

Advanced Hydrogen Research

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Hydrogen (H_2) is considered the energy carrier of the future. It is a versatile and clean energy source that can be produced from a variety of sources, including renewable energy sources such as wind and solar. H_2 is also being used to reduce high CO_2 emissions from metallurgy processes by the direct reduction of such substances as iron ore. While hydrogen production, storage, and conversion have reached a certain technological level, there is still room for improvement and new discoveries. Thermal Analysis can help us better understand material interactions.

BY MEANS OF

THERMAL ANALYSIS

NETZSCH

Challenges in Hydrogen Usage

Hydrogen presents a significant flammability risk, demanding special safety precautions during its production, handling, and utilization. Even low concentrations of hydrogen need to be taken seriously, as ignition can occur when concentrations are only 4% hydrogen in air at room temperature.

Thermal Analysis and Hydrogen

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NETZSCH offers the ability to measure materials in a hydrogen atmosphere along with an accompanying safety concept. Measurements can be conducted in a 100% H₂ atmosphere or mixed with other non-flammable gases like nitrogen (N₂) or argon (Ar). Inert gas is also used to purge the thermobalance. If a failure occurs, the device is flushed with inert gas.

Conduct Reduction and Oxidation Experiments in a Safe Environment

Reduction is used in a variety of industrial processes. Examples include metallurgical processes and the storage and release of H_2 in certain materials. To conduct reduction or oxidation experiments, any STA 449 can be upgraded for use with hydrogen gas. Along with the reduction test, NETZSCH offers the oxidation test under air atmosphere. The STA has to be purged with inert gas in an intermediate state until safe conditions for a gas change are reached. NETZSCH offers the STA 449 hydrogen system as complete solution including the newly developed H_2 Secure control box.

STA 449 *H*₂*Secure* Concept

Defined H₂ Gas Volume

Hydrogen enters at the top of the furnace. H_2 is confined to a defined space above the continuously purged balance chamber.

H₂Secure Box

The central communication box receives gas concentration information and allows or denies gas flow based on set limits.

Monitoring of H₂ and O₂

 H_2 and O_2 gas concentrations are continuously measured to ensure safe handling.

Fail-Safe Security

In the case of a power failure, magnetic valves open up and release inert gas, which removes hydrogen from the system.



Hydrogen Gas Supply Hydrogen can be supplied from an H_2 generator or H_2 cylinder and is connected to the special H_2 gas inlet on the rear of the STA with integrated safety valves.

Optimized Gas Path This provides a precise concentration of gas, e.g., up to 100% hydrogen, while maintaining a protective gas atmosphere at the balance.

3 Continuous Monitoring of Gas Concentrations STA exhaust gas flow is monitored for H₂ and O₂ concentration.

H₂Secure Box

Central communication box to control signals and allow or deny gas flows depending on the H_2 or O_2 limits defined.

The Reversible Nature of Copper (Cu) – Copper Oxide (CuO) Redox Reaction

The example illustrates a cycle experiment exploring the reversible reaction of CuO with hydrogen and air by monitoring the mass changes throughout the process. Initially, CuO undergoes reduction in an H₂ atmosphere, leading to the formation of metallic Cu. Subsequently, in an oxidizing environment, metallic Cu oxidizes again to CuO with the introduction of air. In the following cycles, an increasing loss in the oxidation potential can be observed, indicating degradation of the catalytic capability.

With the help of thermogravimetry, researchers are able to gain insights into reaction kinetics, mechanisms and the thermodynamic properties of oxide-based catalysts, advancing the understanding and optimization of catalytic systems.



Measurement on copper oxide powder (29.975 mg) at 500°C in an alternating hydrogen/air atmosphere

Investigating Hydrogen Absorption and Desorption in Zirconium-Based Getter

The accumulation of hydrogen presents a significant safety concern due to the potential for explosion under specific conditions. To address this issue, hydrogen scavengers, commonly known as getters, play a crucial role in eliminating hydrogen from such environments. Via STA, a zirconium-based getter was analyzed at a rate of 10 K/min in a pure hydrogen environment, and distinct absorption-desorption cycle was observed. The process displayed reversibility with an approximately 50°C hysteresis, showcasing the getter's effectiveness at removing hydrogen and enhancing system safety.



Mass change of a zirconium-based getter (279.5 mg) during heating and cooling under 100% H, atmosphere

Applications and Performance

Technical Specifications

STA 449 + H ₂ Secure Box	
Furnace type supporting H_2 measurements	SiC
Temperature range	RT to 1600°C
Sensor types*	TGATGA-DTATGA-DSC
Thermocouple types*	 P S B
Sensor type for reduction experiments only	W
Optional 4-fold MFC	Possible switching between hydrogen and air atmospheres in one measurement
Hydrogen supply	Supplied by the operator, e.g., hydrogen generator, H_2 cylinder
H_2 and O_2 measuring cell	Included
<i>H₂Secure</i> box	Included
Optional gas outlet treatment	H ₂ dilution
Upgrade of existing STA 449	\checkmark
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* Possibility of reduced life time depending on experimental parameters

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