BIO Web of Conferences **129**, 26040 (2024) https://doi.org/10.1051/bioconf/202412926040
EMC 2024

IN-SITU SYNTHESIS OF FexPy NANOPARTICLES

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Background incl. aims

Iron phosphide is an earth abundant material with several applications, such as an electrocatalyst for the hydrogen evolution reaction (HER) and the oxygen evolution reaction (OER). Water-splitting as a way of producing hydrogen gas is essential to explore due to its low carbon footprint, and using earth abundant catalysts such as iron phosphide is preferred due to being inexpensive, active, and electrochemically stable (1). Therefore, it is of interest to explore methods to manufacture iron phosphide and aim to achieve perfect crystallinity in combination with tuning the different crystalline phases, since they exhibit different catalytic activity. With an environmental transmission electron microscope (ETEM) it is possible to reveal the mechanism of the gas synthesis at an atomic scale in-situ.

Methods

The iron nanoparticles used are created by using spark ablation in a spark discharge generator and deposited on MEMS chips allowing for heating. The ETEM, Hitachi HF-3300S, interfaced with a metal organic chemical vapor deposition (MOCVD) system is the instrument used and the gases induced are PH_3 and H_2. Energy dispersive X-ray (EDX) spectroscopy is used for elemental information and enabled by the SDD X-MaxN 80T detector. Post-experiment data analysis is performed by utilizing FFT spectra obtained from the high-resolution TEM (HRTEM) images and videos from the Gatan OneView IS camera.

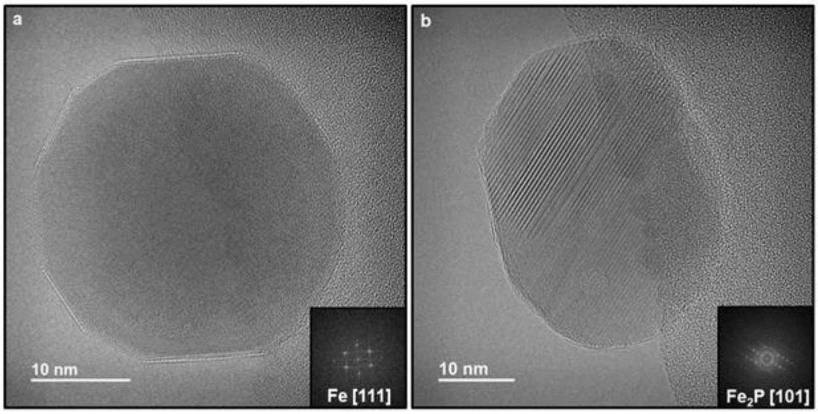
Results

By tuning gas flows and temperature synthesis of different iron phosphide phases were observed directly, currently the phases found are Fe_2P and FeP. The transformation into iron phosphide is nearly instantaneous when PH_3 enters the system, however by using dilution with H_2 it is possible to somewhat slow down the process. It also appears that using pure versus oxidized iron nanoparticles still seems to produce iron phosphide. Moreover, there have been observations of a transformation from FeP to Fe_2P by increasing temperature. Fe_2P has been proven to be very stable in various gaseous and temperature conditions, as well as in air.

Conclusions

Different phases of iron phosphide have been synthesized in the ETEM and dynamics of phase transformations have been investigated. The transition between phases has been observed, as well, indicating that it might be possible to realize phase engineering on iron phosphides even after phosphorus has entered the particles. Our future work will aim towards studying the transformation to iron phosphide further, exploring and characterizing the possible phases and achieving selection of crystal structure in a more controlled manner. By finding a reliable way of creating phases it can pave the way for comparing different phases and for example their catalytic activities in a fair way.

Graphic:



Keywords:

ETEM, Iron Phosphide, nanoparticles, in-situ

Reference:

(1) Xu. S. et.al. J. Mater. Chem. A, 2020,8, 19729-19745