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Development of Fiber Reinforced Compound Bipolar Foils for Fuel Cells

Ali Osman Erdem, Paul Stannek, Marco Grundler, Alexander Nuhn, Stefan Schmidt, Sebastian Schmeer

***Summary* — In the research project »InduRex«, the challenge was to produce graphite polymer bipolar plates with the thinnest possible thickness. The physical and chemical requirements were defined according to the values set by the »Department of Energy«. Within the scope of the project, the continuous production of highly filled foils, which were then successfully structured as bipolar foils and operated in a fuel cell, was successfully implemented. Initial cell tests demonstrated a good cell efficiency at low and medium current densities. For this reason, the approach of creating bipolar plates from compound foils is still being pursued. Now the challenge is to produce bipolar films with a larger active area and to increase the mechanical stability to such an extent that the construction of a multicell stack can be realized. As a result, a metallic bipolar plate design was successfully transferred to a compound foil as part of the »InduRex« project. The problem is that with a larger bipolar foil there is a risk of obtaining low mechanical stability. Therefore, carbon fibers are introduced into the films as part of the research project »Faserverstaerkte Folien« in order to improve the mechanical properties. In addition to continuous production, the thin-walled foils are reinforced with carbon fibers using a hot press. The fiber-reinforced foils will be characterized in the next steps to investigate the influence of the carbon fibers. The aim is to obtain even thinner bipolar plates with consistent properties from the extruded foils in order to reduce the overall weight and volume of the fuel cell.**

***Keywords* — bipolar foils, fuel cell, compound, electrically conductive, highly filled, extrusion**

I. Introduction

Within a fuel cell (FC) stack, bipolar plates (BPP) are the largest volume unit and thus decisively define the resulting size of the stack, the weight and at least 30% of the cost. [1,2]. A low wall thickness of the BPP is therefore desirable, especially for mobile applications, so that metallic BPP are used almost exclusively for automotive FCs. However, metallic BPPs have the disadvantage that their service life is shorter due to corrosion and they have to be coated in advance at high cost. Graphitic or compound-based BPPs, on the other hand, do not need to be coated and currently achieve a longer service life than metallic BPPs [3]. Strategic Analysis Inc. has done a costing exercise for fuel cell systems using metallic and graphitic BPP in [4]. This shows, among other things, that the costs with graphitic BPP result at ~ $3/kW and metallic BPP at ~ $6/kW for light duty vehicles at a produced unit rate of 500,000 systems/year. Thus, FC systems with graphitic BPP meet the Department of Energy (DoE) target for specific cost [5]. However, graphitic BPP are usually significantly thicker (~ 2.3 mm) than metallic BPP (~ 0.1 mm or ~ 0.4 mm formed) due to the „classic" manufacturing processes such as hot pressing or injection molding. Using the production process of foil extrusion presented here, highly filled compound-based unstructured bipolar half-panels (BPHP) can be produced continuously in thicknesses as thin as 0.37 mm. The reduction of the thickness compared to injection or compression molded BPP leads to a significant reduction of the weight and volume of the final FC stack. Also, the reduction of the raw materials needed to produce a BPP saves costs and reduces the carbon footprint. With the addition of carbon fibers, even very thin foils can obtain the required mechanical stiffness.

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II. Methodology

Thin-walled bipolar foils are produced at ZBT on a Brabender KE 30 single-screw extruder with a coupled sheet die and downstream calendaring unit from Saueressig (Fig. 1).

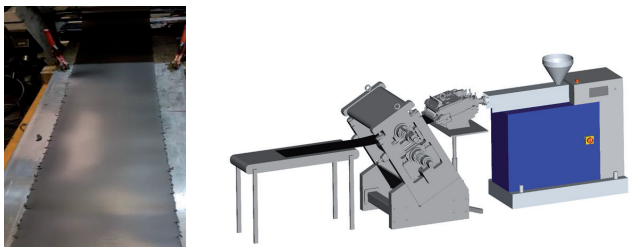

Fig. 1. Calendered bipolar foil and production line

For foil production with the production line shown in Fig. 1, compound material is first produced on a ring extruder (Extricom RE3). In this process, the matrix polymer (polypropylene) is fed into the extruder upstream via a dosing differential scale, melted and loaded downstream with various fillers, mainly graphite and conductive carbon black. The choice of fillers and the ratio of matrix polymer to fillers is decisive for the physical properties of the compound materials and the subsequent bipolar foil. Due to the

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increase in viscosity and the change in mechanical properties with an increase in filler content, the compound formulation has to be adapted to the foil production process.

The previously produced compound is transferred to the feed hopper of the single-screw extruder, melted and conveyed to the sheet die. There, the melt emerges with a width of 250 mm and, due to the variable cleft lip of the die, the thickness can be adjusted between 0.4 mm and 1.5 mm. The downstream calender specifies the final thickness of the bipolar foils via a variable roll gap. The unstructured bipolar foils produced in this way are subjected to numerous characterizations ex-situ (electrical resistance, hydrogen permeation and mechanical parameters). For mechanical characterization tensile tests were performed. Dogbone specimens of 0.4mm thickness were laser cut (speciment type 1B of DIN EN ISO 527-2) and tested in a Zwick 1485 testing machine. Strain measurement was done optically via digital image correlation using the Software GomCorrelate.

Some foils are reinforced with carbon fibers by hot pressing in previous steps to increase mechanical stiffness. These are also characterized ex-situ and compared with the foils without fiber reinforcement to determine any influence of the carbon fibers. The following results relate to two different carbon fiber-reinforced semi-finished products used to reinforce foils:

- 15K carbon fiber roving TR 50S from Mitsubishi, with binder resin (EPIKOTE Resin TRAC 06720) with 8 w% spread to 20 mm width.
- CFR-UD tape Tafnex CF-PP 66 w%, thickness: 0.16 mm

The mold used is a polished 50 mm sample mold consisting of a stamp and guide sleeve, see Fig. 2. The system technology for temperature control and pressure application is a variothermal laboratory press, see Fig. 3. The release agent Frekote 55-NC from Loctite and an additional fabric-reinforced PTFE release foil are used to separate the tools.

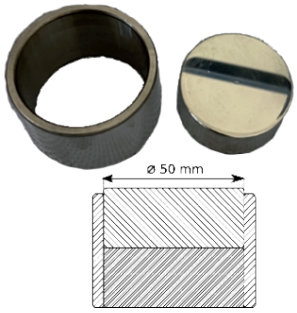

Fig. 2. Polished press body for 50 mm samples

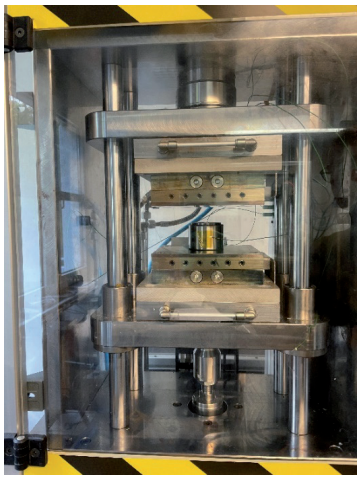

Fig. 3. Laboratory press with pressing tool

The pressing process is divided into three phases: firstly the heating phase (Time: 0 to 5 minutes), secondly the pressing phase with constant temperature and constant pressure (Time: 5 to 15 minutes) and thirdly the cooling phase (Time: 15 to 25 minutes), see Fig. 4.

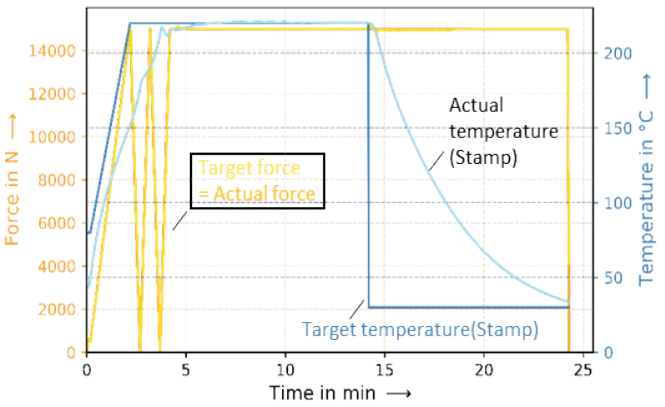

Fig. 4. Variothermal process control

During the first phase, the mold is heated via the heated pressing plates and the pressing force is applied. The mold cavity reacts to the temperature change with a time delay due to its own thermal resistance and adapts to the controlled temperature of the press plates. The tool starts at 40 °C and is heated to 220 °C within 5 minutes. The pressing force is relieved twice during heating to prevent the press plunger from jamming in the tool.

The closed cavity creates a pressure build-up of around 7.6 N/mm² with a pressing force of 15 kN on the samples. This pressure is maintained in the second phase at a constant 220 °C for about 10 minutes.

In the cooling phase, the sample is cooled to 40 °C under constant working pressure, then relieved and demolded. It should be noted that due to the precise control of the press, the measured pressing force overlaps with the specified target force, so that only one line (yellow/orange) is visible in the diagram.

Micrographs:

The molded specimens for the micrographs are embedded in EpoFix Resin plus EpoFix Hardener from Struers. They are then ground on an ATM Saphir 360 grinding disc with water cooling and grit sizes from 180 to 4000. This is followed by polishing stages with 3 and 1 ym on an ATM Saphir 320. The micrographs are taken with a Leica DM6000M reflected light microscope.

In order to also characterize the bipolar foils in-situ in a Baltic Fuel Cell test hardware, flow fields were transferred into the bipolar foils using the embossing die shown in Fig. 5 and a laboratory press from Höfer. For this purpose, the bipolar foil is heated to the melt temperature of the polymer used and then an embossing force of 50 kN is applied to the flow field area of 25 cm².

After the cell made of compound bipolar foils had run successfully, developments for a cell with a larger active area followed. Within the "InduRex" project, it was possible to transfer the design of a metallic bipolar plate to a compound bipolar foil. The decisive factor was to adapt the design in such a way that the cell could be sealed. During the hot press process, it was first necessary to adjust the parameters so that no cracks occurred and the structures were mapped evenly. Analogous to the 25cm² single cell design, the structures were incorporated into the compound foil by means of the hot press process. The following Fig. 5 shows the compound bipolar foil with an active area of 100 cm².

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Fig. 5. Compound foil BPP with an active area of 100 cm²

Initially, the construction and operation of a single cell is planned. With conventional metallic bipolar plates, cooling takes place between the bipolar half-plates. The cooling concept of metallic bipolar plates is not directly transferable to the compound bipolar foils. The compound bipolar foils are structured on one side and embossed flat on the other. This eliminates the cooling function between the bipolar plates. For the single-cell design, cooling was therefore planned through the insulation plate. The cell is sealed with the help of gaskets that are inserted into the grooves in the insulation plate and the dispenser gaskets applied to the bipolar foils. The final assembly is done by threaded rods, which are fastened with nuts. The following Fig. 6 shows the individual components for the assembly of the cell.

Fig. 6. Components for the assembly of the single-cell unit with an active area of 100 cm²

There are also challenges with the single-cell compound bipolar foils. An alternative solution for cooling will have to be found for a multi-cell setup. The presented setup only works as a single cell. It would be conceivable to weld or glue the bipolar foils together. Another alternative would be to emboss the compound foil on both sides to distribute the media on one side and to enable cooling on the other. In the following sections, the embossing of compound foils on both sides will be discussed again.

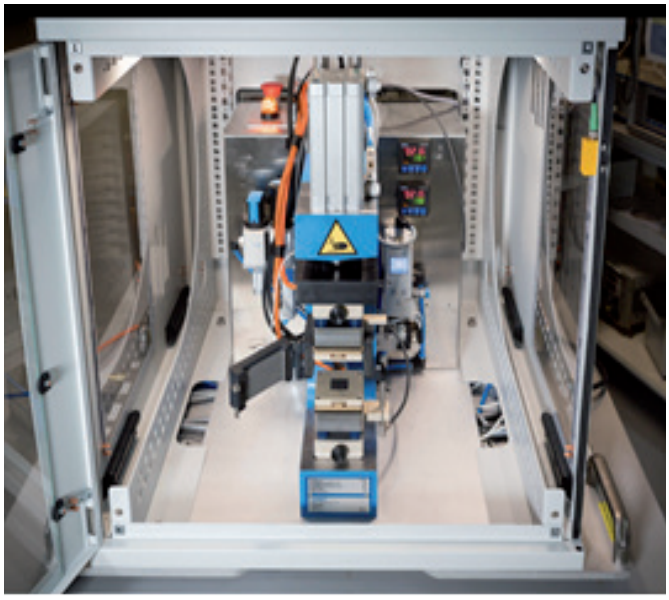
Another problem is the mechanical stability of the compound bipolar foils. The bipolar foils must be tensioned with a defined contact pressure to enable the best possible contact with the GDL. The mechanical strength of the bipolar foils should be sufficient to prevent them from breaking or cracking. This problem increases in the case of an assembly with several cells, because there is a potential risk of misalignment and homogeneous compression of the fuel cell stack is made more difficult. As already mentioned, the „Faserverstarrkte-Folien“ project is attempting to strengthen the mechanical strength of compound films with the help of carbon fibers. Therefore, a 25cm² single cell testwith fiber-reinforced foils is being carried out as part of the „Faserverstaerkte Folien“ project to test the effect of the fibers.

III. Results

Since the beginning of the research project, numerous bipolar foils have been produced from various material formulations and characterized with regard to their physical and chemical properties. Excerpts of the measurement results are presented below.

A. Electrical Resistance of unreinforced foils

The bipolar foils were measured with regard to the area-specific contact resistance relevant for FC applications. A specially constructed test stand was used to measure the resistances. As shown in Fig. 7 the sample is clamped between two gold-plated copper poles (A = 4 cm²) whose contact pressure can be controlled linearly by compressed air. A gas diffusion layer (GDL), whose contact force or pressure-dependent resistance curve is known, is previously applied to each of the measurement poles, whereby the measurement section is oriented very closely to the real resistance chain in a fuel cell [6].



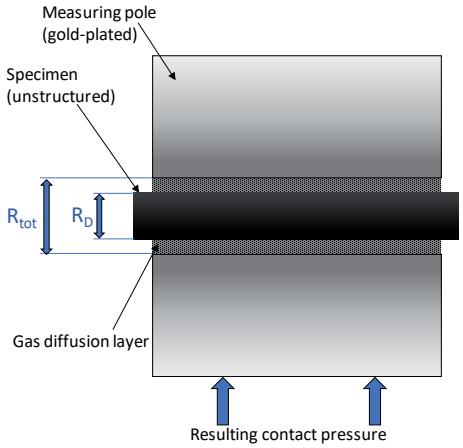


Fig. 7. BePPel resistance test rig (top) and measuring principle (bottom)

For the project “Faserverstaerkte Folien”, in order to reinforce compound foils with fibers, four different compound foils were produced as matrix material with different filler contents. Foils with 23% polymer content, 28% polymer content, 27% polymer content and 30% polymer content were produced. The selected raw materials of Mat.1 and Mat.2 differ from the rest of the foils, whereas Mat.3 and Mat.4 were produced from the same raw materials. These were first measured for area resistivity to determine their suitability as bipolar plate material, as shown in Fig. 8 below. The compound foils were measured in the untreated, ground and then annealed state. It can be clearly seen that by optimizing the material composition and filler content, the area-specific forward resistivity was reduced from, for example, 45 mΩcm² with a polymer content of 23% down to 12 mΩcm². However, the aspect of further

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post-processing or fiber incorporation has to be considered. Therefore, the experiments of fiber insertion are tested with foils of Mat.4. The reason for this is that due to the lower filler content or higher polymer content, the flowability is higher and therefore the fiber incorporation is facilitated.

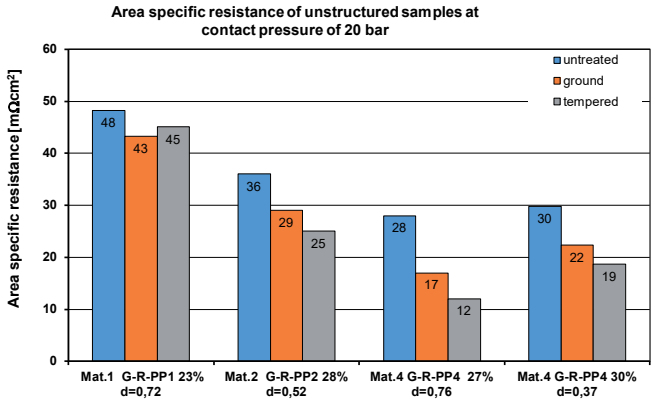

Fig. 8. Area-specific contact resistances of unstructured bipolar foils

B. Mechanical characterization

Stress strain curves of the unreinforced compound foils (Fig. 9) show the strong embrittlement of the polypropylene by incorporation of graphite and carbon black. The values for fracture strain, tensile strength and elastic modulus (measured between 0 and 0.1% strain due to strong non-linearity after 0.1% strain) are 0.48 ± 0.05%, 38.6 ± 1.7 MPa and 11.1 ± 0.4 GPa respectively. Therefore, the fillers reduce the polypropylen's fractures strain drastically from about 700% to about 0.5%. Conversely, the elastic modulus is increased roughly tenfold. Ultimate tensile strength is increased by about 14%.

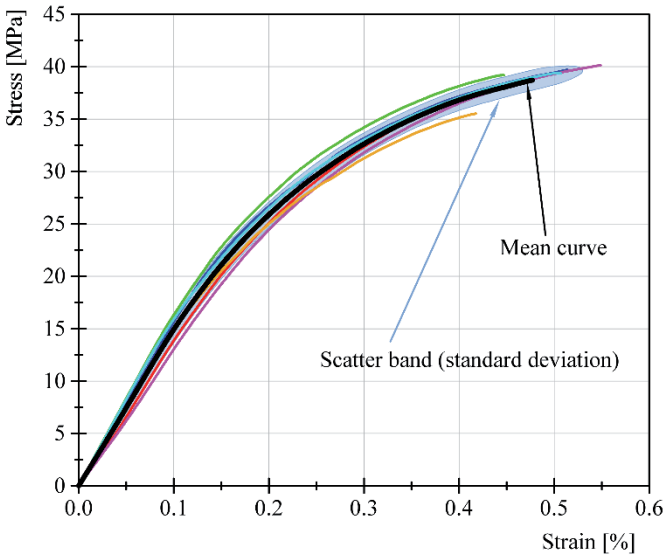

Fig. 9. Stress strain curves of unreinforced compound foils + mean curve and scatter band (standard deviation).

C. Fiber application in the pressing process and micrograph analysis

As part of the project, C-fibers were incorporated in various orientations on and in the compound foils to improve mechanical stability. These samples can be taken from the two following figures.

Fig. 10 shows samples 7 and 13 before impregnation. The UD tapes on the bipolar foil (left) and the spread C-fibers with impregnating powder between the fibers are clearly visible. The powder is milled compound from G-R-PP75%

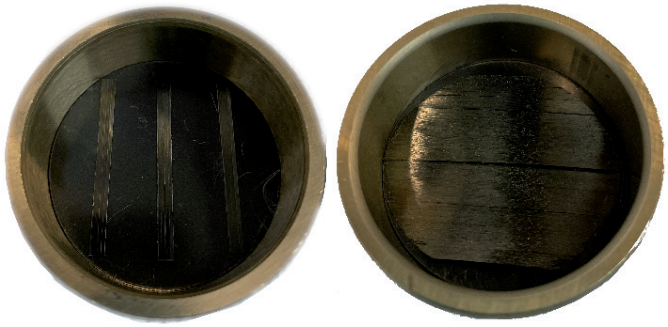

Fig. 10. Sample 7 and 13 before pressing

The compressed and demolded specimens are shown in Fig. 11

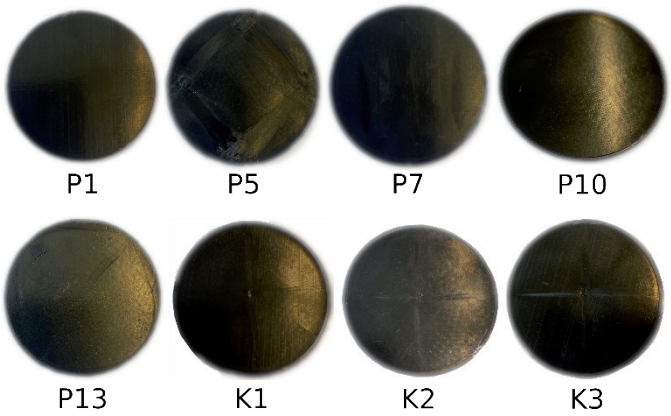

Fig. 11. Fiber-reinforced samples P1, P5, P7, P10, P13 and K1, K2, K3. Diameter of the samples: 50 mm

The following table 1 contains a detailed description of the various samples and their configuration.

Table 1

Produced Fiber Reinforced Compound Foils

Sample	Layer	Fiber type	Fiber size	Press parameters
1	foil + foil	none	none	220°C & 15 kN
5	hashtag + foil + hashtag	CF-PP strip	2.5 mm wide	220°C & 15 kN
7	foil + line + foil + line	CF-PP strip	2.5 mm wide	220°C & 15 kN
10	foil + splayed + foil	splayed C-fibers	whole area	220°C & 15 kN
13	milled + splayed + foil + milled	splayed C-fibers	whole area	220°C & 15 kN
K1	foil + hashtag + foil	CF-PP strip	1.1 – 1.2 mm	220°C & 15 kN
K2	foil + hashtag + foil	CF-PP strip	1.5 – 1.6 mm	220°C & 15 kN
K3	foil + hashtag + foil	CF-PP strip	2.1 – 2.3 mm	220°C & 15 kN

foil | hashtag fiber pattern | line fiber pattern | splayed fiber | milled compound

It can be noted that only the conventional CF-PP tapes achieve good adhesion to the bipolar plate due to their surplus matrix. At the same time, an electrically insulating separating layer is formed, see Fig. 12.

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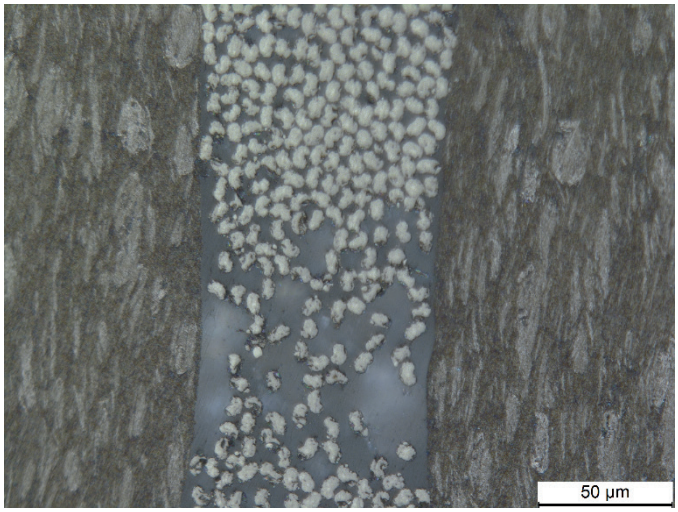

Fig. 12. Micrograph of sample 7 showing the bipolar film at the edges and the interface of the CF-PP tape in the center.

The reduction of this separating layer through the use of spread fiber layers plus milled compound, as in sample 13, shows the lowest fiber-reinforced layer thickness of all samples. Minimum layer thicknesses in the order of up to 2-3 carbon fiber diameters were achieved here, see Fig. 13. Due to the lower proportion of binder resin compared to the conventional UD tape, the C-fibers are much closer together. This leads to poor strength between the fibers.

It can also be seen that the PP matrix flows out of the bipolar plate and partially fills the free spaces between the fibers. However, this effect is not sufficient to achieve a good bond to the bipolar film and between the fibers. However, a relatively high electrical conductivity is achieved, taking into account 100% or full-surface fiber coverage.

Fig. 13. Micrograph of sample 13. The separating layer is approx. 2-3 carbon fiber diameters thick. The bipolar film is located on the sides.

D. Electrical Resistance reinforced foils

In order to investigate the influence of the fibers on the electrical conductivities, the area specific volume resistances of the samples from Fig. 11 were first measured.

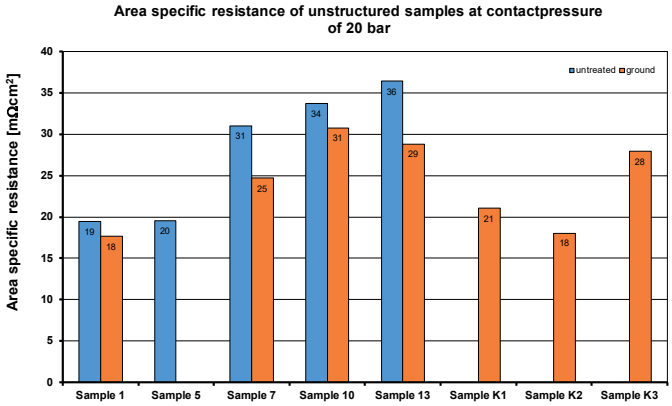

Fig. 14. Area-specific contact resistances of unstructured fiber reinforced foils

In the measurement shown in Fig. 14, two foils were pressed into one sample as the reference sample or Sample 1. The remaining samples are also two foils, but with fibers inserted between the foils.

Analogous to the foils without fiber reinforcement, the samples were each measured untreated and in the ground condition. Due to an uneven surface, the last three specimens could only be measured in the ground condition. Sample 5 could only be measured in the untreated state because fibers detached from the surface after grinding.

It can be seen in the Fig. 14 that the orientation and quantity of fibers significantly affects the area-specific forward resistivity. With the correct arrangement of the fibers and the optimal fiber size, it is possible to keep the resistances very low and in the same range as the reference sample, see sample K2 compared to the sample 1 in Fig. 14.

E. Hydrogen Permeation

The permeation measurements at ZBT are carried out in a cell-realistic manner by applying 100 % hydrogen to one side of a sample material. On the opposite side, the hydrogen concentration increase is measured after a defined time. The bipolar foil is positioned as shown in Fig. 15 and flooded with a hydrogen pre-pressure of 1 bar.

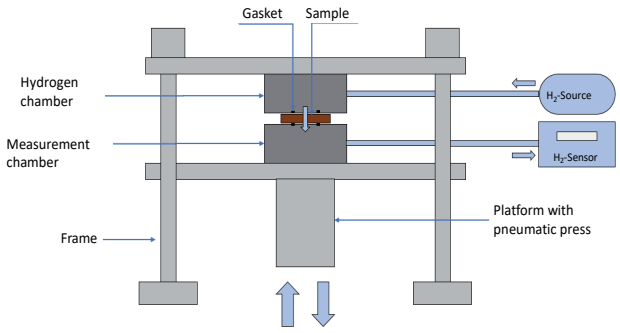

Fig. 15. H2 permeation test rig

To determine the suitability of the compound foils as bipolar plate material, a certain hydrogen impermeability must be achieved. Analogous to the DoE specifications, ZBT determines limit

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values for permeation coefficients in order to evaluate the impermeability. These limit values can be taken from the following table 2. Therefore, the target is to obtain a permeation coefficient less than 10 E-8 cm²/s.

TABLE II

LIMITING VALUES OF PERMEATION COEFFICIENTS FOR SUITABILITY AS BIPOLAR PLATE MATERIAL

Rating	excellent	very good	good	partially acceptable	only sealing material
Perm. coeff. [10E-8 cm²/s]	<0,1	<1	<10	<100	< 1000

The compound foils, which were manufactured for the fiber reinforcement, were tested for hydrogen tightness using the test rig shown in Fig. 15 and the results can be seen in Fig. 16.

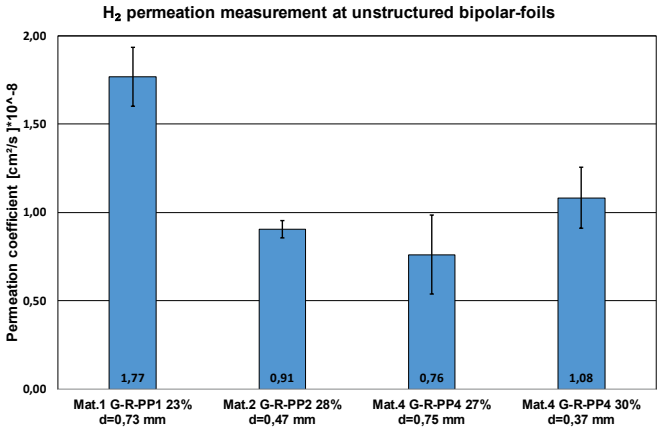

Fig. 16. Permeation coefficients of unstructured bipolar foils

As shown in the figure, all material compositions have a permeation coefficient lower than 10 E-8 cm²/s and even less than or equal to 1 E-8 cm²/s. Therefore, all compound foils are suitable as bipolar plate material in terms of hydrogen impermeability.

First fiber-reinforced samples were also tested for hydrogen tightness and are shown in the following Fig. 17.

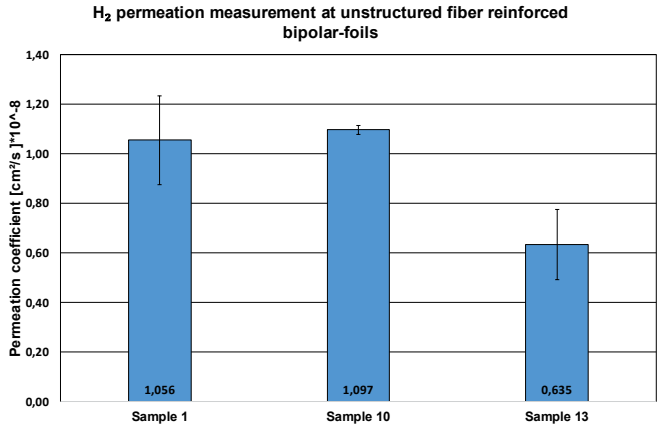

Fig. 17. Permeation coefficients of unstructured fiber reinforced bipolar foils

Analogous to the previous measurement, all samples are in the limit ranges less than or equal to 1 E-8 cm²/s and therefore suitable as fuel cell material.

F. EMBOSSING AND FIBER REINFORCEMEND

Within the project "Faserverstaerkte-Folien", the double-sided embossing of compound foils was first investigated. The foils were inserted between two structured stamps, each consisting of a positive and negative half, and hot pressed with suitable parameters. This made it possible to obtain a structured film with a homogeneous residual wall thickness. Within the project „Faserverstaerkte-Folien", the Leibniz Institute for Composite Materials GmbH was able to demonstrate that it is possible to emboss compound films on both sides and this is shown in the following Fig. 18.

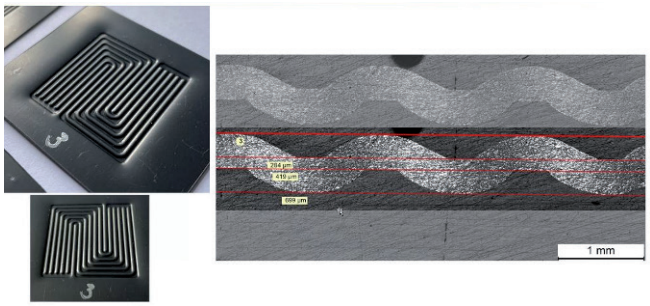

Fig. 18. Both side structured compound foil

To increase the mechanical strength of the compound foil, carbon fibers will be added to the structured foil due the hot press process. In the first steps the challenge is to obtain the right parameters to add the carbon fibers in the compound material without damaging the foil. Afterwards the challenge is to choose the right fibers with the optimal distribution without the deterioration of the function as a bipolar plate. In the following Fig. 19 a structured compound bipolar foil with added carbon fibers is shown.

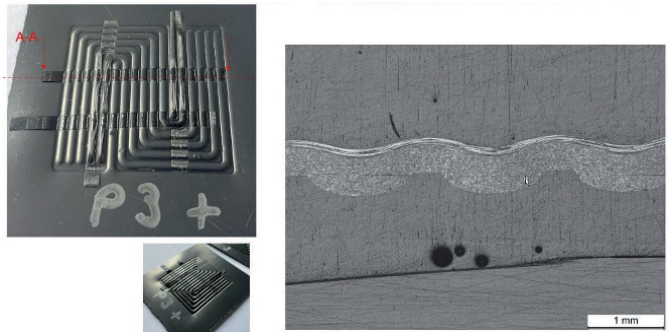

Fig. 19. Both side structured compound foil with carbon fibers

Using carbon fiber reinforcement, it is possible to assemble a fuel cell stack with several cells. However, the main aspect of fiber reinforcement is that it allows thinner foils to be used to reduce the overall weight and volume of the fuel cell stack.

IV. DISCUSSION

With the production process developed at ZBT, it is possible to continuously produce highly filled compound foils with thicknesses between 0.4 mm and 1.5 mm that are suitable for FC applications. The measurement methods and test rigs used had to be adapted to the significantly thinner bipolar foils. The test procedures were successfully modified so that the bipolar foils can be reproducibly characterized both ex-situ and in-situ. The investigations carried out show that the area-specific forward resistances of the most electrically conductive bipolar foils to date are lower than 20 mΩ in the unstructured state. The H2 permeation coefficients are higher compared to metallic and the much thicker injection-molded or hot-pressed compound-based BPHP, but can be evaluated as

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very gas-tight to hydrogen after adjusting the embossing parameters. The drastically low fracture strain of <0.5% demonstrates the need for fiber reinforcement to enable mechanical stability. However, for a comprehensive characterization mechanical tests also need to be performed at the operating temperature of a fuel cell of 80 °C. Strain is expected to increase for higher temperatures but strength and stiffness will drop significantly. Furthermore, the mechanical influence of the fiber reinforcement needs to be appropriately evaluated. Both evaluations will be performed in the course of this project. The fiber reinforcement applied by the IVW appears to implement the desired properties such as electrical resistance or hydrogen permeability and high strength in a favorable trade-off. In addition, locally applied conventional CF-PP tape was shown to be a good way of increasing strength with moderately increasing electrical resistances.

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