

**FA2-MS10-O1****Electron Microscopy Techniques for the Detailed Study of Fuel Cell Electrodes and Components.**

Christina Roth<sup>a</sup>, Frieder Scheiba<sup>a</sup>, Ulrike Kunz<sup>a</sup>, Nathalie Benker<sup>a</sup>, Hartmut Fuess<sup>a</sup>. *<sup>a</sup>Institute for Materials Science, Technische Universität Darmstadt, Germany.*

E-mail: [c\\_roth@tu-darmstadt.de](mailto:c_roth@tu-darmstadt.de)

Transport processes play a significant role for the proper operation of polymer electrolyte membrane fuel cells (PEMFC). In PEMFCs the reactant gases must have access to the catalytically active sites, protons and electrons must be conducted through the electrode and the reaction product water must be removed from the pore system to avoid blocking of the gas diffusion paths. In the current standard electrode design each transport process is realized by a different component. Since the various components influence each other and therefore the electrode properties in a nonconstructive manner, optimization of the electrode structure is far from being trivial.

Porosity and the distribution of the polymer electrolyte appear to be the key parameters for the electrode performance. For the detailed investigation of these parameters transmission electron microscopy (TEM) has been chosen as a suitable tool. However, the sample preparation is crucial to the success of the experiment: the membrane-electrode assembly (MEA) is embedded in an epoxy resin and cut into thin sections by ultramicrotomy using a diamond knife. Details of the procedure can be found in [1] and references therein.

For the analysis of the polymer electrolyte distribution, infiltration of the sample with an epoxy resin has a significant drawback. Since the polymer electrolyte and the embedding resin have almost identical scattering contrast, the polymer electrolyte cannot be distinguished directly. As pores in the electrode structure may be filled by the polymer electrolyte or the embedding resin, it is also not possible to distinguish between open and closed pores (i.e. those filled by the polymer electrolyte). In this paper, we present different approaches to solve the contrast problem and suggest methods to characterize the polymer electrolyte distribution and electrode porosity.

A new imaging routine has been developed in order to avoid significant beam damage of the sensitive sample. By this technique 200 x 200 nm sized parts of the electrode can be imaged and the polymer electrolyte distribution analyzed with the help of the F signal in energy filtered transmission electron microscopy (EFTEM). This approach was very successful in contrast to staining techniques used earlier, and respective results for different MEAs before and after operation will be presented. In addition, first results applying a novel technique using Wood's alloy will be reported, which will help us to image both MEA and gas diffusion layer (GDL) at once in the future.

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

**Keywords: fuel cell electrode; TEM; EF-TEM**

**FA2-MS10-O2****Transmission Electron Microscopy of Electronic Ceramics.** Kevin M. Knowles. *University of Cambridge, Department of Materials Science and Metallurgy, Pembroke Street, Cambridge CB2 3QZ, UK.*

E-mail: [kmk10@cam.ac.uk](mailto:kmk10@cam.ac.uk)

Electronic ceramics based on strontium titanate, SrTiO<sub>3</sub>, and zinc oxide, ZnO, doped with small amounts of other oxide materials are of commercial interest as capacitors and varistors. Such materials are also ideal for fundamental studies on grain boundaries in ceramics because they have relatively simple crystal structures. Transmission electron microscopy (TEM) is a powerful tool for investigating the dopant distribution and, in principle, the space charge at grain boundaries in these materials. It can also provide detailed information through electron diffraction of both grain boundary crystallography and the nature of any second phase particles located at grain boundaries arising as a consequence of doping of the pure materials. This is particularly true for zinc oxide varistor materials where dopant levels of 1–2 wt% of other oxides such as vanadium pentoxide and manganese oxide produce second phase particles which cannot necessarily be identified unambiguously through X-ray powder diffraction. In this paper I will demonstrate using examples from work on these two electronic ceramics how the use of TEM at different length scales and a knowledge of crystallography are together indispensable tools for characterising the grain boundary microstructure of such materials.

**Keywords: grain boundary engineering; precipitation phase formation; transmission electron microscopy**

**FA2-MS10-O3****Transmission electron microscopy of BNT-BT-KNN.**

Ljubomira Ana Schmitt<sup>a</sup>, Jens Kling<sup>a</sup>, Mathis Müller<sup>a</sup>, Hans-Joachim Kleebe<sup>a</sup>, Manuel Hinterstein<sup>b</sup>, Wook Jo<sup>b</sup>, Jürgen Rödel<sup>b</sup>, Hartmut Fuess<sup>b</sup>. *<sup>a</sup>Institute of Applied Geosciences, University of Technology Darmstadt, Germany. <sup>b</sup>Institute for Materials Science, University of Technology Darmstadt, Germany.*

E-mail: [ljuba@st.tu-darmstadt.de](mailto:ljuba@st.tu-darmstadt.de)

Lead-free piezoelectric ceramics (1-x-y)Bi<sub>0.5</sub>Na<sub>0.5</sub>TiO<sub>3</sub>-xBaTiO<sub>3</sub>-yK<sub>0.5</sub>Na<sub>0.5</sub>NbO<sub>3</sub> (0.05 ≤ x ≤ 0.07 and 0.01 ≤ y ≤ 0.03) have been prepared by a solid state sintering method [1]. Preliminary electromechanical measurements revealed a giant strain of 0.45 % at an electric field of 8 kV/mm for composition 0.92BNT-0.06BT-0.02KNN [2].

Therefore the main focus of the following TEM study was directed on this composition. No domain structures, as commonly seen in lead zirconate titanate, were observed in TEM experiments on lead-free ceramics [2]. Only a lamellar contrast was visible within the grains. Selected area electron diffraction revealed no spot splitting but superstructure reflections and satellites due to octahedral tilting and cation ordering. Streaking of reflections in distinct directions