



## Review Article

## Evolution of a GDE setup: Beyond ambient conditions

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## Abstract

Gas diffusion electrode (GDE) setups recently and increasingly received attention as testing platforms for fuel cell catalysts. GDE setups provide realistic reaction conditions while remaining simple and efficient to use. Therefore, GDE setups bridge the gap between rotating disk electrode (RDE) measurements and membrane electrode assembly (MEA) testing. Here, we describe our initial GDE design development to its latest improvements that allow application of high temperatures. We point out experimental challenges we overcame, yet also discuss properly applying our new technique to avoid wrongful use. In particular, we advocate for implementing GDE setups in catalyst stability investigations where the technique so far has been used infrequently.

## Addresses

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## Keywords

GDE setup, Elevated temperature, Elevated pressure.

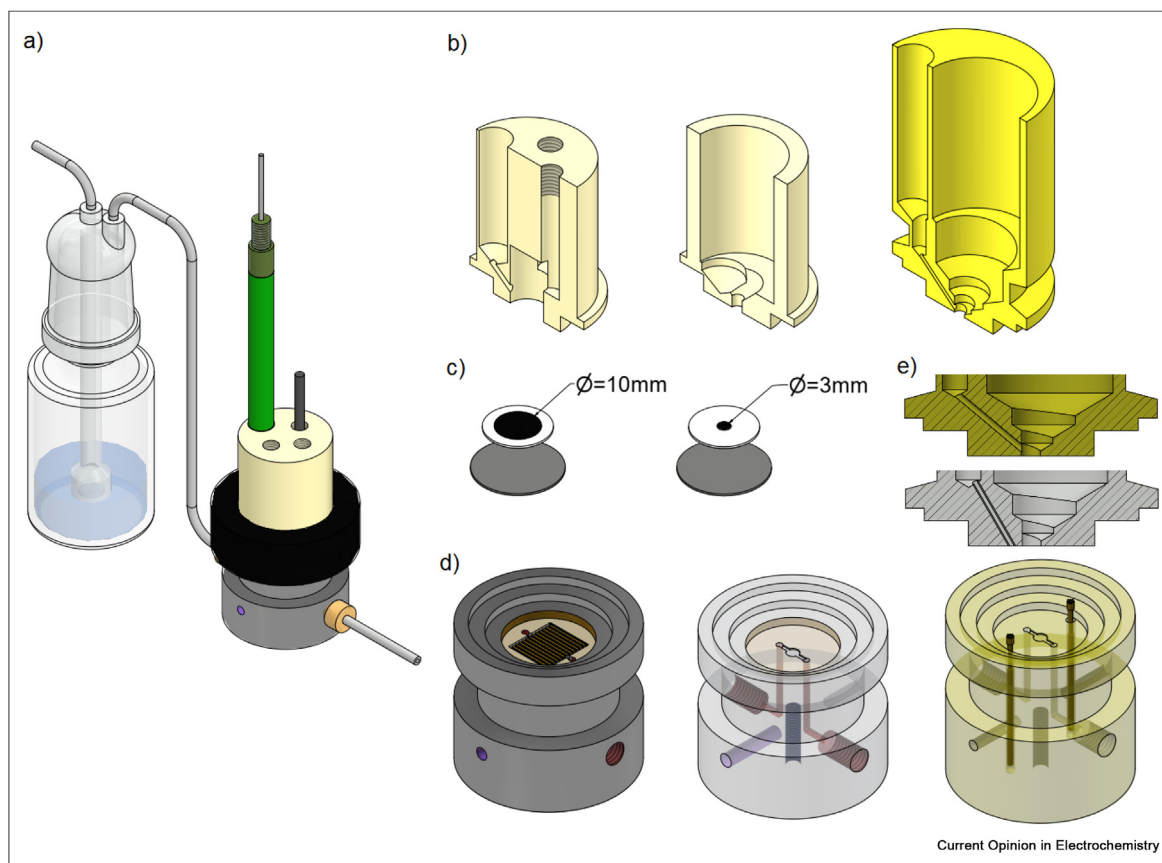
## Introduction

There have been several efforts to improve the reactant mass transport in electrocatalytic measurements. The rotating disk electrode (RDE) is the prevailing standard technique to study fuel cell reactions in basic science—a thin catalyst film is applied to a glassy carbon disk, which is rotated in the liquid electrolyte [1–4]. Thus, a homogeneous reactant flow to the catalyst is established that can be controlled by the rotation rate. When working with gaseous reactants, this technique has a significant drawback as the solubility of a gas in an aqueous electrolyte is low. As a consequence, the

achievable current densities in RDE measurements are limited to around  $6 \text{ mA cm}^{-2}_{\text{geo}}$  (for  $\text{O}_2$  at 1600 rpm) [5]. By comparison in membrane electrode assemblies (MEAs), which represent a single unit of a fuel cell stack, almost three orders of magnitude higher current densities can be reached (i.e., typically  $>4 \text{ A cm}^{-2}_{\text{geo}}$ ) [6]. The low reactant mass transport in RDE measurements severely limits the potential range where reaction kinetics can be investigated; therefore, methods such as the floating electrode have been introduced that allow substantially higher reactant mass transfer [7,8]. These efforts led to several independent designs of setups targeting increasing mass transport [9–13]. Since several articles and reviews on the application of GDE setups and more recently an inter-laboratory comparison of the use of different GDE designs exist [14–22], we do not discuss the general use of GDEs.

Instead, we selectively describe the “evolution” of the GDE setup designed in our research group. We used our initial GDE design [10] (Figure 1a) to study ethanol oxidation by simulating conditions of high temperature proton exchange membrane fuel cells (HT-PEMFCs). Therefore, the upper compartments contained hot (up to  $160 \text{ }^\circ\text{C}$ ), concentrated phosphoric acid. The catalyst coated gas diffusion layer (GDL) was in direct contact with the phosphoric acid. To illustrate the challenges that came with introducing our new setup, we briefly discuss the initial design and stepwise advancements, then our latest work applying the GDE setup at elevated temperatures using aqueous electrolyte in the upper cell compartments. Working at the same temperature as fuel cells or other conversion devices operate is an important advancement, particularly for degradation studies. Although many applications of GDEs concentrate on activity measurements, GDEs also offer the opportunity to considerably improve and expand catalyst degradation studies. Such studies are still scarce [23–29]. However, GDE setups are particular suitable for studying catalyst degradation. They offer the same, straight-forward methods for determination of the electrochemically active surface area as RDE measurements yet apply a catalyst layer that has the same thickness and properties as in an MEA. Therefore, it can be easily coupled to ex-situ characterization techniques, such as X-ray diffraction (XRD) or small angle X-ray scattering (SAXS) that require a certain catalyst amount. Another important advantage over standard RDE measurements to investigate degradation is that

Figure 1



(a) The basic gas diffusion electrode setup, see text for more specifics. (b) cross section of the different upper parts designed. (c) catalyst coated gas diffusion layer covered by a membrane (d) different versions of lower parts. (e) Upper parts with differently positioned ends of the Luggin capillary; above the membrane and at the membrane.

the catalyst does not have to be in direct contact to the liquid electrolyte. This may have a significant influence on the observed degradation mechanisms as it is for example known that the type of electrolyte influences Pt dissolution [30,31]. The advantages of GDE over MEA measurements, are not only the ease of the measurements and the accurate control of the working electrode conditions, also it is important that by applying accelerated stress tests (ASTs), degradation can be studied providing statistics and standard deviations and not only single data points. Additionally, spatial heterogeneities of the catalyst layer, which would not be observable in a full-sized MEA due to averaging, can instead be observed with the smaller sized GDE. A discussion concerning the choice of differently sized working electrodes (geometric area) can be found in Ref. [22]. Ultimately operando studies with realistic GDE setups will become state-of-art as well [32].

#### Gas diffusion electrode setup: Basic design

Our initial GDE design was developed with the goal to be easily and quickly assembled and modified [10]. Since setups can never be perfect for all applications, we implemented a modular design with improvements based on changing requirements. In the following section, we give a few examples how the design has changed over time and which achievements have been reached.

We show a basic GDE setup in Figure 1. The setup consists of a gas humidifier, typically we use a simple gas bubbler filled with water, and the GDE electrochemical cell. The electrochemical cell is comprised of a lower and an upper part. The upper part has compartment(s) filled with liquid electrolyte; it houses the counter electrode (CE) and reference electrode (RE). The lower part has extruded gas lines and a gas flow field. It also facilitates the electrical contact to the working

electrode (WE): a catalyst coated GDL, i.e., the GDE. The GDE WE is sandwiched between the two parts. Additionally, in [Figure 1](#), we show different examples of the upper and lower parts and discuss them below.

### Gas bubble formation and minimizing the solution resistance

The most difficult problems to address when designing GDE setups are related to high current densities. High current densities cause ohmic heating which can significantly increase the local temperature which can affect the apparent reactivity. High current densities also result in massive gas formation at the counter electrode and a significant voltage offset due to the solution resistance between RE and WE.

In the first design, we used a 10 mm GDE with an upper part, where the volume of the liquid electrolyte was kept small and the CE and RE were kept in separate compartments ([Figure 1b](#)). We placed a small hole, known as the Luggin capillary—near the bottom; it connected the two compartments. The separation of the compartments avoided that gas evolution at the CE disturbed the contact between WE and RE. If the WE-RE contact breaks, the potentiostat loses control and pushes the compliance current through the system—the result is severe ohmic heating possibly burning the electrode. However, during steady state measurements at large overpotentials, we observed large current fluctuations. The low electrolyte volume together with the massive gas evolution created gas bubbles that temporarily caused the contact between WE and CE to break and consequently stop the reactions. We addressed this issue in a second version.

In the second version, we wanted to maximize the electrolyte volume. We designed an undivided upper part and placed the RE and CE next to each other. Additionally, we also reduced overall current by making the GDE 3 mm-diameter and simplified the gas flow field ([Figure 1c](#)). Although this cell combination results in fewer issues with gas bubbles, consequently it led to varying positions of the RE which caused the apparent solution resistance to vary greatly between measurements. We therefore designed a third version with two compartments and large electrolyte solution volume.

The measured WE potential—the voltage difference between the RE and the WE—is the sum of the polarization of the electrode interfaces and the voltage drop over the resistance of the solution between WE and the end of the Luggin capillary when a current is passing. The voltage drop over the resistance can thus be considered to be an unwanted voltage error proportional to the current. Whether the controlled quantity is the current, state of the art for large current systems, or voltage, common for small current systems, this voltage error is equally present. Even in highly conductive

electrolytes, a non-neglectable solution resistance is common. In the third design version, a cell resistance of about 1–2  $\Omega$  was typical. To put that in context, a total current of 0.1 A (i.e., 1.4 A cm<sup>-2</sup><sub>geo</sub> for a 3 mm electrode) results in a voltage offset (iR drop) of 0.1–0.2 V. This is typically a voltage difference in which the kinetic current of a reaction changes by one to two orders of magnitude; for the ORR Tafel slopes of 60–120 mV dec<sup>-1</sup> are reported [[6,15,18](#)]. Consequently, the determination of what causes an observed current profile can be ambiguous towards large current densities as the voltage error can dominate. Therefore, electrode polarization potential uncertainties should be at most a few millivolts.

To reduce voltage error, the potentiostat can use an active potential compensation or data can be corrected after measurement. In principle, active potential compensation is preferable because post measurement corrections cannot correct for dynamic effects. If using post correction, steady-state instead of potentiodynamic measurements should be performed [[15](#)]. Both these techniques rely on determining the system solution/cell resistance with high precision.

Different strategies determine solution/cell resistance from recording full frequency electrochemical impedance spectroscopy (EIS) to single frequency impedance measurements at a frequency high enough to measure only solution resistance. The preferred method depends on the WE geometric area [[22](#)]. Generally, the larger the WE the more precise the iR drop needs to be determined. In our GDE setup using a small WE area, the absolute currents are relatively small and a single frequency at 5 kHz can determine the solution resistance. An advantage of using a single frequency is that the solution resistance can be monitored continuously during the measurement since it can be superposed onto other probing techniques, such as voltage/current ramping. However, determining the solution resistance much better than  $\pm 10\%$  is unlikely under operational conditions. As a result, using our previous calculation values, we still have an error of about  $\pm 10$  mV, which grows with the current, also after correction or compensation.

Minimizing the solution resistance via the cell design is therefore paramount. In contrast to having a small liquid volume between the end of the Luggin capillary and the membrane ([Figure 1e](#)), by moving the end of the Luggin capillary directly to the membrane, we reduce the solution resistance in our cell to less than 0.1  $\Omega$ . The estimated voltage error is thus less than 10 mV without compensation or post correction. The remaining small voltage error can easily be removed by post correction. Yet, a too small solution resistance can impede with the control circuit of the potentiostat and result in a loss of control during voltage control mode. In such a case, the control circuitry needs to be slowed down or current control measurements performed.

### Implementing a membrane between catalyst layer and liquid electrolyte

For measurements, we typically use a catalyst coated hydrophobic GDL as GDE. Producing such an electrode is straight forward and easily achieved by drop casting, vacuum filtration, or spraying of the catalyst. Such a GDE is good for catalyst screening and catalyst layer optimization. For GDE measurements, the result depends on the quality of the catalyst layer and not solely the intrinsic properties of the catalyst itself, especially when large current densities are applied. In our initial design, the GDE separated the reactant gas and the electrolyte: viscous concentrated phosphoric acid [10]. However, we narrowed the gap between GDE and MEA measurements by introducing GDEs covered by ion-conductive membranes as separation to an aqueous electrolyte [14].

Typically, covering a catalyst coated GDL with an ion-conducting membrane entails some form of pressing to establish good adhesion between the catalyst layer and the membrane for maximal performance. We use a simple in-house developed press for hot and cold pressing. As proton exchange membrane (PEM), we use relatively thick and robust Nafion membranes (Nafion 117). In contrast, anion exchange membranes (AEMs) are typically more fragile and pressing easily rips the membrane. When using AEMs, we apply no pressure [23,33–35].

In principle, catalyst coated membranes (CCMs) can also be used in the setup. CCMs are by nature more complicated to fabricate yet more suitable for catalyst layer optimization since the same fabrication methods for MEA studies are used.

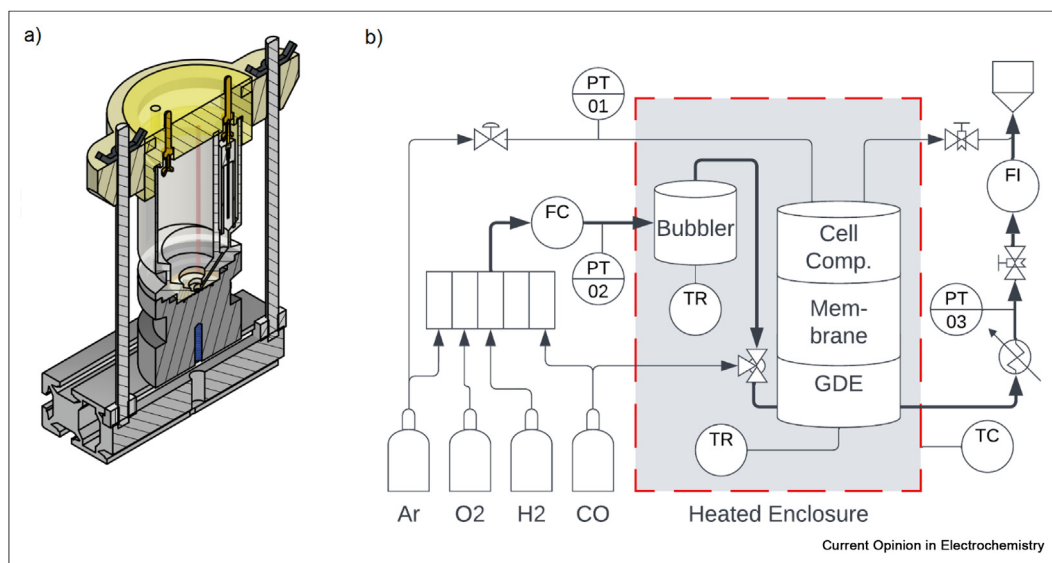
The hydrophobic GDL can lose its hydrophobicity if exposed to large positive potentials and can then easily be flooded. A flooded GDL cannot transport gas and as an effect the maximal current density is significantly reduced. An advantage of using a membrane is that it better blocks electrolyte from leaking into the GDE. Instead, a membrane covered GDE requires a humidified reactant gas feed or the catalyst layer slowly dries out and performance lost [14]. Further, we also notice that the catalyst needs activation, which is more effective using oxygen gas; thus, creating water in the catalyst layer, instead of flushing the GDE with Ar. Similar observation can be done with a MEA.

As preparation methods of GDE can be similar to those used for MEAs, the results from GDE setup experiments would be technical relevant also for a MEA.

### Beyond ambient conditions using aqueous electrolytes

Our most recent design efforts for improving the GDE setup were to reach fuel cell operando conditions ( $\geq 80\text{ }^{\circ}\text{C}$ ) using aqueous electrolyte in the upper part. Our experience with the initial design showed a non-closed/non-pressurized system only practically operates up to  $60\text{ }^{\circ}\text{C}$  due to electrolyte evaporation. We used our experience with pressurized RDE setups operated at elevated temperatures and pressure, which were successfully applied in several studies but are cumbersome in its use [36–38]. To pressurize the GDE setup, we designed a lid to fit the upper part (Figure 2a). For RE, we used a platinum wire inside a glass tube with trapped hydrogen gas—an electrode known as trapped hydrogen electrode (THE).

Figure 2



(a) Pressurized GDE cell, (b) setup schematics.

We show the system schematic in Figure 2b. To sustain the elevated temperature and pressure, we custom made a bubbler from polysulfone (PSU). We placed the GDE cell and the gas bubbler in a heated enclosure. A flow controller limits the gas flow and a valve at the outlet sets the pressure. With this setup, even temperatures exceeding 100 °C are feasible.

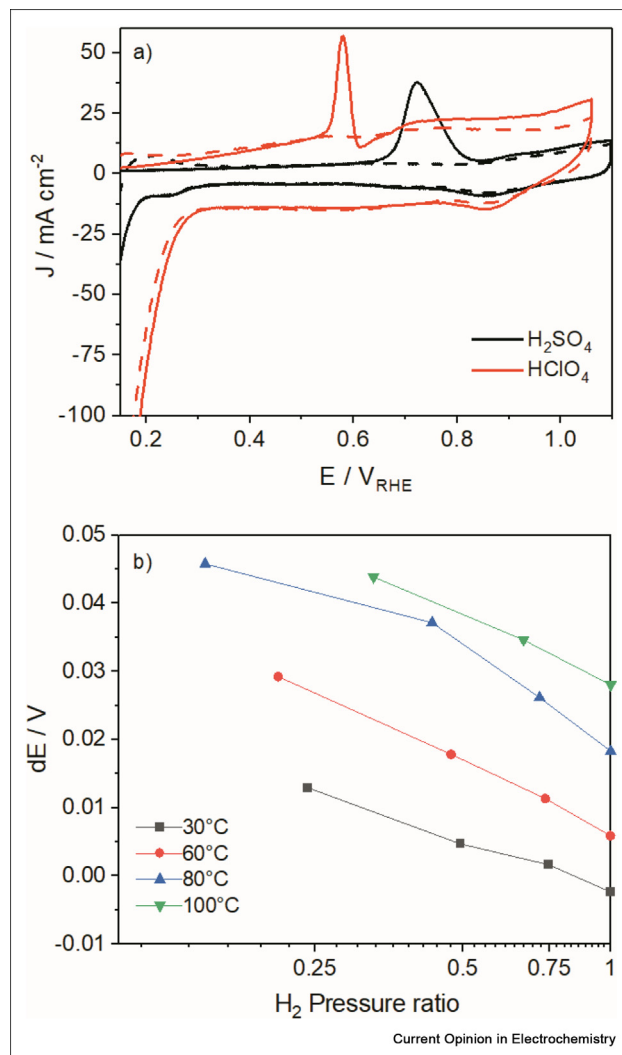
### Working with a pressurized GDE setup

Applying such elevated temperatures allows studying the impact of temperature on degradation phenomena and also its impact on basic electrochemical measurements. However, at temperatures above 60 °C the stability of the aqueous electrolyte must be considered. In Figure 3a, we show CO stripping curves recorded at 80 °C when the aqueous electrolyte in the upper part was sulfuric and perchloric acid, respectively. The measurements we recorded with perchloric acid exhibit substantial artifacts, such as a reduced, unsymmetric CO stripping peak and larger double layer capacitance, which leads to considerable differences in the CO stripping charges and therefore the apparent ECSA values [using the normalization factors of room temperature measurements (i.e., ca. 60 and 30 m<sup>2</sup> g<sup>-1</sup><sub>Pt</sub> in sulfuric and perchloric acid, respectively)]. Our findings indicate the perchloric acid starts decomposing and affects the catalyst and support under these conditions [39]. Instead, using sulfuric acid exhibits more typical features of the CO stripping curves.

Furthermore, when working with pressurized parts, it is important to recognize that the conditions at the RE can be substantially different from those at the WE. In other words, the reversible hydrogen evolution/oxidation potential the WE experiences can be substantially different from the RE experiences in the upper part. To demonstrate, we flushed the WE (Pt/C) with hydrogen and cycled the electrode slowly. Afterwards, we changed the pressure in the gas lines while we kept the pressure in the upper part constant at 4 bar. The resulting change in pressure ratio between the parts led to a potential difference between RE and WE at 0 current (Figure 3b). Without analyzing these trends in detail, it can be seen that only at around room temperature (here 30 °C) the reversible hydrogen potential of the THE and at WE are the same. A pressure difference or higher temperatures leads to systematic deviations between the RE (THE) and the WE reversible hydrogen potential. Since the potentiostat uses the RE as control, yet the investigated reactions occur at the WE, these deviations need to be accounted for when planning experiments or analyzing the recorded data.

Varying the operation temperature also requires refined or adapted normalization procedures for the determination of the ECSA [40]. For example, in standard electrochemical degradation studies, upon applying an AST

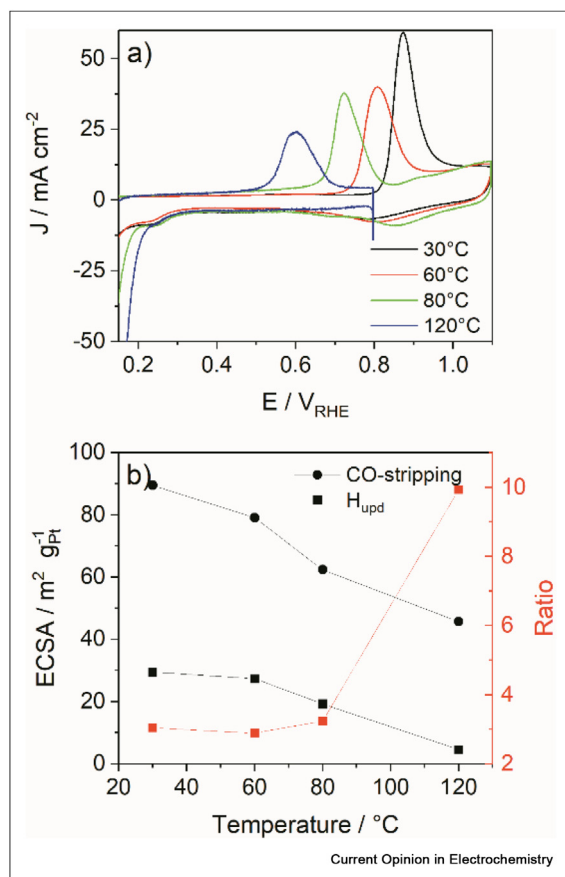
Figure 3



(a) We recorded CO stripping curves at 80 °C with the upper part filled with aqueous H<sub>2</sub>SO<sub>4</sub> (black line) and HClO<sub>4</sub> (red line), respectively. The dashed curves show the CV recorded in Ar atmosphere directly after the CO stripping measurements. The pressures in the upper and lower parts were 4 and about 2 bars, respectively. (b) We plotted the potential difference between the reversible potential for hydrogen evolution/oxidation reaction that we measured with a Pt/C working electrode at different temperatures relative to THE as a function of the hydrogen partial pressure ratio between upper and lower part. We maintained the pressure of the upper part at 4 bar, while we changed the pressure in the gas lines from 1 to 2, 3, and 4 bar. The electrolyte was 1 M aqueous H<sub>2</sub>SO<sub>4</sub>.

the change in ECSA is often used to probe the degree of degradation [41]. To determine the ECSA of Pt based catalysts from electrochemical measurements, either CO stripping experiments are performed or the H<sub>upd</sub> charge is determined, which does not necessarily lead to the same values [42–44]. In Figure 4a, we demonstrate that the CO stripping charge of a Pt/C catalyst is strongly temperature dependent. The temperature dependence

Figure 4



(a) We recorded CO stripping curves at different temperatures. The upper part filled with 1 M  $\text{H}_2\text{SO}_4$  and pressurized with 4 bar; the lower part between 1 and 3 bars, depending on temperature. (b) We determined apparent ECSA by CO stripping charge and  $H_{\text{upd}}$  area, respectively, as a function of the applied temperature. In addition, we plotted the ratio between the two values in red.

can be assigned to a temperature dependent CO coverage on Pt/C related to two effects. First, the saturation coverage (under CO pressure) is temperature- (and pressure-) dependent. Second, before recording the CO stripping measurement, the atmosphere at the catalyst changes from CO gas to inert Ar gas. During the gas exchange the adsorbed CO can desorb from the catalyst, but not re-adsorb. Thus, the CO coverage can slightly decrease with time. At low temperatures, this effect can be neglected; however, not at elevated temperatures. Nevertheless, the GDE has a significant advantage over classical measurements in this respect. In RDE measurements, all CO gas needs to be removed from the aqueous electrolyte which requires up to 20 min. In the GDE setup, the CO gas is exchanged in the gas phase, which is achieved in a matter of seconds. But the temperature dependence of the observed CO stripping charge requires temperature-dependent charge normalization values (for room temperature, typically a charge between 390 and 420  $\mu\text{C cm}^{-2}_{\text{Pt}}$  is

assumed for the two electron process [3,40,43]). Integrating the charges indicates the value is constant only up to ca. 60  $^{\circ}\text{C}$  and rapidly decreasing at higher temperatures. When keeping the temperature constant, an alternative to an “absolute” ECSA determination is to monitor the relative loss in ECSA during an AST.

Furthermore, the temperature dependence of the  $H_{\text{upd}}$  charge and the CO stripping charge are not identical. In Figure 4b, we show that we determined substantially lower  $H_{\text{upd}}$  charges than CO stripping charges effect might be partially related with insufficient catalyst cleaning as we avoided longer potential excursions to clean the catalyst surface despite the fact that it is known that the measured  $H_{\text{upd}}$  area is highly sensitive to surface contaminations [42]. We also observed the hydrogen evolution starts at substantially earlier electrode potentials at higher temperatures, which might shift the ratio of  $H_{\text{upd}}$  and  $H_{\text{Opd}}$  and distort the ECSA determination. Furthermore, the adsorption energies of  $H_{\text{upd}}$  atoms and CO molecules are quite different which is known to complicate the measurement of the ECSA of Pt-alloys via the  $H_{\text{upd}}$  charge [43]. At 120  $^{\circ}\text{C}$ , the monometallic Pt/C  $H_{\text{upd}}$  region is mostly undetected. Hence at elevated temperatures, determining the ECSA via CO stripping or other more robust methods is essential. Since the pressurized cell is a closed setup and the determination method should minimally influence the investigated electrochemical processes, other methods, such as metal upd, are not feasible [40]. Consequently, we regard CO stripping as the best alternative and recommend its use.

### Future outlook

Developing new performance and cost optimized GDEs or CCMs for fuel cell applications is a time- and cost-intensive processes. Each catalyst layer can consist of nearly infinite combinations of active phase, catalyst support materials, ionomer, porosity and so forth. Therefore, materials, amounts, as well as manufacturing techniques require elaborate optimization. In addition, these GDEs or CCMs must work together in the stack as a whole. Although all fuel cell-related issues cannot be addressed and studied using a 3-electrode GDE setup, we strongly suggest many can. Our simple setup provides a variety of insightful data for optimization, especially since it now functions at fuel cell operando conditions. Therefore, the GDE approach bridges basic and applied research for fuel cell catalysts.

In the next step, the ability to perform experiments in parallel as it is commonly done for batteries could greatly enhance scientific throughput, which is also important for stability investigations enabling statistics. Our small GDE setup and economical instrumentation needed to control it, is well suited for such parallel

measurements. In addition, pressurized setups for operando measurements at elevated temperatures is a great asset for investigating temperature effects.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

### Data availability

Data will be made available on request.

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- \* of special interest
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