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The role of surface area and stability-aspects of nickel-molybdenum HER electrocatalysts for the Alkaline Water Electrolysis

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Summary. Hydrogen by alkaline water electrolysis is gaining increasing interest due to favourable scalability [1]. In order to compete with the production rates of competing technologies, improving the electrocatalyst-activity is a commonly aspired approach. For this matter, the role of the surface area and electrocatalyst-stability aspects need to be addressed.

Abstract. In contrast to other technologies, a variety of non-noble electrocatalysts are suited for the alkaline water electrolysis. Among the electrocatalysts for the hydrogen evolution reaction (HER), nickel-molybdenum electrocatalysts are often reported to deliver high activities [2]. However, it is disputed whether the high activity of nickel-molybdenum electrocatalysts stems from intrinsic properties or from high surface areas. Moreover, the stability of these alloys remains uncertain [3,4].

The role of the surface area of nickel-molybdenum and nickel electrocatalysts is often disregarded. Furthermore, an agreement on the procedure to evaluate the electrochemical active surface area (ECSA) is not existent. Commonly applied methods to evaluate the ECSA include measurements of the double layer capacitance, either through non-faradaic cyclic voltammetry [1] or electrochemical impedance spectroscopy [5,6], or through the formation of α -nickelhydroxide for nickel electrocatalysts [6]. A comprehensive study on the correlation between different approaches to assess the ECSA, as well as the relation between the ECSA and the activity of nickel-molybdenum HER electrocatalysts is missing yet needed.

We investigated the outcome of the different procedures to evaluate the ECSA. The different methods correlate well if EIS is conducted at HER overpotentials (η_{HER}) greater than 200 mV. For $\eta_{\text{HER}} < 200$ mV, the results from EIS were influenced by a pseudocapacitive effect and thereby overestimated the ECSA. Lead by this observation, we present a relation between the overpotential at 10 mA/cm² and the ECSA for a variety of nickel and nickel-molybdenum electrocatalysts. We further included nickel and nickel-molybdenum electrocatalysts reported in the literature, which applied comparable procedures to evaluate the ECSA. We find that the overpotential generally decreases in a semi-logarithmic trend, indicating that the intrinsic activity of nickel and nickel-molybdenum HER electrocatalysts are not significantly different when compared to platinum. Nonetheless, the overpotential-ECSA relation of nickel-molybdenum electrocatalysts does not refute the theory about a higher intrinsic activity over nickel electrocatalysts. In addition and in order to address stability concerns, we investigated the stability of nickel-molybdenum during intermittent operation *in situ* by means of spectroelectrochemical Raman, using an in-house developed setup.

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