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Characterization of aluminum and nickel thermochemical diffusion for synthesis of alkaline water electrolysis electrodes

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Many of the renewable energy sources, such as wind and solar, are fluctuating daily and seasonally. Hence, load management is of high importance when such energy sources are used [1]. Using the excess electrical power from renewable energy sources to produce hydrogen via water electrolysis, offers new alternatives for load management without emission of greenhouse gases. Water electrolysis constitutes only 4% of global hydrogen production due to its relatively high price, but the other available hydrogen production processes emit CO_2 [2]. The increasing legislations towards reducing the CO_2 emission worldwide, encourages further development of water electrolysis technologies [1]. Alkaline water electrolysis is the standard large-scale and the most mature technology of water electrolysis. There is a large interest for improving the electrodes in terms of efficiency, durability and cost. We have developed electrodes of high efficiency, durability and stability, by thermo-chemical diffusion of Al-Ni and selective leaching of aluminum [3]. Various electron and ion microscopic characterizations techniques are applied in this study to elucidate on the mechanism of diffusion, formation of new phases and microstructure evolution to optimize the process of the electrode synthesis.

The electrodes were synthesized in the following manner. Commercially available nickel plates with a thickness of 0.5 mm which cathodically degreased for 2 min were used as substrate. The substrates were heated and etched by Ar sputtering and aluminum plasma vapor deposited in a non-reactive DC-magnetron sputtering mode using a CC800/9 SinOx coating unit from CemeCon AG. For thermochemical diffusion, the Al-Ni specimens were heat treated in an atmospheric furnace for various times at 610°C.

EDS, EBSD and ion channelling contrast imaging (ICCI) are performed in an FEI Helios NanoLab™ 600 dual beam FIB/FEG-SEM, equipped with an EDAX-TSL EBSD system and a Hikari camera. The SEM was operated at 15 kV and a probe current of 5.5 nA. An FEI Titan 80–300ST field-emission-gun TEM, equipped with an EDS (Oxford Instruments, X-Max^N 80 mm^2 silicon drift detector) and a spherical aberration probe corrector. The TEM was operated in scanning TEM (STEM) mode at 300 kV and the images were acquired using a high-angle annular dark-field (HAADF) detector. STEM-EDS was carried out with an interval of 10 nm from a region of 100x200 nm^2.

Fig.1 shows the microstructure of the as-deposited Al on Ni. The Al layer has a columnar microstructure in which the microstructure consists of fine grains in the vicinity of the substrate and by increase of distance from the substrate some grains outgrow the rest. Notably, the microstructure of Al is not fully dense and no epitaxial relation between the Ni substrate and the Al deposited layer is detected. Upon 10 min. heat-treatment at 610°C, two diffusion layers form in the vicinity of the Ni-substrate (Fig.2). EDS analysis showed that the chemical composition of those layers are Al_3Ni_2 and Al_3Ni. The rest of the Al layer has still a columnar microstructure and on some of the grain boundaries and triple lines a new phase has formed. EBSP revealed that the newly formed phase is $AlNi_3$. Based on the EDS results after heat treatment for 120 min., $AlNi_3$ and AlNi diffusion layers form close to the substrate and the rest of layer is composed of Al_3Ni_2. TEM results showed that γ-Al_2O_3 also forms at the top Al_3Ni_2 layer where the material has highest interaction with the atmosphere of the furnace (Fig.3).

In summary, the grain boundary diffusion plays a major role in formation of various Al-Ni intermetallics, and by controlling the time of heat treatment the desired phases can be obtained.

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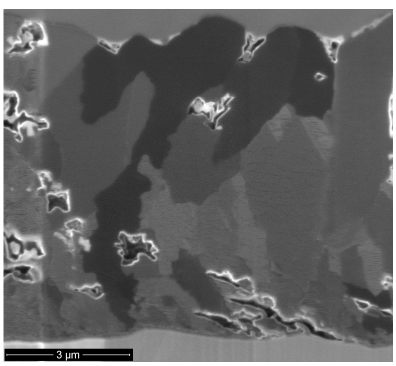

Figure 1. Ion channeling contact image of an aluminum layer deposited on a nickel substrate

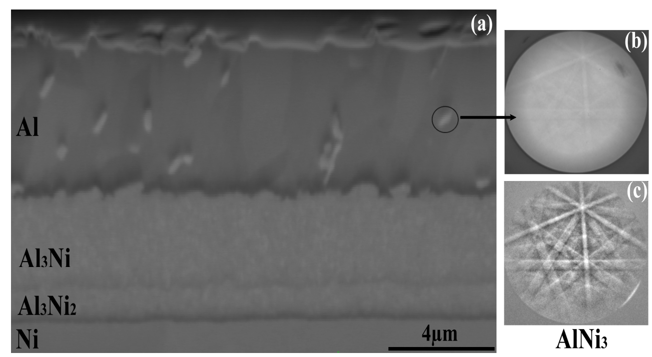

Figure 2. (a) Forward scatter electron image of the Al-Ni couple after heat treatment for 10 min at 610°C. (b) EBSP of a new phase forming on Al boundaries. (c) Same as (b) after background subtraction.

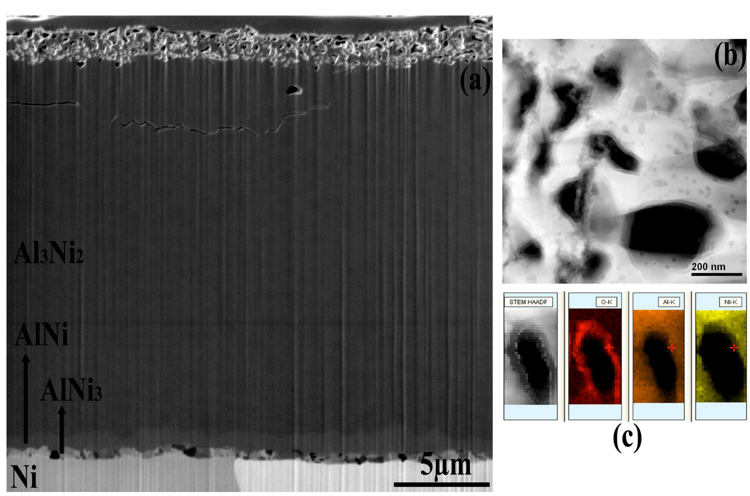

Figure 3. (a) Ion channeling contact image of the Al-Ni couple after heat treatment for 120 min. at 610°C. (b) STEM-HAADF image of a porous layer lying on the top surface of the Al_3Ni_2. (c) STEM-EDS elemental maps of an area adjacent to a pore.

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