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## Fuel cell research with neutron imaging at Helmholtz Centre Berlin

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### Abstract

This paper demonstrates the capabilities of the new instrument CONRAD II at Helmholtz Centre Berlin for the investigation of fuel cells. The performance gain of CONRAD II with respect to its predecessor instrument, CONRAD I, is demonstrated and different examples for in-operando measurements of polymer electrolyte membrane fuel cells are given. Furthermore, an application example for the high resolution detection system recently developed by the group is demonstrated which includes a three-dimensional measurement of the water distribution in a small fuel cell with a width of about 14 mm by means of neutron tomography.

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### 1. Introduction

In-operando investigation of the water distribution in polymer electrolyte membrane fuel cells (PEFC or PEMFC) is one of the most important application fields of neutron imaging [1-3]. Liquid water plays a crucial role in PEMFCs [4]. The membrane is only proton conductive above a certain level of humidity [5-7]. Therefore,

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dehydration of the membrane must be prevented. However, liquid water accumulations in the porous gas diffusion layers and in the flow field channels of a fuel cell strongly affect the gas flow and, therefore, the gas supply of the catalysts [8]. Excessive accumulations of liquid water have to be prevented, i.e. a quick water removal is desirable. For these reasons, fuel cell research relies on techniques that allow for non-invasive visualization of the liquid water distribution in an operation fuel cell at high spatial and temporal resolution.

Neutrons can easily penetrate metals like aluminum and steel while they are strongly scattered by hydrogen [1, 3, 9, 10]. This allows for investigation of water in fuel cells without the necessity to change the fuel cell setup and, therefore, without altering the properties of investigated fuel cells.

Since the first neutron imaging measurements were done in 1999 by Bellows et al. this research field has quickly expanded [11]. Later also X-ray imaging was introduced into this field [12-16]. Today in-operando imaging of fuel cells became an important research field being followed by an increasing community of fuel cell researchers [17-24].

One of the limiting factors is the availability of beam time at neutron imaging stations. Only a few neutron imaging instruments in the world are optimized for the investigation of fuel cells. Therefore, most of this research is focused at a few facilities. The instrument CONRAD/V7 at the Helmholtz Centre Berlin for Materials and Energy (HZB) is among the most important ones on this field.

With the installation of the new CONRAD II several improvements were implemented. Most important are the much larger beam size and increased flux, both being of special importance for the research on fuel cells. Here, we give a brief overview on recent activities on this field at CONRAD II.

## 2. Experimental setup

### 2.1. Neutron Imaging Facility

The measurements were performed at the CONRAD II or its predecessor instrument, CONRAD I, at the BER II research reactor (Helmholtz Centre Berlin for Materials and Energy, HZB, Berlin, Germany) [25]. The instrument is located at the end of a curved neutron guide. The flux at the end of the guide (after the cold source upgrade) is about  $2.7 \times 10^9$  neutrons per  $\text{cm}^2$  and sec. The spectrum consists of cold neutrons with a wavelength larger than about  $1.8 \text{ \AA}$ . Due to the curved neutron guide neutrons with higher energies as well as high energy photons are mostly eliminated. The high flux and the pure cold neutron spectrum provide high image qualities.

For the here presented measurements two different detection systems were used: a high resolution setup for small fuel cells that is described below and an adaptable optical system for larger field of views of up to  $200 \times 200 \text{ mm}^2$ . Both setups are described in literature [26, 27]. For measurements with the large field of view setup a  $100 \text{ }\mu\text{m}$  thick  $6\text{LiF}$  scintillator was used as detector screen converting incident neutrons into visible light.

Further experimental details like exposure times or  $L/D$  ratios are given in the text.

### 2.2. Fuel Cell Setup

The investigated fuel cells were developed at the Centre for Solar Energy and Hydrogen Research (ZSW Ulm, Baden-Württemberg, Germany). The dimensions of the large fuel cells were about  $140 \times 140 \text{ mm}^2$  with an active area of  $100 \times 100 \text{ mm}^2$  (see figure 1). The cells were equipped with carbon fiber-based gas diffusion layers [28] and different types of flow fields (for details see text below). A humidification system was used for the cathodic gas stream [29].



Fig. 1. The investigated PEM fuel cell of the ZSW Ulm. The size was about 140x140 mm.

For the tomographic studies using the high resolution neutron imaging set up a small fuel cell was designed by the Centre for Solar Energy and Hydrogen Research (ZSW Ulm) [30-32]. It provides an active area of about  $9 \times 60 \text{ mm}^2$ . The fuel cell setup is shown in figure 2. The middle part of the cell is made of aluminum that allows high transmission of neutrons. It has a round-shaped cross section with a diameter of 14 mm that provides good mechanical stability. It fits into the field of view of the high resolution imaging setup allowing for tomographic investigations of the cell. For further details on the used operating parameters we refer to the literature (see above).



Fig. 2 The investigated PEM fuel cell optimized for high resolution tomography in front of the high resolution detection system. The round-shaped scintillator can be seen in the center of the image.

### 2.3. High Resolution Neutron Imaging Setup

A dedicated imaging setup was used for measurements at high spatial resolutions. It consists of an optical magnifying lens system and an optimized Gadox scintillator screen. Pixel sizes of  $3.2 \text{ }\mu\text{m}$ ,  $6.8 \text{ }\mu\text{m}$  and  $13.5 \text{ }\mu\text{m}$  are possible. However the maximum field of view is limited by the size and shape of the scintillator screen. It is typically about  $12 \times 12 \text{ mm}^2$  or a little larger when the whole area of the round-shaped scintillator screen is used. The front part of the detection system and the scintillator screen can be seen in figure 2. The details of this setup are described by Williams et al. [26].

## 3. Results

### 3.1. Radiography of large fuel cells

One of the major drawbacks of the old CONRAD I instrument was the limited field of view and the inhomogenous spatial flux distribution due to the limited neutron flight path of the instrument of about 5 m (i.e. the distance between pinhole and detector). Figure 3(a) shows a flat field image and an image of a large fuel cell (figure 3 (b)) measured at the CONRAD I instrument before the upgrade program was started. The cell hardly fit into the beam. Even when count rates in the center of the image were already sufficient the exposure time had to be

further increased by about an additional order of magnitude just to ensure sufficient count rates at all locations within the active area of the cell. An approach to overcome these limitations was the use of a focusing neutron guide as described by Tötze et al. [33, 34]. However, the focusing device also decreased the neutron flux and increased the beam divergence and, therefore, the pixel blurring, i.e. the spatial resolution was decreased.

The instrument upgrade of CONRAD could eliminate this drawback by elongating the neutron flight length to 10 m and by installing of a novel neutron guide with larger beam divergence. As the result a significant gain in beam size and a much more homogenous flux distribution have been achieved at CONRAD II. When investigating fuel cells of aforementioned size it allows a uniform illumination of the whole cell area. Figure 4 (a) shows a raw image taken from such a fuel cell together with the flat field corrected image in figure 4 (b).

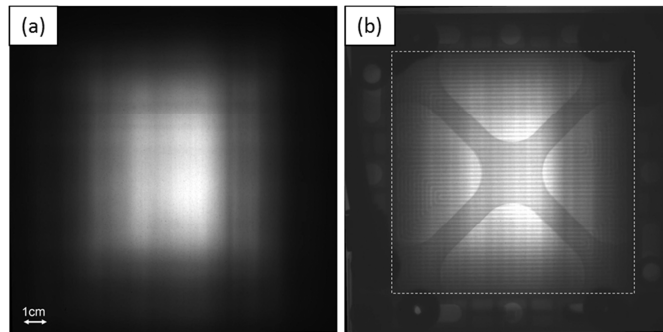


Fig. 3. (a) Flat field image (b) Neutron radiograph of a PEM fuel cell of the type shown in figure 1. The measurements were taken before the upgrade.

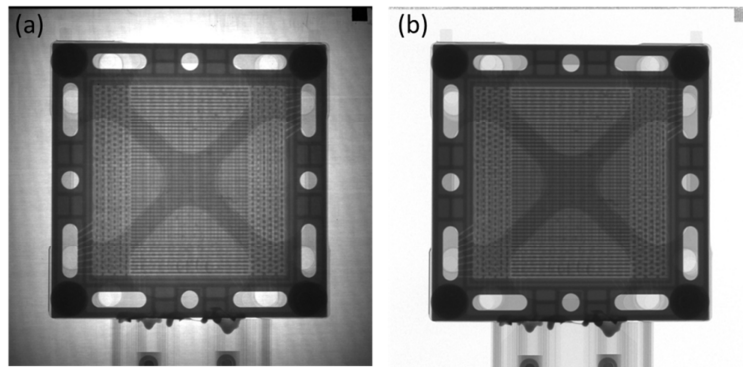


Fig. 4. Neutron radiographs of a fuel cell of the same size as the one shown in figure 3 taken after the upgrade of the CONRAD/V7 facility. (a) image without dark and flat field correction. (b) after dark and flat field correction.

The instrument is frequently used to study the water distribution in operating PEMFCs. A typical measurement of such a cell is shown in figure 5. It was operated at a current density of  $1 \text{ A/cm}^2$ . Overall measurement time for each radiographic projection image was about 16 s (12 s exposure time and 4 s read-out time). A 3 cm pinhole was used resulting in a  $L/D$  ratio of about 330. The pixel size was about  $100 \text{ }\mu\text{m}$ .

The images display changes in the liquid water distribution (white) with respect to the dry reference state of the fuel cell during a time period of about 1 min. Most water agglomerations present in the flow field channels at the upper and middle third of the cell area remain fairly constant in both size and position. However, significant water accumulation is observed within channels at the bottom third (marked by red arrows in figure 5).

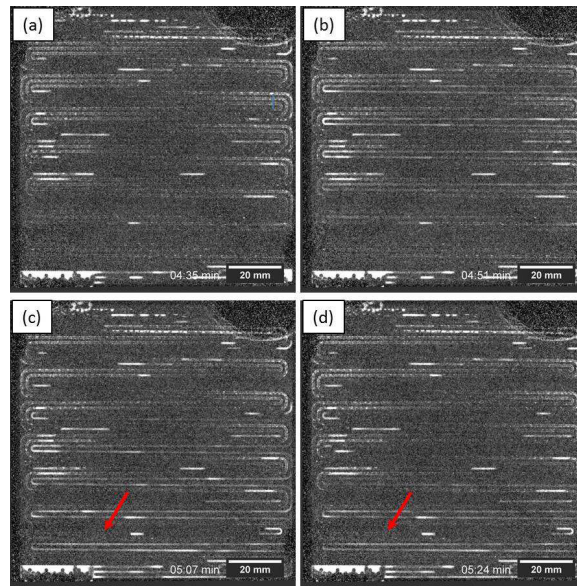


Fig. 5. Time series of neutron radiographs revealing the in-operando water distribution of a PEM fuel cell with an active area of  $100 \times 100 \text{ mm}^2$ . Acquisition time per image was about 16 s.

In-operando water distributions strongly depend on the geometry of the employed flow field. For this reason, neutron imaging assisted optimization of flow field designs and geometries has become an important subject in fuel cell research. In the example shown in figure 6 three different flow-field designs are compared: a pattern structure flow field (figure 6 (a)), a meander-shaped flow field (figure 6 (b)) and a straight channel flow field (figure 6 (c)). Water distribution and water transport dynamics are completely different. This has strong effects on the overall operating properties of the fuel cells. The meander-shaped flow field was identified to have the best overall performance, which might be partially attributed to a more uniform water distribution, resulting from improved water removal capability of such a flow field. This is attributed to the fulfilment of certain pressure drop criteria. Details of this study were recently published by Klages et al. [35].

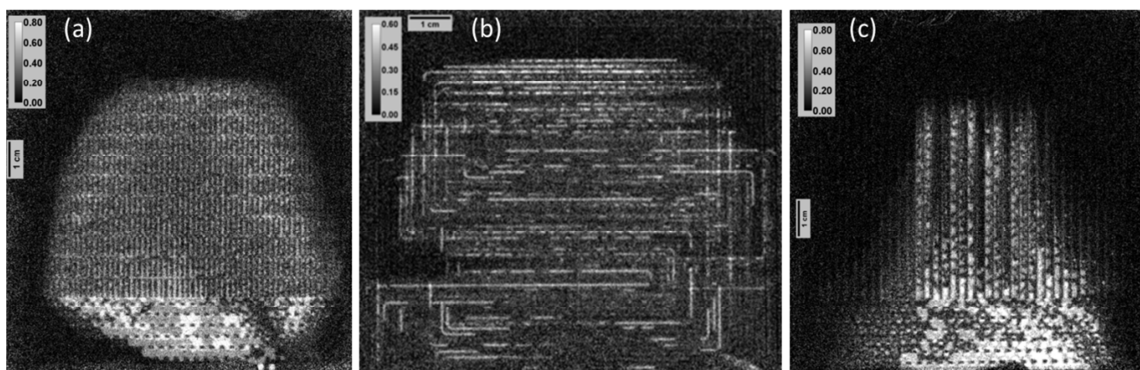


Fig. 6. Comparison of three different flow field geometries. The water distribution appears white. The measurements were performed at the new CONRAD II instrument [35].



### 3.2. High resolution radiography and tomography

The next examples demonstrate the advantages of high resolution radiography and tomography for the investigation of water distributions. A dedicated fuel cell with rectangular cross section (of about  $14 \times 5 \text{ mm}^2$ ) was designed to meet the requirements of the CONRAD II high resolution setup (for details see above).

The first example shows a so-called “in-plane” radiographic measurement of this cell (figure 7)[36]. The measurement mode allows for analyzing anode and cathode water amounts separately. The first image (figure 7 (a)) shows a radiograph of the dry cell before cell operation. The image in figure 7 (b) was taken some minutes after starting operation. Some dark spots can be found at the location marked by arrows in the flow field channels of the cathode, which is located on the right side in the image. This is water that starts to accumulate, as expected, on the cathode side first. The water accumulation can be quantitatively derived from the logarithmized quotient of the images shown in figures 7 (b) and (a) eventually divided by the attenuation coefficient of water. The resulting image (shown in figure 7 (c)) yields the 2D liquid water distribution in terms of local thickness of water transmitted by the incident neutron beam.

A similar fuel cell setup was subject of tomographic investigation. During tomography, the cell was rotated in 800 equidistant steps over an angular range of  $360^\circ$ . At each angle position 3 radiographic projection images were taken and, subsequently, processed to a single median filtered image in order to eliminate white spots. The acquisition time of a single radiograph comprised 6 s exposure and 4 s read-out time resulting in a measurement time of about 30 s per projection angle and a total acquisition time of about 8 h for the entire tomographic scan. A 2 cm pinhole at a distance of 10 m from the measure position was used ( $L/D = 500$ ). The achieved spatial resolution was restricted by the beam blurring caused by the limited  $L/D$  ratio and the sample-scintillator distance (about 2 cm) rather than by the capabilities of the detection system. We calculated a beam blurring of about  $40 \mu\text{m}$  determining the spatial resolution to about this value.

A cross sectional view of the cell extracted from the reconstructed tomogram is displayed in figure 8. In this slice no water was found in the anode and cathode channel, respectively (see white arrows). However, some residual water accumulations were found in the surrounding cooling flow fields on both sides (see arrows in figure 8). These water droplets have diameters of around  $50\text{-}100 \mu\text{m}$ . Smaller droplets appear larger due to the blurring caused by the limited spatial resolution.

This example demonstrates that the  $L/D$  ratio [3] needs to be increased in order to exploit the maximum spatial resolution capability of the detector system (which is around  $10\text{-}15 \mu\text{m}$ ). However, this increases the required measure time to about one day per tomogram.

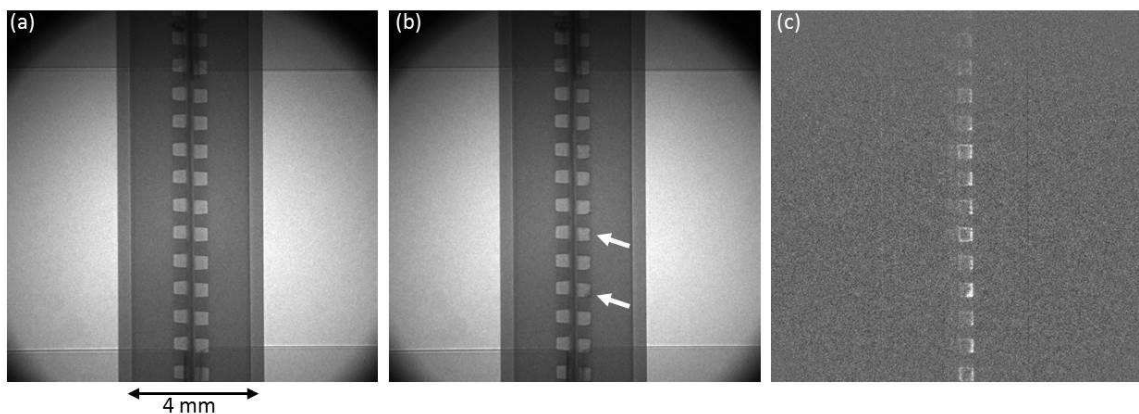


Fig. 7. (a) Neutron radiograph of a small PEMFC in dry condition. (b) The same cell during operation. (c) Normalized image (image (b) divide through image (a)) showing the water distribution.

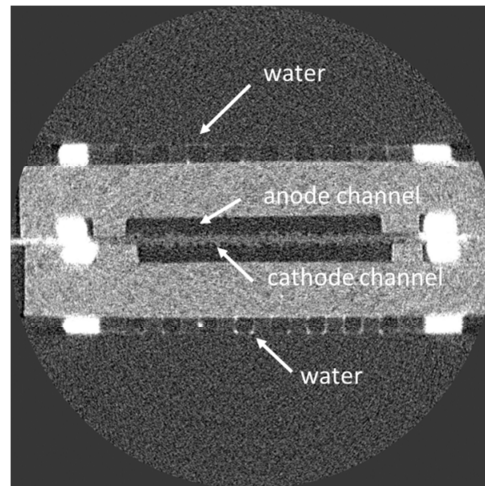


Fig. 8. Tomographic cross section through a specially designed fuel cell. Cross sectional size of the cell is about 14 mm x 5 mm.

#### 4. Conclusions and Outlook

In conclusion, the new CONRAD II instrument provides much better conditions for fuel cell researchers compared to its predecessor, CONRAD I. The increased beam size and higher flux allow for larger fuel cell samples, faster radiographic measurements and also better tomographic measurements in future [37-39]. The novel high resolution neutron imaging setup promises a much more detailed mapping of the water distribution in fuel cells. However, exploitation of the maximum possible spatial resolution requires high  $L/D$  ratios and, therefore, long exposure times.

In future the capability of the facility will be extended by an imaging setup providing a field of view of  $300 \times 300 \text{ mm}^2$ . This is of particular interest for the research on very large fuel cells and on fuel cell stacks. Furthermore, other fuel cell types like high temperature PEMFCs may be investigated in future [40-42].

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