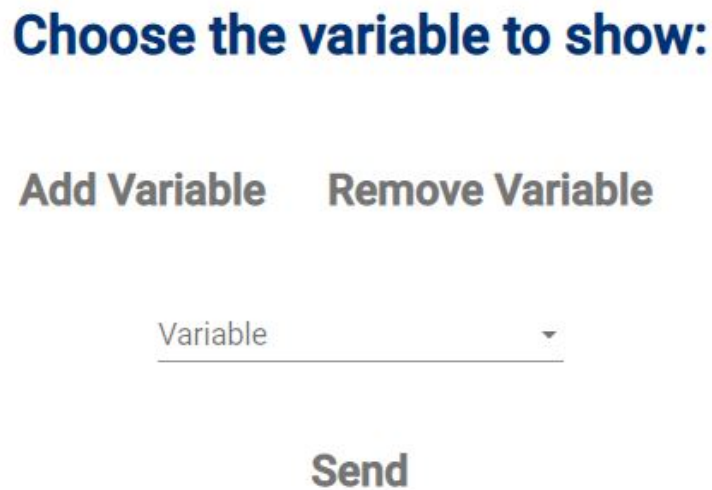


Figure 3.25: User logs section

3.3.3 Log out

By pressing the "log out" section, the *User* will be logged out of the system.

3.4 Teachy Script Editor

This section is devoted to the software *Teachy Script Editor*, which can be used by the *Administrator* to produce script files to be uploaded in the Script section 3.2.6 of *Teachy*. In fact, *Teachy Script Editor* has been specifically created for the *Teachy* project and involves commands and actions suitable for testing the cell.

3.4.1 Generalities

After downloading and installing the editor software, the *Administrator* can launch it. In figure 3.26 the homepage is shown.

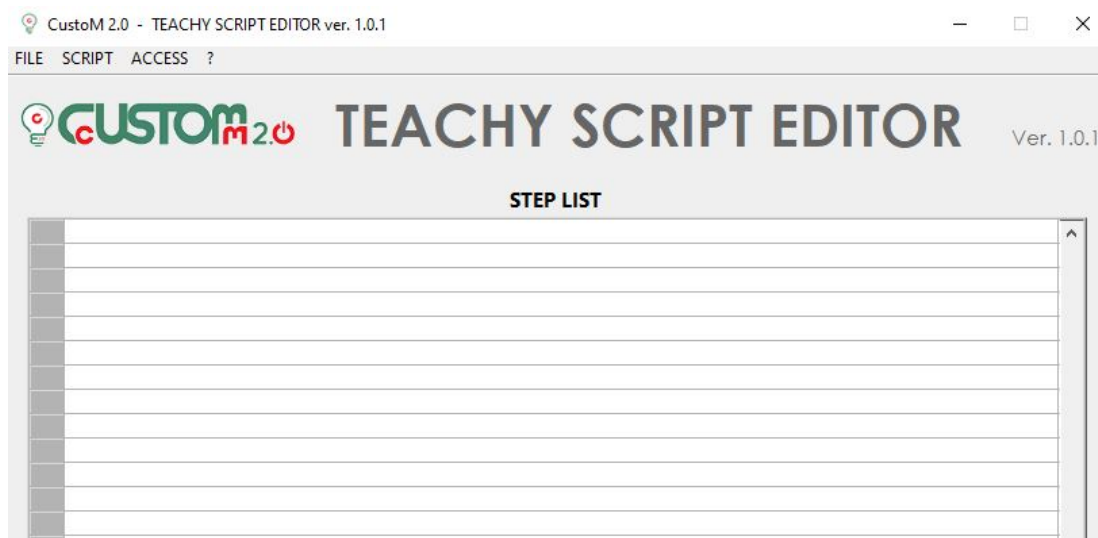


Figure 3.26: General Homepage

As visible, three main drop-down menus are present in the top part:

- **ACCESS.** Through this menu three main actions are available: the operator can login, logout and have access to the account list. The login is necessary each time the software is launched: the operator must insert the given username and password.
- **SCRIPT.** This menu allows to manage the steps of the script. The possible actions are reported in figure 3.27. Through "Add step" the operator has access to the available commands, that will be analysed in section 3.4.2. When a new step is added, it will be inserted in the empty rows below the inscription "STEP LIST" (figure 3.26). A step

can be selected by clicking on the correspondent row. When no step is selected, new step will be added below the last-created step. When a step is selected the new step will be added above it.

Through "Remove step", "Edit step", "Copy"/"Paste" the operator can respectively remove, edit and copy/paste the selected step.

Finally, it is also possible to add comments between rows and check the syntax for discover possible errors.

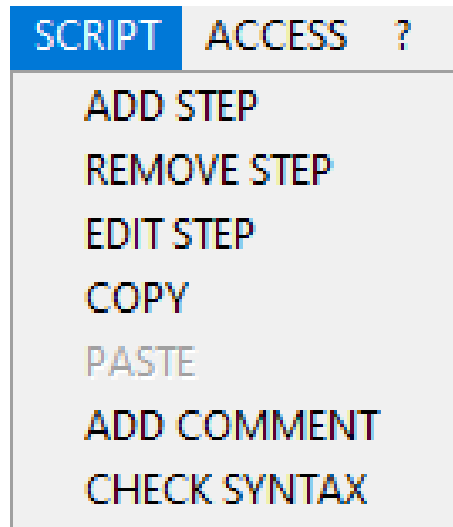


Figure 3.27: Script drop-down menu

- **FILE.** This menu allows the operator to save the current script or open/create a new file.

3.4.2 Commands

The available commands will be analysed in the following sections. As mentioned before, the commands are accessible by pressing on "Add step" from the *SCRIPT* menu (figure 3.27).

There are five possible commands: Wait, IV Curve, Set Variable, Eurotherm RLD, HSDB Log.

After setting the required parameters the operator should press "OK" to add the new step.

3.4.3 Wait

The "Wait" command allows to set a waiting time. The operator should set the time in seconds. A waiting time can be useful in many circumstances, allowing the system to stabilize between two subsequent actions.

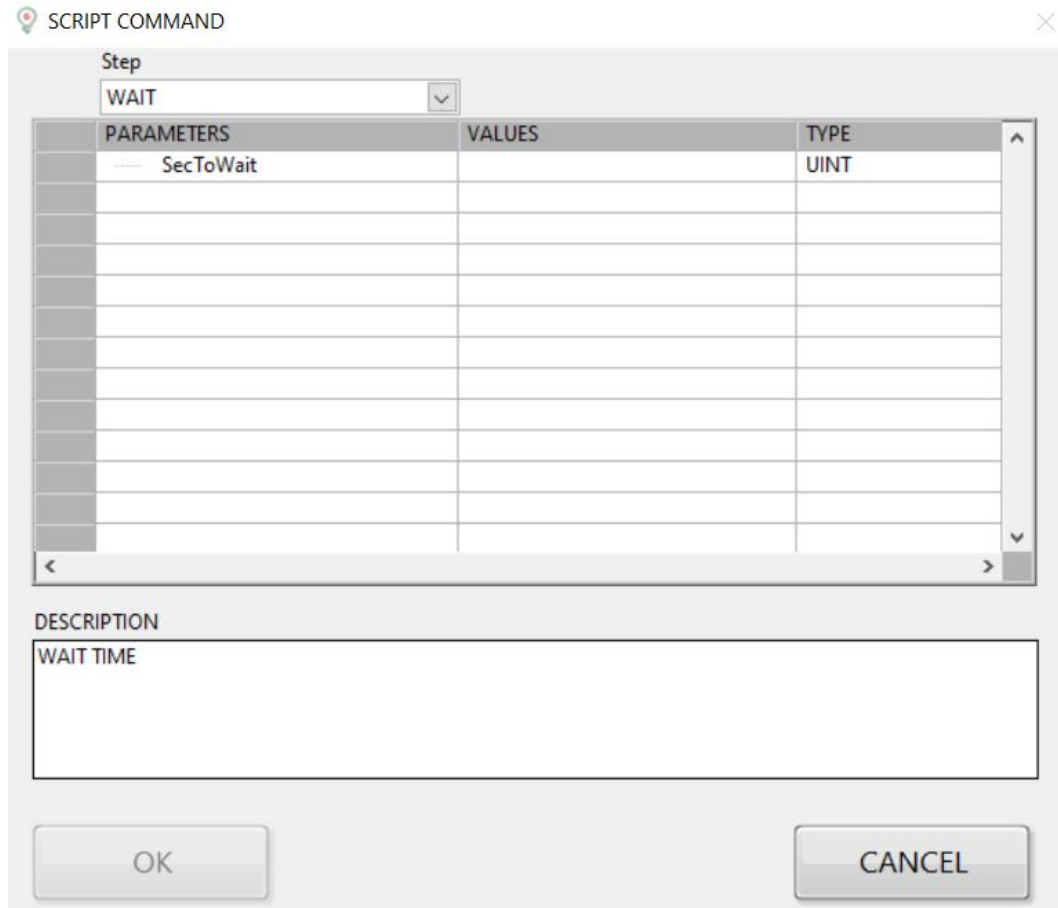


Figure 3.28: Wait command

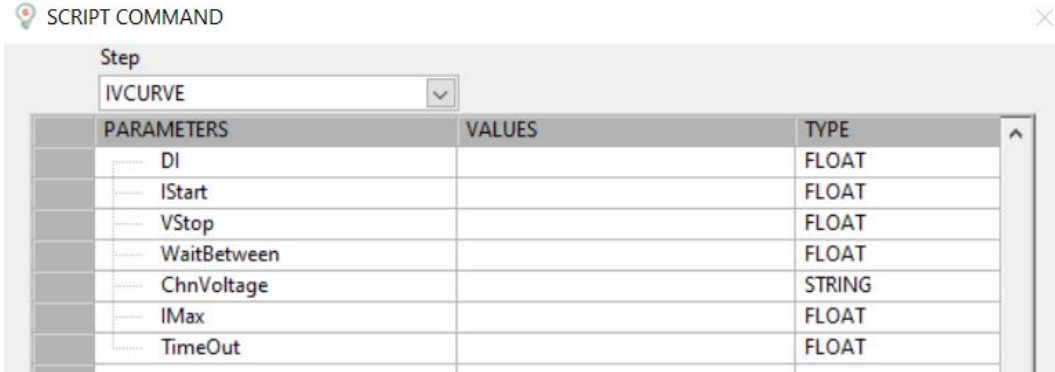
3.4.4 IV Curve

The "IV Curve" Command is specifically designed for the Polarization test of the fuel cell, which consists in increasing the fuel cell's current step by step, keeping track of the voltage decrease. The polarization test is automatically implemented and the operator should only insert the required parameters:

- **DI:** the current increment at each step, expressed in [A].

- **IStart**: current's starting value, expressed in [A]. Usually, it corresponds to the open circuit voltage, so 0 [A].
- **VStop**: minimum voltage value, after which the Polarization ends, expressed in [mV].
- **WaitBetween**: waiting time between two subsequent steps.
- **ChnVoltage**: the name of the voltage variable (Vcell.pv).
- **IMax**: maximum current value, after which the Polarization ends, expressed in [A].
- **TimeOut**: maximum duration of the polarization test, expressed in seconds.

The Polarization test will end when voltage or current or time reach their limit values, imposed by the operator (Vstop, Imax, TimeOut).



The screenshot shows a 'SCRIPT COMMAND' dialog box with a 'Step' dropdown menu set to 'IVCURVE'. Below the dropdown is a table with three columns: 'PARAMETERS', 'VALUES', and 'TYPE'. The table lists the following parameters and their types:

PARAMETERS	VALUES	TYPE
DI		FLOAT
IStart		FLOAT
VStop		FLOAT
WaitBetween		FLOAT
ChnVoltage		STRING
IMax		FLOAT
TimeOut		FLOAT

Figure 3.29: IV Curve command

3.4.5 Set Variable

"Set Variable" command allows to set the value of a generic ".sp" variable. The operator is requested to insert:

- **VarName**: the name of the desired variable, which can be retrieved in the section "Variable List" of *Teachy*.
- **Value**: the variable's set point. The unit of measurement employed must be compliant with the one specified in the "Variable list" table in *Teachy*.

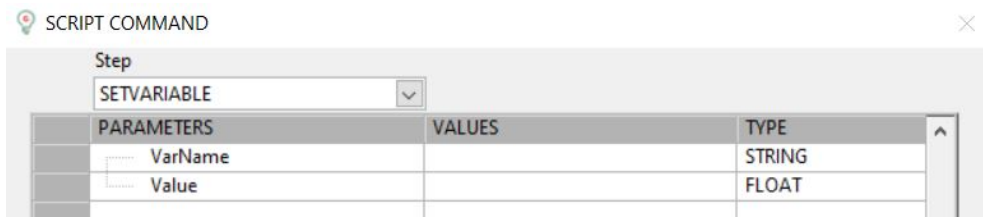


Figure 3.30: Set Variable command

3.4.6 Eurotherm RLD

"Eurotherm RLD" command is specifically designed for communicating with Eurotherm unit. As outlined in section 2.4.3, the Eurotherm conveys a signal to the oven's Electronic load, according to the temperature set point and ramp imposed by the operator. The electronic load will convey the required power to the oven's resistances, allowing to reach the desired temperature in the desired amount of time.

The "Eurotherm RLD" command requires three parameters:

- **Ramp:** the temperature ramp, expressed in [°C/h]. The ramp can not be too high, in order to avoid thermal shocks on cell and other components. A common value is 30 [°C/h].
- **Level:** the temperature set point, expressed in [°C]. When testing an electrode-supported cell, the operating temperature is around 850 [°C].
- **Dwell:** time to wait after reaching the set point temperature.

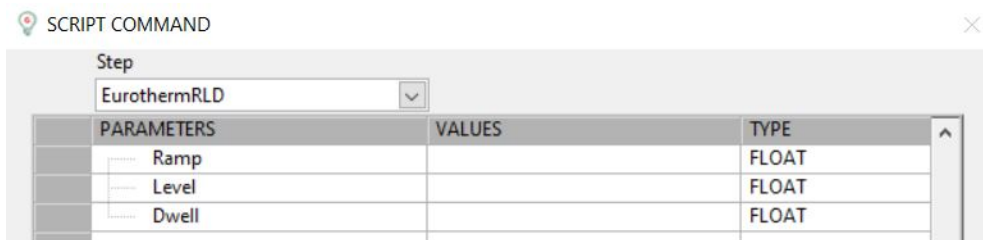


Figure 3.31: Eurotherm command

3.4.7 HSDB Log

When a script is running in *Teachy* environment, the normal log (DB Log) is automatically enabled for all variables. On the other hand, the fast log (HSDB

Log) should be manually activated, thanks to the command "HSDB Log". The required parameters are:

- **TimeToLog**: time duration of the fast log.
- **Group**: the name of the group of variables which should be logged fast. In fact, the fast log should be enabled only for a restricted group of variables. In *Teachy*, the variables can be associated to a group through the section 3.2.3 and only during the maintenance mode.

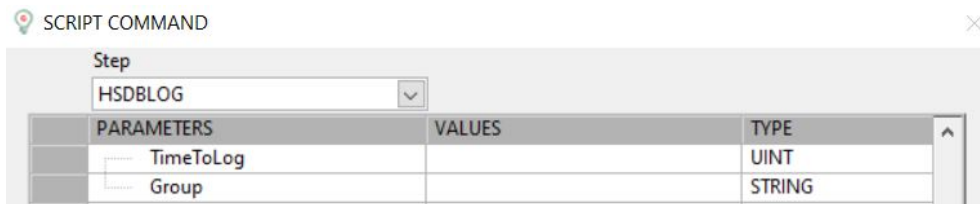


Figure 3.32: HSDB Log command

Chapter 4

Experimental session: Solid Oxide Fuel Cell testing

In order to assess the correct operation of the test rig, a complete experimental session has been carried out. The whole procedure will be outlined in this chapter, providing a guide for future tests.

4.1 SOC description

The tested SOC is provided by *Fuelcellmaterials*: it is an electrolyte supported planar solid oxide cell of 5x5 [cm] (*NextCellTM*). Its configuration is represented in figure 4.1. The dense cell-supporting electrolyte, made of *HionicTM* scandium stabilized zirconia, has a nominal thickness of 150 μm . This patented electrolyte assures both high strength and high ionic conductivity [34]. The anode and cathode electrodes have a multi-layered composition of, respectively, NiO-GDC/NiO-YSZ and LSM-GDC/LSM, with a nominal thickness of 50 μm .

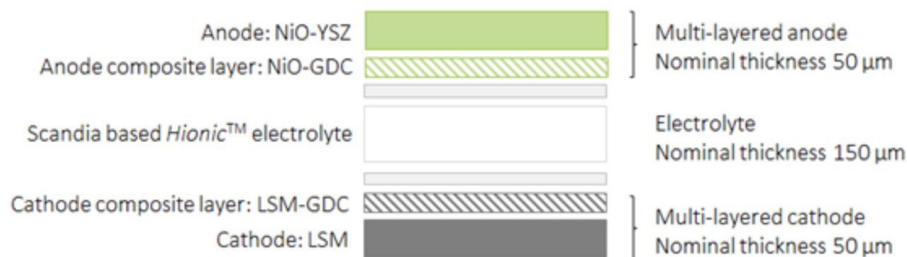


Figure 4.1: *NextCellTM* configuration [ne]

The cell shows good performances in a temperature range between 750-850 [°C]. Polarization and power density curves at 800 [°C], provided by the company, are reported in figure 4.2.

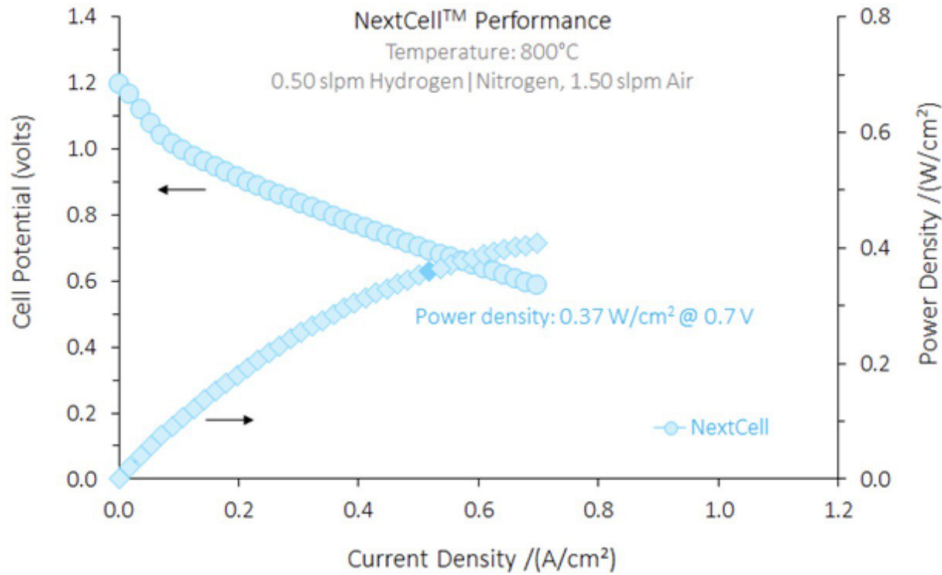
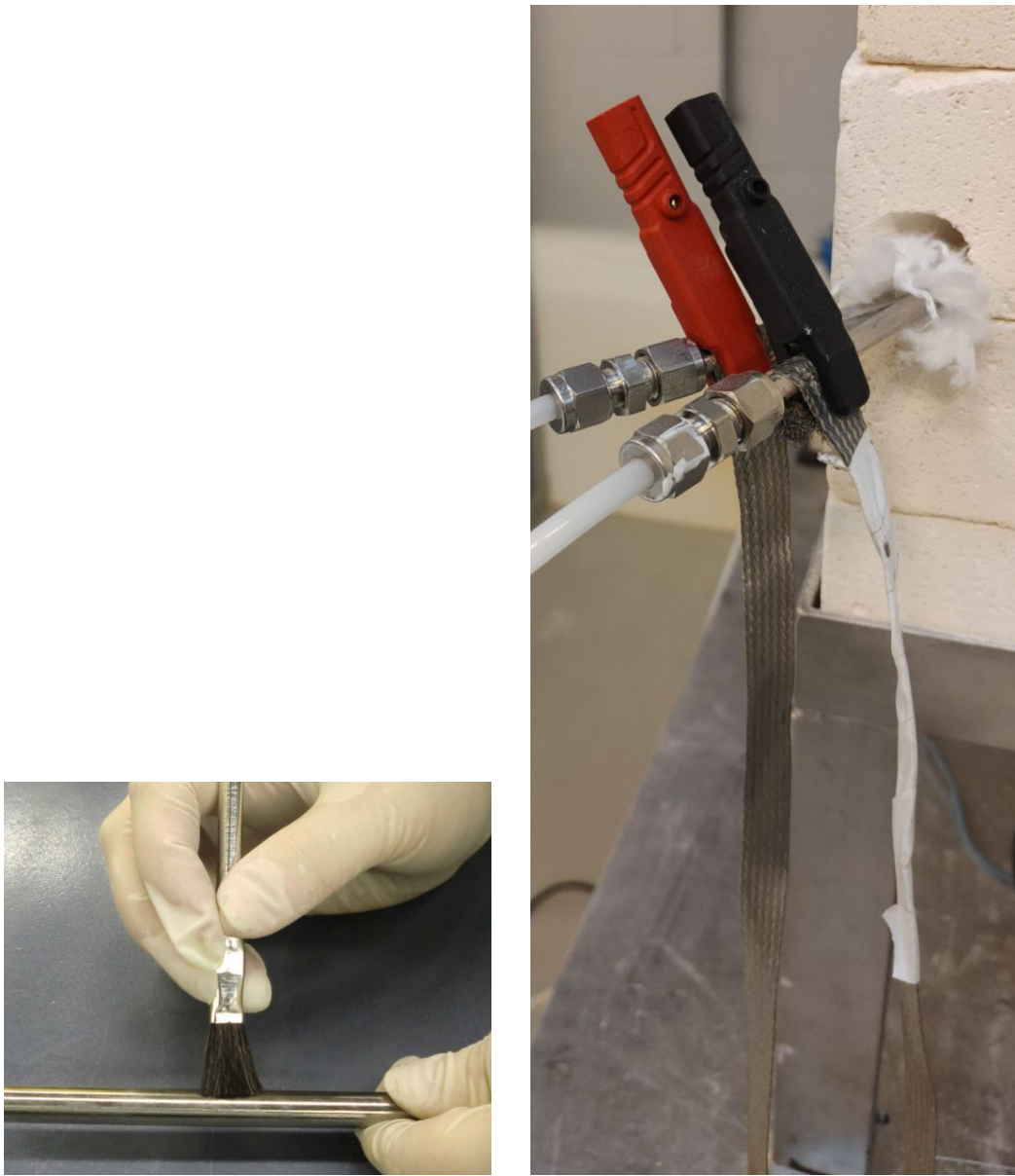


Figure 4.2: *NextCell*TM performances supplied by the company []

4.2 Physical assembly

The steps performed to achieve a correct experiment assembly are reported in this section, according to *Fuelcellmaterials* manual [35]. The main components which will be employed are: 2 cell's housings, *NextCell*TM SOC, platinum wires, cathode silver mesh, anode nickel mesh, 2 sealing frames, copper leads, weights.

1. At first, the operator should check both integrity and clean-up of each component.
2. The operator has to apply a coating of silver-ink, which is provided in the *Fuelcellmaterials* kit, at the outlet ends of the housing's pipes (figure 4.3a). Then, a current lead has to be wrapped and clamped around the end of each pipe, to allow current transmission to the electronic load (figure 4.3b).
3. The operator has to dismount the oven, by carefully removing the refractory bricks. At this point, a basement-brick should be inserted inside the oven, in



(a) *Silver ink application on gas manifold [35]*

(b) *Current leads stapled and clamped to the end of the outlet pipes*

Figure 4.3: Current leads application

bottom-centered position. Its function is to support the cell's housing.

4. The fuel-electrode housing should be placed on a stable surface and a superglue has to be applied on its corners (figure 4.4). At this point, the sealing frame

has to be taped to the housing: the holes for gas distribution must not be covered.



Figure 4.4: Superglue application on the fuel-electrode [35]

5. The operator has to place the nickel mesh on the cell's housing, with the raised pattern facing up, being careful to not cover the gas distribution channels.
6. A platinum wire should be obtained: it has to be long enough to cover the distance from the cell's housing to the furnace's outlet. The operator has to bend it in order to form a hook and place it in the center of the nickel mesh. At the housing's outlet, the wire should be covered with an insulating cloth. In figure 4.5, the up-to-now described configuration is represented.
7. The cell can now be placed in the housing, with the anode compartment (green colour) facing the nickel mesh and the platinum wire.
8. The second platinum wire should be placed above SOC's cathode (black colour), with an analogous procedure to the anode one. It is important for the two wires to not enter in contact: the insulating cloth is employed for this reason, as long as for isolating the wires from the furnace's atmosphere.
9. Cathode silver mesh has now to be placed above the cathode: mesh's channels should be parallel with respect to the housing's pipes.
10. The second sealing frame should be positioned on the cell's boundary, without covering the silver mesh.

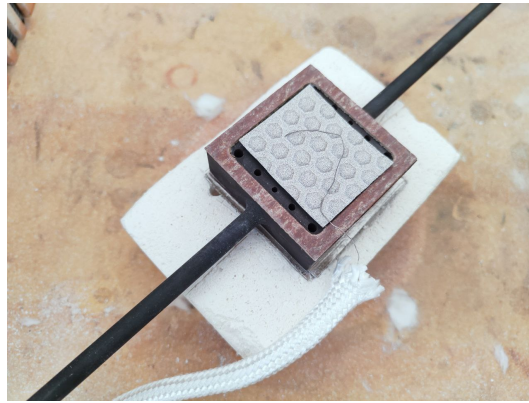


Figure 4.5: Fuel-electrode housing with nickel mesh, sealing and platinum wire.

11. The obtained configuration has now to be placed inside the furnace, above the previously positioned basement brick. Afterwards, it has to be closed with the air-electrode housing, by align it very carefully. In figure 4.6 the complete housing configuration is shown.



Figure 4.6: Complete housing configuration positioned inside the oven

12. The operator has now to close the oven by correctly placing the weights above the housing and by re-positioning the bricks. Proper holes are present in order to assure the passage of the inlet and outlet pipes: the operator should add proper insulation material to avoid heat dissipation. Insulation should be

placed, if needed, also between the two pipes, to prevent any contact. Also the voltage wires must be led out of the furnace, passing through the refractory bricks.

13. Final connections are now required. Voltage wires and current leads must be adequately connected to the electronic load, as shown in figure 4.7. Fuel-electrode inlet pipe has to be connected to the fuel-mixture line through a flexible tube enveloped in a heating resistance (used to prevent water condensation in case of humidified mixture). Air-electrode inlet tube has to be connected with the air line. Fuel-electrode outlet pipe must be connected to a condenser and then to the hood. Air electrode outlet pipe should be conveyed to the hood.

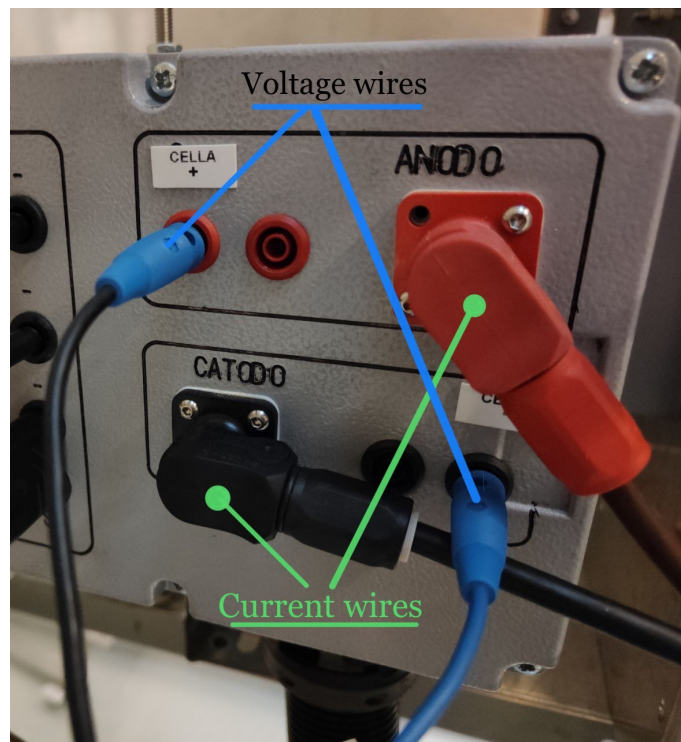


Figure 4.7: Connection of voltage and current wires to the electronic load

4.3 Testing steps and Script development

4.3.1 Conditioning and reduction procedures

Before starting the actual test procedure, cell conditioning and reduction must be carried out, always referring to *Fuelcellmaterials* manual [35]. *Teachy script Editor* will be exploited to create a script file to be uploaded and launched through *Teachy* environment. At first, the oven must be programmed to reach cell's operating temperature:

- The air flow towards the cathodic compartment of the cell is set to 150 [Nml/min].
- A dry flow of 150 [Nml/min] of N_2 is set for the anodic compartment.
- Afterwards, a ramp rate of 60 [°C/h] is fixed for the oven up to 300 [°C], followed by a second ramp of 30 [°C/h] up to 850 [°C]. At the end of the second ramp, the oven is programmed to maintain the temperature for 6 hours, allowing to perform the experiment section.

Since this procedure requires roughly 23 hours, the oven should be allowed to heat overnight.

1	SETVARIABLE(MFC5 AIR 2000.sp,150);
2	SETVARIABLE(MFC7 N2 1000.sp,150);
3	EurothermRLD(60,300,0);
4	EurothermRLD(30,850,6);

Figure 4.8: Script steps for oven's start up

When the desired operating temperature is reached, the reduction procedure can be performed:

- Maintaining the cell at open circuit condition (no current), the flow rate of H_2 in the fuel mixture is brought to 50 [Nml/min], leaving unadulterated the N_2 content.
- At the same time, the air flow to the cathodic compartment should be increased up to 250 [Nml/min].
- A waiting time of 30 minutes should be set, allowing the system to stabilize.

- At this point, the H_2 content in the fuel mixture is gradually increased, decreasing the N_2 percentage: a fuel mixture of pure hydrogen (225 [Nml/min]) is desired. The air flow rate at the cathode is incremented concurrently, up to 750 [Nml/min]. Between two subsequent steps a waiting time of 10 minutes is imposed, allowing the system to adapt to the new operating condition.
- Finally, when the condition of 225 [Nml/min] of H_2 at the anode and 750 [Nml/min] of air at the cathode is reached, a waiting time of about 30 minutes is imposed: the reduction procedure is completed.

1	SETVARIABLE(MFC5 AIR 2000.sp,150);
2	SETVARIABLE(MFC7 N2 1000.sp,150);
3	SETVARIABLE(MFC1 H2 1000.sp,50);
4	SETVARIABLE(MFC5 AIR 2000.sp,250);
5	WAIT(1800);
6	SETVARIABLE(MFC1 H2 1000.sp,75);
7	WAIT(600);
8	SETVARIABLE(MFC7 N2 1000.sp,100);
9	SETVARIABLE(MFC1 H2 1000.sp,125);
10	SETVARIABLE(MFC5 AIR 2000.sp,375);
11	WAIT(600);
12	SETVARIABLE(MFC7 N2 1000.sp,50);
13	SETVARIABLE(MFC1 H2 1000.sp,175);
14	SETVARIABLE(MFC5 AIR 2000.sp,500);
15	WAIT(600);
16	SETVARIABLE(MFC7 N2 1000.sp,0);
17	SETVARIABLE(MFC1 H2 1000.sp,225);
18	SETVARIABLE(MFC5 AIR 2000.sp,625);
19	WAIT(600);
20	SETVARIABLE(MFC5 AIR 2000.sp,750);
21	WAIT(1800);

Figure 4.9: Script steps for cell's reduction procedure

4.3.2 Polarization test

Before starting the actual test, an activation procedure is performed manually through *Teachy* environment. The cell is subjected to $0.5 [A/cm^2]$ ($12.5 [A]$) for

a couple of hours [4], until voltage level becomes stable. In this way, the oxygen which is still bounded to nickel is removed and the electric contact among grid and electrode is enhanced [4].

The polarization test can be programmed through *Teachy Script Editor*, thanks to the specific designed command (explained in section 3.4.4):

- For this test section, a flow rate of 225 [Nml/min] of pure dry H_2 is set for the anode. A flow rate of 750 [Nml/min] of air is sent to the cathode. Anyhow, for future tests, it is meaningful to plot the polarization curve under different operating mixtures and humidification factors.
- The HSDB LOG is activated for the group of variables which includes cell's current and voltage. In this way, voltage and current values are recorded more frequently, which is useful for the computation of the polarization curve.
- The i-V curve command is inserted. Starting from OCV condition (0 [A]), current will be incremented of 1 [A] every 60 seconds. Maximum current value is set to 13 [A], minimum voltage value to 650 [mV] and maximum time to 4000 [s].

1	SETVARIABLE(MFC5 AIR 2000.sp,750);
2	SETVARIABLE(MFC1 H2 1000.sp,225);
3	HSDBLOG(4000,TensioneCella);
4	IVCURVE(1,0,650,300,Vcell.pv,13,4000);

Figure 4.10: Script steps for cell's polarization test

4.4 Results

The results extrapolated through *Taechy* are reported in figure 4.11 and discussed in this section.

The test bench has been found to work properly, allowing to complete the entire experiment procedure. Anyhow, the resulting polarization and power curves highlight a deficit in SOFC's performance. For comparative purposes, the 850 [°C] curves provided in the data-sheet are also reported in figure 4.12, in blue colour. Although they have been performed under different operating conditions (different air flow rates), the gap with respect to the curves derived by our test is too high. In fact, the data-sheet's OCV is equal to 1.2 [V], while the one obtained through the test is 1.096 [V]. Moreover, the voltage drop obtained through the test is very

high compared to the one expected. As visible by comparing figures 4.11 and 4.12a, during the test the voltage of 600 [mV] is reached at 0,2 [A/cm^2], against the 0.8 [A/cm^2] (roughly) of the data-sheet curve. Of course, these voltage values result in a poor power density.

The voltage deficit can be derived by an imprecise mounting of the cell in the housing or by possible damages in the cell itself. Moreover, an uneven temperature distribution is present in the furnace: the actual cell's temperature could be lower than 850 [°C]. More tests should be performed in order to investigate the error's source.

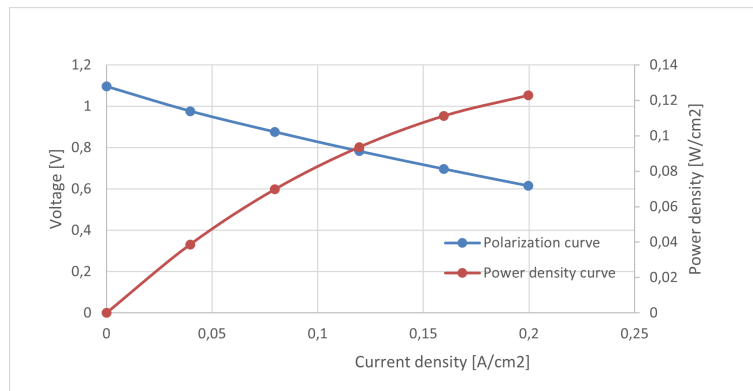
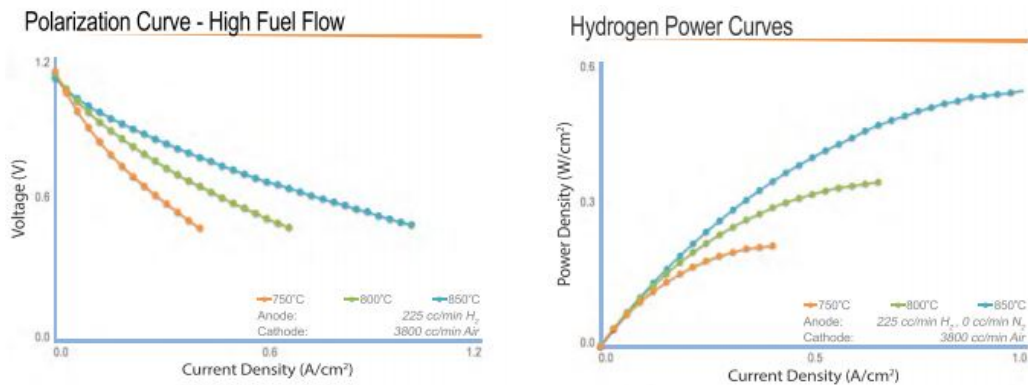


Figure 4.11: Polarization and power density curves computed at 850 [°C] with 225 [Nml/min] of H_2 at the anode and 750 [Nml/min] of air at the cathode.



(a) Polarization curves provided in the data-sheet under different operating temperatures [34] (b) Power density curves provided in the data-sheet under different temperatures [34]

Figure 4.12: Data-sheet curves

Chapter 5

LabVIEW

5.1 Introduction

LabVIEW software has been employed to program the CompactRIO of the previously analysed Test Bench. To fully understand and investigate *LabVIEW* environment, a simpler system has been realised. In particular, it involves less instruments than the actual test bench and a CompactDAQ, which is directly connected to a local PC. *LabVIEW* software has been exploited to manage and control the new system, similarly to what was done with the actual test bench. The aim of this chapter is to discuss the realised system.

5.2 Hardware description

In this section, employed devices and their physical connections will be described. Despite the lower grade of complexity with respect to the actual test bench, the new system allows to understand the connections between measuring/control devices and National instruments.

The main instruments employed are:

- *Bronkhorst EL-FLOW Select F-201CV* mass flow controller [18]. This MFC is designed to work with CO_2 and can withstand a maximum flow rate of 5000 [Nml/min]. The AI/AO signals range between 4-20 [mA]. The supply voltage must range between +15-24 Vdc.
- *Bronkhorst EL-FLOW Seelect f-111b* mass flow meter [36]. This MFM is designed to operate with H_2 and can withstand a maximum flow rate of 2000 [Nml/min]. The AO signal ranges between 4-20 [mA]. The supply voltage must range between +15-24 Vdc.

- Thermocouples K and B.
- *National module NI-9265* [37]. It is an analog output module, with an output range of 0-20 [mA], suitable for the connection with the mass flow controller.
- *National module NI-9208* [38]. It is an analog input module, with input range of +/- 20 [mA], suitable for the connection with both the mass flow controller and the mass flow meter.
- *National module NI-9213* [39]. It is a module specifically designed for connection with thermocouples, with a voltage range of +/- 78 [mV].
- *National CompactDAQ* [40]. It is the data acquisition platform where the national modules are placed. It is directly connected with a local PC through a USB cable, to allow data exchange and powering up. Therefore, the system under analysis can be managed only locally, differently from the test bench.
- Local PC with *LabVIEW* software [41].
- Transformers used as power supply.

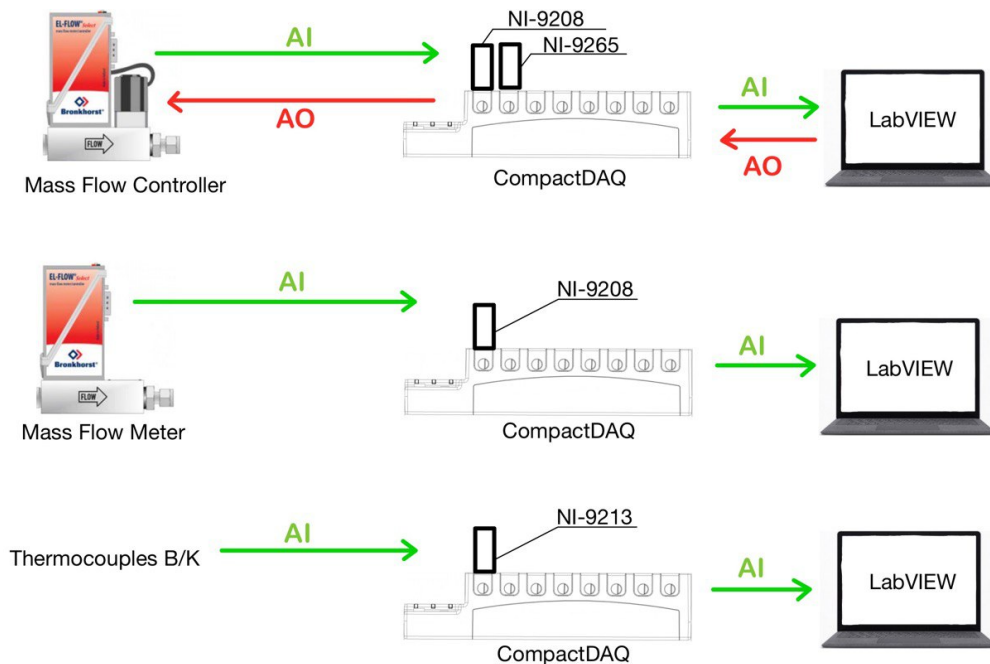


Figure 5.1: Scheme of devices data exchange [42], [16]. AI/AO signals are intended with respect to National modules and PC.

In figure 5.1 a schematic diagram represents AI/AO signals exchange in the system. As visible, the two thermocouples and the mass flow meter send AI signals to the specific National modules, that transfer them to the local PC through the compactDAQ. On the other hand, the mass flow controller both sends AI signals and receives AO signals. Therefore, the operator can:

- Control the mass flow rate of MFC;
- Monitor the temperatures measured by the two thermocouple elements;
- Monitor the mass flow rates of MFC and MFM.

The physical connections among measuring/control devices and National modules have been realised by connecting the respective terminals through male-male jumper wires. The pin-out diagrams of the National modules [43], [44], [45] and the hook-up diagrams of MFC and MFM [46] have been consulted, in order to perform correct connections.

The mass flow meter has been connected to the National module NI-9208 as represented in figure 5.3. As prescribed, the analog output terminal (2) and the 0V sense terminal (8) of the mass flow meter have been connected respectively to the Analog input terminal (2) and to the COM terminal (9) of the National module. COM terminal is the Common reference connection to isolated ground.

The MFM has been powered up through a transformer, connected to a proper power supply (+15-24Vdc). As prescribed, the 0V power terminal (4) and the 0V sense terminal (8) of the MFM have been connected together to the negative terminal of the transformer. The +Us (7) terminal has been connected to the positive terminal of the transformer.

The Mass flow controller has been connected to the National modules NI-9208 and NI-9265, as represented in figure 5.2. The connections to NI-9208 and to the powerup-transformer are analogous to the ones of the MFM. For the connection to NI-9265 module, as prescribed, the Analog Output terminal (3) and the 0V sense terminal (8) of the MFC have been connected respectively to the Analog output terminal (0) and to the COM terminal (1) of the National module. NI-9265 requires separate powering up: also in this case a transformer has been exploited by connecting Vsup (8) and COM (9) terminals of the NI module with the terminals of the transformer.

Thermocouples connections have been performed by connecting the positive and negative wires of each thermocouple to the positive and negative connections of the National module NI-9213.

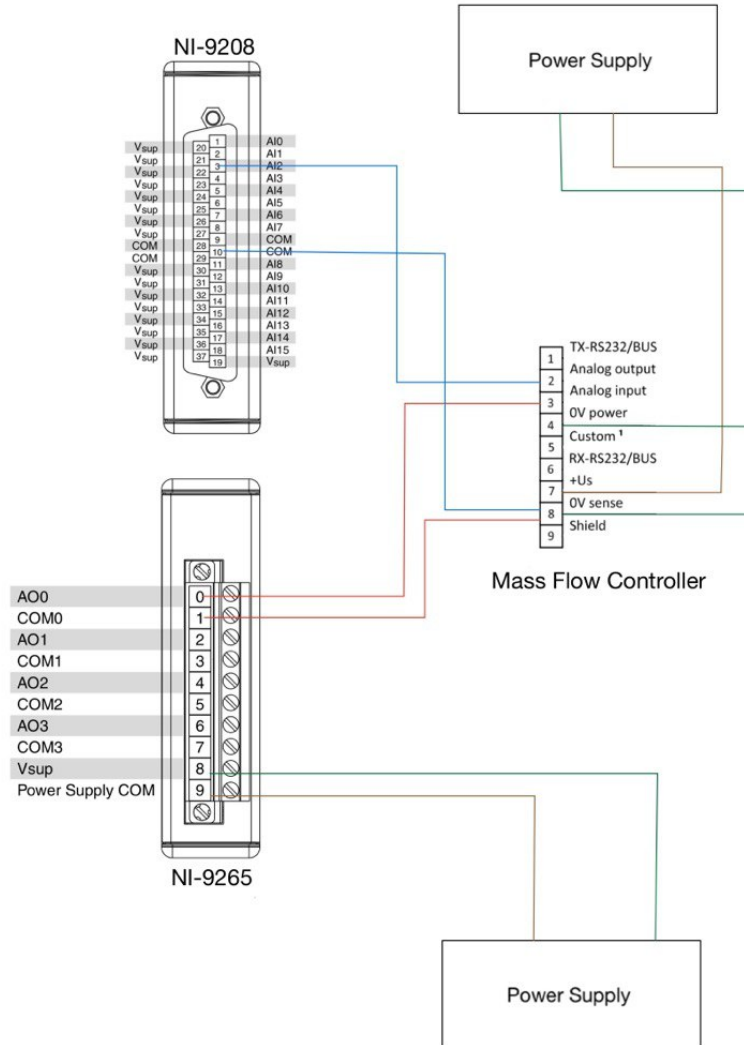


Figure 5.2: Physical connections of the mass flow controller with National modules and power supplies [43], [45] [46]

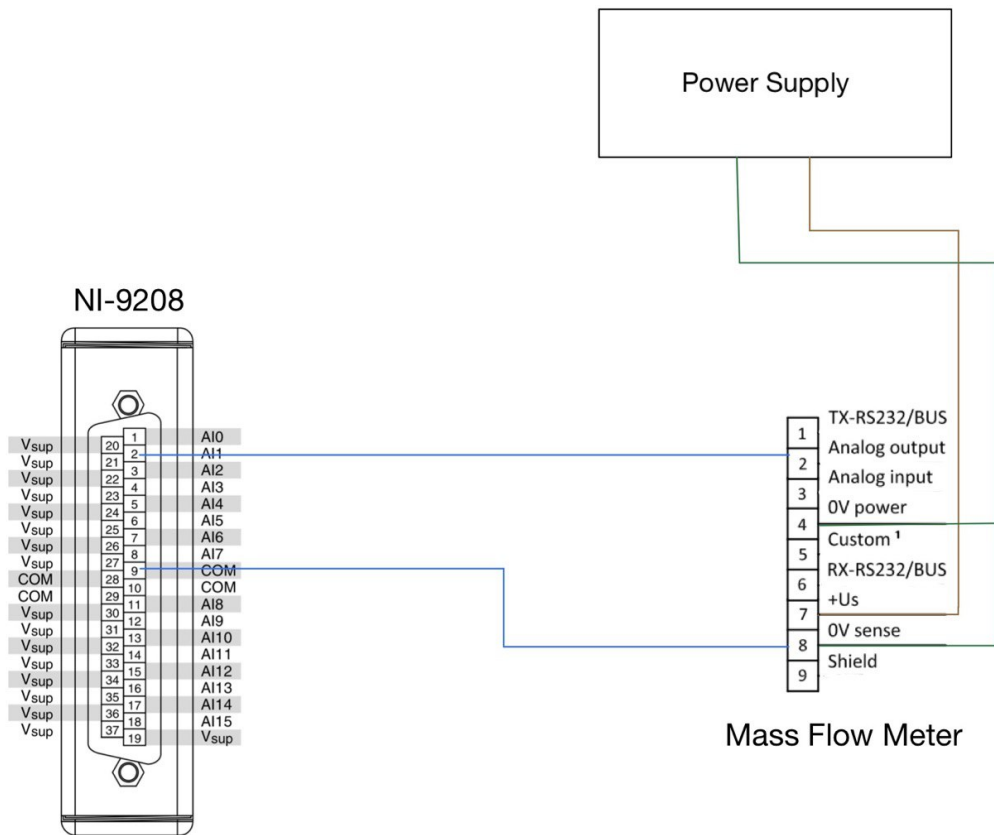


Figure 5.3: Physical connections of the mass flow meter with National module and power supply [43], [46]

5.3 Software description

The user interface is available on a local PC, connected to the CompactDAQ unit. *LabVIEW* software has been employed to manage the realised system, allowing data acquisition, conditioning and controls implementation.

The current section is intended to briefly describe how the system has been implemented in *LabVIEW* and how to manage it.

LabVIEW programming environment [47] is mainly divided into two subsections:

- **Front Panel:** represents the user interface. It involves indicator and control blocks. The first ones are used to display data, charts and other information. The second ones are used to insert input data.

- **Block Diagram:** it is used to elaborate and condition data through a graphical source code. The commands and indicators employed in the Front Panel appear as input and output terminals in the Block Diagram, allowing data exchange between the two subsections. Besides the terminals, also function blocks, connection wires, constants and other blocks can be employed.

5.3.1 Data Acquisition and Conditioning

The first step towards system's implementation on *LabVIEW* is to acquire data from the measuring devices. The *DAQ Assistant Block* must be inserted in the Block Diagram to meet this request. The block must be initialized by specifying, for each measuring device, the type of acquired input signal and the specific National module which acquires it.

Thermocouple's signals are automatically converted in [°C] and do not need any further conditioning. On the other hand, mass flow meter and controller signals must be converted from [A] to [Nml/min]. The conversion can be performed in the *Block Diagram* through mathematical operators and constants. In performing the conversion, the signal range and the mass flow rate range, specified in section 5.2, must be used.

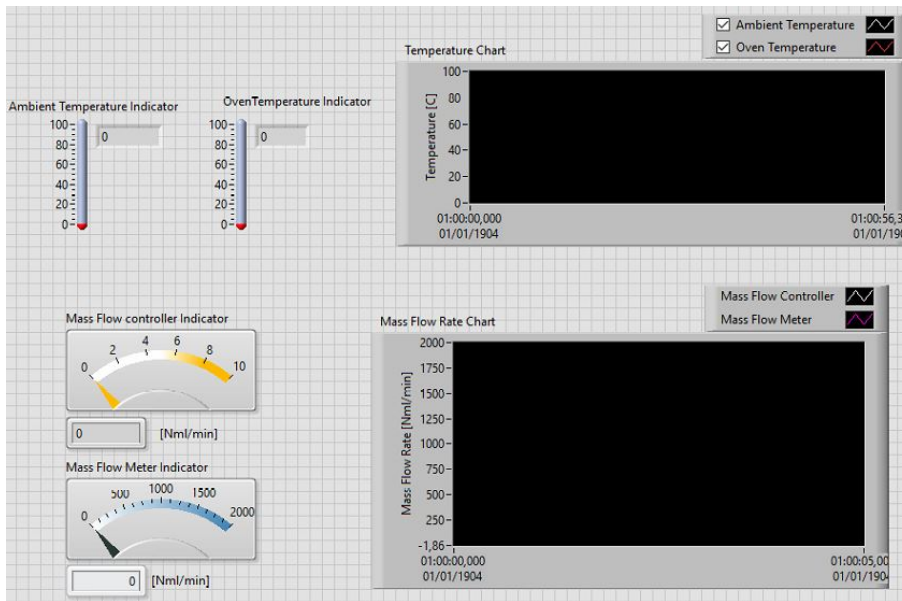


Figure 5.4: Front panel: Indicators are positioned on the left, Waveform charts on the right.

The acquired and converted data must be displayed to the operator. In the Front

Panel, which represents user's interface, instantaneous values can be visualized through proper indicators. Data's history can instead be plotted in Waveform Charts.

As visible in figure 5.4, indicators are positioned on the left, reporting temperatures of B/K thermocouples and mass flow rates of MFC/MFM. On the right, Waveform charts are present, plotting temperatures and mass flow rates changing in time.

Once inserted in the Front Panel, indicators and charts appear as terminals in the Block Diagram and must be properly connected to the *DAQ Assistant* outputs.

The acquired signals can be written and stored on a .xlsx file. In the Block Diagram, "Write to Measurement File" block can be exploited for this purpose, by connecting it to the *DAQ Assistant* output.

5.3.2 MFC control

The control of the mass flow controller should now be implemented. A second *DAQ Assistant* block must be inserted in the *Block Diagram*. This time, it must be initialized to generate an analog output signal of "current" type. The National module and the physical channels sending the signal must be specified. Finally, signal range and scale unit must be inserted.

The *DAQ Assistant* block must receive as an input the AO current signal expressed in [A]. In the front panel, a control block is inserted to allow the operator to set the desired mass flow rate in [Nml/min], as shown in figure 5.5. In the Block Diagram, the control block appears as a terminal: through constants and mathematical operators, the conversion from [Nml/min] to [A] is performed.

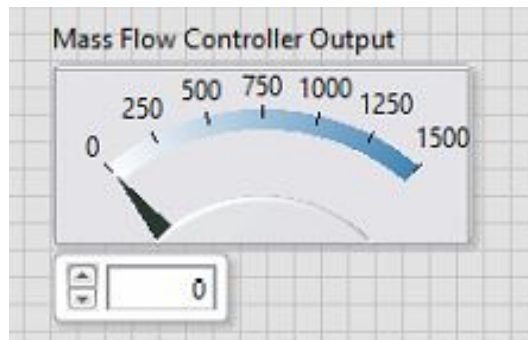


Figure 5.5: Control block for the mass flow controller: the operator can set the desired mass flow rate

5.3.3 While Loop and implementations

A *While Loop*, including all the above mentioned blocks, should be inserted in the Block Diagram. *While Loop* allows to repeat the code until an exit condition is met. In this specific case, it has been chosen to allow the operator to set a time duration of the experiment section: the expiration of that time coincides with the exit condition of the *While Loop*.

Further implementations have been developed:

- An indicator has been inserted in the Front Panel, displaying the volume of gas passed through the mass flow controller from the beginning of each experiment section.
- An alarm-indicator block has been inserted in the Front Panel, to warn the operator when the temperature measured by thermocouple K is too high. In normal operation it is a green button and it becomes red when the limit is exceeded.
- A second alarm-indicator has been inserted in the Front Panel, to warn the user whether the mass flow rate command imposed to the MFC exceeds the upper limit of 5000 [Nml/min]. In normal operation it is a green button and it becomes red when the limit is exceeded.
- When the temperature measured by thermocouple K is outside of a predefined range, the mass flow rate flowing in the MFC is stopped.

In figure 5.6, the final configuration of the Front Panel is reported.

5.4 Testing and applications

The system has been tested in all its components, in order to check the correct operation.

Mass flow controller and mass flow meter have been connected in series to the laboratory's gas manifold. They have been fed with N_2 . Since it is not the gas they are designed for, the use of N_2 can lead to a small measuring/control error: anyhow, it is acceptable for the target of this experiment. Through LabVIEW, MFC control and MFM monitoring has been performed.

Thermocouple K, designed for lower temperatures, has been tested at ambient condition, eventually heating it up manually.

Thermocouple B, designed for higher temperatures, has been inserted inside the oven of the analysed test bench. In particular, it has been positioned in a central position, to monitor the temperature of the cell during the oven's heating up.

System management and monitoring has been found to work for all components.

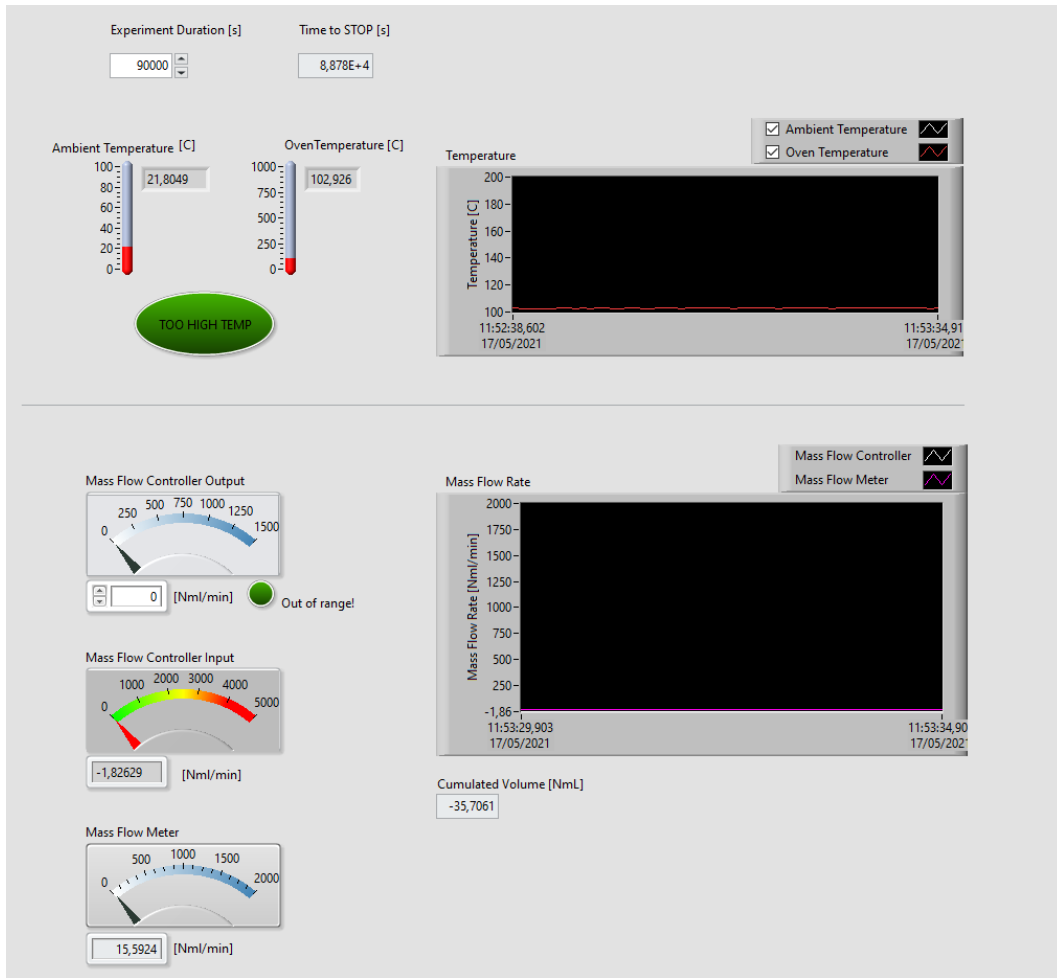


Figure 5.6: Front Panel final configuration

It can eventually be exploited and adapted to different applications. In particular, thermocouple B can be implemented in the *Politecnico* test bench in order to monitor cell's temperature during tests.

Chapter 6

Conclusions

The present work provided a user guide for the utilization of an experimental test-bench, designed for remote testing and characterization of single solid oxide cells. An example of complete acquisition and control system for mass-flow controllers and thermocouples based on National Instruments hardware and LabView software has been implemented to provide insights on the acquisition and control architecture of the test-bench.

The main conclusions regarding this work are inherent to possible future improvements: in fact, being a just-born test bench, many adjustments can be implemented to better exploit it.

At first, a larger number of experiments sections should be carried out. Until now, most of the work has been devoted to the test bench development and to *Teachy* software testing and improvement. Therefore, it is now important to better investigate possible problems and solutions in solid oxide cell testing. As explained in Chapter 1, many test and characterization techniques can be implemented with this experiment configuration. Moreover, other cell's typologies could be used (for example, anode supported SOCs).

Another important step consists in implementing a temperature control of the solid oxide cell. For this purpose, a thermocouple should be placed in the central part of the oven. The simple system developed and explained in Chapter 5 can be exploited: thermocouple B has successfully been tested for monitoring cell's temperature inside the oven and could be integrated in the test bench. It would also be interesting to plot the temperature gradient inside the oven, since it is affected by an uneven temperature distribution: the central part reaches the set point temperature slower compared to the walls, where the resistances are placed. Regarding the mass flow controllers: the oldest ones, generally affected by a lower accuracy, could be replaced. Appendix C can be consulted to detect the less precise ones. Moreover, MFC9 (dedicated to CO_2) is currently unavailable and should be substituted. Mass flow meters can be also added on gas streams exiting the cell for

a continuous measure of the flow, useful to provide a leakage estimation. A more complex implementation could finally be the remotization of control valves, which are currently manually-controlled; same considerations can be done for the test bench switching-on.

Appendix A

P&id diagram of the test bench

In figure A.1 the p&id diagram of the analysed test bench is reported.

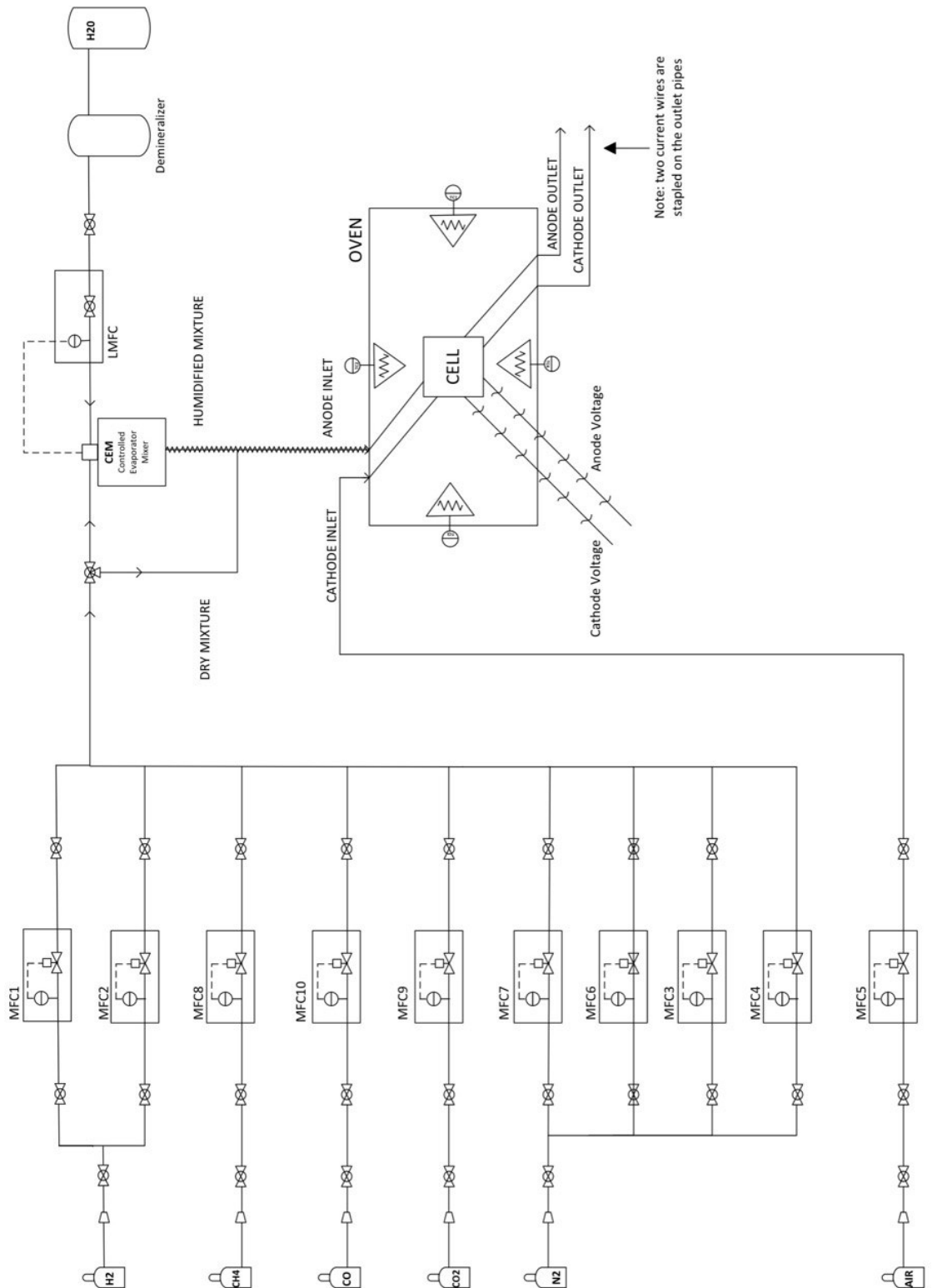


Figure A.1: P&id diagram of the test bench

Appendix B

ATEX document

To consult the ATEX document, professor and researcher Domenico Ferrero can be contacted at the following email address:
domenico.ferrero@polito.it

Appendix C

Mass Flow Controllers calibration

In the following tables the percentage errors of each mass flow controller are computed for three different flow rates, according to the procedure explained in section 2.2.3. MFC9 is not present since it is currently unavailable. In each table: *Set point value* refers to the flow rate inserted by the operator; *Measured value* refers to the value measured by *Definer 220* flow meter; *Read value* refers to the value measured by the mass flow meter itself; *Set point error* is the percentage error in the MFC control, computed as $\frac{Measuredvalue - Setpointvalue}{Setpointvalue} * 100$; *Read error* is the percentage error in the MFC measurement, computed as $\frac{Measuredvalue - Readvalue}{Readvalue} * 100$.

Table C.1: Mass Flow Controller MFC1

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50,00	55,95	52,00	11,90	7,60
100,00	102,50	101,80	2,50	0,69
300,00	302,80	302,00	0,93	0,26

Table C.2: Mass Flow Controller MFC2

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50,00	55,50	50,00	11,00	11,00
100,00	102,50	100,00	2,50	2,50
300,00	296,70	301,00	-1,10	-1,43

Table C.3: Mass Flow Controller MFC3

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50,00	47,70	50,00	-4,60	-4,60
100,00	99,10	101,00	-0,90	-1,88
300,00	299,50	301,00	-0,17	-0,50

Table C.4: Mass Flow Controller MFC4

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50,00	57,50	51,00	15,00	12,75
300,00	299,80	301,00	-0,07	-0,40
500,00	491,50	501,00	-1,70	-1,90

Table C.5: Mass Flow Controller MFC5

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
200,00	210,20	202,40	5,10	3,85
280,00	292,00	282,40	4,29	3,40
400,00	413,50	403,20	3,38	2,55

Table C.6: Mass Flow Controller MFC6

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50,00	56,50	50,00	13,00	13,00
100,00	108,40	100,00	8,40	8,40
300,00	307,70	300,00	2,57	2,57

Table C.7: Mass Flow Controller MFC7

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50,00	51,20	51,00	2,40	0,39
100,00	101,30	101,00	1,30	0,30
300,00	300,30	301,00	0,10	-0,23

Table C.8: Mass Flow Controller MFC8

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50,00	51,40	50,30	2,80	2,19
100,00	99,90	100,20	-0,10	-0,30
200,00	198,50	200,00	-0,75	-0,75

Table C.9: Mass Flow Controller MFC10

Set point value [Nml/min]	Measured value [Nml/min]	Read value [Nml/min]	Set point error [%]	Read error [%]
50	60,75	50,30	21,50	20,78
100	111,40	100,40	11,40	10,96
300	310,20	300,40	3,40	3,26

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