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Influence of Contact Pressure on Performance and Crossover in Zero-Gap Alkaline Water Electrolysis

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ABSTRACT

Performance and product gas purity in alkaline water electrolysis are influenced by the mechanical contact pressure between electrodes and diaphragm in zero-gap cells. This relation is addressed here using a self-designed contact pressure cell, which allows us to adjust the contact pressure directly during cell operation. It is found that the performance in non-welded lab cells can depend significantly on contact pressures. This mainly depends on varying contact resistances, but also on the deformation of the electrodes and diaphragm. The latter leads to increased crossover, which is also visible in a reduced bubble point pressure. Welding the electrode to the flow field reduces the impact of the contact pressure on performance, but not on the crossover. Optimizing and controlling contact pressure impacts in zero-gap cells is essential, and the presented cell simplifies future studies in this field.

1 | Introduction

Alkaline water electrolysis (AWE) is described as a mature technology to split water into hydrogen and oxygen with electric energy. AWE is characterized by a long lifetime and relatively inexpensive components [1]. To improve the impact of hydrogen and AWE on the global energy transition, further technological development is necessary to decrease hydrogen costs. In particular, the gas purity at part load, dynamic operation in combination with renewable energy sources, and maximum current density need to be improved [2].

AWE cells consist of two electrodes (anode and cathode) separated by a porous diaphragm (mostly made of zirconia and polymeric binders [3–6]) and an ion-conducting electrolyte (usually potassium hydroxide in water). Due to the porous nature of the diaphragm, gas crossover is a serious concern in AWEs, which reduces the hydrogen production rate and purity [7].

Crossover occurs due to concentration and pressure gradients between the anode and cathode, leading to diffusive, convective, and electroosmotic transport, which results in gas contamination of H₂ with O₂ (hydrogen-to-oxygen volume fraction, HTO) and O₂ in H₂ [8]. Such impurities reduce the value of the product and can lead to safety issues due to the explosion limits of 4 vol% H₂ in O₂ and 6 vol% O₂ in H₂ [9, 10]. Overpotentials of the electrodes, mass transport losses, and ohmic losses increase the cell voltage compared to the reversible cell voltage and reduce the efficiency [11]. Ohmic resistances consist of the ionic resistance inside and outside the diaphragm (porosity, thickness, electrolyte conductivity, gas bubbles), as well as other electric bulk and contact resistances of the components [11].

When the electrodes and diaphragm are in direct contact, the cells are designated zero-gap cells, as illustrated in Figure 1. Compared to gap cells, a shorter ionic transport distance between the electrodes reduces the ohmic losses [12]. Zero-gap cells need

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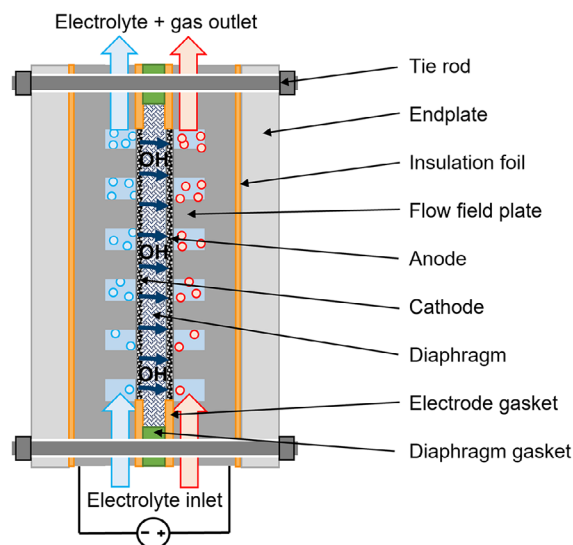


FIGURE 1 | Structure of a zero-gap AWE cell (based on the concept of Karacan et al. [16]).

flow fields and porous electrodes to supply the electrolyte over the active area and remove the gas [8, 13–15]. To ensure a zero-gap structure, the flow field and electrodes are pressed onto the diaphragm. This compression force is usually applied by tie rods compressing the cell (see Figure 1). The amount of force or the contact pressure between the flow field, electrode, and diaphragm could influence the cell behavior.

1.1 | Contact Pressure and Cell Performance

There is very little literature investigating the direct influence of contact pressure on cell performance in AWEs, but there are studies on anion exchange membrane (AEM) and proton exchange membrane (PEM) electrolysis, which feature a polymer membrane instead of a porous diaphragm. For AEM electrolysis, Ahn et al. [17] found the optimum contact pressure at approximately 4.1 MPa. In their setup, decreasing interfacial resistance was reported to improve the performance with increasing contact pressure. In contrast, Xia et al. [18] found the optimum contact pressure in their AEM cell to be approximately 0.5 MPa. Thus, the optimal contact pressure seems to be specific to the cell setup and is an important parameter when optimizing cell performance. In PEM electrolysis, contact pressure influences three main effects: contact resistances, water content of the membrane, and mass transport properties of the porous transport layers (PTLs) [19–21]. Increasing contact pressure reduces contact resistances between the PTL, flow field, and membrane [22–24]. At high contact pressure, water is pressed out of the membrane, which reduces the ionic conductivity [20]. As an example, Borgardt et al. [21] determined the optimal contact pressure for their setup at 2.5 MPa. On the other hand, high contact pressure can damage the PTL and membrane and lead to poor mass transport [25].

In AWE, Hartert et al. [26] investigated the effect of electrode compression on cell performance in a zero-gap cell. The compression was adjusted by the cathode gasket thickness, but the actual contact pressure and contact pressure distribution were not measured. It has been shown that increasing compression of

the electrode leads to mass transport limitations, which impair performance [26]. In one of our earlier studies [27], we developed a specialized differential test cell that, in addition to other features, allows for adjusting contact pressure on the active area. For a one-cell configuration featuring a UTP220 diaphragm and catalyst-coated mesh electrodes welded to the nickel foam flow field, the influence of contact pressure on cell performance was measured. Increasing the contact pressure for the first time improved the cell performance, while it was almost constant with the contact pressure after compressing the components multiple times.

At the level of AWE components and cell configurations, various studies offer results that can be linked indirectly to the influence of contact pressure. The effect of electrode spacing is investigated by varying the gap from a few millimeters to a zero-gap configuration [12, 28–32]. According to Phillips et al. [12], the ohmic resistance of the cell can be reduced in a zero-gap setup by 30% compared to a 2-mm gap. In gap cells, a lower gap width means a lower cell resistance, but gas removal is crucial because gas bubbles can block parts of the electrode or increase the path length for ionic transport [32, 33]. Small gaps of 200 μm outperformed zero-gap gaps in cells with perforated plate electrodes [31]. In gap or small-gap configurations, the main share (up to 95% depending on the electrode geometry) of the current density appears on the front side of the electrode facing the diaphragm [30, 33]. In zero-gap cells, this front side of the electrode is partly deactivated due to its contact with the diaphragm. A porous electrode can compensate for this by providing additional active sites within its structure [33]. The structure and morphology of cell components may change when they are mechanically compressed, which alters their properties. Electrodes were optimized, for example, regarding active sites and bubble entrapment [12, 34]. Both factors could be influenced by deformation at high mechanical contact pressure. Xia et al. [18] investigated the wetting behavior and physical properties of AEM electrodes and PTLs (thickness, contact resistance, and bulk resistance) after compressing them to a specific mechanical contact pressure. Decreasing thickness and resistances were measured for higher contact pressures. Excessively compressing components such as the porous electrode or the flow field can lead to poor transport of electrolyte and gas [11, 17, 18]. Hindered mass transport inside the electrode and the trapping of gas bubbles covering active sites of the catalyst are reported to decrease the cell performance [12, 17]. In particular, a mechanically less stable flow field (e.g., metal foam) could significantly change its properties due to contact pressure. Compressing the diaphragm will change its thickness and can thus also affect other properties, such as pore size, porosity, tortuosity, and electrolyte content. These parameters influence the ionic conductivity of the diaphragm [35, 36]. de Groot et al. [35] found that in zero-gap cells, a large ohmic contribution exists, apart from the measured diaphragm resistance. They assumed that this could be caused by an uneven current distribution, local gaps between the electrode and diaphragm, or gas bubbles in the electrolyte [35]. The presence of local gaps could be related to a low contact pressure or an uneven distribution.

On the interface between the components (e.g., flow field and electrode), contact resistances are reported to be contact pressure dependent [37]. Welding the electrode to the flow field can

avoid contact resistances. Welded configurations are common in industrial electrolyzers, whereas in lab cells, the electrodes are usually only stacked [11, 16, 17, 38, 39].

1.2 | Contact Pressure and Gas Crossover

In addition, regarding gas crossover, there is very little literature investigating the direct impact of contact pressure in zero-gap AWE cells. In PEM electrolysis, Martin et al. [40] and Stähler et al. [41] revealed that hydrogen permeation reacts very sensitively to compression of the gas diffusion layer and membrane. Increasing the contact pressure and the compression of the inner components leads to increased crossover.

For AWE, one study found an increasing HTO from 0.8% at 0.2 MPa to 2.0% at 2.0 MPa, measured for a one-cell configuration featuring catalyst-coated mesh electrodes and a UTP220 diaphragm [27]. Another paper analyzes the effect of electrode compression on the hydrogen crossover in AWEs but does not link it to the contact pressure [26]. The HTO value was found to increase from approximately 0.5% at almost zero compression to 3.5% at 16% compression (0.5 A cm^{-2} , UTP220). They assume that a thinning of the diaphragm and more bubble penetration increases the diffusive and convective crossover. The impact of gap size was evaluated regarding the crossover in gap cells. Barros et al. [42] found the cathodic gap to be an important parameter. A decreasing gap width toward zero-gap was found to increase the crossover. They confirmed that supersaturation on the surface of the diaphragm plays a crucial role. The contact pressure in zero-gap cells potentially also affects this crossover mechanism.

Damaging the diaphragm at high contact pressure will probably also increase crossover. One indication of the gas separation capability of a diaphragm is the first bubble point pressure measurement, which is widely used for diaphragm characterization [43]. In short, the first bubble point is the pressure at which a gas, often nitrogen, is pressed upward through the wet diaphragm, indicating the maximum pore size. In AWE, a strong and stable separator with a high first bubble point pressure is desired to minimize gas crossover [43, 44]. When diaphragms are affected by high contact pressure, the first bubble point might change.

Overall, the optimal contact pressure in AWEs will depend on the type and structure of the electrode, flow field, and overall cell design, as observed previously for other electrolysis technologies [17–25]. Contact pressure has a major influence on cell performance and gas crossover in PEM and AEM electrolysis but has not been systematically investigated for AWEs thus far. Unlike PEM or AEM electrolyzers, typical industrial alkaline electrolyzers often have open flow channels instead of conventional PTLs [45–50]. They also differ significantly in the structure of their flow fields or electrodes, such as porous foam electrodes, perforated metal sheets, or expanded metals [47, 51, 52]. Laboratory cells may deviate even further from commercial systems, particularly regarding the flow field geometry [38]. As a result, investigating the influence of contact pressure is more complex than for PEM and AEM electrolyzers, and the effects of various cell configurations must be carefully investigated. This work takes a first step toward addressing this gap, contributing to comparable testing and scale-up of AWE cells.

2 | Design of the Contact Pressure Cell

In standard zero-gap cells, as shown in Figure 1, the inner components are stacked between two endplates and clamped by tie rods [16, 38]. The compression of the inner components and thus the contact pressure is usually adjusted by adapting the gasket thickness to the electrode and diaphragm thickness. It is usually assumed that the gaskets (mostly made of PTFE) are much harder than the electrodes and the diaphragm and act as a hard stop for cell compression [12, 16]. In this setup, the contact pressure can only be measured in a preliminary test for a specific cell configuration (electrode, diaphragm, flow field, and gasket thickness), for example, by using contact pressure-sensitive films. If the cell is assembled with new components for operation, the contact pressure could be different, for example, due to component tolerances, initial electrode thickness, thermal expansion, or water uptake of the diaphragm. To adjust the contact pressure on the active cell area or the gap width in gap cells, the cell must be disassembled to change the gasket thickness or to add some spacers, leading to reproducibility issues [12].

To overcome these issues and to investigate the influence of mechanical contact pressure in zero-gap cells, a new type of test cell was developed, a designated contact pressure cell. This test cell allows us to measure and adjust the contact pressure in a zero-gap cell during operation. Controlling and optimizing the contact pressure could help to improve the reproducibility and cell performance regarding current density and gas purity. A schematic illustration of the working principle of the contact pressure cell is given in Figure 2a. Instead of the conventional flat endplate, the contact pressure cell has a cell frame and a backplate. The cell unit is placed inside this frame and consists of an electrode attached to the electrode body, which is a massive stamp including the flow field. This cell unit is movable inside the cell frame and can be positioned and pressed against the diaphragm by one adjustment screw in each backplate (yellow arrows in Figure 2a). As the cell unit and the cell frame are two separate parts, the contact pressure in the active cell area is independent of the contact pressure in the gasket area. A force sensor can be placed between the adjustment screw and the cell unit to measure the applied force. Dividing the measured force by the electrode area yields the mean contact pressure on the electrodes. Even without such a force sensor, it is possible to calibrate the contact pressure depending on the torque or rotational angle of the adjustment screw. Alternatively, the preload of the screw can be estimated by mechanical calculations. The tie rods only apply the needed contact pressure to the diaphragm gasket to prevent leaks but do not influence the contact pressure in the electrode area.

An explosion view of the contact pressure cell is shown in Figure 2b. The electrode (5 cm^2 , $22.4 \times 22.4 \text{ mm}^2$) and electrode body have round corners to allow sealing of the unit. A circular O-ring gasket is used between the cell unit and the frame, retaining its movability. It is possible to use different types of flow fields, electrodes, and diaphragms in the contact pressure cell. For a different flow field or electrode type, only the cell unit will be replaced. It is possible to weld the three components of the cell unit (electrode body, flow field, and electrode) together before cell assembly to form a single component, avoiding contact resistances. Different diaphragms can be used by adjusting the

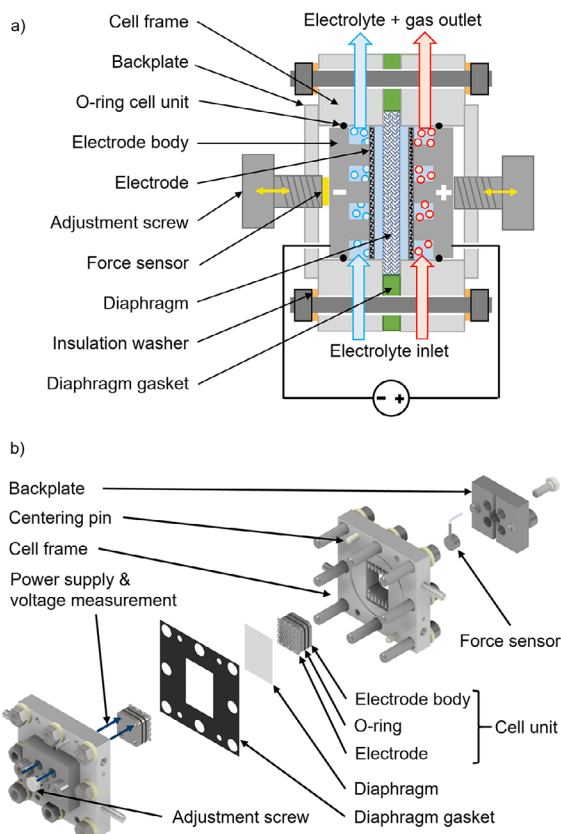


FIGURE 2 | Design of the contact pressure cell: (a) schematic structure of the cell, (b) exploded view of the CAD model.

thickness of the diaphragm gasket accordingly. The electrolyte is supplied and removed through the cell frame. The cell unit is electrically contacted from the back side. Insulation washers were used to avoid a short circuit via the tie rods.

3 | Experimental

The experiments with the contact pressure cell are divided into three groups. The contact pressure adjustment for the active area is first validated before measuring the effect on the cell performance. The influence of contact pressure on the crossover is analyzed by hydrogen in oxygen measurements, computer tomography (CT) imaging, and first bubble point measurements of the diaphragm after compression.

3.1 | Contact Pressure Adjustment

The nominal contact pressure on the cell area σ_N is calculated as the measured force F divided by the active cell area A (5 cm^2). The force applied to the active cell area is measured by an ultraminiature load cell (force sensor type 8416 from Burster Sensors and Process Instruments), which has a measurement range up to 2 kN with a deviation of 0.5% [53]. This measurement range allows us to set mean contact pressures up to 4 MPa.

$$\sigma_N = \frac{F}{A} \quad (1)$$

To validate the actual force application to the active cell area, pressure measurement films from FujiFilm Prescale were used to visualize the contact pressure distribution inside the contact pressure cell. The results were analyzed by Fujifilm FDP8010E software. A color bar represents the local contact pressure from 0.2 to 10 MPa. To measure such a wide range of contact pressures, three types of films were combined [54]:

- 0.2–0.6 MPa (LLLW FujiFilm Prescale ultra super low pressure)
- 0.5–2.5 MPa (LLW FujiFilm Prescale super low pressure)
- 2.5–10 MPa (LW FujiFilm Prescale low pressure)

For this preliminary test, contact pressure-sensitive films were added to a real cell assembly between the anodic cell frame and the diaphragm gasket (450- μm PTFE gasket, Zirfon UTP500 diaphragm). The anodic and cathodic halves of the contact pressure cell are assembled individually, each including the frame, cell unit, backplate, and adjustment screw. The force sensor is installed at the cathode side between the cell unit and the adjustment screw. Both adjustment screws are first set in such a way that the upside of the electrode is aligned with the cell frame. This procedure ensures that the diaphragm is not bent and damaged at the edge of the cell frame before compression. The anodic adjustment screw is then slightly loosened before mounting the cell. The diaphragm, diaphragm gasket, and contact pressure sensitive films are stacked on the cathodic half of the cell, followed by the anodic half. The cell is screwed together with tie rods (crosswise 2, 5, and 8 Nm). The contact pressure will be adjusted by the anodic adjustment screw and measured on the cathode side. A fresh diaphragm and new contact pressure-sensitive films were used for each experiment.

We investigated a nickel foam flow field (Recemat BV type Ni-1723.03) and nickel foam electrode (Recemat BV, 500 g m^{-2} , $300 \mu\text{m}$ initial thickness) at forces of 400, 800, and 1600 N for 2 min each. With the milled parallel flow field (2-mm channel and 2-mm rib width), the experiment was repeated for 800 N with nickel foam electrodes and with mesh electrodes from De Nora (anode: nickel expanded metal mesh; cathode: nickel woven metal mesh). Pictures of the components are shown in Figure 3.

3.2 | Influence of Contact Pressure on Cell Performance

The test station for the electrochemical experiments was built in-house [16]. A schematic illustration of the test station and the installation of the contact pressure cell is shown in Figure S1. All electrochemical experiments were conducted with 30 wt% KOH (Merck EMSURE 1.050339025; batch number MB2292633) at 80°C . Anolyte and catholyte gas separator tanks and the cell frame were heated to control the cell outlet temperature on the anode side to 80°C . Preheated electrolyte (50 mL min^{-1}) was fed to each anode and cathode by membrane pumps (KNF Simdos 10). The electrolyte leaves the cell together with the produced gas to the gas separator tanks. The electrolyte cycles for the anode and cathode are separate, but the tanks are connected by a balancing line. The electrolyte tanks are made of Monel, and the tubing is PTFE. The potentiostat (BioLogic BCS-815) is connected to the

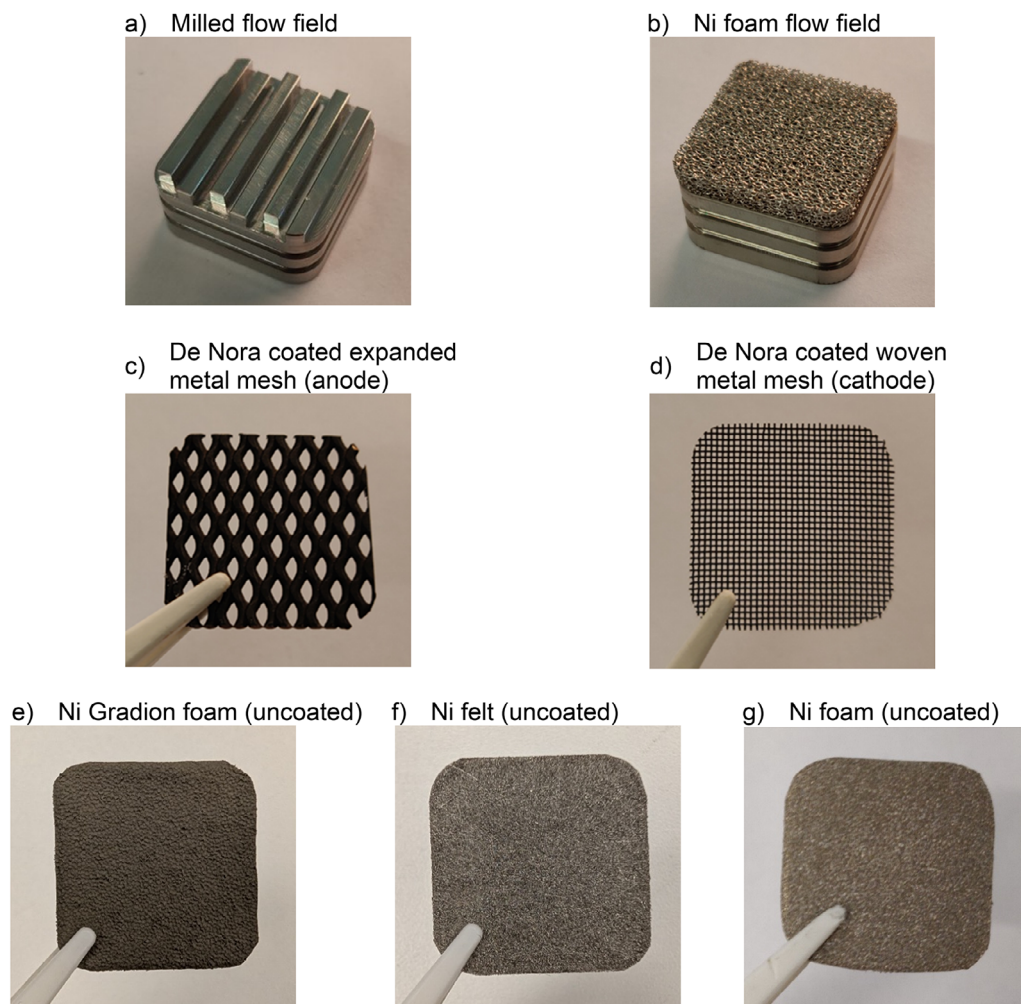


FIGURE 3 | Different flow fields and electrode types for the contact pressure cell.

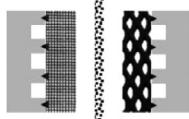
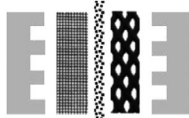
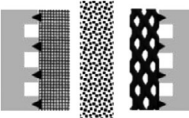
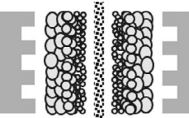
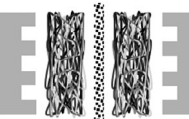
electrode body by 4-mm banana plugs individually for power supply and voltage measurement (blue arrows in Figure 2b). Two cell assemblies were operated in the same test station in parallel.

The different flow fields and electrodes for the contact pressure cell used in this work are summarized in Figure 3. The electrode body either contains (a) the milled flow field or (b) the electrode body is flat, and the foam flow field is stacked on top. Either catalyst-coated mesh electrodes from De Nora (anode: AENRG standard—coated on nickel expanded metal mesh; cathode: NRG-R standard—coated on nickel woven metal mesh), (e) uncoated nickel Gradion foam electrodes from Alantum (initial thickness 1.66 mm), or (d) uncoated nickel felt electrodes (Bekaert type 2Ni30-050, initial thickness 0.5 mm) were used. The electrode body, flow field and electrode are either loosely stacked on top of each other and only fixed by the contact pressure or are welded together. Fresh Gradion foam (Figure 3e) or felt (Figure 3f) electrodes and new diaphragms were used for each assembly. The commercial catalyst-coated electrodes from De Nora (Figure 3c,d) were used repeatedly, as they are considered stable under regular operation conditions. As diaphragms, Zirfon UTP220 (thickness $220 \pm 30 \mu\text{m}$ [4]) or UTP500 (thickness $500 \pm 50 \mu\text{m}$ [3]) from AGFA were used. The diaphragm gaskets are made of PTFE and are 10% thinner than the diaphragm, that is, $450 \mu\text{m}$ for UTP500 and $200 \mu\text{m}$ for UTP220.

The cell performance is measured using the described components for six different configurations, which are summarized in Table 1. In the milled flow field, the catalyst-coated mesh electrodes are either loosely stacked or welded to the flow field ribs. The electrodes were tested with a UTP220 or UTP500 diaphragm. The Ni Gradion and Ni felt are only assembled in a stacked configuration. The sketches in Table 1 show the order and arrangement of the components, which are also described by the corresponding abbreviations (flow field type, electrode connection, electrode type, and diaphragm). The black triangles represent the welding of the electrodes to the flow field, and the gap between the components means they are loosely stacked in a zero-gap configuration. For all experiments, the cell is initially assembled with a low contact pressure of 0.1 MPa (50 N) to ensure a zero-gap assembly.

The operation of the electrolysis cells starts with conditioning of the electrodes for 20 h at 2.0 V, which was performed before each experiment to mitigate the influence of the electrode history. Cell conditioning is important, especially when new components are used. The cell performance can change considerably in the first hours of operation [16, 38]. To measure the cell performance, potentiostatic I - V curves were measured from 1.5 to 2.0 V, with a step width of 0.1 V and a holding time of 3 min per step, and the mean current value was measured over the last minute. Since

TABLE 1 | Different cell configurations for performance measurements.

Abbreviation (flow field-connection-electrode-diaphragm)		Flow field (mill/foam)	Connection (weld/stack)	Electrode (mesh/NF/NGR/felt)	Diaphragm (220/500)
Mill-Weld-Mesh-220		Milled	Welded	Catalyst coated mesh	UTP220
Mill-Stack-Mesh-220		Milled	Stacked	Catalyst coated mesh	UTP220
Mill-Weld-Mesh-500		Milled	Welded	Catalyst coated mesh	UTP500
Mill-Stack-Mesh-500*		Milled	Stacked	Catalyst coated mesh	UTP500
Mill-Stack-NGR-220*		Milled	Stacked	Ni Gradion	UTP220
Mill-Stack-Felt-220*		Milled	Stacked	Ni felt	UTP220

Note: Three configurations were measured twice. The others, marked with an asterisk (*), are single measurements.

the uncoated nickel Gradion and nickel felt electrodes achieve significantly lower current densities at 2.0 V, all I - V curves with these components were measured up to 2.4 V. Electrochemical impedance measurements were conducted before each I - V curve at 1.5 V from 10 kHz to 1 Hz with a 10 mV amplitude. The high-frequency resistance (HFR) of the cell was read off at the highest frequency with a negative imaginary resistance.

The contact pressure is increased stepwise from 0.1 to 3.2 MPa (50–1600 N) in a first compression, and the I - V curve and HFR are measured for each pressure step. Between the I - V curves, the cell voltage is kept at 1.5 V to change the force of the adjustment screw without a full shutdown of the cell. After reaching the maximum pressure of 3.2 MPa, the adjustment screw is completely loosened to zero force, and the next compression cycle is started from 0.1 MPa. Again, the I - V curve and HFR are measured for each pressure step. Six compression cycles were recorded in total. The first three compression cycles were measured in 1 day, and the pressure was released to 0.1 MPa overnight, with a cell voltage of 1.5 V. Compression cycles four to six were recorded on the next day, again starting at 0.1 MPa.

For three of the cell configurations in Table 1, two separate experiments were conducted. Fresh diaphragms were used in

the second assembly, while the catalyst-coated mesh electrodes were reused from previous experiments, as well as the other cell components, such as the flow field stamps and the cell frame. The second run was carried out in a different lab and test station, with different temperature controls and balances of plant materials (Monel tubes instead of PTFE tubes). Testing the same components in different labs and test stations is known to yield different cell performances [38]. Reasons for deviations can be differences between the test conditions (e.g., cell and ambient temperature, flow rate, iron content, purity, and electrolyte concentration), as well as the diaphragm batch or electrodes with a different history, aging, or activation state. Conducting the second experiment in a different test station and different lab ensured that the results were robust against such influences.

With the milled flow field, catalyst-coated mesh electrodes (welded and stacked), and UTP500, a preliminary test to validate the measurement procedure was carried out with an old diaphragm batch, well beyond the recommended period of use. This resulted in a significant degradation rate and unclear trends over time and between the contact pressure cycles. This measurement is not included in further data analysis, and the result for “Mill-Stack-Mesh-500” is therefore only a single measurement. The “Mill-Weld-Mesh-500” experiment was repeated separately.

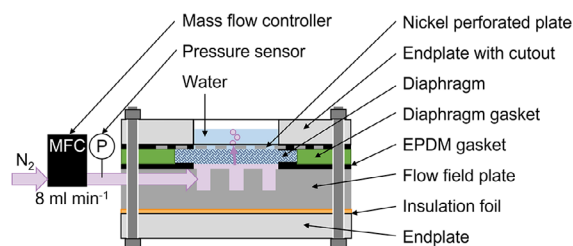


FIGURE 4 | Schematic illustration of the bubble point test setup.

To allow a comparison between the different configurations, the relative change in the current densities over the contact pressure is calculated. For each configuration and for different voltages, the average current density from Cycles 3, 5, and 6 for each contact pressure step is divided by the value at 0.1 MPa. This method allows us to compare the different cell configurations, even if the absolute current densities of the configurations are far apart. If the contact pressure causes a change in resistance, then a proportional voltage change should occur at a constant current. To compare the various configurations not only at the same voltage values, but also for a constant current, an individual reference voltage was extracted for each configuration corresponding to a reference current density of 1 A cm^{-2} at 0.1 MPa. This voltage was obtained by linear interpolation or extrapolation of the I - V curves at 0.1 MPa (Compression 3, 5, and 6) between 1.9 and 2.0 V (for Gradion and felt between 2.2 and 2.4 V). The relative change in current density with increasing contact pressure was then determined at this voltage for all compression steps.

3.3 | Influence of Contact Pressure on Gas Crossover

3.3.1 | Gas Purity Measurements

To measure the gas crossover (H_2 in O_2), the outlet of the oxygen gas separator tank was fed to a Micro GC 490 from Agilent Technologies (molecular sieve column). Apart from connecting the GC, the test station configuration remains identical to that used for cell performance measurements. Figure S1 shows a schematic illustration of the test station. The electrolyte cycles for the anode and cathode are separate, but a balancing line between the tanks is used to avoid differences in level and pressure.

For the crossover experiments, we used a separate cell assembly with fresh diaphragms and started with a minimum contact pressure of 0.1 MPa (50 N). The experiments were performed with fresh nickel foam electrodes (Recemat BV, 500 g m^{-2} , $300 \mu\text{m}$ initial thickness; Figure 3g) and foam flow field (Recemat BV, type Ni-1723.03), and in a second assembly, the catalyst-coated mesh electrodes with a milled flow field, both in a stacked configuration. In contrast to the cell performance measurements in potentiostatic operation, the gas crossover measurements were in galvanostatic operation. The cell was operated at 250 mA (0.05 A cm^{-2}) for 5 h, 500 mA (0.1 A cm^{-2}) for 2 h, and 1 A (0.2 A cm^{-2}) for 1 h. Depending on the current and the respective gas production rate, the measurement signal takes some time to stabilize. After 5, 2, and 1 h, the measured value at the GC tends toward a stable value. While changing the

contact pressure with the adjustment screw, the current was fixed at 250 mA to avoid the effects of a shutdown. The contact pressure was increased from 0.1 to 0.2, 0.4, 0.8, 1.6, and 3.2 MPa, measuring the gas purity for each of the three applied currents. After loosening the adjustment screw, this set was repeated one more time, followed by a third compression at 0.1, 0.8, and 3.2 MPa.

3.3.2 | CT of the Diaphragm

To check the diaphragm samples after compression and electrochemical performance measurements, two samples of the UTP500 and one UTP220 sample were scanned in the computer tomograph (CT) Xradia Versa 410 from Carl Zeiss AG. The CT scan provides a 3D model (tomogram) of the sample. The CT parameters are given in Table S1. A $10 \times 10 \text{ mm}^2$ section of the active cell surface was scanned to detect plastic deformation and possible damage to the diaphragm. After disassembling the cell, the diaphragm samples were cleaned and stored in water before the CT scan.

3.3.3 | Bubble Point Measurement

To evaluate the diaphragm integrity, the first bubble point pressure of selected samples (see Tables S2 and S3) was measured. If the diaphragm is damaged, the bubble point pressure will decrease due to large “pores,” causing increased gas crossover. If the diaphragm is just compressed and thus the porosity decreases, the bubble point pressure could also increase. Unused samples as a reference (*Pristine*) and samples from the electrochemical measurements (contact pressure cycles at 80°C in 30 wt% KOH), which were exposed to electrochemistry and contact pressure (EC&CP), were tested. In addition, *CP only* samples (contact pressure cycles at room temperature in water, not treated with any electrochemical reactions) were used to solely investigate the impact of contact pressure and number of pressure cycles without any electrochemical stress. The pressure steps, similar to previous experiments, were held for 1 min each, except that the highest pressure was held for 10 min.

Figure 4 illustrates the test setup for assessing the first bubble point pressure of the entire active area (5 cm^2) of the diaphragm. The bubble point test cell consists of two stainless steel endplates that are mounted around a nickel plate with a 5 cm^2 serpentine flow field, where the diaphragm to be tested is placed on top. The upper endplate of the test cell has a 5 cm^2 cutout, which is filled with wetting liquid water. The first bubble point is determined by introducing a constant nitrogen gas volume flow of 8 mL min^{-1} by a mass flow controller into the dead-end flow field below the diaphragm. The steadily increasing pressure is measured close to the cell inlet with the pressure sensor. To avoid deformation of the diaphragm and its pores by the increasing differential pressure between both sides of the diaphragm, a nickel perforated plate is placed on top of the diaphragm. EPDM gaskets are used to ensure gas tightness at elevated pressures. When reaching the bubble point pressure, the first continuous stream of bubbles in the water on top of the diaphragm can be observed visually, as well as by a slope decrease in the recorded pressure over time. To evaluate the

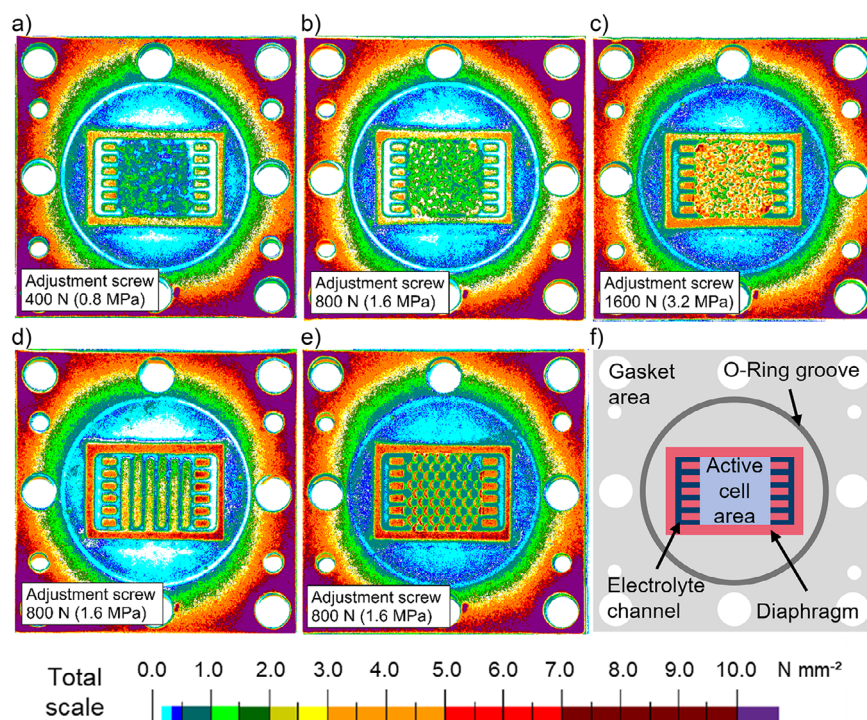


FIGURE 5 | Measurement of the contact pressure distribution: foam flow field and Ni foam electrodes at (a) 400 N, (b) 800 N, and (c) 1600 N at the adjustment screw. Contact pressure distribution for the milled flow field and (d) Ni foam electrodes and (e) De Nora metal mesh electrodes at 800 N. (f) Characteristic areas in the contact pressure measurements.

gas tightness of the setup, a PTFE sheet was used instead of the diaphragm. At 4.5 bar, the pressure loss was less than 0.05 bar h^{-1} over 53 min, which is satisfactory since the first bubble point is determined within a few minutes.

4 | Results and Discussion

The contact pressure cell enables the direct control of the contact pressure on the electrode area, independent of the force needed to compress the sealings for tightness. The contact pressure can be adjusted from the outside, even under operation. The contact pressure adjustment is first validated by contact pressure distribution measurements. Then, the effect of contact pressure in the active area on cell performance and gas crossover is evaluated.

4.1 | Contact Pressure Adjustment

To validate the contact pressure adjustment and to evaluate the homogeneity of the applied contact pressure on the active cell area, FujiFilm pressure-sensitive foils were installed in the contact pressure cell. The contact pressure distribution is presented for five different configurations (Figure 5): Featuring a foam flow field and foam electrode at (a) 400 N, (b) 800 N, and (c) 1600 N of the adjustment screw, as well as (d) the milled flow field with foam electrode and (e) expanded mesh electrode for each 800 N. The characteristic areas that are visible in all five contact pressure measurements and result from the cell design are illustrated in Figure 5f. Outside the electrode area, the contact pressure distribution is similar for all five measurements and

independent of the adjustment screw. Contact pressure values in the range of 0.5–10 MPa are measured. The force on the gasket is applied by the eight tie rods. The cell frame is bent under the load of the outer tie rods. This leads to increasing contact pressure in the gasket area from the inside out. The cell frame contains an additional O-ring groove around the diaphragm to improve the sealing performance of the flat gaskets. This groove was empty for the contact pressure distribution measurement, leading to a very low contact pressure in this area. The diaphragm creates a rectangular high contact pressure imprint around the active cell area and the flow channels because it is initially thicker than the gasket. The PTFE gasket acts as a hard stop for diaphragm compression. A continuous increase in contact pressure in the diaphragm area is measured from Figure 5a–e. While the gasket was reused, a fresh UTP500 diaphragm was used in all five measurements. Plastic compression of the gasket might increase the contact pressure on the diaphragm around, but not within, the active area. Under the electrolyte channels, the contact pressure is almost zero.

In the active cell area, the contact pressure is different for the different cell assemblies. The measurements with the foam flow field and foam electrode (Figure 5a–c) differ only in the adjustment screw force (400, 800, and 1600 N), resulting in different contact pressures in the active cell area. For these three configurations, the distribution is relatively homogeneous. For the lowest force, the contact pressure in the active cell area is in the range of 0.2–2 MPa, and for the highest compression between 2 and 6 MPa. The calculated mean contact pressure values of 0.8, 1.6, and 3.2 MPa (adjustment screw force of 400, 800, and 1600 N divided by the 5 cm^2 active cell area) are in

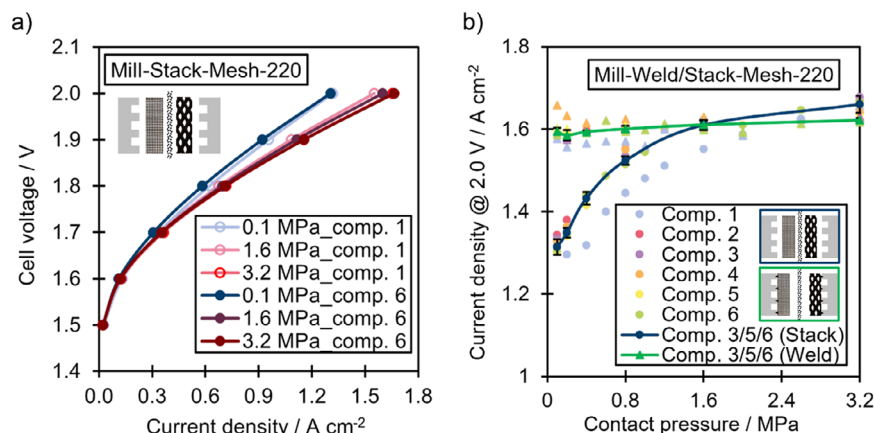


FIGURE 6 | Influence of contact pressure on cell performance: (a) I - V curves for selected contact pressures and compression cycles (UTP220, catalyst-coated mesh electrodes, stacked, milled flow field). (b) Current density at 2.0 V over contact pressure in stacked versus welded configuration (UTP220, catalyst-coated mesh electrodes, milled flow field). Round marks indicate stacked, and triangular marks indicate welded configurations. Operating conditions: 80°C, 30 wt% KOH, 50 mL min⁻¹.

line with the contact pressure observed on the film. For the milled flow field and foam electrode (Figure 5d), the channel-rib-structure of the flow field is visible in the contact pressure distribution. High contact pressures were measured above the ribs. The foam electrode is not mechanically stable enough to transfer contact pressure to the channel area. Combining the milled flow field with the expanded mesh electrode (Figure 5e), the mesh structure leads to a characteristic imprint. The channel-rib-structure of the flow field is not visible because the mesh electrode is mechanically more stable than the foam. Contact pressure peaks up to 7 MPa were measured, which could lead to local damage of the diaphragm.

From this first visualization, it is concluded that the mean contact pressure over the active cell area can be manually adjusted independently of the sealing area. The pressure distribution depends on the cell configuration. Due to the flow field or electrode type, the contact pressure distribution and range can be different and more or less homogeneous. This has to be taken into account when analyzing the contact pressure influence on cell performance and crossover measurements.

4.2 | Influence of Contact Pressure on Cell Performance

We used a contact pressure cell to investigate the influence of contact pressure on cell performance. The cell was assembled with different configurations regarding the flow field, electrode, and diaphragm. The components were either stacked or welded together. Several contact pressure cycles were measured, which is not possible with existing standard test cells.

First, a reference configuration is investigated: Two cells are assembled with the milled flow field, catalyst-coated mesh electrodes and a UTP220 diaphragm, one in the stacked and one in the welded configuration. As described in the experimental section, I - V curves are measured for different contact pressure steps and in six compression cycles. The I - V curves for the stacked configuration are given in Figure 6 for contact pressures of 0.1,

1.6, and 3.2 MPa in the first compression cycle and in the sixth compression cycle. The I - V curves for the welded configuration are shown in Figure S2.

For the stacked configuration, current densities increase with higher contact pressure. The difference is visible as a different slope in the high-current region, dominated by ohmic resistances. Increasing the contact pressure decreases the contact resistance between the flow field and electrode. In the low current region, the I - V curve is independent of compression and contact pressure. Thus, neither the catalyst activity nor the size of the electrochemically active area significantly changed during the experiment.

For the welded configuration, no influence of contact pressure on cell performance is visible in either the low or the high current region (see Figure S2). For high contact pressure, both cell configurations reach a similar current density of approximately 1.6 A cm⁻² at 2 V.

The experimental results are similar to those of Ritz et al. [27], who also used a UTP220 diaphragm with the same catalyst-coated mesh electrodes welded to a nickel foam flow field instead of a milled one. In the first compression cycle, they observed that increasing contact pressure greatly improved cell performance. This was explained by compression of the foam flow field, which removed local gaps and flattened the initially wavy electrode structure caused by the welding spots. After multiple compression cycles, they found a stable contact pressure-independent performance, even with the same current density of approximately 1.6 A cm⁻² at 2 V. The lower influence in the first cycle in our experiment can be explained by the different flow field types, which are incompressible and initially much flatter.

For further analysis, the current density value at 2.0 V is extracted from the I - V curves for all six compression cycles and contact pressure steps (Figure 6b). Almost no change in current density with the contact pressure is visible for the welded configuration, while the increase is strong for the stacked configuration. For both configurations, the first compression cycle shows lower

current densities than later cycles. This behavior was observed for all tested cell configurations. The first compression probably improves the alignment of the electrodes, removes any remaining gaps, and could lead to plastic deformation of the components, resulting in higher current densities from Compression 2. From the third compression cycle, the current density might change with the contact pressure but hardly changes with the number of cycles, and the current density values are stabilized. To distinguish between the first compression and the stabilized behavior after several pressure cycles in further data analysis, the current density over the contact pressure is averaged for compression Cycles 3, 5, and 6. Error bars are included to show the standard deviation between these three cycles. The small error bars indicate stable and mechanically reversible behavior after multiple compressions and no significant aging of the components within the test series. The second compression was omitted in this calculation to ensure sufficient separation between the behavior during the first compression and stable cell behavior after several compressions. Compression 4 is also excluded, as it is the first measurement on the second day (see Section 3). This implies a significantly higher relaxation time regarding mechanical (low contact pressure) and electrochemical stress (low voltage, no I - V curves) before Compression 4 and consequently a different behavior. This deviation is also visible in Figure 6b, especially for the first measurement points of Compression 4.

The effect of the contact pressure is highest at high current density, suggesting that it is mostly due to changes in ohmic resistance. We calculated the ohmic resistance from the I - V curve slope in the ohmic region between 1.9 and 2.0 V (see Figure S3a). In the stacked configuration and averaged over Compressions 3, 5, and 6, the I - V curve slope decreases by $60.9 \text{ m}\Omega \text{ cm}^2$ from 259.3 to $198.3 \text{ m}\Omega \text{ cm}^2$ from 0.1 to 3.2 MPa, respectively. In the welded configuration, the value hardly changes, decreasing by only $3.9 \text{ m}\Omega \text{ cm}^2$ from 204.4 to $200.5 \text{ m}\Omega \text{ cm}^2$. Comparing how resistance changes with contact pressure for the welded and stacked configurations estimates the contact resistance between the electrode and the flow field. Although many factors influence cell resistance, these configurations should only differ regarding these specific contact resistances. Here, this difference in resistance amounts to $57.0 \text{ m}\Omega \text{ cm}^2$.

In addition to the analysis of the ohmic resistance as indicated by the I - V curve slope, the HFR was obtained from the impedance measurements (at 1.5 V, 10 mV amplitude). The HFR demonstrated a marked enhancement, with an average increase of $6.7 \text{ m}\Omega \text{ cm}^2$ from 89.1 to $82.3 \text{ m}\Omega \text{ cm}^2$ in the stacked configuration and an average increase of $2.0 \text{ m}\Omega \text{ cm}^2$ from 84.8 to $82.9 \text{ m}\Omega \text{ cm}^2$ in the welded configuration. These observations were made over the range of 0.1–3.2 MPa and averaged for Cycles 3, 5, and 6 (see Figure S3b). The discrepancy in HFR change between welded and stacked samples was found to be $4.7 \text{ m}\Omega \text{ cm}^2$. A selection of complete impedance spectra is available for reference in the Supporting Information, specifically in Figure S4. The observed HFR is significantly higher than the nominal separator resistance of $50 \text{ m}\Omega \text{ cm}^2$ (at 80°C and 30 wt%), which has previously been attributed to bubble effects apart from contact resistances [4, 55].

The differences between the HFR and the slope of the I - V curves have also been documented in previous studies [35, 55]. One

potential explanation for these discrepancies is the assumption of a constant Tafel slope during the fitting process. However, in practice, the Tafel slope is typically found to be current density dependent. This phenomenon appears to be evident in the current context, as the calculated I - V curve slope diverges markedly from the HFR. As the current density increases, the resistance measured by impedance spectroscopy typically increases due to the presence of gas bubbles in the electrode and diaphragm [55]. In this study, we will compare the changes in the HFR and I - V slopes over contact pressure for welded and stacked configurations for the same components (electrodes and diaphragm). The variation in resistance observed among the two configurations can be attributed to the disparity in contact between the electrode and the flow field. The observed discrepancy in the resistance change (welded vs. stacked) at 1.5 V in the HFR and at 1.9–2 V in the I - V curve indicates a nonohmic contact resistance that is subject to variation with current or voltage. This issue merits further investigation.

Variations in contact resistance depending on cell compression have been observed by some authors [17, 55–58]. Phillips et al. [58] reported that the contact resistance decreased by approximately $3 \text{ m}\Omega \text{ cm}^2$ with increasing contact pressure between a nickel flow field and a foam electrode. In contrast to the novel contact pressure cell, their setup had no well-defined contact pressure on the active area. Despite the unclear contact pressure and the different electrodes, the value agrees well with the observed improvement of approximately $6.7 \text{ m}\Omega \text{ cm}^2$ between 0.1 and 3.2 MPa for two electrodes in the stacked configuration measured by the impedance measurement. However, contact resistance might be insufficient to explain the slightly decreasing HFR with increasing contact pressure for the welded configuration. Elastic deformation of the components might be an explanation, but this requires deeper investigation.

In addition to the analysis of the reference configuration, which features a milled flow field, catalyst-coated mesh electrodes, and a UTP220 diaphragm, the other cell configurations mentioned in Table 1 of the experimental section are evaluated. The results for the relative change in current density at selected voltages with increasing contact pressure on all considered cell configurations are presented in Figure 7. In addition to the current density values directly extracted from the I - V curves at constant voltages, data for an individual reference voltage are extracted by linear interpolation from the I - V curves corresponding to a current density of 1 A cm^{-2} at 0.1 MPa for each configuration.

A subset of the configurations is measured in duplicate at disparate test stations. This approach is employed to ascertain the extent to which the results are resilient to variations stemming from the test configuration (see Figure 7a,b). The different conditions yielded different current densities in the second set of measurements, with offsets ranging from 0.2 to 0.3 A cm^{-2} at 2 V for both stacked and welded configurations. The data from all experiments can be found in Figure S5. Apart from this offset, the overall trend of the curves is similar in both experiments. The current density exhibited an upward trend in parallel with the increase in contact pressure for the stacked configuration. Conversely, the welded configuration displayed no substantial

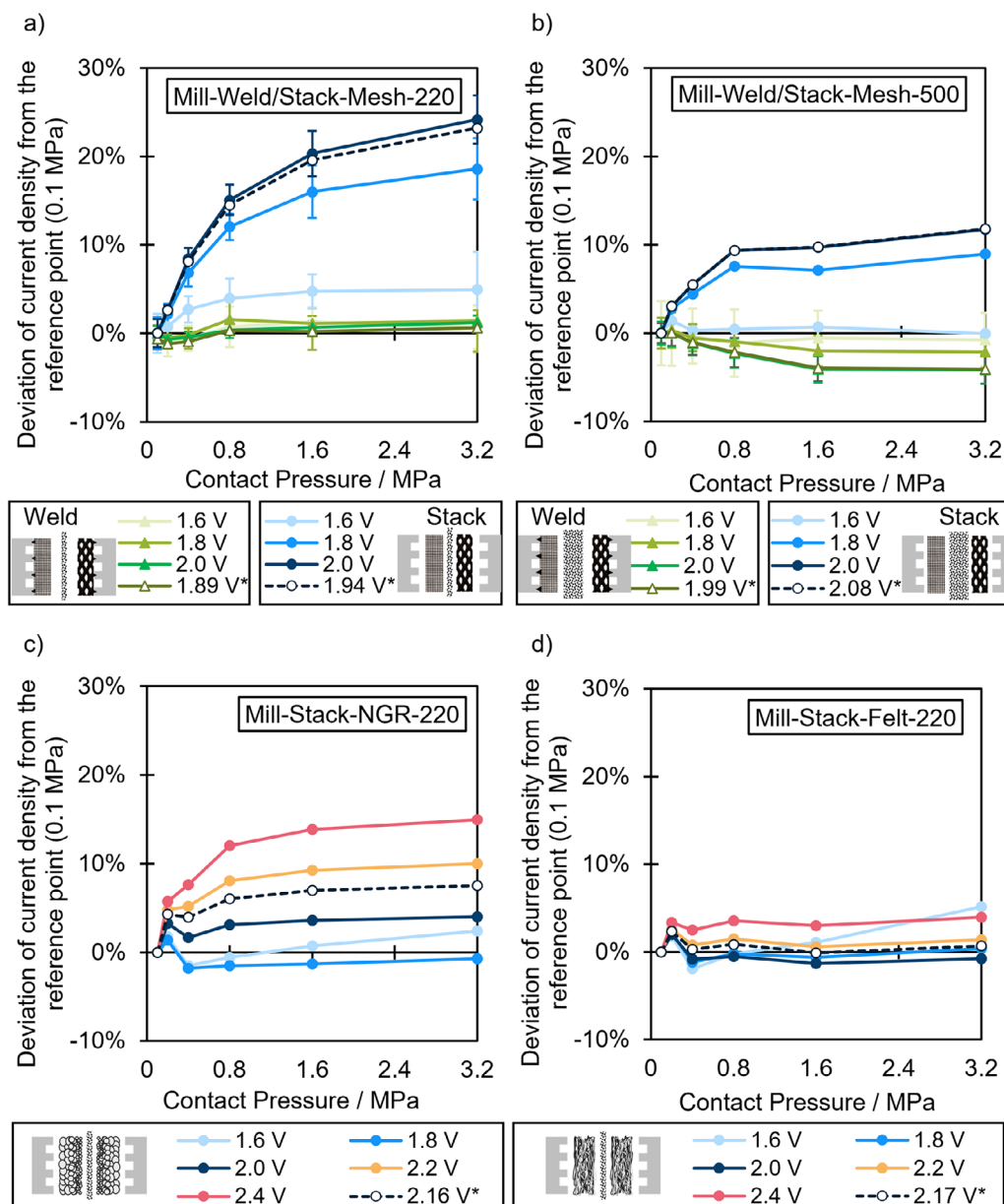


FIGURE 7 | Influence of contact pressure for different cell configurations: Deviation of current density from the reference point at low contact pressure (0.1 MPa) for different voltages and averaged for compression Cycles 3, 5, and 6. The individual voltage corresponding to a current density of 1 A cm^{-2} at 0.1 MPa is marked with *. The operating conditions were 80°C , 30 wt% KOH and 50 mL min^{-1} .

influence from contact pressure. The relative change in current density is compared to the individual reference current density at 0.1 MPa. This allows for the reduction of external effects and the merging of the two measurement sets. Despite the disparity in absolute current density between the two experiments, when evaluated relative to the respective reference point, the cell behavior exhibited notable similarities. The results from the experiments that were repeated are plotted in Figure 7a,b, with their mean value and error bars representing the standard deviation between the two measurements. The narrow error bars, with a standard deviation of approximately $\pm 3\%$, suggest that the relative change in current density with contact pressure is reproducible. The observed discrepancy in current is attributed to variations in conditions across different setups, rather than being a consequence of contact pressure.

For all considered configurations, the relative influence of the contact pressure is greater at higher voltages or currents. Increasing the contact pressure from 0.1 to 3.2 MPa changed the current density depending on the cell configuration in the range of +24% (Mill-Stack-Mesh-220 at 2.0 V) and -4.1% (Mill-Weld-Mesh-500 at 2.0 V). This underlines the importance of controlling, understanding, and optimizing the contact pressure in zero-gap AWE cells depending on the specific cell components. In the following paragraphs, the results for the different cell configurations in Figure 7 are compared, aiming to understand the effect of the individual components.

For both UTP220 and UTP500 with catalyst-coated electrodes in the stacked configuration, the current density increases with increasing contact pressure (Figure 7a,b). The main performance

improvement is from low to moderate contact pressures up to approximately 1.6 MPa, where contact resistances are effectively reduced. The improvement is higher for the thinner UTP220. The higher ionic resistance of the thicker UTP500 in relation to the improvement of the contact resistance with the contact pressure leads to a smaller increase in current density. Interestingly, with UTP220, the current density appears to improve up to the maximum force of 3.2 MPa, whereas with UTP500, no further improvement occurs beyond approximately 0.8 MPa. This indicates an additional effect that counteracts the performance improvement resulting from decreasing contact resistance.

In the welded configuration, the current density is not significantly influenced by increasing the contact pressure for UTP220, whereas the current density decreases for UTP500. When the mesh electrode is pressed to the diaphragm, a clinging and intrusion of the mesh structure into the diaphragm takes place. This behavior is visible in the CT images presented in Figure S6. The electrode clinging to the diaphragm might deactivate or mask parts of the electrode or deteriorate the electrolyte and gas transport there. The deterioration observed with UTP500 (welded electrodes) occurs mainly at higher voltages and currents, which suggests that transport is impaired at the electrode and diaphragm interface. A full deactivation of active sites should affect all current density regions. A deactivation or masking of contact points of the electrode with the diaphragm is also described in the literature comparing zero-gap and small-gap electrolysis cells [30, 33]. Compressing the diaphragm at high contact pressure always results in a smaller interelectrode distance and thus reduced cell resistance. However, porosity and other structural parameters could also change. For UTP500 with welded electrodes, the clinging and intrusion effects seem to dominate as the diaphragm is thicker and softer. For the UTP220 with welded electrodes, the decrease in interelectrode distance seems to balance the change in diaphragm properties and the clinging and intrusion effect.

The stacked and welded configurations with the same diaphragm differ only in the presence of contact resistance. If we compare the configurations—once for UTP220 and once for UTP500—we would expect the difference between the welded and stacked configurations to be similar at the reference voltage (with a constant current of 1 A cm^{-2}). This is approximately the case in Figure 7a,b. At 3.2 MPa, the difference between welded and stacked UTP220 is approximately 22%, and the difference is slightly smaller for UTP500, at approximately 16%.

With the nickel Gradion electrodes (Figure 7c), the performance improves in the low contact pressure region up to 0.8 MPa and is then relatively constant. At a voltage of 2.0 V, the improvement is approximately 4%, which further increases to 10% and 15% at 2.2 and 2.4 V. In addition to reduced contact resistances, the Gradion electrode is compressed plastically and elastically. This brings more active sites closer to the diaphragm. Starting with an initial thickness of 1.66 mm, the electrode is plastically compressed by 46% to approximately 0.9 mm (measured after the experiment). Such a high compression of the porous Gradion foam electrode could generally cause mass transport limitations within the structure. However, this was not evident in the experiment.

For the nickel felt electrodes (Figure 7d), the contact pressure has almost no effect on the cell performance, even up

to the higher voltage of 2.2 V. Only at 2.4 V, there is a small increase of approximately 4% between 0.1 and 0.2 MPa, which is then constant for higher contact pressure values. Interestingly, we see a minor improvement at 1.6 V by 5% at 3.2 MPa. With the nickel felt, the difference between the first and later compression cycles was large, especially in the low contact pressure region in Compression 1 (see Figure S5). Since the felt is relatively stiff and is hardly compressed in the investigated pressure range, initial gaps between the diaphragm, electrode, and flow field might be removed in the first compression cycle, improving the alignment of the electrodes. After the test, the electrode still had approximately the same original thickness of 0.5 mm, and no plastic deformation was observed.

The two uncoated nickel electrodes, both stacked and with UTP220 diaphragms, show distinctly different behavior. This suggests that factors other than the material pairing in the contact area with the flow field influence cell behavior. For the two pure nickel electrodes, the effect of contact pressure on cell performance is generally much lower than for the coated electrodes, even with the same diaphragm (UTP220). In the low current region, due to the poor activity of the uncoated electrodes, the kinetic losses dominate over any deviation in ohmic resistances. However, for higher current densities or even at the reference voltage corresponding to 1 A cm^{-1} , the improvement is 23% with the coated mesh electrodes, but only 7.5% with the nickel Gradion and below 1% with the nickel felt. This suggests that the change in contact resistance depends not only on the material pairing but also on the electrode morphology and the deformation and compression behavior of the various electrodes. In addition to contact resistance, compressible electrodes may alter bulk resistance with contact pressure and mass transport within the electrode. Different types of electrodes have very different contact areas with the diaphragm, which probably affects cell performance in various ways. Due to the three-dimensional electrode structure of Gradion and felt, only a lower share of current flows in the direct contact area to the diaphragm. As the Gradion and felt electrodes are relatively dense and have fine pores, the smooth surface facing the diaphragm results in a more even contact pressure distribution with fewer contact pressure peaks than with the mesh electrodes (see Figure 5). This means a lower intrusion and local compression of the diaphragm, probably reducing the influence of these types of electrodes.

In summary, we observed three main effects of contact pressure on cell performance: change in contact resistance, diaphragm compression, and electrode compression or deformation. The contact resistance appears to be partially nonohmic, and the resistance increases with cell voltage. This was determined by comparing stacked and welded assemblies with the same components. Contact pressure has other effects in addition to contact resistance. This was observed for the UTP500 and mesh electrodes in the welded configuration, where the performance decreased with the contact pressure. It was also observed for the felt electrodes in the stacked configuration. The performance did not improve with the contact pressure there, which revealed a counteracting effect. For diaphragm compression, a homogeneous pressure distribution means less change in diaphragm properties and consequently less change in current density. Local contact

pressure peaks mean higher local compression and a greater influence of contact pressure. If the electrode is compressible, contact pressure brings the active sites closer to the diaphragm. This means a larger influence of contact pressure compared to hardly compressible electrodes. The electrode clinging to the diaphragm probably deactivates or masks parts of the electrode or deteriorates the electrolyte and gas transport there. From the different behavior observed in the cell configurations, it becomes clear that the contact pressure acts upon all the components and their interfaces in the cell. Thus, it influences the contact resistances between the flow field and electrodes, the contact area or intrusion between electrodes and diaphragm, and the elastic deformation of the electrodes and the diaphragm and probably other yet overlooked parameters. How strong the effects are depends strongly on the cell configuration and can result in an overall improvement or deterioration of the cell performance with increased contact pressure. The new cell design enables future work on understanding and optimizing the contact pressure and its different effects.

Regarding the tested cell configurations in this work, with stacked electrodes, a high contact pressure is needed for the highest current density. For welded electrodes, a low contact pressure is sufficient, reducing the risk of mechanical damage to the diaphragm. While contact resistances between components are often present in lab cells, the components in commercial electrolyzers are often welded together. Using different test cells and different components, the influence of varying contact pressure should be considered to improve the reproducibility of results. Welding the electrodes is probably even more important in long-term operation. Growing oxide layers on the anode could lead to increasing contact resistance over time and a higher degradation rate of the stacked configuration compared to the welded configuration. Preload relaxation, viscoelastic creep of the diaphragm and gaskets, or temperature cycles will probably reduce the contact pressure over time. However, the long-term behavior of the different configurations is not within the scope of this work.

4.3 | Influence of Contact Pressure on Gas Crossover

In addition to the change in current density with contact pressure, the influence on product gas quality and the gas separation capability of the diaphragm is investigated. This is done with gas purity measurements in the contact pressure cell, bubble point measurements, and CT imaging.

4.3.1 | Gas Purity Measurements

The influence of contact pressure on gas crossover was investigated for the UTP500 diaphragm with nickel foam electrodes and catalyst-coated mesh electrodes in a stacked configuration. Unfortunately, it was not yet possible to measure the crossover with the UTP220 in the contact pressure cell. Either before or within the measurement series, the crossover exceeded 2% of H_2 in O_2 , which is a safety limit for the measurement (50% of the lower explosion limit [9, 10]). Despite the specified cell assembly procedure, it is not always guaranteed that the diaphragm will

remain in the center of the cell and will not be bent or even sheared at the edge of the active cell area for all compression conditions. Thus, the thin UTP220 can be pierced, especially during high compression, leading to failure. This indicates that cell compression is even more critical with UTP220 diaphragms than with UTP500 diaphragms but could not be studied here due to safety limitations.

For the nickel foam electrode and UTP500, the results from the gas purity measurements are summarized in Figure 8 for (a) 0.05 A cm^{-2} , (b) 0.1 A cm^{-2} , and (c) 0.2 A cm^{-2} . The HTO value decreases with increasing current density, which has also been reported in the literature [7, 36, 41, 42, 59]. Increasing current means a higher production rate, diluting the presumably constant crossover flux due to diffusive and convective transport. In the first compression of the cell, the HTO increases with increasing contact pressure. After releasing that contact pressure to start the second compression cycle, the crossover is much higher than in the first compression. Increasing the contact pressure again leads to an even higher HTO. In the third compression, the HTO stays constant at this high level.

In a different test setup (mixed electrolyte cycles) and test cell, Lee et al. [5] measured slightly higher HTO values for UTP500 and foam electrodes (2.5% H_2 in O_2 for 0.05 A cm^{-2} , 1.2% for 0.1 A cm^{-2} , and 0.5% for 0.2 A cm^{-2}). The electrolyte mixing, in contrast to our setup featuring separated tanks and a balancing line, could explain the difference in HTO.

The results of the gas purity measurements with the UTP500 and catalyst-coated mesh electrodes are shown in Figure 8d–f. Again, the crossover decreases with the current density, increases with the contact pressure and number of compression cycles, and stays at a high level in the third compression. Ritz et al. [27] also observed the effect of increasing crossover with increasing contact pressure for the same electrodes but with a UTP220 diaphragm and a nickel foam flow field.

With the coated mesh electrodes, the crossover is lower than for the foam electrodes, although the same type of diaphragm was used. This is probably due to the different electrode structures, which result in different contact pressure distributions, different mass transport characteristics, and thus a different gas oversaturation profile. Simulations have shown that concentrations and current density can differ substantially depending on the local environment, for example, between the rib and channel of the flow field [60]. In addition, gas bubble entrapment is a known issue for foam electrodes depending on their pore size [61]. Trapped gas bubbles could influence crossover, especially if the diaphragm is damaged due to high contact pressure. The structure of the mesh electrode is much coarser than that of the fine foam electrode and leads to a different contact area with the diaphragm and contact pressure distribution. The mesh electrodes lead to an inhomogeneous pressure distribution, including peaks up to 7 MPa at the medium average pressure of 1.6 MPa (see Figure 5e). In contrast, for the foam electrode, the distribution is more homogeneous, with a maximum contact pressure of approximately 3.5 MPa at an average pressure of 1.6 MPa (see Figure 5b). A more homogeneous distribution means that a greater area of the diaphragm is affected by contact pressure. To determine which load type has a greater

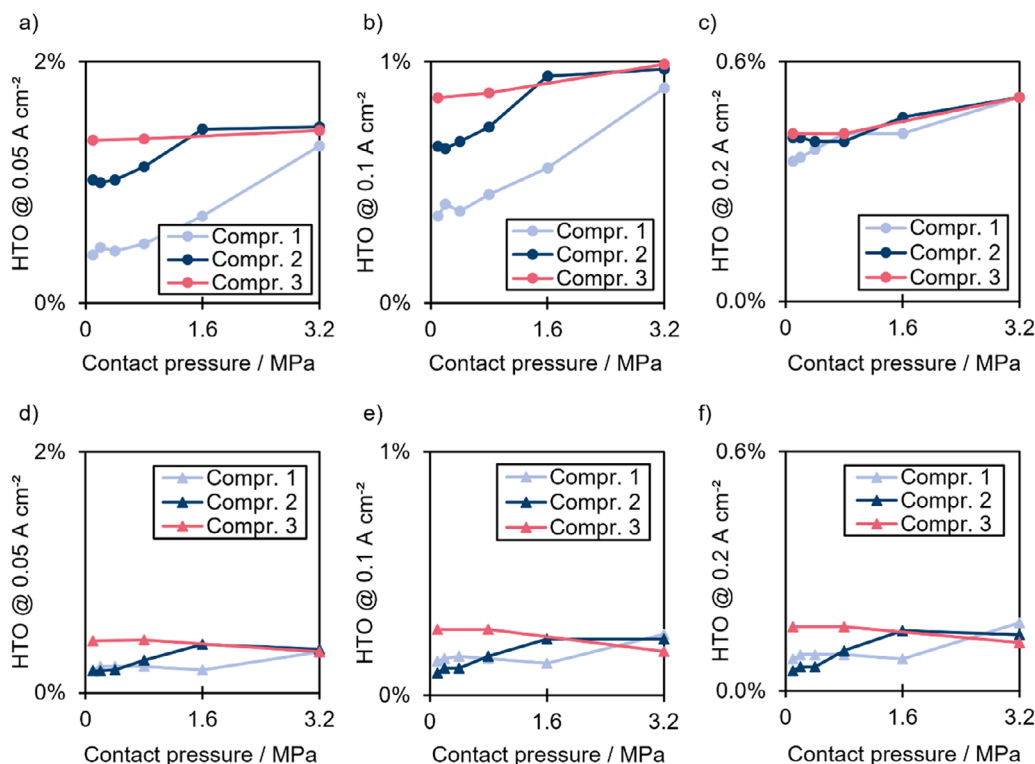


FIGURE 8 | Change in gas crossover due to mechanical contact pressure and compression cycles: HTO for the Ni foam electrode at (a) 0.05 A cm⁻², (b) 0.1 A cm⁻², and (c) 0.2 A cm⁻². HTO for the catalyst-coated expanded mesh electrode at (d) 0.05 A cm⁻², (e) 0.1 A cm⁻², and (f) 0.2 A cm⁻². Measurement conditions: 80°C, 30 wt% KOH, 50 mL min⁻¹.

influence on the crossover, we calculate the relative increase in crossover with contact pressure using the results from Figure 8. The HTO at 0.05 A cm⁻² increases in the first compression from 0.1 to 3.2 MPa by a factor of 1.8 for the mesh electrodes and 3.3 for the foam electrodes. At 0.2 A cm⁻², the increase is slightly larger for the mesh (Factor 2.1) and smaller for the foam (Factor 1.5). As the relative increase is similar for the two different electrodes, both high local compression and low compression across the entire area appear to affect the crossover.

The increasing crossover at higher contact pressure is in accordance with the study by Hartert et al. [26], where higher electrode compression promoted the crossover. It is also in line with the observations from Barros et al. [42], who measured a much higher crossover in zero-gap cells compared to a small-gap configuration. de Groot et al. [35] state that in industrial electrolyzers, manufacturing is not accurate enough to ensure a true zero-gap assembly over the whole area. In addition, in a small test cell and especially with porous electrodes, a low overall contact pressure could cause a combination of some true zero-gap areas with high contact pressure and more small-gap areas. Increasing the overall contact pressure increases the share of zero-gap areas as well as the local contact pressure. Interestingly, a dependence of the crossover on the contact pressure is also observed in PEM electrolysis cells, which do not have a gap [40, 41]. Thus, the multiple factors leading to the observed trend will need further investigation.

While the previous findings explain the increase in crossover with increasing contact pressure, they do not explain the global

increase between the first, second, and third compressions. We suspect that the electrode is clinging to the diaphragm in the first compression. The diaphragm is plastically deformed and potentially damaged due to this first high contact pressure. Releasing the contact pressure does not lead to full recovery of the diaphragm due to permanent damage. The resulting changes in the diaphragm are analyzed using CT scans and bubble point measurements.

4.3.2 | CT Scan of the Diaphragm

Figure S6 shows CT images of the diaphragms in different electrode-diaphragm configurations and after compression in the contact pressure cell (6 × 3.2 MPa and cell performance measurement). As the CT images were recorded postmortem, only the plastic deformation of the diaphragm was visible. Unfortunately, the resolution of the CT scan is not sufficient to reveal the internal pore structure, which potentially changes with the contact pressure.

The structure of the mesh anode and cathode electrode is stamped into the diaphragm. For the foam electrodes, the fine structure of the foam is visible in the 3D image. The plastic deformation of the diaphragm and the electrode intrusion reduce the effective thickness of the diaphragm and explain the increasing crossover. For the foam electrode, only the diaphragm surface seems to be affected by contact pressure. The intrusion is much lower than for the mesh electrode. At the same time, the surface is stressed in many spots, while for the mesh electrode, large parts of the surface remain unaffected.

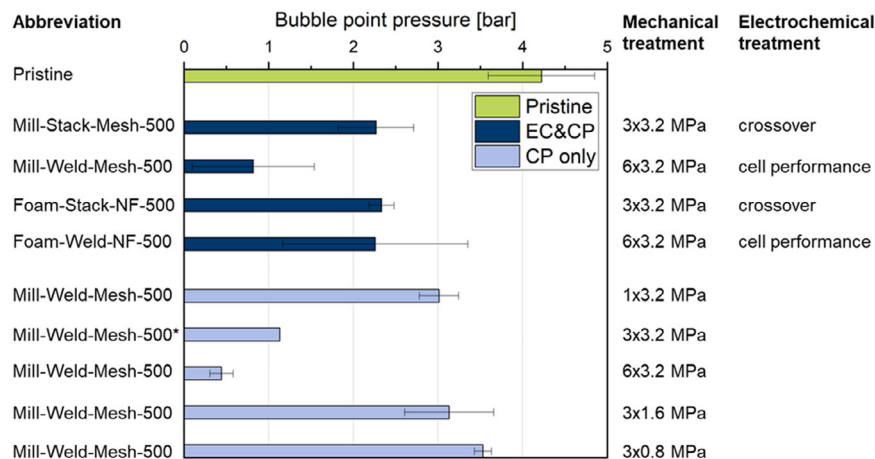


FIGURE 9 | Bubble point pressure of UTP500 diaphragm samples in different configurations. *Only the first measurement is shown because the first bubble point pressure dramatically decreased due to severe material washout and pore widening in the subsequent measurements.

4.3.3 | Bubble Point Measurement

To support the results from crossover experiments, the first bubble point pressure is measured, giving an indication of the gas tightness of the diaphragm. As a *pristine* sample, three unused pieces of UTP500 were measured three times each (green bar in Figure 9). The mean bubble point pressure of UTP500 was 4.2 bar, with a standard deviation of 0.6 bar. According to Washburn's formula (Equation S1), with water at room temperature as the wetting liquid (surface tension of 72.75 mN m^{-1} [62]), a contact angle of Zirfon and water of 75° [5], and a maximum pore diameter of 184 nm for UTP500 [36], the calculated first bubble point is 4.1 bar, close to the experimental value. The bubble point values of pristine samples will now be compared with those of mechanically and electrochemically stressed samples. The tested samples are given in Table S2, and the results are presented in Figure 9.

The *EC&CP* samples (dark blue bar in Figure 9) are from the crossover and performance experiments, in which they were subjected to mechanical and electrochemical stress. The diaphragms of the crossover experiments, which experienced three compression cycles up to 3.2 MPa with a long holding time (9 h) at the maximum contact pressure, indeed have a reduced first bubble point pressure of 2.3 bar. These results align with the increasing crossover with varying contact pressure. This indicates that the high contact pressure or the number of pressure cycles harms the diaphragm. The diaphragms from the performance experiments experienced twice the number of contact pressure cycles for a shorter time (i.e., a total of 20 min at maximum contact pressure). The respective samples with mesh electrodes show an even lower first bubble point pressure of less than 1 bar. Therefore, an increased number of pressure cycles causes more damage to the diaphragm. The larger standard deviations of the cell performance samples are due to sample-to-sample variations. These variations could be caused by material differences or slight differences in plane parallelism and orientation of the anode and cathode stamp to each other, resulting in more or less heavily stressed areas, as shown in Figure 5e. For foam electrodes, on the other hand, the trend of a decrease in the first bubble point with the number of pressure cycles is barely visible. The

foam electrodes apply a more homogeneous contact pressure, as shown in Figure 5b,e. This results in a lower maximum local contact pressure and causes less deformation of the diaphragm, correlating with a higher first bubble point.

Example pressure curves, which are given in Figure S7, also show that the gas throughput in the first bubble point test depends on two factors. First, the first bubble point itself, which is the starting point when the first pore opens. Second, the amount of gas depends on the number of open pores. In the pressure curves, this is visible as a greater pressure slope decline after the first bubble point is reached. There are cases where the slope change is higher for homogeneously compressed foam samples compared to locally compressed mesh samples. Regarding the higher measured HTO with the foam electrodes, this can be another possible explanation, in addition to an increased supersaturation on the diaphragm surface with the denser foam electrode. In addition, the crossover mesh sample is surprisingly intact for a mesh electrode sample at 3.2 MPa.

To evaluate whether the diaphragm is damaged more with increasing maximum contact pressure or more compression cycles, additional samples (*CP only*), without any previous electrochemical treatment, were tested. Figure 9 shows a decreasing first bubble point pressure with increasing contact pressure (light blue bars), so elevated contact pressure mechanically damages the diaphragms (compare $3 \times 0.8 \text{ MPa}$, $3 \times 1.6 \text{ MPa}$, and $3 \times 3.2 \text{ MPa}$). In addition, as assumed above, the first bubble point decreases with an increasing number of contact pressure cycles (compare $1 \times 3.2 \text{ MPa}$, $3 \times 3.2 \text{ MPa}$, and $6 \times 3.2 \text{ MPa}$). This underlines that the reduction of the bubble point is probably a result of the contact pressure itself, rather than being caused by other factors relating to electrochemical operation.

Regarding UTP220, the trend is similar. The mean first bubble point values and standard deviations are given in Figure S8 (tested samples are summarized in Table S3). Here, most samples are measured only once because gas crossover measurements were focused on UTP500. The mean first bubble point pressure of the *pristine* UTP220 diaphragms was 1.9 bar, with a standard deviation of 0.8 bar. All diaphragms from different contact

pressure experiments showed dramatically decreased mean first bubble point values below 0.3 bar. The *CP only* samples tend to show a slightly lower first bubble point than the *EC&CP* samples, even though they were not stressed electrochemically or by elevated temperatures. This might be related to the shorter regeneration time after each pressure increase step. However, this would require further investigation. Nevertheless, the markedly reduced first bubble point indicates that the thinner UTP220 is more sensitive to contact pressure than the thicker UTP500. The damage caused by high contact pressure or by stress on the edge of the active area when only one electrode stamp is moved is probably the reason why crossover measurements were unsuccessful with the thinner UTP220. The required stop of crossover measurements with UTP220 for safety reasons can be explained by greater damage by compression due to the reduced thickness, visible in nano-CT images, as well as a dramatically reduced first bubble point pressure for all compressed samples. This underlines that exact control of the contact pressure is essential when testing thin diaphragms.

In summary, an increasing contact pressure as well as an increasing number of contact pressure cycles deteriorate the diaphragm, resulting in a reduced first bubble point pressure and increased crossover. The significant increase in crossover with rising contact pressure highlights the importance of controlling and optimizing contact pressure in electrolytic cells and stacks. This applies to all operating conditions of the electrolyzer, including the assembly process. Elastic elements in the cell can help maintain a low, constant contact pressure across all operating conditions and over time. Whether high contact pressure during long-term operation also affects the aging and degradation of the diaphragm is beyond the scope of this work.

5 | Conclusion

A contact pressure cell was developed to investigate the influence of mechanical contact pressure on cell performance and gas crossover in zero-gap AWEs. In this new cell design, the contact pressure in the active cell area can be independently adjusted during operation by an adjustment screw and can be measured by a force sensor. Visualizing the contact pressure distribution for different contact pressures and cell configurations confirms the contact pressure adjustment. Using a porous foam flow field and a foam electrode improves the contact pressure distribution, while a channel-rib-flow field or the shape of a mesh electrode is visible in the pressure distribution.

It was found that cell performance and gas crossover are influenced by the mechanical contact pressure. The influence of contact pressure on cell performance is strongly affected by the cell configuration, such as the flow field, diaphragm, and electrode type, as well as the welding or stacking of the electrode on the flow field. A high impact of contact pressure on cell performance was found for the stacked configuration under high current. The contact resistances between the electrode and flow field decrease with increasing contact pressure and improve the current density. For the components considered here, increasing the contact pressure improved the current density by up to 24% compared to a low contact pressure case. Welding the electrode to the flow field mitigates this effect and leads

to higher current densities, even at low contact pressures, or the current density actually decreases by up to 4% at high contact pressure. For high contact pressures, the welded and unwelded (stacked) configurations deliver similar performance. High contact pressure is observed to result in clinging and intrusion of the electrode into the diaphragm. This affects cell performance by blocking active sites. In addition, increasing contact pressure was found to increase gas crossover by damaging the diaphragm, which was also confirmed by measuring the first bubble point pressure of the diaphragm. Welding the electrodes to the flow field reduces the influence of contact pressure, and a low contact pressure is ideal for both high current density and low crossover. In stacked configurations, the requirements for performance (high contact pressure for low contact resistance) and crossover (low contact pressure to prevent diaphragm damage) are conflicting.

This study shows that mechanical contact pressure is an important parameter and must be taken into account in cell and stack development and testing. Depending on the cell configuration, electrode type, and diaphragm, the influence of contact pressure can vary drastically. Optimizing and controlling this parameter will improve the current density, gas crossover, and the reproducibility of experiments. Scale-up may alter the contact pressure distribution. Applying high or precisely defined contact pressure unchanged over time and over a large area means considerable demands on the mechanics of the electrolyzer. The new contact pressure cell developed in this work enables us to systematically investigate the effects related to contact pressure.

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Data Availability Statement

The research data, including experimental raw data and the CAD files of the contact pressure cell, are openly available at the Forschungszentrum Jülich data repository [63].

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

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