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Operando surface X-ray diffraction studies of electrodeposited Co oxide thin films during oxygen evolution

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Water splitting is one of the most promising routes for renewable energy conversion and storage. Iron group metal oxides, especially Co oxides, are promising candidates for noble metal free catalysts for the oxygen evolution reaction (OER). In order to design efficient and scalable catalyst materials the relation between the surface structure and the catalytic properties are of fundamental importance. In this work we performed operando studies of electrodeposited $\text{Co}_3\text{O}_4/\text{Au}(111)$ and $\text{CoOOH}/\text{Au}(111)$ thin films during oxygen evolution using surface X-ray diffraction (SXRD). The SXRD measurements were done in our novel operando XRD cell which allows measurements during gas evolution and current densities of up to 30 mA/cm^2 . As samples thin epitaxial (111) oriented Co_3O_4 and (001) oriented CoOOH films were used. SXRD measurements during potential cycles into the OER regime reveal a clear reversible decrease in film thickness of about 1 nm for the Co_3O_4 film between 0.3 V and 0.6 V whereas no structural changes were observed in the case of CoOOH . The onset potential of OER is the same for both films which suggests that the topmost layer of Co_3O_4 is converted to CoOOH and the active phase for the OER is the same in both cases. Since the CoOOH thickness is constant (within 0.05 nm), only changes in the surface termination of the catalyst seem to occur in the OER regime.

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