#### #01130

# **3D** Pyrolyzed Carbon Modified with g-C3N4@Ni Electrodeposit for Electrocatalytic Hydrogen Generation

## B. Advanced catalytic materials for (photo)electrochemical energy conversion IV

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### Abstract

Hydrogen, derived from water electrolysis, has emerged as a promising alternative to fossil fuels, offering a clean and recyclable energy solution. However, despite its remarkable attributes, the widespread adoption of hydrogen faces significant challenges because of its viability as a practical solution. One of the major obstacles is the affordability of water electrolyser systems, which play a crucial role in the production of hydrogen through water electrolysis. Herein, we propose the fabrication of a free-standing 3D electrode based on stereolithography 3D printing of polymer which can be functionalized via a series of post-processing treatments. By subjecting the 3D polymer to a thermal decomposition process via vacuum furnace annealing, we were able to transform it into a conductive and stable electrode material. To enhance the electrochemical performance of the electrodes, post-processing has been conducted involving nickel electroplating and chemical vapour deposition (CVD) of graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>), derived from melamine. These steps were aimed at exploring the potential of further improving the conductivity and catalytic properties of the 3D electrodes. Different diamond lattice-based crystal structures such as tapsterite, rod-connected diamond (RCD) and F-rhombic dodecahedron (FRD) are investigated offering controllability over the electrode surface area and geometries. Cyclic voltammetry measurements were conducted to evaluate the electrochemical behaviour of the 3D graphitic electrode. The CV curve exhibits a smooth and continuous shape without significant sharp peaks or irregularities. The current response shows gradual changes as the potential is scanned within the chosen range. One notable observation from the CV analysis is the increased area under the curve after post-processing of the electrode. This expanded area indicates improved electrochemical performance and suggests an enhanced active surface area of the electrode. The absence of sharp peaks suggests that the electrode possesses a capacitive behaviour and can facilitate efficient charge storage and transfer processes. To gain insights into the surface morphology and elemental composition of our sample, we performed scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) analysis. The rate of thermal decomposition was found to affect the morphology of the pyrolyzed carbon. Furthermore, the EDS spectra of post-processed electrodes indicated the presence of nickel and carbon when compared to the pre-processed carbon. The detection of nickel suggests successful electroplating, while the presence of nitrogen indicates the incorporation of carbon nitride during CVD steps. Further, this work explores the electrochemical performance of the 3D electrode via electrochemical measurements using rotating disk electrodes. Moreover, crystallographic analysis will be performed using XRD spectroscopy.