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Specific Procedures of TEM Sample Preparation for Fuel Cell Electrodes and Components. Ulrike Kunz[a], Frieder Scheiba[a], Nathalie Benker[a], Hartmut Fuess[a], Christina Roth[a]. [a]*Institute for Materials Science, Technische Universität Darmstadt, Germany.*
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Fuel cells are efficient energy converters, which may play an important role in the world's future energy infrastructure. The main bottleneck in the commercialization of the most promising low-temperature fuel cell (PEMFC), however, is its high cost and poor durability. The noble metal platinum typically used as catalyst in both fuel cell electrodes makes the PEMFC technology highly expensive. Tremendous research efforts therefore deal with the degree of platinum utilization, since less than 50% of the platinum content of the electrodes contributes to the reactions, so far.
Transmission electron microscopy (TEM) is a promising tool to study the electrode porosity and the distribution of the polymer electrolyte, which are key parameters for the fuel cell performance and long-term stability. However, the sample preparation is crucial to the success of the experiment, as will be reported in this work. The membrane-electrode assembly (MEA) is embedded in Araldit 502© and cut into ultrathin sections by an ultramicrotome using a diamond knife. Details of the procedure can be found in [1] and will be described with particular emphasis on the preparation parameters used.
In addition, the adaptation of a technique using Wood's alloy will be reported, which can be applied to image both, the membrane-electrode assembly (MEA) and the gas diffusion layer (GDL) as complete composite structure in the future (Fig. 1).

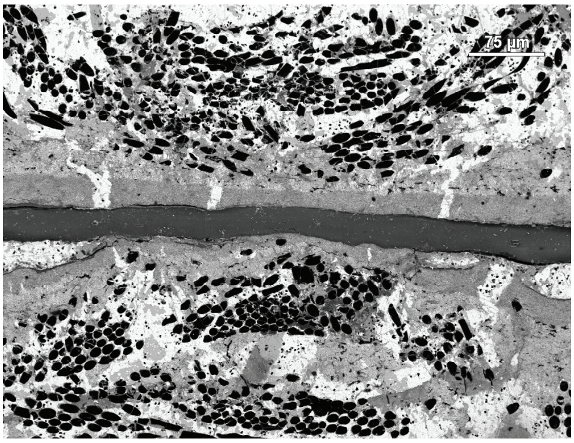

Fig. 1. Composite MEA-GDL structure infiltrated by Wood's alloy.

[1] Scheiba F., Benker, N., Kunz, U., Roth, C., Fuess, H., *J. Power Sources.* 177, **2008**, 273.

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Structural Investigations of MEAs for Fuel Cells Via Electron Microscopy. Susanne Zils[a], Nathalie Benker[b], Christina Roth[a]. [a]*Institute for Materials Science, Technische Universität Darmstadt, Germany.* [b]*Institute of Applied Geosciences, Technische Universität Darmstadt, Germany.*
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In order to maximise the performance of polymer electrolyte membrane fuel cells (PEMFC) it is crucial to increase the accessibility of the active noble metal particles and the contact between the electron- and ion-conducting parts of the membrane electrode assemblies (MEAs). Hence, the structural investigation of the MEA is an important step towards the systematic optimisation of PEMFCs. Up to now, the main part of the investigations has been carried out by both scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [1]. The structural characterisations via SEM and TEM have to take place in ultra high vacuum and therewith do not allow observation at operating conditions. In this work the environmental scanning electron microscopy (ESEM) is used in addition to SEM and TEM in order to obtain significant information about the MEA structure at quasi in-situ conditions. This technique allows studying the membrane electrode assemblies in different gas atmospheres as well as at different relative humidities and temperatures. These aspects render the possibility to adjust conditions in the sample chamber close to those in working fuel cells.
Noble metal catalyst nanopowders, proton conducting membranes and membrane electrode assemblies manufactured with different preparation techniques have been structurally characterised via TEM, SEM and ESEM. Long term single cell tests have been carried out in order to obtain information about the correlation between the manufacturing techniques and the performance of the corresponding fuel cells. First results show a degradation of the electrodes (crack formation) and at some areas a partially ablation of the electrodes from the membrane for MEAs tested in the long term single cell tests. The MEAs observed in the ESEM at quasi in-situ conditions showed a degradation but no ablation of the electrodes. The ESEM experiments carried out on the proton conducting membranes and MEAs lead to the conclusion, that the crack formation might be induced by the swelling of the membrane. Further ex-situ and in-situ experiments shall allow us to gain information about the influence of ice formation on the electrode structure and the electrode membrane interface.
In combination, the structural investigations by electron microscopy and the single cell tests allow to draw conclusions about the influence of the electrode structure and defects in the electrode and at the electrode membrane interface on fuel cell performance.

[1] F. Scheiba, N. Benker, U. Kunz, C. Roth and H. Fuess, *J. Power Sources*, **2008**, 177, 273.

Keywords: fuel cells; ESEM; TEM characterisation