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Dynamic microscopy of energy conversion processes

JS-SFB.09

Benefit and challenge of in-situ ETEM experiments - electron beam induced potentials

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Environmental TEM (ETEM) represents a powerful tool for atomic resolution studies of materials in selected gas atmosphere. It enables, for example, the in-situ investigation of catalytic materials in their active state and, equipped with EELS, allows for observation of the atomic and electronic surface structure during reactions. However, the interpretation of such investigations also poses a great challenge in terms of comparability with conventional (ex situ) experiments: Inter alia, the question how the electron beam affects catalytic activity needs to be addressed.

In this contribution, the electron beam driven oxygen evolution at a $\text{Pr}_{0.68}\text{Ca}_{0.32}\text{MnO}_3$ manganite electrode in water vapor atmosphere is analyzed where oxygen evolution has been monitored by means of a side reaction of a sacrificial substance with evolved oxygen (Fig.1). Using a sample holder equipped with an STM tip, we present a bias-controlled ETEM experiment at $\text{Pr}_{0.05}\text{Ca}_{0.95}\text{MnO}_3$ in contact with water vapor where an electron beam initiated degradation process has been stopped by applying an electric potential. The role of the electron beam as the driving force for chemical reactions in the ETEM is investigated in the framework of electron beam induced electric potentials caused by secondary electron emission (Fig. 2). A quantification of these potentials on the basis of off-axis electron holography experiments combined with electrostatic modeling as well as a theoretical consideration of the secondary electron yield is presented. The impact of the gas species and pressure on beam induced potentials is addressed.

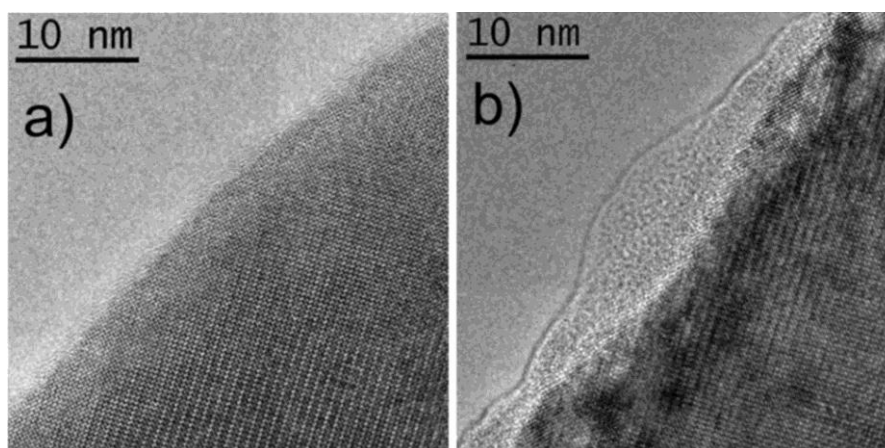


Figure 1. Crystalline PrCaMnO₃ TEM sample (a) in the initial state and (b) after 120 s in 0.3 mbar He₂/H₂O/SiH₄ gas under electron flux of 11000 e/Å²s. Electron irradiated sample regions are covered by amorphous SiO₂ which originates from the side reaction of SiH₄ with the evolved oxygen [S. Mildner et al., J. Phys. Chem. C 119 (2015)].

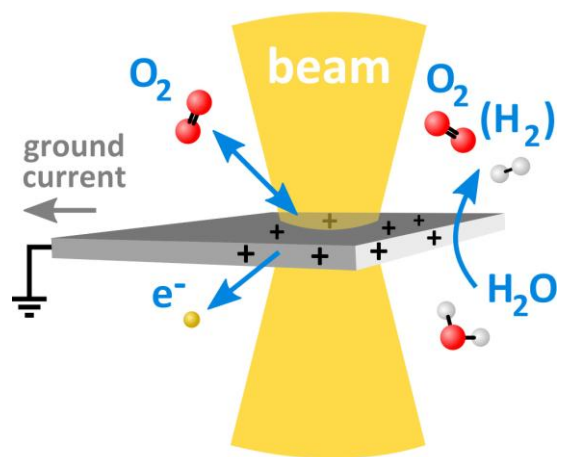


Figure 2. Scheme of TEM sample charging and potential buildup by secondary electron emission as the driving force for water splitting reaction in the ETEM [S. Mildner et al., J. Phys. Chem. C 119 (2015)].