KRATOS ANALYTICAL
A SHIMADZU GROUP COMPANY

Technical Note

High Temperature Gas Reaction Cell

MO477(A)

Keywords High temperature, high pressure, reaction cell, catalyst

Part Number WX–665 / WX-679

Overview

The high temperature gas reaction cell allows sample modification under high temperature and high pressure conditions. The reaction cell is a small volume, quartz chamber with facility for gas introduction, purging and evacuation. Maximum operating temperature is 1000˚C. Maximum operating pressure is 30 bar. The accessory has integrated sample and gas handling allowing transfer under UHV conditions directly to the sample analysis chamber or via the surface science station, depending on which chamber the reaction cell has been mounted.

Gas reaction cell key features

- Operating temperature-pressure ranges: 1000˚C at 1 bar, 300˚C at 30 bar
- Oxidation / reduction studies of heterogeneous catalyst materials

Introduction

The value of determining surface chemistry of catalyst materials by X-ray photoelectron spectroscopy (XPS) was realised soon after the first commercial spectrometers were available in the early 1970s. Surface characterisation of heterogeneous catalysts continues to be an important application of the technique.

A perceived limitation of XPS and surface analysis techniques for characterisation of these materials is the requirement to operate at ultra high vacuum. This contrasts to real catalytic working conditions of atmospheric or even higher pressures and elevated temperatures. The high temperature gas reaction cell is an attempt to bridge the so-called ‘pressure-gap’ allowing the material to be exposed to high pressure gases at elevated temperatures.

The high temperature gas reaction cell allows samples to be oxidised or reduced and transferred to the analysis chamber for characterisation by XPS and other surface characterisation techniques available on the AXIS photoelectron spectrometers. After characterisation the samples may be returned to the reaction cell for further treatment and then re-analysed. Typical heterogeneous catalyst characterisation experiments are conducted as a series of temperatures in either an oxidising or reducing environment.

AXIS spectrometers have the sensitivity to allow spectroscopic investigation of real catalyst system with loadings as low as 0.01wt% (dependent upon exact system under investigation). The combination of an *in-situ* catalyst cell and modern XPS instrument is an attractive one to many in the field of catalysis.

Vacuum envelope & reaction cell

The gas reaction cell is shown in Figure 1. At the heart of the device is the reaction vessel formed completely from fused quartz. The cell is a completely inert containment vessel in which reactions can occur. The small volume quartz reaction cell is contained within a stainless steel vacuum envelope. All internal parts of the cell and the powder sample stub are fabricated from quartz or are gold plated stainless steel to improve their high temperature chemical resistance. The cell incorporates three gas lines; the gas inlet, gas outlet and gas purge inlet. These connections are fed via UHV compatible Swagelok unions connected to the gas handling panel.

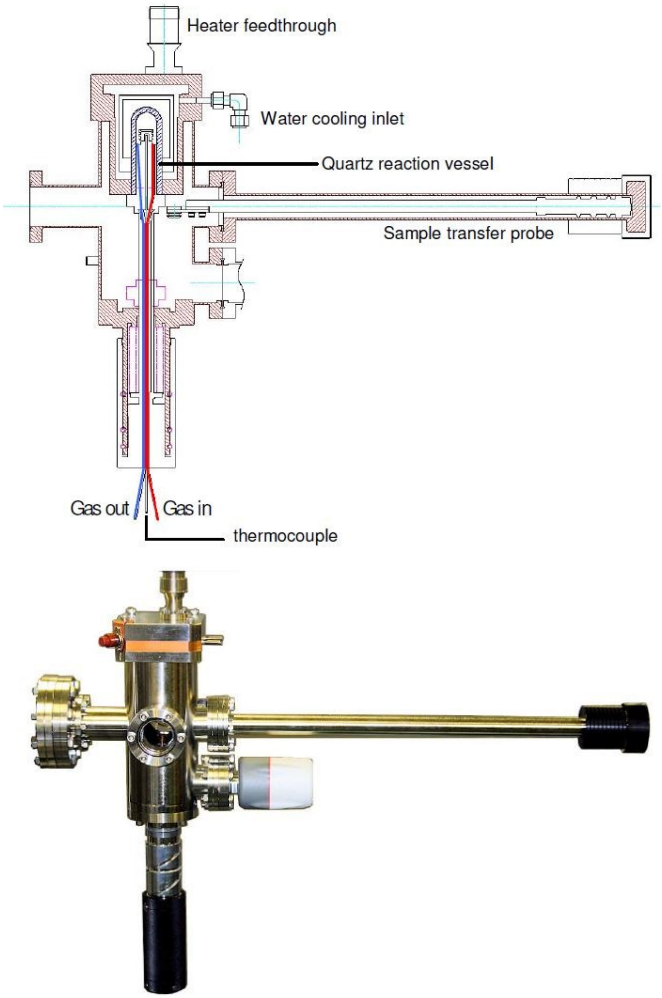

Figure 1: Schematic cross section (top) and photograph (bottom) of the high temperature gas reaction accessory.

Indirect, radiative sample heating is achieved using a boron nitride heater element directly above the reaction cell. The heater is controlled by a PID temperature controller. During operation, the sample holder temperature is measured by a thermocouple in intimate contact with the rear of the stub so as to minimise temperature lag between measured and actual sample temperature.

Water cooling is integrated into the vacuum chamber and a thermal cut-off switch ensures that the chamber temperature remains safe, even when the reaction cell temperature is at maximum operating temperature of 1000˚C. Cooling of the cell door seal is achieved by forced air. The cell door seal temperature is measured and interlocked to prevent overheating of the seal during use.

Samples are mounted on a special 13 mm diameter quartz stub. Sample transfer from the reaction cell to the analysis or surface science station chambers is by a magnetically coupled transfer arm. The high temperature gas reaction cell has part number WX-665 when mounted directly to the sample analysis chamber of the AXIS Supra$^+$ and WX-679 when mounted on the surface science station. The function of the high temperature gas reaction cell is identical, the difference being in the sample transfer dimensions.

Samples are introduced into the cell either directly through a viton sealed hinged door on the front of the vacuum chamber or from the AXIS main instrument.

Gas Handling

The gas handling panel is shown in figure 2. Gases are introduced into the reaction cell using bellows-sealed precision leak valves and fine stainless steel capillary tubes

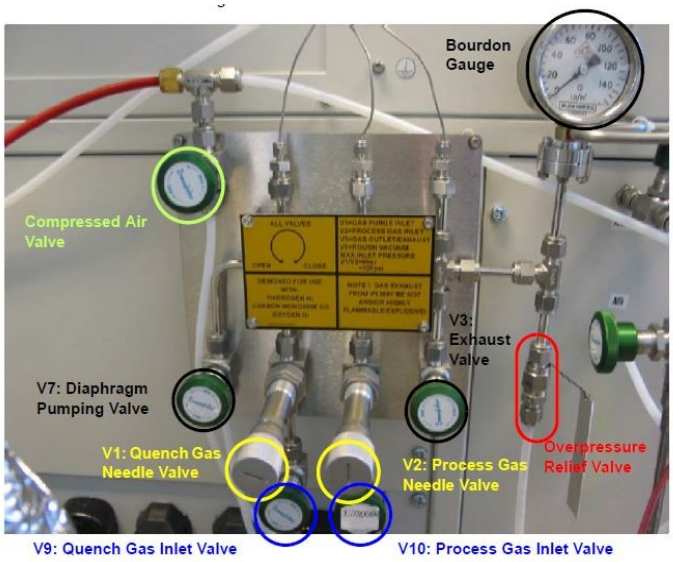

Figure 2: Gas handling panel mounted at the rear of the instrument.

The cell can be run in a static mode where gas is back filled into the chamber or dynamically where the needle valves control gas flow. The reactant gas exits the chamber via the exhaust capillary where it can be either vented safely or analysed directly by an external mass spectrometer (not supplied). The pressure within the cell is measured using a Bourdon gauge. Safety is designed in with an over pressure relief valve.

The gas reaction cell and UHV envelope are rough pumped by a dry diaphragm pump and turbo molecular pump. The safe pressure / temperature operating regimes are shown in Figure 3.

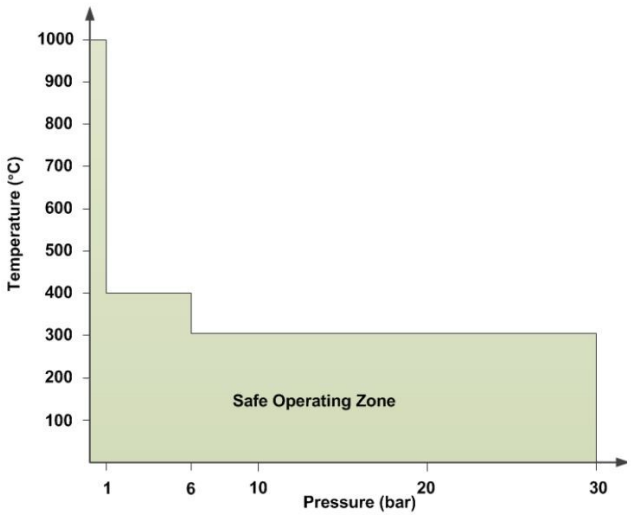

Figure 3: Safe temperature/pressure conditions for operation of the gas reaction cell.

The standard pumping configuration of this accessory is not designed to pump corrosive gases. If experimental requirements dictate the use of corrosive gases please consult Kratos for options.

Allowed gases with standard configuration include:

- Hydrogen (H_2)
- Oxygen (O_2)
- Carbon monoxide (CO)
- Methane (CH_4)
- Ammonia (NH_3)

Recommended purge gas: Argon (Ar)

Pressure in the UHV chamber is measured using a cold cathode, wide range gauge.

Summary

The gas reaction cell is an accessory for AXIS spectrometers providing the ability to carry out high temperature, high pressure surface modification of samples or pre-treatment of (in general) powder samples. These samples are subsequently transferred under UHV conditions into the sample analysis chamber for surface characterisation.

Related applications notes online:

MO402 Oxidation of cobalt hydroxide

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