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Phase-controlled formation of NixPy catalyst using environmental TEM for potential application in CO2 reduction

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Background:

The urgent need to mitigate climate change has driven intensive research into efficient strategies for reducing CO2 emission and potentially removing them from the atmosphere. Carbon capture and storage represents a promising approach, together with conversion of CO2 produced in industrial processes and from the environment into useful products and fuels via CO2 reduction reactions. Earth-abundant transition metal phosphides, i.e. nickel-phosphide (NixPy) catalysts, offer several advantages, including, cost- effectiveness, switchable selectivity for a range of reaction products, phase-dependent catalytic activity and potential for facilitating CO2 transformation into value-added products for renewable energy [1–3]. However, the development of this system as a tunable catalyst for CO2 reduction reactions is in its early stages, requiring a comprehensive understanding of synthesis processes to attain control over phases, size, shape/facets, and catalytic activities. Structural evolution that occurs during reactions are still largely unknown, and the relationship between facet structure and product selectivity remains poorly understood. This study aims to investigate the phase-controlled formation dynamics of nickel phosphide nanoparticles and their stabilities at higher temperatures using dedicated in-situ transmission electron microscopy (TEM) coupled with chemical vapor deposition (CVD) system.

Method:

The experiments were conducted using environmental transmission electron microscopy (ETEM) combined with a customized gas handling system. Size-selected nickel nanoparticles (10-30nm) were deposited onto micro-electro-mechanical system (MEMS) heating chips by an aerosol-phase fabrication method. These nanoparticles were then exposed to PH3 gas inside the TEM at appropriate temperatures up to 700°C to transform them into nickel-phosphides. Phase transformation of the nanoparticles was induced by changing the reaction parameters such as reaction temperature and PH3 gas flow inside TEM. The dynamic processes were followed by high-speed high-resolution imaging (up to 300 fps) during gas exposure. Structural information was obtained using power spectra of acquired high-resolution images, while composition was monitored by energy dispersive X-ray spectroscopy. The gas composition inside the microscope was monitored by an integrated residual gas analyzer.

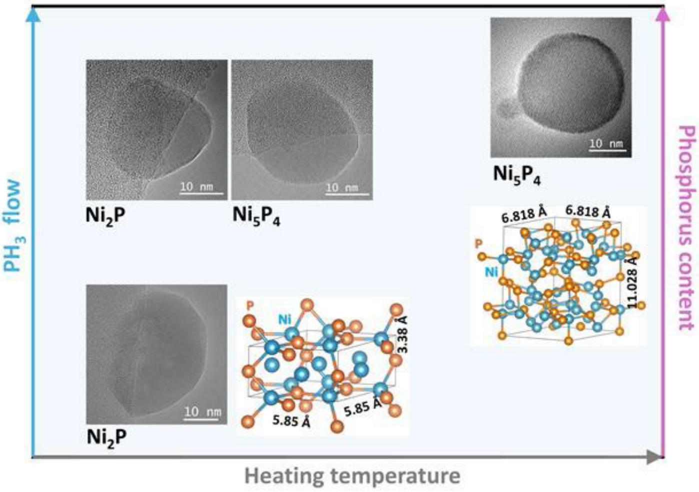
Result:

Our Preliminary results focus on the formation dynamics of phase-controlled nickel phosphide nanoparticles. We investigated the role of reaction parameters, such as phosphene gas flow and heating temperature, on the phase transformation of nanoparticles. Two distinct phases were achieved with varying PH3 flow and temperature. Lower PH3 flow (0.3 sccm) at 300°C resulted in Ni2P phase formation, while higher PH3 concentration at elevated temperatures led to Ni5P4 phase formation. Intermediate experimental conditions, such as higher PH3 flow at lower temperature, resulted in a mixture of Ni2P and Ni5P4 phase particles. All the phases showed high thermal stability. These findings suggest that higher PH3 flow at elevated temperatures favors the incorporation of more phosphorus into the nickel phosphide nanoparticles. According to literature, both phases Ni2P and Ni5P4 are interesting for catalytic activity for hydrogen evolution reaction [4] and CO2 reduction [2], respectively.

Conclusion:

This work demonstrates an in-situ solid-gas phase strategy for synthesizing nickel phosphide nanoparticles with controlled phase by varying PH3 gas flows and reaction temperatures. Such insights are critical for designing phase-engineered NixPy catalyst for sustainable CO2 conversion technologies, offering significant implications for addressing global climate challenges. The next step in this work would be to investigate these phases for CO2 reduction and to monitor the structure evaluations occur during the catalytic reactions.

Graphic:



Keywords:

Nickel phosphide, Phase engineering, In-situ

Reference:

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