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Formation of desert rose structures in vacuum plasma sprayed electrodes for alkaline electrolysis

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Introduction

The EU FCH-JU RESelyser project is concerned with the development of high pressure, high efficiency and low cost alkaline water electrolyzers that can be operated variably and intermittently to meet the demands for integration into energy networks relying on fluctuating renewable energy. The project utilizes NIAiMo alloy electrodes produced at the German Aerospace Center (DLR) by vacuum plasma spraying (VPS). VPS results in a heterogeneous microstructure consisting of a multitude of intermetallic phase sub domains and pores. Prior to electrolysis operation the electrodes are activated by leaching of Al and some AI containing intermetallic phases leaving micrometer pores and nanometer dendritic pores increasing the surface area available for the electrolysis reactions. Post mortem analysis of the electrodes revealed “desert rose” like nano flakes on the surfaces and in the pores. Earlier microscope analysis of Raney-Nickel electrodes for alkaline electrolysis (Ref. 1, 2, 3) has also reported on very fine unidentiﬁed nano structures on electrode surfaces. This study seeks to investigate the nature and the formation of these nano structures.

Results and discussion

Analyses of cross sections and electrode surfaces revealed desert rose like nano flake structures on the surfaces and in the pores of several electrodes, depending on the electrode history. The size of the faceted flakes varied from tens of nm to a couple of µm where the thickness varied from a few nm to ~20 nm. The particles were too fine for reliable EDS analysis in SEM. However, high oxygen and Al contents were indicated in the flakes. Similarly, the NIAiMo powder leached for Al showed emerging desert rose structure after storage in distilled water.

Experimental

Vacuum plasma sprayed NIAiMo alloy electrodes were characterized before and after electrolysis operation and after storage in distilled water for various length of time. For comparison, NIAiMo raw powder for VPS spraying, powder after leaching out Al, and the leached powder stored in distilled water for 40 days were also analyzed. The microstructural investigation was carried out applying a FEGSEM (Zeiss Supra 35) and HRTEM (JEM-3000F).

Evaluation of the ﬂake size of the desert rose structure as function of sample history indicated that the formation of the desert rose structure was related to the electrolysis operation involving exposure to KOH and ~70°C as well as the duration of storage in distilled water at room temperature.

Conclusion

The desert rose structure on the NIAiMo electrodes for alkaline electrolysis occurred only after storage in water and/or electrolysis operation; not on the “as VPS sprayed” or on the “by leaching AI activated” electrodes. The size of the flakes appeared to be more affected by the time of electrolysis/exposure to KOH at elevated temperature than by the time of storage in water. This data and our preliminary study of the chemical composition of the nano structured flakes lead us to suggest the following mechanism for the formation of the desert rose structure: Incomplete leaching of Al during activation of the electrodes can leave some AI that may dissolve in water and precipitate as oxidized species. In KOH at elevated temperature this could be more pronounced. The implications, positive or negative, for the application and performance of the electrodes would depend on the electronic and catalytic properties of the precipitates, and whether the precipitation takes place during operation or at breaks in the operation.

References