

A Microwatt-Resolution Calorimeter for Studying the Reaction Thermodynamics of Nanomaterials at High Temperature and Pressure

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OVERVIEW

A microwatt resolution calorimeter for the analysis of nanomaterials

- Permits the means for stable, sensitive calorimetry at high temperature and pressure
- Useful for probing catalysis, phase transformations, and thermochemical energy storage

BACKGROUND

Calorimetry is a powerful technique used to study reactions involving nanomaterials, providing valuable insights into their thermodynamic properties, reaction kinetics, and stability. Calorimetry measures the heat flow associated with a chemical reaction or physical process. Calorimetry of reactions involving nanomaterials is of great current interest, but requires high-resolution heat flow measurements and long-term thermal stability. Such studies are especially challenging at elevated reaction pressures and temperatures. Calorimetric reactors enable direct measurement of reaction thermodynamics and kinetics but conventional calorimeters are generally inadequate for the analysis of milligram-scale samples at elevated temperature and pressure. As such, a need exists for improved methods for studying the reaction thermodynamics of nanomaterials.

INNOVATION

Researchers at the University of Michigan have developed a new type calorimetry instrument capable of measuring thermodynamics of milligram amounts of material on the microwatt scale. This instrument can resolve the net change in the amount of gas-phase reactants due to surface reactions in an operating range from room temperature to 300 °C and reaction pressures of 10 mbar to 30 bar. This instrument enables high heat flow resolution for measuring the enthalpy of reactions between gas-phase reactants and milligram scale nanomaterial samples. The calorimetric resolution is shown to be $<3 \mu\text{W}/\sqrt{\text{Hz}}$, with a long-term stability $<4 \mu\text{W}/\text{hour}$. The performance of the instrument is demonstrated via a set of experiments involving H₂ absorption on Pd nanoparticles at various pressures and temperatures. For this specific reaction, we obtained a mass balance resolution of 0.1

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$\mu\text{mol}/\sqrt{\text{Hz}}$. Results from these experiments are in good agreement with past studies establishing the feasibility of performing high resolution calorimetry on milligram scale nanomaterials, which can be employed in future studies probing catalysis, phase transformations, and thermochemical energy storage.