A Sample Cell for in situ EXAFS Measurements

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A sample cell has been built in our laboratory to carry out EXAFS experiments on supported metal catalysts. The cell has the capability of heating the sample to 800 K in the presence of specific gases or in vacuo and then performing EXAFS measurements in situ over a temperature range of 77 K to 600 K.

I Introduction

Supported metal catalysts are one class of materials in which it is essential to be able to heat the sample in vacuo or in the presence of specific gases and then perform the X-ray absorption measurements in situ. It is also desirable to make the EXAFS measurements over a wide temperature range to study dynamic effects (i.e. the Debye-Waller factor), the temperature dependence of gas chemisorption or structural changes during catalytic reactions.

This paper describes a sample cell with the capability of heating the sample up to 800 K and then performing X-ray absorption measurements in situ over a temperature range of 80 K to 600 K. Two versions of the cell have been fabricated at the Eindhoven University of Technology and used at the Stanford Synchrotron Radiation Laboratory (SSRL) during dedicated runs of February 1981 and February 1982.

II Description of the cell

A schematic diagram of the EXAFS cell is shown in fig. 1. The cell consists of two compartments (1: sample treatment; 2: measurements). The sample is transferred between the two compartments by a movable inner tube (3), which in its highest position separates compartment 2 from 1 by an O-ring seal (4).

The sample is a self-supporting wafer approximately 0.1 to 0.5 mm thin made by pressing the catalyst powder in a metal die. The sample holder (5) is attached to the base of the movable tube.

The sample can be heated using a heater (6) attached to the base and cooled via nitrogen gas or liquid passing through

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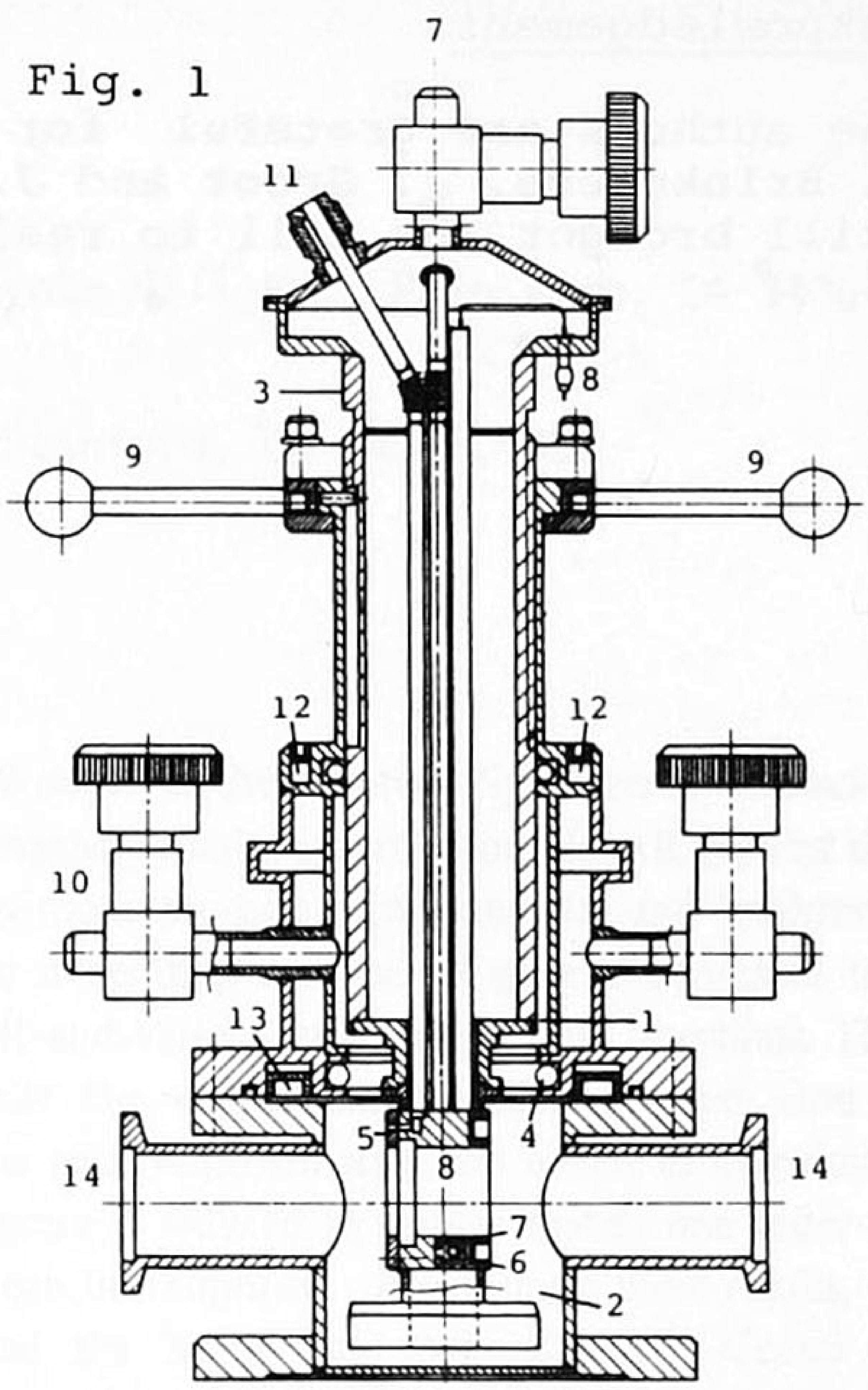
channels (7). Temperature can be measured or controlled using a thermocouple (8) attached to the base.

Sample treatment is performed with the movable tube locked in its highest position using the locking ring (9). The sample chamber can be evacuated through the vacuum valve (10) or exposed to specific treatment gases through the inlet (11). During sample heating the walls of the chamber are cooled by flowing water through the cooling channels (12,13). In this way the beryllium windows (14) are not exposed to high temperatures.

Fig. 1



X-ray absorption measurements can be carried out with the movable tube in its lowest position. Measurement temperatures between 77 K < T < 300 K are achieved by controlling the flow of liquid nitrogen or cold nitrogen gas through the cooling channel (7) and by using the heating element. It takes about 15 minutes to cool the sample from 300 K to 80 K. It is also possible to heat the sample in this position up to 600 K without damaging the beryllium windows (max. window temperature ∿ 325 K).

Summary

The EXAFS cell described above has the following properties:

I Sample treatment in situ
- reduction, oxidation,
- evacuation ($p \sim 10^{-5}$ Torr),
- adsorption of gases (CO, O_2, H_2, hydrocarbons).

II EXAFS measurements during catalytic reaction.

III Sample temperature variable 77 K < T < 800 K.
(maximum sample temperature during EXAFS experiment 620 K)

IV Rapid and easy sample mounting.

V Sample present as self-supporting wafer prepared by pressing the catalyst powder at high pressure in a metal die.

With this in situ cell EXAFS measurements can be performed on samples under vacuum or exposed to gases. The combination of the variable sample temperature and the possibility of exposure to gases allows us to pretreat the sample at high temperature and to measure in situ the EXAFS spectrum as a function of temperature.

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