

# NETZSCH

Proven Excellence.

## Advanced Hydrogen Research

BY MEANS OF  
THERMAL ANALYSIS



### Hydrogen Fuel for Tomorrow

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.

### 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

NETZSCH offers the ability to measure materials in a hydrogen atmosphere along with an accompanying safety concept. Measurements can be conducted in a 100%  $H_2$  atmosphere or mixed with other non-flammable gases like nitrogen ( $N_2$ ) 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<sub>2</sub>Secure* Concept

### Defined H<sub>2</sub> Gas Volume

Hydrogen enters at the top of the furnace. H<sub>2</sub> is confined to a defined space above the continuously purged balance chamber.

### Monitoring of Gas Concentrations

H<sub>2</sub> and O<sub>2</sub> gas concentrations are continuously measured to ensure safe handling.



### *H<sub>2</sub>Secure* Box

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

### Fail-Safe Security

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

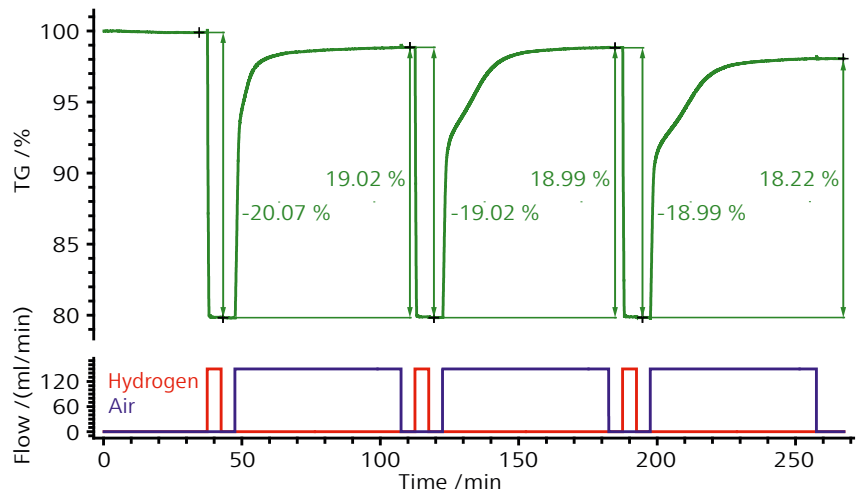
## SET UP



- 1 Hydrogen Gas Supply**  
Hydrogen can be supplied from an H<sub>2</sub> generator or H<sub>2</sub> cylinder and is connected to the special H<sub>2</sub> gas inlet on the rear of the STA with integrated safety valves.
- 2 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 H<sub>2</sub> and O<sub>2</sub> concentration.
- 4 *H<sub>2</sub>Secure* Box**  
Central communication box to control signals and allow or deny gas flows depending on the H<sub>2</sub> or O<sub>2</sub> limits defined.

## The Reversible Nature of Copper – Copper Oxide Redox Reaction

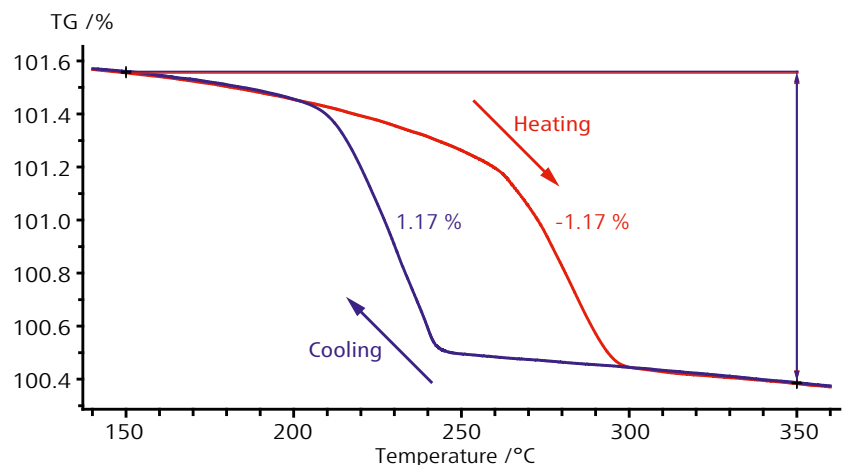
The example presented illustrates a cycle experiment exploring the reversible reaction of copper oxide with hydrogen and air by monitoring the mass changes throughout the process. Initially, copper oxide undergoes reduction in a hydrogen atmosphere, leading to the formation of metallic copper. Subsequently, in an oxidizing environment, metallic copper oxidizes back to copper oxide 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.



Thermogravimetric reduction-oxidation cycles of 29.975 mg copper(II) oxide powder at 500°C

## 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<sub>2</sub> atmosphere

# Applications and Performance

# Technical Specifications

STA 449 + H <sub>2</sub> Secure Box	
Furnace type supporting H <sub>2</sub> measurements	SiC
Temperature range	RT to 1600°C
Sensor types*	<ul style="list-style-type: none"><li>▪ TGA</li><li>▪ TGA-DTA</li><li>▪ TGA-DSC</li></ul>
Thermocouple types*	<ul style="list-style-type: none"><li>▪ P</li><li>▪ S</li><li>▪ B</li></ul>
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 <sub>2</sub> cylinder
H <sub>2</sub> and O <sub>2</sub> measuring cell	Included
H <sub>2</sub> Secure box	Included
Optional gas outlet treatment	H <sub>2</sub> dilution
Upgrade of existing STA 449	✓

\* Possibility of reduced life time depending on experimental parameters

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