Chapter

Polypropylene Compounds for PEM-Electrolyzer Plates: Materials, Processing Methods, and Analysis of the Materials

Venkatesan Prassad, Thorsten Hickmann, Engelke Martin, Klemp Eric and Derieth Thorsten

Abstract

Titanium and graphite-filled composites together with a polymer, such as polypropylene, are a suitable material for bipolar plates in PEM electrolyser applications. Similar to pure titanium metals, titaninium and graphite composite plates have quite good properties when mixed with polypropylene (PP). The plates have relatively low electrical resistance and can withstand the aggressive electrochemical environment encountered in an electrolyser. The main challenges here are resistance to oxygen, hydrogen, and complete. The conditions in operation are extreme. Above all, the comparatively high voltage and the fact that pure water is used generally cause problems for the materials. Besides pure titanium, composite materials can also be used. This chapter summarizes the most important requirements for the titanium-PP composite material. This material is used for bipolar plates. This is the basis for the characterization methods of titanium-based bipolar plates. The modern PP-based composites and their general properties are described, with a focus on improved long-term stability. In this chapter, the material fillers such as titanium and graphitic powders are analysed. The last deals with the way the electrical conductivity of the materials is measured and with *in situ* operation.

Keywords: bipolar plate (BPP), titanium compound, graphite compound plates, recycling, titanium plate, polypropylene (PP)

1. Introduction

Polymer electrolyte electrolyzers (PEMEL) are electrochemical reactors. They are used for creating hydrogen from electrical energy through chemical reactions [1]. Generally, the reaction takes place at a temperature level of around 70°C and in an aggressive media, where hydrogen and oxygen is produced. The main area of the reaction is located in the stack, which consists of the elements: membrane, gaskets porous transport layer, and bipolar plates. The bipolar plates are made of titanium or titanium composite, which is necessary for the electrochemical electrical atmosphere.

PEMEL has reached a high level of technical maturity. The systems are available on the market through many suppliers. However, due to a high remaining cost structure—partly due to a lack of economies of scale—the profitable market introduction of PEMEL a not so far at a price level, so that there are still on the way to being accepted widely in the market. The main cost driver of the system is the stack, and here are the abovementioned components, which means also the bipolar plates.

Here, the idea is to replace the pure titanium with a mixture of plastic and titanium powder, namely the polypropylene composite titanium plates. The bipolar plate is part of the complete system. It is necessary to look at some characteristics, such as total dimensions, total weight, and thermal and electrical properties. In other words, the system is determined by the membrane unit and the bipolar plate technology [2].

In general, there are two different types of bipolar plate technologies. The one based on metal and other one based on titanium powder as a conductive component. The latter can be a mixture of polymer and pure Ti-powder. The compound plates with titanium integrediantes have some advantages since it is possible to use them also for small-volume production. As a counterpart, the pure metal plates are inherently prone to corrosion under aggressive oxidizing conditions in a PEME.

Considerable effort has been spent in the development of protective coatings for steel plates, both precious metal coatings and, preferentially, cost-effective coatings and surface treatments. However, for PEMEL applications, the corrosion problem of pure metal plates appears to be challenging now, so that composite-based materials are the preferred material.

2. Technical requirements of bipolar plates

Technically, the bipolar plate of the PEMEL has to fulfill the following functions [3, 4]:

- The plate is responsible for electrical current.
- The plate is usually a heat-conductor and can distribute the coolant.
- The plate is also called a separator plate. It distinguishes gases of neighbored cells.
- The plate is responsible for the transport of product water into the cell within the flow field.
- The plate provides mechanical stability for the stack.

Based on these basic functions of each component, the technical requirements for plates are described in the next section.

Due to the corrosive conditions of acid contact, presence of oxygen, electrochemical potential, and respectively high temperature, in most applications titanium plates and titanium and sometimes graphite powder [3] or titanium powder plates with polymer binders are used in these PEMELs.

Composite plates are a superior material regarding their stability under corrosive conditions. However, composites require thicker plates than metal plates, resulting in more weight and more volume of the stack. In terms of cost, for sure the MEA is

Technical property	Units	Targeted value
Plate weight	$\frac{kg}{kW}$	< 0.4
Electrical conductivity Depending on type	$\frac{S}{cm}$	> 100
Corrosion resistance	$\frac{\mu A}{cm}$	< 1
Flexural strength	MPa	> 34
Temperature reliability Thermo-mechanical test	°C	> 70
Acid uptake Depends on application or technology		Low

Table 1.
Benchmarks for bipolar plates in redox-flow applications defined by DoE [8] and experiences from customer requirements from Eisenhuth GmbH & Co. KG.

considered to be the dominating part of the stack, however, the bipolar plates tend to be underestimated both regarding their technical requirements and their contribution cost structure.

The chemical conditions for the materials used in PEMEL are challenging [3]. Most systems are operated between 40 and 90°C in a liquid of deionized water. Besides the PEMEL technology, there are also some other technologies (alkaline or anionic exchange membrane technology). In this context, we will focus on PEM technology and not on alkaline technology.

Due to these harsh conditions in a PEMEL, graphite or also titanium-based bipolar plates with polymeric binders can be used in the applications in PEMEL stacks [4]. The graphite composite plates are a suitable material in terms of stability under the above-mentioned corrosive conditions, especially when used on the hydrogen side, since is not so abrasive. The titanium based powder is used on the Oxygene = cathode side of the PEMEL, where it is more aggressive than the oxygen side [5].

Graphite or titanium composite-based bipolar plates are manufactured with two elements: filler and polymer. The plates contain fillers like graphite and/or other electrically conductive carbons incorporated in polymers performing as a gluing binding matrix. The second option is to use titanium powder.

The production technologies for the plates can be either compression molding or injection molding or continuous extrusion. All the production processes are very sensitive to process parameters and need to be carefully controlled. The objective is to manufacture bipolar plates in large volumes and high quality more or less like standard plastic parts [6, 7].

Since the definition of compound materials is not been so far published, we stick here to the recommended characteristics of properties defined for fuel cells for composite bipolar plates: the department of energy (DoE) figures are shown in **Table 1** for bipolar plates [8]. Most of the targeted values can be directly transferred to the PEMEL technology.

3. General concepts of bipolar plate manufacturing

As mentioned before, the composite bipolar plate has polymer on one side and filler on the other side. Generally speaking, the filler content is above 80 wt.% and

< 20 wt.% binder polymer [6]. The function of the titanium or carbon filler is to provide electrical and thermal conductivity.

Therefore, a three-dimensional percolating carbon structure is required. Usually, the main carbon component of the plate is either titanium powder or synthetic graphite. For producing plates, several options are possible [7]:

- Compression molding
- Injection molding
- Plate extrusion
- Foil extrusion

3.1 Binder polymers

Different concepts of polymer binders can be applied to bipolar plates. At this point, it is helpful to give a small overview.

In order to understand the different types of polypropylene, it is helpful to look at the thermoplastics categories. Thermoplastics are divided into two groups: commodity thermoplastics, this group covers the major plastics, such as polyethylene, polypropylene, polystyrene, and polyvinyl chloride. Engineering thermoplastics are the second thermoplastics group. This group is often described as materials for electrical and mechanical engineering applications. These include polysulfone, nylons, polycarbonates, acetal, and ABS terpolymers [9].

According to Karger-Kosic/Barany [10], the polymer is well known existent in the market for many years. Due to its low price, polypropylene (PP) is a commodity, which means it is very often used worldwide. The PP has excellent properties and is also acid resistant.

The formula for polypropylene is as follows (**Figures 1** and **2**):

or in short-form.

According to Serban [12] is a polymer that is the basis of CH3 and H is very often repeated. In addition to that, PP is a thermoplastic polymer is often combined with different fillers (UV-resistance, bending properties etc.) in a huge range of different applications [14].

In this chapter, we will focus on polypropylene for the reasons of pricing and good characteristics. However, polypropylene is a closer distinction necessary, since there are several types existing.

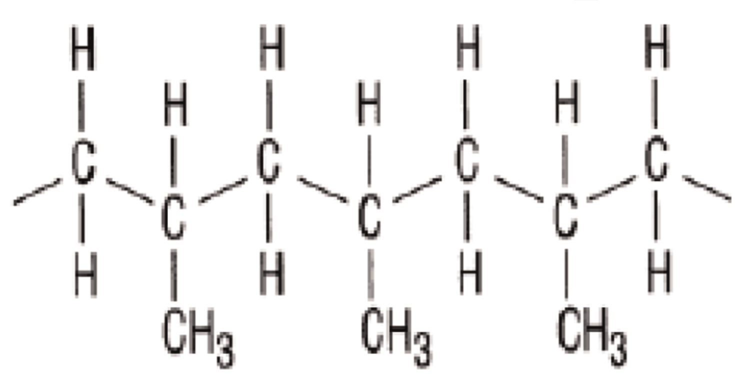

Figure 1.
The formula of polypropylene (long version) [11].

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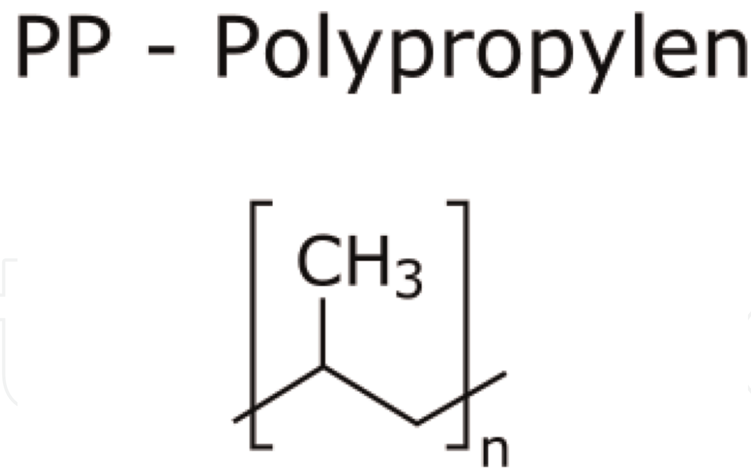

Figure 2.
Formula of polypropylene (short version) [11].

Generally spoken, there are three different types of polypropylene [14]:

1. Homopolymer: Polypropylene containing only propylene monomer in a semi-crystalline solid form.

2. Random copolymer: Polypropylene containing ethylene as a comonomer in the PP chains at levels in the range of 1–8% and this is referred to as a random copolymer (RCP).

3. Imapct compolyers: Polypropylene containing an ethylene content of 45–65%.

Not only in the hole market, but also for fuel cells and electrolyzer, homopolymer PP is the most widely used polypropylene material. It is followed by random copolymers, whereas impact copolymers are not very often used, due to the reason for mechanical behavior [14].

For the homopolymers, it has the characteristics such as a fairly high at a high level of stiffness, but a lower impact strength. As far as the melting point is concerned. It is lower. Random copolymers are ethylene/propylene copolymers that are produced in one process step by using more than 90% of polypropylene and 7% of polyethylene. For mechanical and process reasons, the approach of using a PP in a compound as a hompolymer and as a copolymer [13].

3.2 Graphite materials and fillers

Polypropylene is normally insulator with a surface resistivity of around 1014–1018 Ohm X cm. The conductivity can be achieved by adding conductive fillers into it. The electrical conductivity is realized with carbon black, graphite, or titanium powder [15].

In **Figure 3**, it can be seen that the structure of the material highly depends on the chemical composition. It can be seen that the polymer is represented by the black particles, whereas the titanium powder is represented in the white articles. As mentioned before, the complete processing chain—from the raw material to the molded plate—has to be carefully controlled to ensure the consistency and reproducibility of the bipolar plates [16].

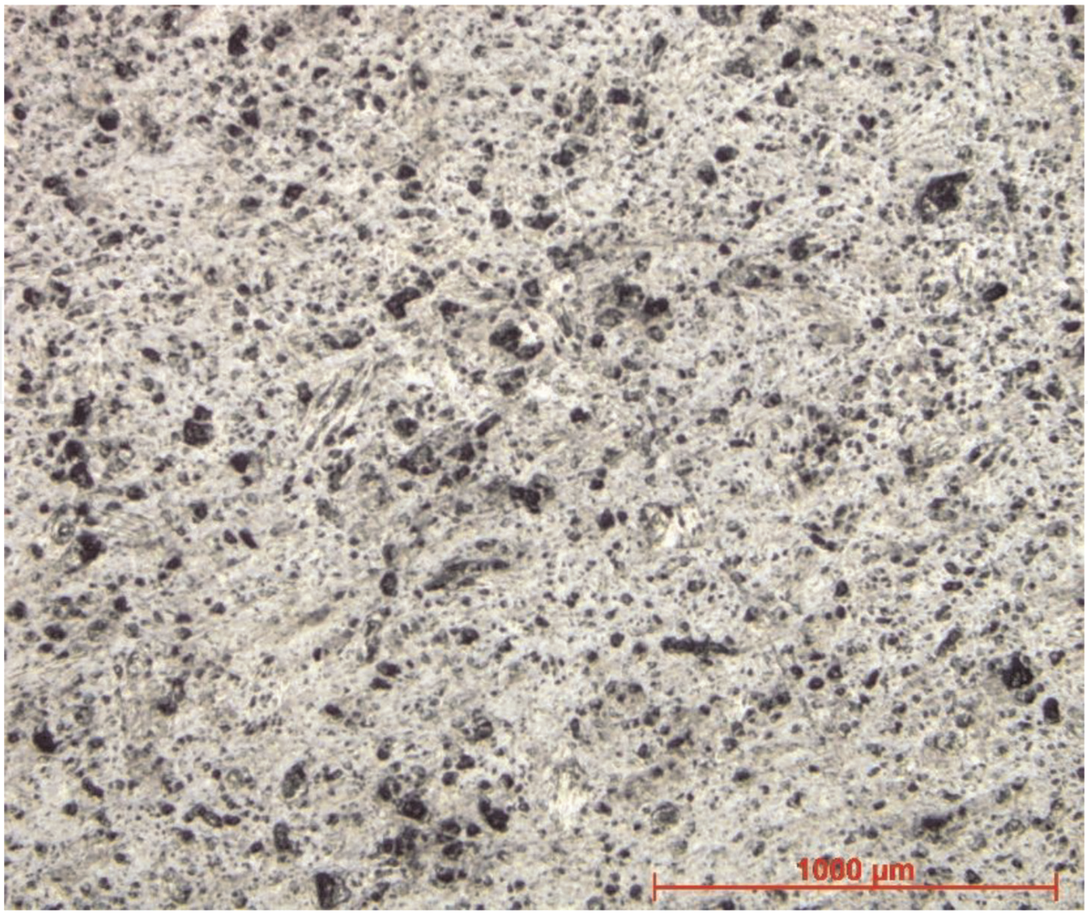

Figure 3.
Polarization microscopy of the polished surface from the bipolar plate with 80 wt.% graphite content. Particles (black) are locatable in a polymer matrix (white).

For the composition of the compounds, there are some aspects that need to be considered:

- High purity

  Since the electrolyzer as well as the fuel cell membranes and catalysts are highly sensitive against contamination with iron ions, it is crucial to have a very well-composed material.

- A higher range of different particle sizes. A good mixture of different particle sizes helps to receive better electrical conductivity [14, 17].

3.3 Titanium powder as fillers

Parallel to the graphite fillers, titanium powders can be used with different particle sizes. The morphology test was performed on the manufactured composite blank plates after trimming them to a thickness of 3 mm using a CNC milling machine. The morphology test results are displayed in **Figure 4**.

From the appearance, we can easily anticipate that all the PP composite plates have a smooth finish and the grain structure also has good adhesion with the polymers, resulting in a very minute or no pores.

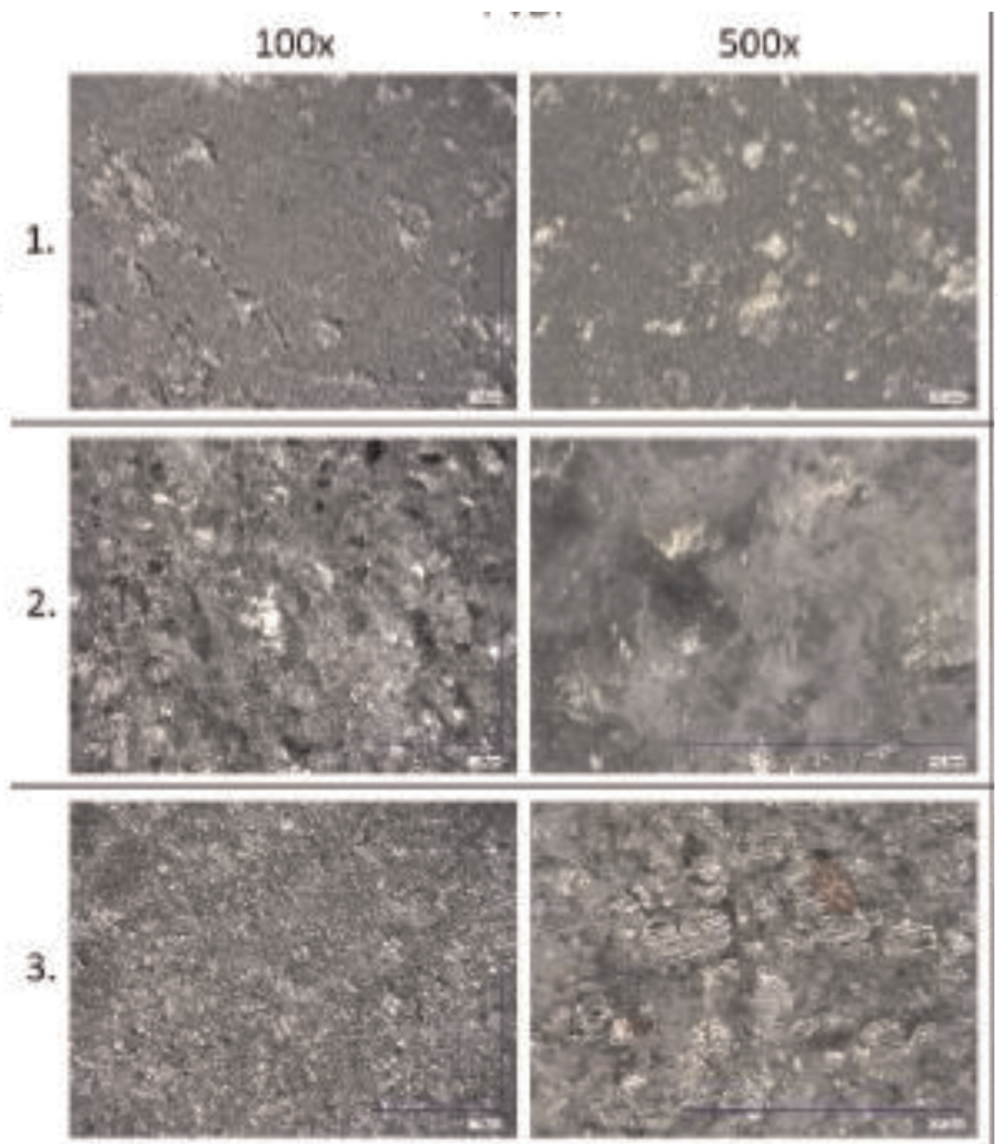

Figure 4.
Polarization microscopy. It shows the polished surface from PP-compound bipolar plate with 82 wt.% titanium content.

3.4 Recycling

At status quo, the amount of waste caused by the production of bipolar plates—an inhomogeneous system consisting of plastic and carbon—is significantly higher when compared to a fully implemented commodity plastic production process [18], since it has shown also for redox-flow batteries [16].

This topic is becoming a mega topic of the twenty-first century. The usage of secondary materials as the feedstock of the graphite compounds is only at its beginning. [16] Some of these processes generate to some degree useful carbon materials [19, 20].

These circumstances and opportunities result in increasing development of recycling methods with the consequence of a property upgrade of the carbon by combining lower general production costs [16]. In the best case, these carbons are suitable for bipolar plates. In **Figure 5**, the principle of the different recycling opportunities are described.

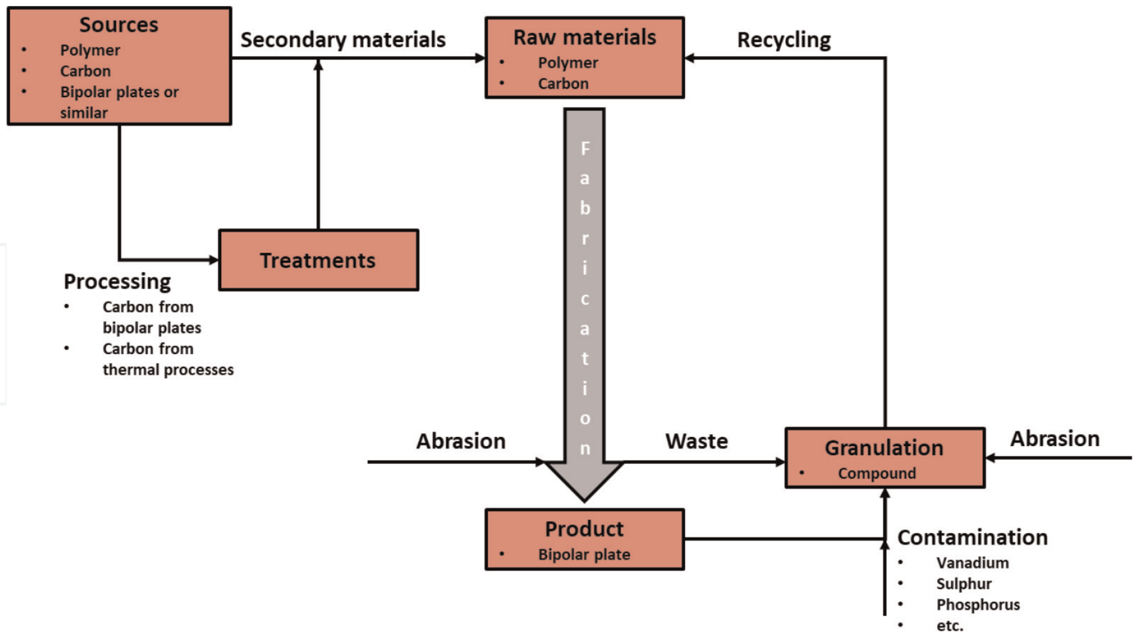

Figure 5.
Scratch of a flow diagram for resources from recycling and secondary sources. The primal structure is from plastic treatment [16, 18, 19].

Figure 5 shows the barriers and technical challenges of the recycling process. The two most important factors are the contaminations of the material and secondly the changes in material properties caused by multiplied processing.

4. Characterization data of PEMEL bipolar plate materials

In order to have an adequate characterization of the bipolar plates. Several test methods are well-established for bipolar plates and some are presented below. The list of test methods is not considered to be complete.

For the trials, we used a nucleated and controlled homopolymer with a fairly High Melt Flow Index of 55 g/10 min. The hompolymer is additionally characterized by high fluidity. Therefore, it normally can be used for high-speed injection of thin wall articles. This polymer-material was mixed with a titanium powder of TOHO Japan, type TC 150 [20] with a particle as follows:

The particle size distributions show (**Table 2**) that the homogeneity of the material is fairly high. Between the smallest group (Dx10) and the biggest particle group (Dx 90) is only a range of 83 μm [20].

In fact, the electrical conductivity in-plane is one of the most important properties of the bipolar plate. It is clear that most electrolyzer and fuel cell (component) laboratories have access to electrical conductivity testing equipment. However, there is no generally standardized test method for electrical conductivity for bipolar plates for electrolyzers and fuel cells so far.

For this reason, it is necessary to realize an in-plane measurement and a through-plane measurement. All these effects are important to make a final decision on the usage of such bipolar plates [16].

During the last 3–4 years, it can be stated that there is a consensus in the market that the 4-point measurement method. When combining it with a CNC-axis system, **Figure 6**) then it is a semiautomatic system.

	Dx 10 (μm)	Dx 50 (μm)	Dx 90 (μm)	Space-density (m^2/KG)
Value	45.3	78.1	129	84.25

Table 2.
Particle size distribution of used material [20].

Figure 6 shows the measurement device of the in-plane conductivity system. With this technology, a single measurement is possible, but it is also possible to measure the hole plate by a size up to 300×750 mm. This type of conductivity mapping is in the meantime an established method for measuring the conductivity in the plane [16].

Figure 7 shows a plate that is completely measured all over the plate by taking different probes with a distance of 20×20 mm. This plate is a PPG86-based plate produced by plate extrusion. The border area of the plate parallel to the flow direction during the extrusion process seems less conductive.

Irregularities in the conductivity are in some processes unavoidable because of the process-depending-orientation of the particles through different processing

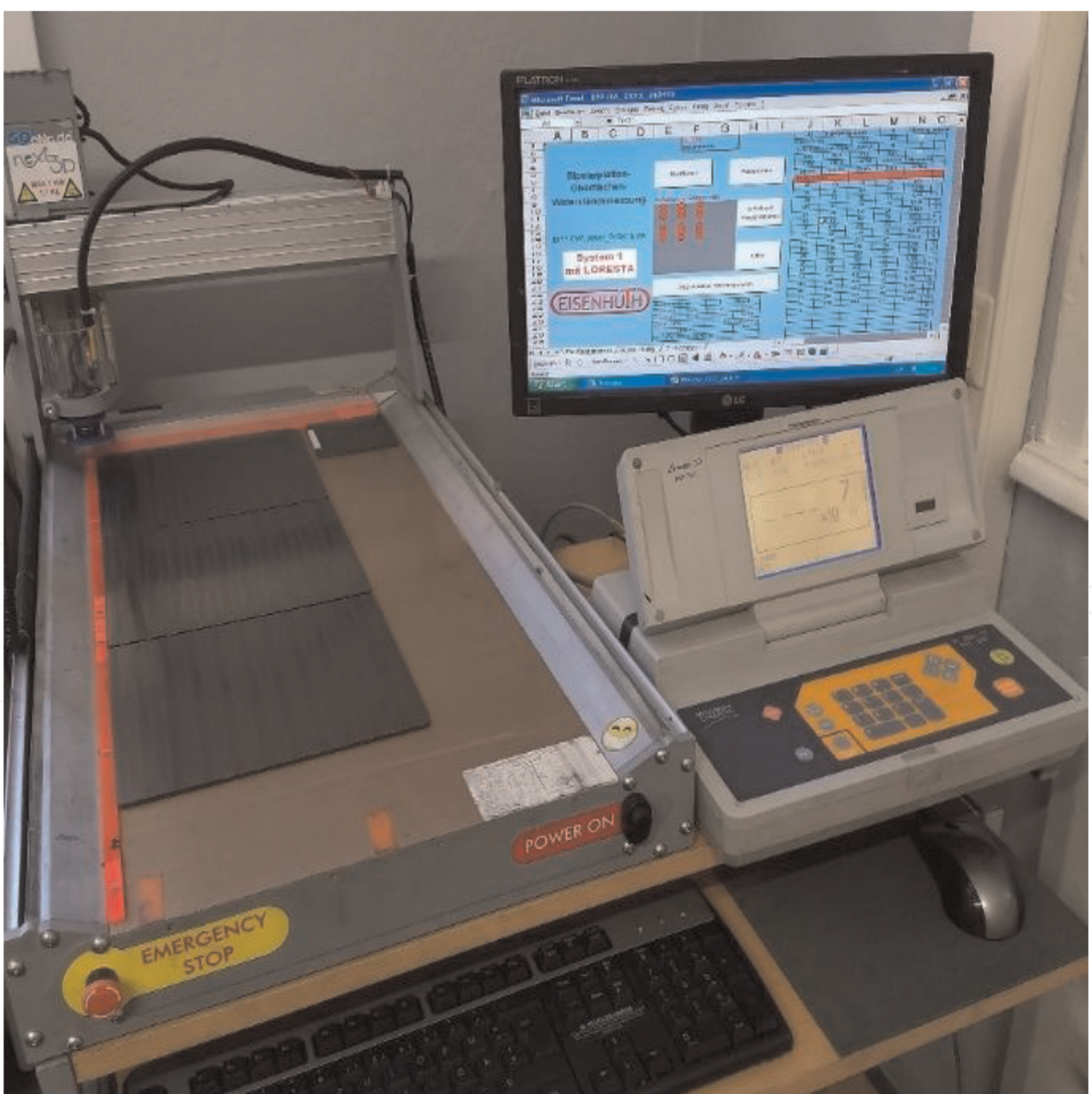

Figure 6.
Testing device and method for electrical conductivity measurement at Eisenhuth GmbH & Co. KG.

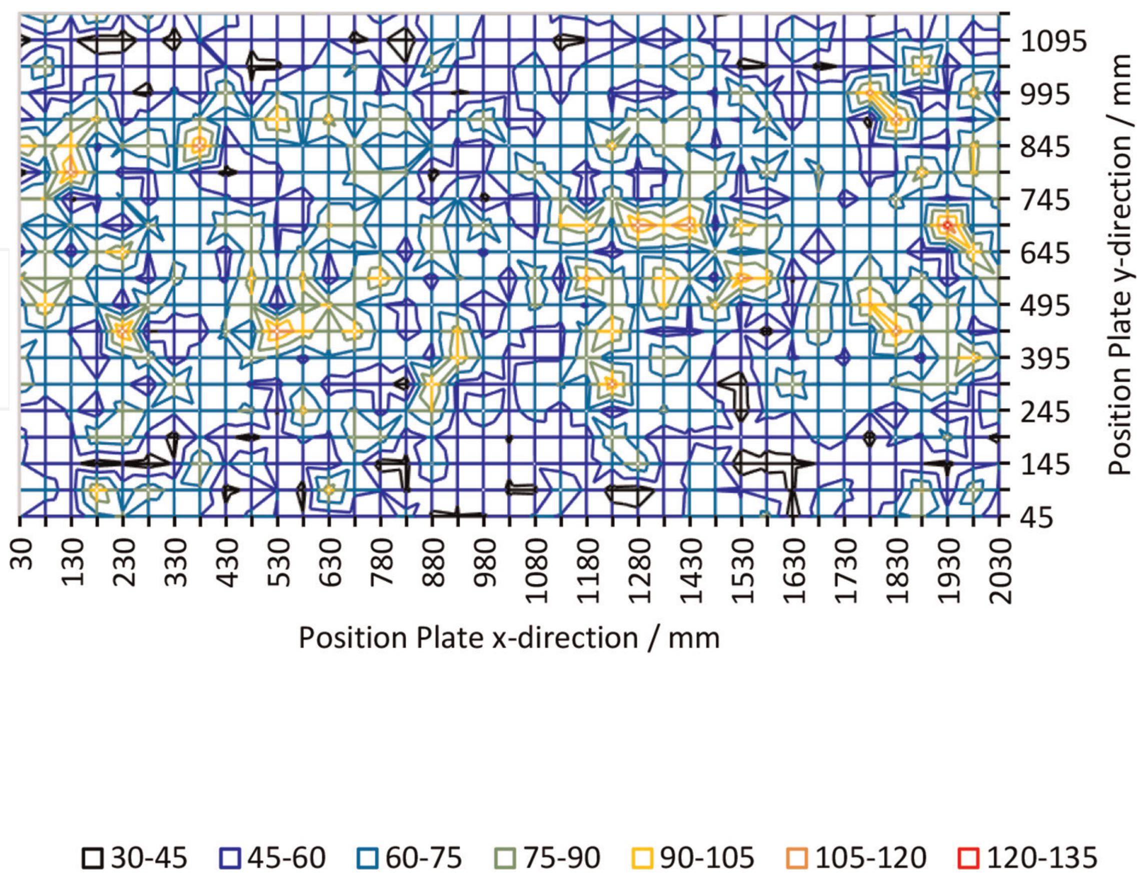

Figure 7.
In-plane conductivity mapping of a PPG86-based bipolar plate made by extrusion by another company that is also active in the field of PEMEL.

influences. For example, in injection molding, the filler particles orientate differently from the core to the surface of the produced parts, which results in different conductivities measured in-plane or through-plane.

In addition, the regions which will be filled lastly in injection molding show a higher average conductivity compared to the gate region [16].

For titanium and graphite-based plates, it is necessary to have a good mixture of the different elements. A very important issue is a homogeneous distribution of conductivity with only minimal deviations between different points on the bipolar plate.

The material shows a higher resistivity in the outside areas, caused by the manufacturing process. The results are corrected with finite size corrections for 4-point probe measurements [17].

After the mixture, an endless production of titanium sheets was realized with a thickness of 0.9 mm. The mixture ration of the material was between 75% titanium powder and 83% titanium powder, which is also identified in **Figure 8**. The material was measured. The results can be seen in **Figure 8**.

The values of the in-plane measurements can be summarized as follows:

- From the production point of view, all sheets were easy to produce.
- All the materials reach the recommended target value of 100 S/cm given by the Department of Energy (DoE) [8] and are shown in **Table 1**.

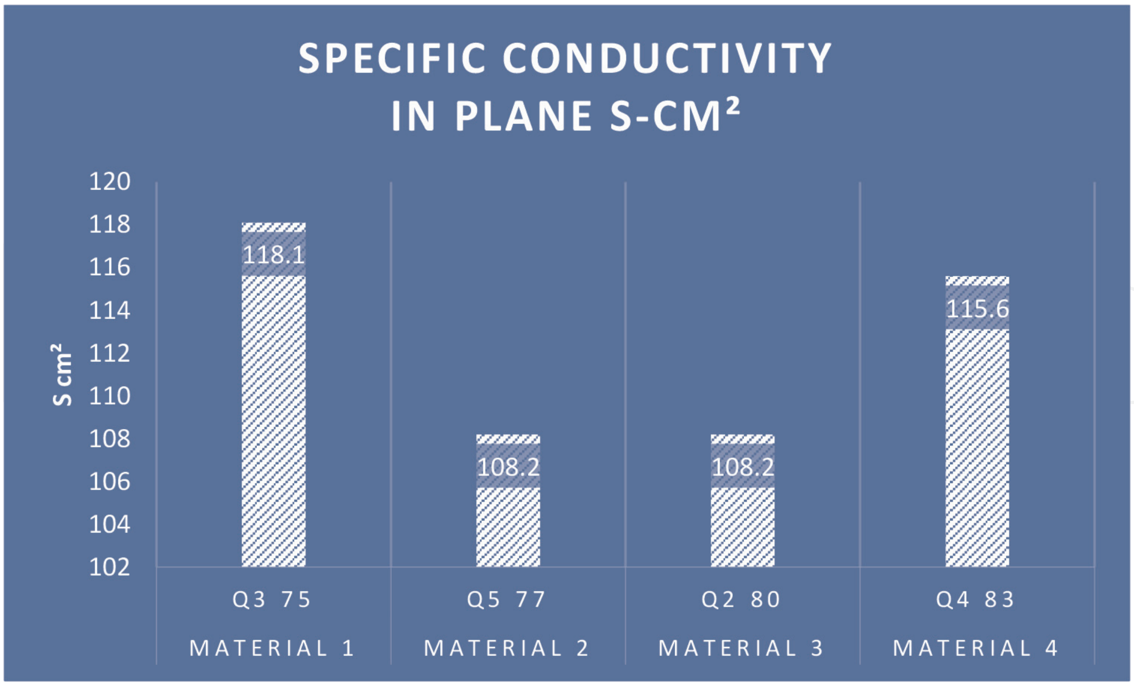

Figure 8.
In-plane conductivity of the measured titanium compound sheets.

- It is surprising that the material with the highest polymer content, Q3 75, has also the best electrical conductivity in the plane.

4.1 Electrical conductivity measurements (through-plane)

After explaining the in-plane conductivity, the second measurement, the trough plane conductivity is an important value to describe the electrical conductivity of a plate.

For all composite materials, it can be confirmed that the trough plane conductivity is lower than the in-plane conductivity. When looking at the two measurement methods, there are some common aspects, but there is no direct correlation between the two measurement types.

In **Figure 9**, examples of through-plane results are presented, with the key messages that (a) through-plane conductivity tends to be systematically lower than in-plane. For reason of reproducibility, the standard tension testing machine is used [16].

As mentioned above in an isotropic material, in-plane and through-plane conductivities are supposed to be equal, but it is not the case. One explanation for this effect is that during the molding process of titanium or graphite platelets are oriented preferentially parallel to the sample's surface, thus transporting the electrons faster within the in-plane direction than through-plane.

The values for the trough plane resistivity can be seen in **Figure 9**.

Figure 10 shows the materials with different polymer ration between Q3 75 (25% PP) and Q4 83 (17% PP). In addition, here the results can be summarized as follows:

- From a production point of view, all sheets were easy to produce.

- There is no correlation between in-plane conductivity and through-plane conductivity.

Figure 9.
Through-plane measurement system.

- The values are not reaching the recommended values given by the DoE [8] and are summarized in **Table 1**.
- It is surprising that the material with the highest polymer content, Q3 75, has also fairly low electrical resistivity through-plane.

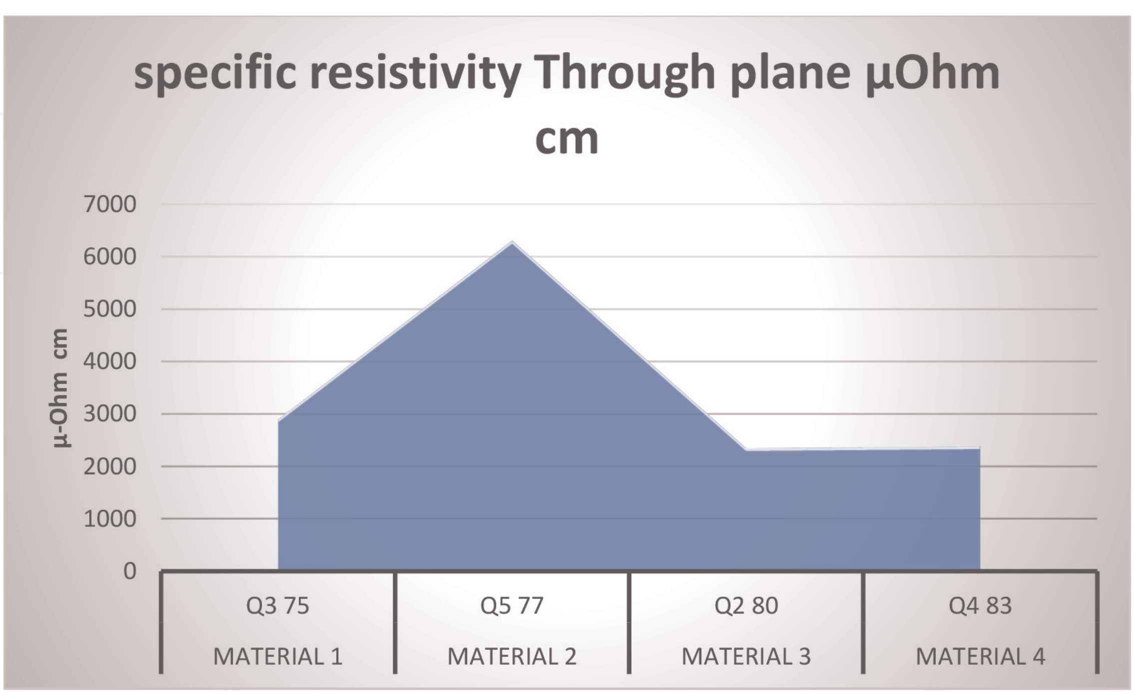

Figure 10.
Through-plane resistivity measurement.

4.2 Corrosion tests

Electrochemistry is primarily concerned with the relationship between chemical and electrical processes, for example, at the phase boundaries between electrolytes and solid surfaces. The conversion of electrical energy to chemical energy takes place primarily in the electrolyzer.

During the electrolyzer process, electrochemical corrosion takes place. This is the natural reaction at phase boundaries between, for example, metals and electrolytes, such as water-containing minerals [19]. Ions are dissolved in the electrolyte, with the help of which electrical charge can be transferred.

In an electrochemical corrosion reaction, the metallic properties of the attacked metal are lost through oxidation [20]. The reaction will occur spontaneously in base metals, such as titanium under atmospheric conditions, because this reaction is thermodynamically favorable.

The basic principle is that the driving force (difference in electrode potentials between the anodic and cathodic areas of the metal surface) of the corrosion process becomes greater the higher the energy gained. Electrochemical corrosion occurs when there is a closed circuit between the aqueous medium and the metal, as well as contact with a possible side reaction.

This is ensured by the current electron current in the metal and the proton current in the medium. In the process, the electron migrates from the anode to the cathode, at whose phase boundaries oxide elements, such as ions or oxygen, are reduced in the medium. The positively charged metal ions flow out of the lattice structure of the bulk material into the medium, so that charge neutrality is maintained. This is the positive particle flow and represents the corrosion process of the metal [21].

The corrosion behavior of compound materials can be understood by using corrosion tables, literature references as well as test results [22]. The corrosion tests of the bipolar plate material cannot be made by general tests but must be suitable for the environmental conditions of the PEM electrolysis in order not to run the risk of observing different corrosion processes.

For the corrosion tests (**Figure 11**), a 0.5 molar sulforic acid solution is used and the tests run at an operating temperature of 60°C. The solution is saturated with oxygen. This results in a pH value of approx. 1 ± 0.02 and thus represents an overall worst-case consideration for the material test. The compilation of corrosion flows can be found in **Table 2**. Assuming that the surface of the sample is subjected to uniform surface corrosion, the material removal can be reflected by the corrosion rate.

In **Table 3**, it can be seen that the corrosion current density values of all compound materials, according to the United States Department of Energy (DOE) standard, are less than 1 $\mu A/cm$-2. [8]

In addition, the corrosion rate of the PP compound is at a low level. The corrosion rate can be determined from the corrosion currents of the materials, whereby the material removal is extrapolated over 1 year. Using Faraday's law, an estimate of corrosion in terms of material removal can be made. In the case of PP, the corrosion rate is lying at 0.094+/−0.03 μm/year. The reason for this is that titanium and graphite are–generally spoken–highly resistant to corrosion [20–23].

4.3 *In situ* tests

Eisenhuth has realized a small flexible setup-system (**Figure 12**), in order to realize different tests on the electrolyzer system.

Figure 11.
Representation of the real three-electrode arrangement of the structure of corrosion cell of the company Meisenberg.

Compound material	Corrosion potential in mV	Corrosion current in μA/cm^2
Titanium (0–100) + Polypropylene (PP)	391,496+/−7.5	0,014+/−3.6
Titanium (0–100) + Phenolic Resin	230,115+/−5.4	0,042+/−2.4
Titanium (TC 150) + Phenolic Resin	161,341+/−0.9	0,05+/−4.5
Titan (0–100) + Polyester resin	−65,342+/−20.3	0,105+/−17.2
Titan (0–100) + Bakelite Resin	−242,374+/−23.2	0,185+/−14.1

Table 3.
Corrosion of different compound materials in 0.5 mol per liter H2SO4 at 60°C.

With this complete system (**Figure 12**) and test bench (**Figures 13** and **14**), it is possible to test different materials in a continuous atmosphere. This test bench has the following features:

- Emerald control software control for electrolysis R&D.
- Anode and cathode pressure control.
- DI water recirculation.

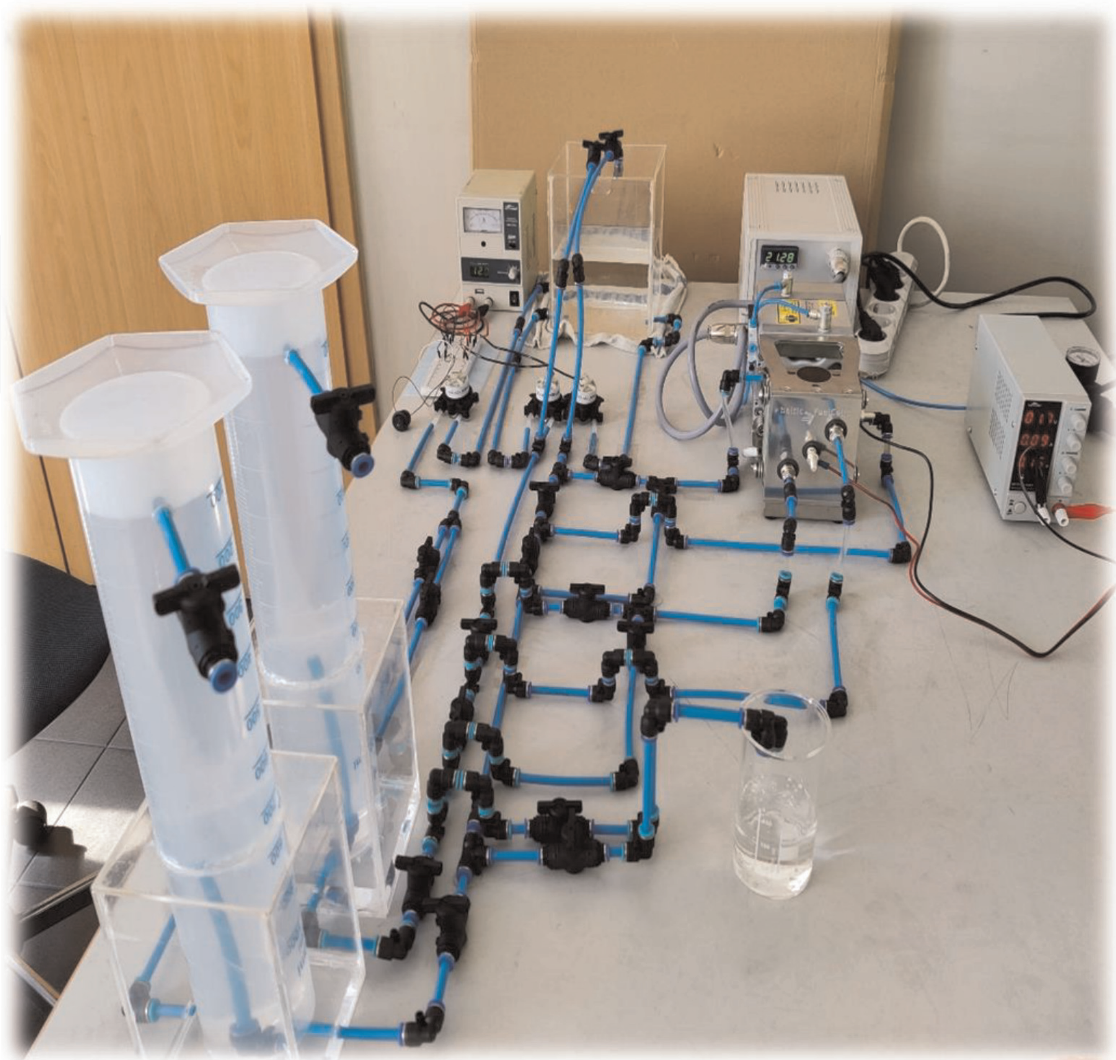

Figure 12.
Electrolyzer test bench.

- Cell voltage monitoring.
- Data acquisition system (cell temperature monitoring).
- Power supplies up to 100A.
- Constant current/voltage.
- Applications for PEM electrolysis technology.

The corresponding plate is initially installed in the test chamber (= single-cell stack) (**Figure 13**).

The test chamber is installed in the test system of the Baltic fuel plant (**Figures 13** and **14**). Here, it is possible to compare the different materials and get results in a real PEMEL atmosphere and give a link to the standard polymers as well as the functioning of the technology [24].

With the *in situ* test bench, all the *ex situ* results could be reaffirmed. That shows that the *ex situ* tests are a good basis for a pre-evaluation of the total system.

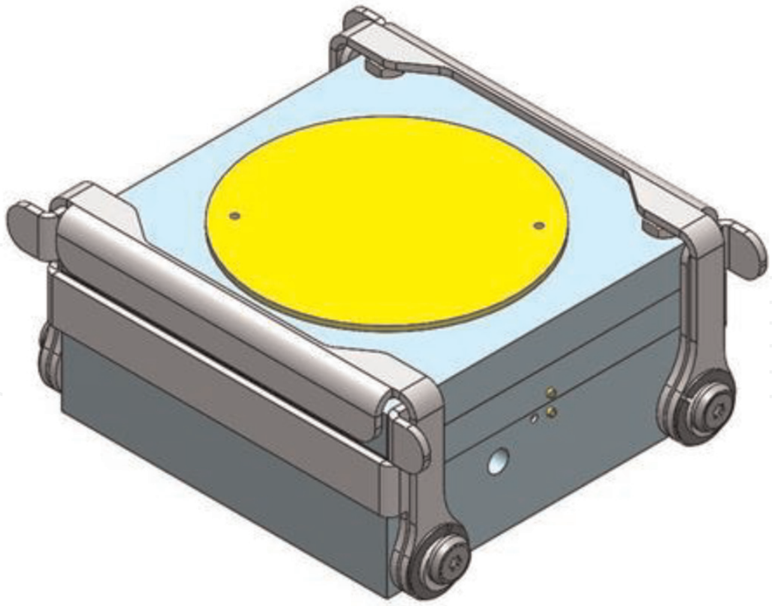

Figure 13.
Test chamber for a single-cell electrolyzer test system.

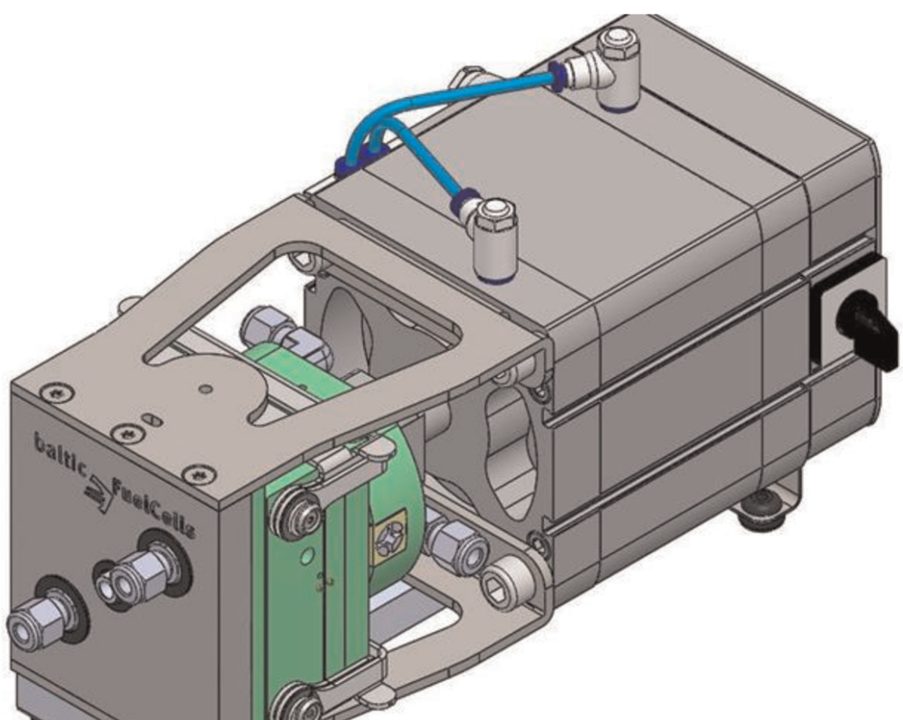

Figure 14.
*Baltic fuel cell test stand, where the single cell system (**Figure 13**) is integrated.*

5. Conclusions

In the chapter, the composite bipolar plates based on polypropylene were presented. The fillers, such as graphite or titanium, are the basis for a bipolar plate in a PEMEL system. The results were presented in connection with the current research topics at Eisenhuth. The main aim, however, is to reduce the production cost for bipolar plates and, as a consequence for the complete electrolyzer system. This is an important lever since the bipolar plate is one of the most used components in the electrolyzer stack.

Reduced cost of the system combined with good performance can be achieved by modifying the bipolar plates, such as

- Using graphite compound plates instead of pure titanium.
- Using titanium-compound plates instead of pure titanium.

By using graphite, it might be difficult to operate a PEM electrolyzer at high voltages, since at a voltage of higher than 2 V corrosion of the graphite material might occur. It is only possible to use graphite on the hydrogen side. For the oxygen side, titanium composite is a good choice. For better flexibility in the total system, usually, titanium or here titanium compound is used in the electrolyzer. As far as corrosion values and in-plane conductivity are concerned, the titanium-compound shows good results.

The proposed targets for material properties are not always fully achieved, but that progress in materials research is possible. It could be shown that the substitution of carbon composite or titanium composite materials is an option to bring the material cost down.

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Conflict of interest

The authors are part of the company Eisenhuth GmbH & Co. KG, which produces bipolar plates made of graphitic compounds and gaskets for fuel cell, redox-flow battery, and heat exchanger purposes.

The shown data are part of the acknowledged public funded projects. The conclusions and statements made are based on the experiences of the authors in their specific working fields in the said company.

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